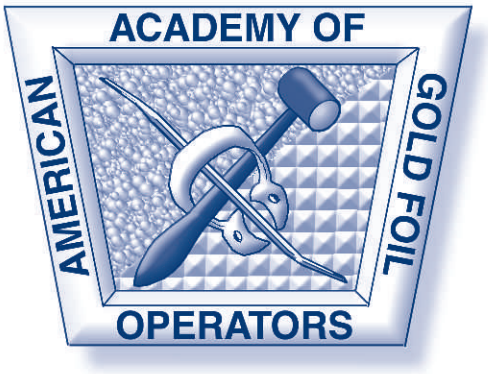
OPERATIVE DENTISTRY



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Buonocore Memorial Lecture

Water Treeing in Simplified Dentin Adhesives–Déjà Vu?

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N Hiraishi • CKY Yiu

Franklin R Tay

INTRODUCTION

Over the last decade, the classic concept of three-step bonding to dental tissues has developed rapidly to more user-friendly, simplified adhesive systems. These comprise the two-step etch-and-rinse, two-step self-etch and one-step self-etch adhesives. The last category of adhesives has been further reduced to single-bottle systems for simpler and faster applications. They carry, along with their simplicity, some characteristics that are detrimental to their efficacy in providing long-lasting bond stability. Simplified dentin adhesives are similar to resin-modified glass-ionomer cements in their high affinity for water. Such hydrophilicity renders these adhesives very permeable and denies their ability to hermetically seal dentin. Additionally, the water flux across simplified adhesives may compromise bonding in certain circumstances and their durability in the oral environment. This paper presents evidence of water transport across simplified adhesive systems and resin-modified glass-ionomer cements and relates them with clinical implications of the phenomenon.

Adhesive dentistry has come a long way since the development of adhesive monomers by Dr Oskar Hagger (1948), which were first used by McLean and Kramer (1952), and the acid-etching of dental hard tissues by Dr Michael Buonocore (Buonocore, 1955; Buonocore, Wileman & Brudevold, 1956). The availability of dentin adhesives has profoundly improved the quality of conservative work performed in pediatric and geriatric dentistry, prosthodontics, and lately, in endodontics. Today, we salute the commercialization of different classes of etch-and-rinse (total-etch) and self-etch adhesives (Van Meerbeek & others, 2003). Simplified versions of these adhesives have made bonding simpler, faster and more user-friendly (Perdigão, Duarte & Lopes, 2003). The latest one-step self-etch adhesives are now being marketed as single-bottle versions. They represent remarkable scientific and entrepreneurial achievements by research scientists, clinicians and manufacturers.

In all the words spoken and written about dentin adhesives, one theme constantly recurs: how long do these man-made bonds last? This question is becoming

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increasingly challenging to answer, as the increasing hydrophilicity and step-reduction in contemporary simplified adhesives render the adhesives very permeable and susceptible to water sorption and movement. These are alarming concepts. But the realities we face should trigger alarms. It is not the intention of this paper to provide an extensive review on the bonding of these adhesives to dentin. Rather, evidence of water movement across resin-dentin interfaces, which are associated with the use of the simplified dentin adhesives, will be illustrated and discussed. Once identified, steps may be taken to minimize the negative impact of water sorption, while maximizing the convenience of self-etching systems.

Permeability of Simplified Dentin Adhesives

Contemporary dentin adhesives are rendered very hydrophilic in order to improve their bonding to intrinsically wet substrates such as dentin. The incorporation of high concentrations of hydrophilic and/or ionic resin monomers in these adhesives increases their osmolalities and attraction of water, leading to increased water sorption (Tay & Pashley, 2003a). As simplified total-etch and self-etch adhesives lack non-solvented resin coatings (Cheong & others, 2003), they behave as permeable membranes (Tay & others, 2002a) that permit rapid, through-and-through water movement across the polymerized adhesives (Tay & others, 2004a). Dentinal fluid transudation across simplified dentin adhesives has been shown to occur when they were bonded to non-carious, deep, vital human dentin (Chersoni & others, 2004; Tay & others, 2004b). This phenomenon could be duplicated *in vitro* using bonded, extracted human teeth (Elgalaid & others, 2004). The entrapment of water droplets that emerged from the adhesive surfaces by slow-polymerizing, chemical-cured resin composites (Tay & others, 2003a; Carvalho & others, 2004) accounts for the apparent incompatibility when these composites were coupled to acidic adhesives in the presence of non-amine-type ternary catalysts (Suh & others, 2003).

The fact that fluid transudation rapidly occurs across polymerized dentin adhesives implies that there are interconnected porosities or channels within the adhesives that are responsible for rapid water movement within these adhesive membranes. This rapid water movement differs from the relatively slower diffusional processes across bulk resin. Unfortunately, these very minute channels cannot be seen with standard electron microscopical techniques, as the water is lost after desiccation of the specimens and the channels collapse when specimens are examined under vacuum. This prompted the immersion of bonded specimens in electron-dense tracers, such as silver nitrate, before they were subsequently prepared for morphologic examination. Once silver nitrate is reduced to metallic silver grains, it remains trapped in those sites regardless of whether the specimen is dehydrated or not.

Potential Pathways of Fluid Transport in Polymerized Adhesives

Ultrastructually, water entrapment in simplified dentin adhesives may be visualized as water-filled channels. Using ammoniacal silver nitrate (pH 9.5) as a tracer, Tay and Pashley (2003b) reported the existence of different forms of silver-filled, interconnecting channels within the adhesive layers of some commercial brands of two-step etch-and-rinse and one-step self-etch adhesives. The authors used the term "water trees" to describe these silver-filled water channels within the adhesive layers to distinguish them from the nanoleakage silver deposits (Sano & others, 1995) that were observed within the bulk of the dentin hybrid layers created by these adhesives. These water channels, which were revealed by silver staining, were seen when very thin, undemineralized sections (ca 1/100 mm thick) of the resin-dentin interfaces were examined using transmission electron microscopy (Figure 1A). Similar to nanoleakage, they could not be seen in demineralized sections that were stained with uranyl acetate and lead citrate (Figure 1B). Water trees were predominantly located along the surface of the hybrid layer, extending into the overlying adhesive layers. Other water channels were seen as "reflecting" downward from the junction between the adhesive and the resin composite. Occasionally, through-and-through channels that were oriented perpendicular to the hybrid layers could also be observed spanning the entire thickness of the adhesive layer.

Water trees were initially reported using more technically advanced transmission electron microscopy. However, they can be readily observed when silver-stained resin-dentin interfaces are examined using the backscattered mode of a scanning electron microscope. This is illustrated very nicely by the myriad of "unreported" water trees with resin-dentin interfaces in a recent nanoleakage paper. In this paper, Li, Burrow and Tyas (2003) demonstrated the existence of water trees in simplified self-etch adhesives such as Prompt L-Pop and simplified total-etch adhesives such as Single Bond, although these entities were not mentioned explicitly by the authors. By examining extracted specimens of vital human teeth bonded with a simplified total etch adhesive (Excite DSC), Ferrari and Tay (2003) have shown that water trees are not *in vitro* artifacts and that these potential water channels do exist following *in vivo* bonding procedures (Figure 1C).

Rapid water transport via water channels created within the adhesives is by no means the only way in which water traverses through the resin-dentin interfaces. The transport of ions and small molecules across an amorphous polymer matrix may still occur via dif-

fusion in the absence of physically-detectable water channels, albeit more slowly. Based on the dynamic percolation theory, water and ions can move across a polymer matrix by hopping from one hydrophilic domain to another (Dürr & others, 2002). This kind of ion hopping mechanism has been commonly observed in polymeric ionic conductors in which ionic salts are incorporated in a polar polymer that possesses significant ionic conductivity (Frisch & Stern, 1983). The diffusion of penetrant molecules through a polymer matrix occurs via micro-cavities of different sizes that are formed and destroyed almost instantaneously on an atomic time scale of picoseconds in the polymer (Müller-Plathesup, Laaksonen & van Gunsteren, 1993). The more hydrophilic the polymer, the greater the ease of formation of these micro-cavities, because of the reduced energy required to disrupt the adjacent polymer chains (Soles & Yee, 2000). In the context of dentin adhesives, these hydrophilic domains were seen as very small, discrete silver grains (Figure 1D) that were present in the adhesive after immersion in ammoniacal silver nitrate (Tay, Pashley & Yoshiyama, 2002c).

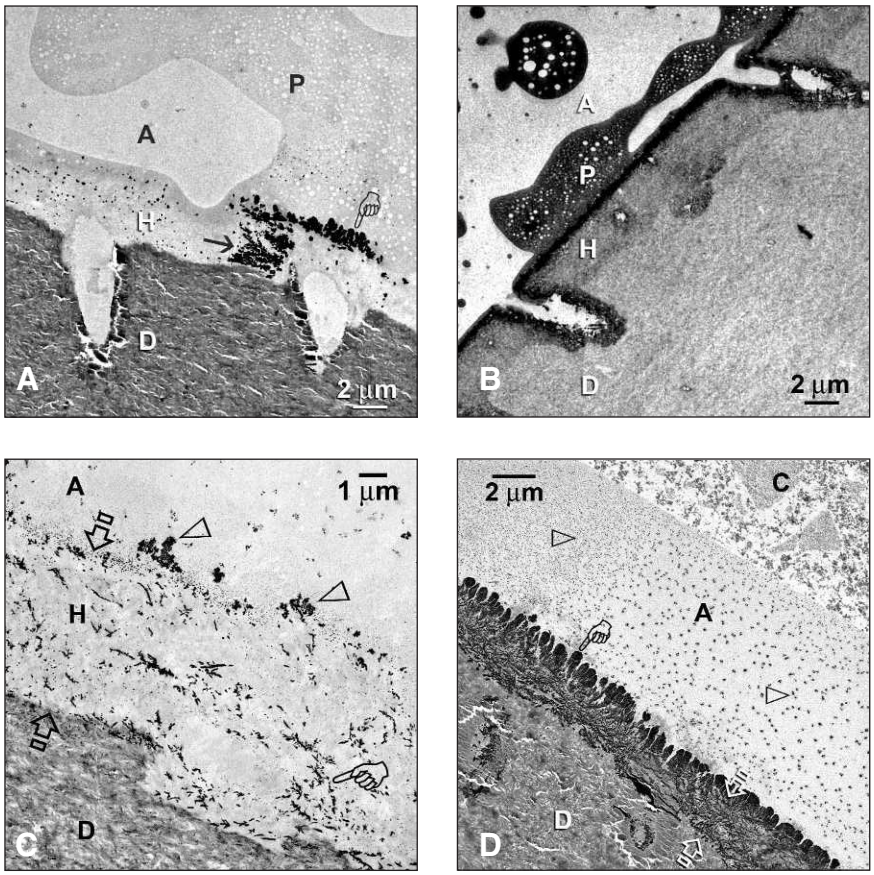

Figure 1*A. Differentiation between nanoleakage (arrow) and water trees (pointer) in a two-step etch-and-rinse adhesive containing polyakenoic acid copolymer (P). B. Both nanoleakage and water trees could not be identified in conventionally stained section of the same adhesive. P: stained polyalkenoic acid copolymer. C.* In vivo *specimen of another two-step etch-and-rinse adhesive confirming the existence of nanoleakage (pointer) within the unstained hybrid layer (open arrows) and water tree formation (open arrowheads) along the hybrid layer surface. D. Differentiation between discrete silver grains (open arrowheads) that represent water diffusion through an adhesive, and water trees (pointer) on the surface of a hybrid layer with extensive nanoleakage (open arrows) in a one-step self-etch adhesive. A gradient of water diffusion could be seen from the lower right to the upper left of the micrograph along the adhesive layer.*

**Labels common to all figures: C: resin composite; A: adhesive; H: hybrid layer; D: dentin. All micrographs are unstained, undemineralized, silver-impregnated sections examined with transmission electron microscopy unless stated otherwise.*

Historical Perspectives

Tay and Pashley (2003b) did not invent the term "water trees." They borrowed this term from the field of electrical engineering to reflect the similarities of the water channels seen in dentin adhesives with the microscopic tree-like channels that were identified in aged electrical insulation cables after water sorption. This well-known phenomenon was first reported in 1969 at the IEEE-NEMA Electrical Insulation Conference, Boston, MA, USA, in a paper titled "Deterioration of water-immersed polyethylene coating wire by treeing" (Miyashita, 1969). Water treeing is a well-recognized degradation phenomenon in the dielectric insulation cable industry that is responsible for the water-induced deterioration of polymer insulation of electrical cables after aging (Moreau & others, 1993). Water trees in polyethylene-coated cables are submicroscopic, self-propagating, water-filled tracks that are formed electrochemically by the oxidation of the hydrophobic polymer into more hydrophilic moieties, followed by the condensation of moisture within the hydrophilic, electro-oxidized regions (Ross, 1998). Similar to water trees in dentin adhesives, the original water trees in polymer insulation cables were seen only after immersion of polyethylene in silver nitrate solution (Fan & Yoshimura, 1996; Stepp & others, 1996). They could not be seen in conventional microscopy, as they collapsed after dehydration. Despite the fact that polyethylene is one of the most moisture-resistant polymers available, water has been identified as a contributing factor to this unexpected failure in performance. Water trees, once initiated from microvoids along the surface of the polymer, continue to propagate as hydrophilic tracks in a fractal manner within the hydrophobic polymer (Ding & Xing, 1996). They are converted into electrical trees as a result of lightning surges that result in a rapid deterioration in the cable insulation properties and ultimate

cable failure (Lewis, 1990; Dissado & Fothergill, 1992).

Unlike water trees in polymer-insulated cables that are formed after localized electrochemical transformation of the hydrophobic polymers, hydrophilic resin monomers and water are both present in self-etch and some total-etch dentin adhesives. This led Tay and Pashley (2003b) to hypothesize that water trees in dentin adhesives, together with nanoleakage within the hybrid layers, represent water-rich interfacial regions from which the leaching of hydrophilic resin components may occur readily and expedite the degradation of resin-dentin bonds.

Morphogenesis of Water Trees

Two theories have been proposed on the morphogenesis of water trees within dentin adhesive layers—the "remnant water" theory and the "water flux" theory. It was initially thought that water trees were morphologic expressions of water that was incompletely removed from water contained in simplified dentin adhesives. The rationale for such an assumption was that water trees were almost exclusively identified from resin-dentin interfaces bonded with ethanol-based adhesives and were rarely observed in acetone-based adhesives before aging and water sorption. It is also known that the inclusion of 2-hydroxymethacrylate (HEMA) in these adhesives makes it difficult to remove water completely from these adhesives. Using different HEMA-water concentrations, Pashley and others (1998) observed that the addition of HEMA to water lowered the rate of evaporation of water from the water-HEMA mixtures in a manner proportional to its effect on lowering the vapor pressure of water, making it more difficult to remove the last amounts of water. These ongoing experiments further showed that when experimental self-etch adhesives created with HEMA and acidic monomers were solvated in an ethanol/water mixture, as much as 40% of the solvent mixture remained after 60 seconds of air-blast evaporation. Up to 32% of the solvent mixture still remained upon further evaporation for an additional 60 seconds (a total of 120 seconds) (Yiu & others, unpublished results). It is speculated that this is also due, in part, to hydrogen bonding between ethanol, water and polar resin monomers in the adhesive.

The "remnant water" theory, however, does not account for four aspects of water tree formation:

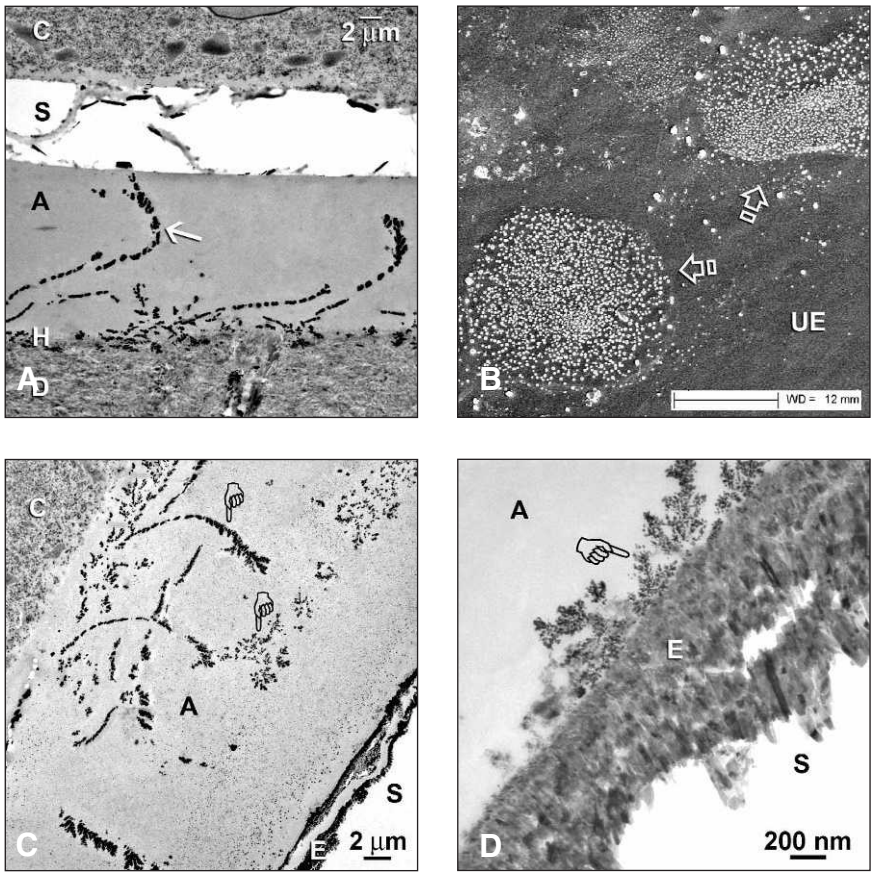

Figure 2*A. Through-and-through water tree formation (arrow) from the resin-dentin interface of a one-step self-etch adhesive. S: empty space caused by fracture of the adhesive during sectioning. B. SEM micrograph of a replica of the author's incisor, showing fluid transudation (arrows) along the surface of the uncut enamel (UE). C. Cut enamel (E) bonded with a 4-META containing, single bottle one-step self-etch adhesive showing water trees (pointers) within the adhesive. S: empty space. D. A higher magnification view showing the origination of some water trees (pointer) directly from the enamel surface (E). S: empty space.*

1. *Uneven Distribution*

One would have expected that, by random TEM sectioning of a myriad of resin-bonded dentin specimens conducted over a five-year period, that the distribution of water trees within the adhesive layer would be normal or Gaussion. Interestingly, water trees were seldom identified from the central part of an adhesive layer, and the frequency of observing through-and-through water tree formation (Figure 2A) across the entire cross section of a 5-20 µm thick adhesive layer was less than 10%. Indeed, water trees were predominantly identified from the surface of the hybrid layer. Being mostly vertical in their orientation (perpendicular to the surface of the hybrid layer), they were often interconnected with the horizontal or oblique reticular patterns of nanoleakage that were observed in the hybrid layer (Carvalho & others, 2005).

2. *Substrate Dependence*

Although water trees were frequently seen in sound dentin bonded with one-step self-etch adhesives, they were not observed when disks of resin composite were used as a bonding substrate if the adhesives were adequately air-dried before bonding (King & others, 2005). Residual water probably exists as water clusters that are attached to the hydrophilic domains of the adhesive via hydrogen bonding. This form of bound water could be seen in the form of discrete silver grains within the polymerized adhesive. As the solvents in the dentin adhesives are evaporated in the same manner when bonding two composite disks together, the absence of water trees when composites are bonded to composites suggested that water trees contribute minimally by residual water derived from the dentin adhesives.

Controversy exists regarding whether the water trees found in bonded enamel are derived from the adhesive. A previous study has shown that there is a slow outward flow of fluid through the enamel of vital human teeth (Bergman, 1963). This can easily be demonstrated by taking an impression of one's own incisors using a slow-setting polyvinylsiloxane wash (Figure 2A). This phenomenon occurs more readily in young teeth. Although there have been no reports that this fluid flow adversely affects enamel bonding, it is possible that some of this fluid may be trapped by the polymerized adhesive (Figure 2C). One can see from the higher magnification view of the previous TEM micrograph that some of the water trees were originated from the surface of the enamel smear layer (Figure 2D). This suggests that at least some of the water trees that are seen in bonded enamel have been derived from water that is expressed out of the enamel tissues.

3. *Reverse Water Trees*

Similar to real trees that are found in nature, water trees in dentin adhesives are manifested in different sizes and forms. Some of the water trees are like birch trees with a wide canopy that spreads out in a fractal manner (Figure 3A). Others resemble pine trees and

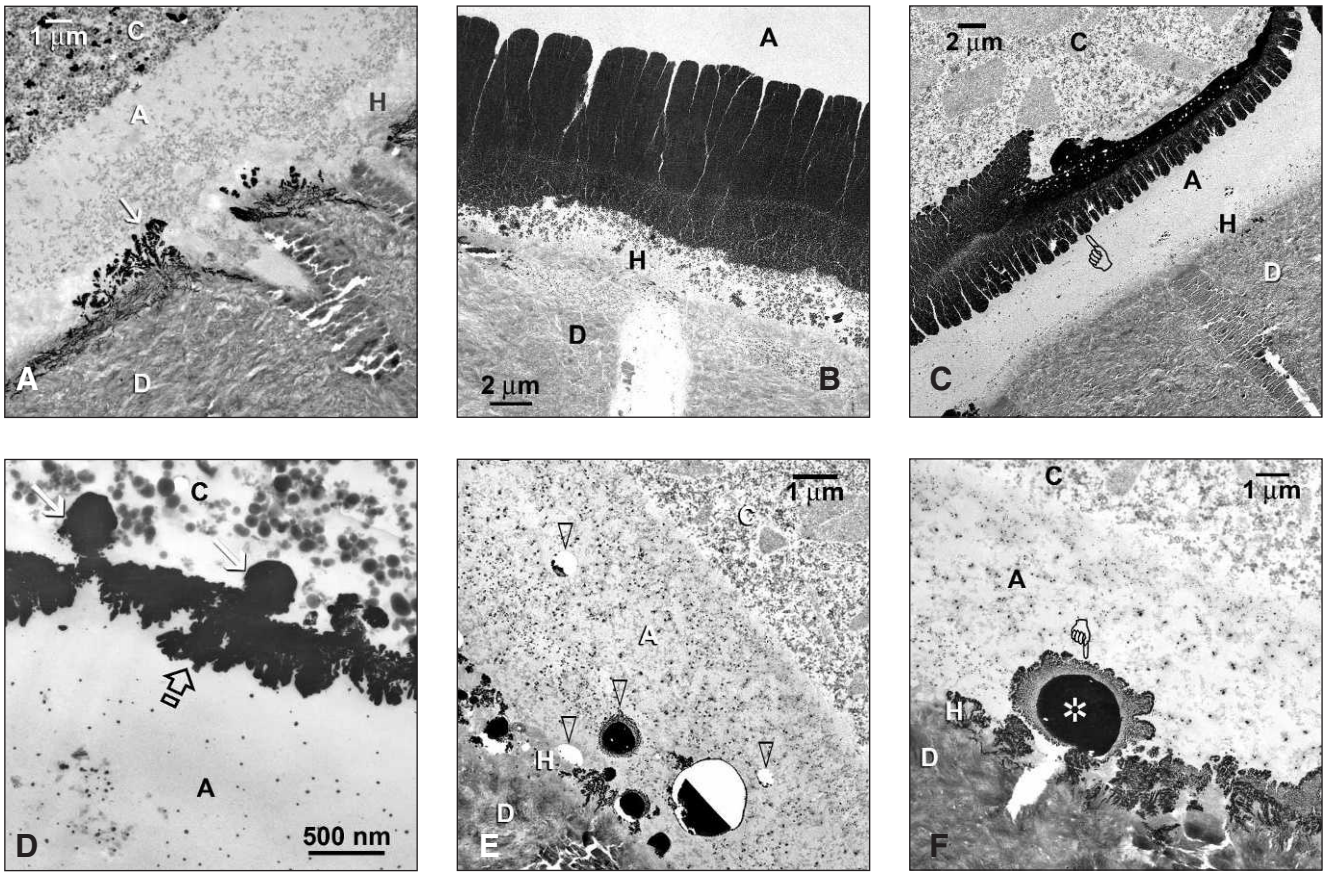

Figure 3*A. A canopy-type water tree (pointer) from the resin-dentin interface of a two-bottle one-step self-etch adhesive containing nanofillers. B. Columnar-type water trees from the resin-dentin interface of a single bottle one-step self-etch adhesive. C. Reverse water trees (pointer) that are opposite in direction to those water trees seen in Figures 3A and 3B. D. Reverse water trees (arrow) are frequently connected to water blisters (arrows) that are trapped between the adhesive and the composite. E. Water droplets (open arrowheads) formed within the adhesive layer of a fumed silica-filled, single-bottle, non-HEMA containing one-step self-etch adhesive. F. Emanation of a water droplet (asterisk) from a tubular orifice in the adhesive described in Figure 3E. Secondary water tree formation (pointer) along the periphery of the water droplet produced a sun burst effect around the droplet.*

are more columnar (Figure 3B). Nevertheless, all exhibit a definitive orientation, in the sense that the location from which they originate can usually be traced. Normally, water trees have points of origin from the surface of dentin, with their branching pointing upward into the adhesive. In the case of reverse water trees, they appear to originate from water trapped at the interface between the adhesive and the overlying resin composite; they spread downward, with their "branches" pointing toward the dentin (Figure 3C). Reverse water trees are frequently affiliated with water blisters that are trapped along the adhesive-composite interface (Figure 3D).

4. *Secondary Water Trees Derived From Discrete Water Droplets*

The entrapment of water droplets is commonly observed in single-bottle versions of one-step self-etch adhesives such as Brush&Bond, iBond and G-Bond (Tay, Pashley & Peters, 2003; De Munck & others, 2005a) (Figure 3E). These water droplets were surmised to represent phase separation of water from adhesive components after the evaporation of more volatile solvents such as ethanol or acetone (Van Landuyt & others, 2005). When these water droplets were examined without the adjunctive use of a tracer solution, they appeared as clear, circular holes within the adhesive layer. However, when these specimens were previously immersed in a silver nitrate tracer solution prior to laboratory processing, water trees could be seen radiating circumferentially from the periphery of these water droplets, creating a fuzzy sun burst effect around the silver-impregnated droplets (Figure 3F).

Water can exist as free and bound water within a polymer matrix (Klotz & others, 1996). The existence of water trees succinctly indicates the presence of free water within the adhesive matrix. However, it must be stressed that the retention of residual water within a hydrophilic adhesive does not necessarily result in the generation of physical water-filled channels within an adhesive. When a polymer contains hydrophilic or polar functional groups, water molecules can exist as bound water that are weakly attracted to the hydrophilic and/or polar domains of the polymer matrix via hydrogen bonding or van der Waal's forces. With the use of FTIR spectroscopy, three molecular water species have been identified with epoxy resin matrices after water sorption. The S_0 molecular species (Cotugno & others, 2001), or the portion of water that can be frozen at 0°C (Ping & others, 2001), was associated with bulk or free water that occupied the free volume of the polymer matrix. The S_1 species, or the portion of water that can be frozen below 0°C, represent either self-associated dimmers or water molecules with weak hydrogen bonding along the secondary hydration shells. Conversely, the S_2 molecular species, or the non-freezable portion of absorbed water molecules, is firmly bound to polar sites along the polymer network and exhibit high plasticizing efficiency. Hydrogen bonding between the water molecules and the polar hydroxyl, carboxylate or phosphate groups of polymer networks will disrupt the interchain hydrogen bonding (VanLandingham, Eduljee & Gillespie, 1999), altering the molecular structure and increasing the segmental mobility of the polymer chains (Musto & others, 2002). These changes are reflected by reductions in the mechanical properties and the decline in glass transition temperatures (T_g) of polymerized resins (Nogueira & others, 2001).

The above description applies to a well-polymerized, physically non-porous epoxy resin polymer matrix that is subjected to water sorption. Nevertheless, introduction of the concept of bound water serves to point out that within limits, residual water that is bound to the hydrophilic domains within the dentin adhesive can disrupt interchain hydrogen bonding during polymerization of the adhesive matrix (Lagouvardos & others, 2003). Thus, the different patterns of water tree formation within the adhesive may be regarded as historical traces of free water movement along the relaxed/disrupted regions of a polymer matrix during the initial gelation stage of the adhesive as it is being polymerized.

As water trees seldom exist when water-containing adhesives are used to bond composites to composites, it is unlikely that water tree formation is caused by water that is present within the adhesive formulations. Two questions, which remain to be answered, are: a) where is the water derived from and 2) what triggers the movement of this additional source of water through the disrupted sites of an adhesive matrix during the gelling phase of polymerization. Both of these questions can be reasonably addressed with the "water flux" theory.

The "Water Flux" Theory

Vital dentin, particularly, deep vital dentin, is highly permeable, because of the presence of relatively short and wide dentinal tubules and normal positive pulpal tissue pressure (14 cm H_2O or 10.3 mm Hg; Ciucchi & others, 1995). Three types of fluid movement may occur through dentin: evaporative, osmotic and convective water fluxes. Evaporative water flux may be induced by air blasts (Goodis, Tao & Pashley, 1990), such as those that occur during air-drying of a crown preparation. The presence of the smear layer and smear plugs offers little resistance to evaporative water loss. Dehydrating dentin with an air blast or with absorbent paper will generate capillary forces that induce rapid outward fluid movement from dentinal tubules (Matthews, Showman & Pashley, 1993). Because non-vital dentin also contains water, evaporative water flux may occur irrespective of the tooth's vitality status, even when

smear plugs are retained within the dentinal tubules. Although it is mandatory to remove solvents from solvated adhesives before light-curing, the same air-drying process induces outward evaporative water flux from the smear-layer-covered dentin (Hashimoto & others, 2004a).

Moreover, the high concentrations of water-soluble ionic monomers in the presence of water may also induce osmotic water flux from deep dentin (Panopoulos, Gazelius & Olgart, 1983; Pashley, 1985; Pashley & others, 1996), if the osmolatities of these comonomers exceed the osmolality of dentinal fluid (Pashley, Horner & Brewer, 1992). This is very likely to occur just prior to polymerization. The authors have calculated the concentration of HEMA and other hydrophilic monomers in solvated adhesives as the solvents are evaporated. Just prior to light curing, the concentrations can be between 2.5 and 3.5 moles/L, which is very hypertonic. That is, 2500 to 3500 milliosmoles/kg (mOsm/kg) compared to the osmolality of plasma, which is 290 mOsm/kg. These high solute concentrations (8.6 to 12 times as concentrated as plasma) mean that their water concentration is much lower than the water concentration in dentinal fluid, thus causing osmotic fluid movement from dentinal fluid into these concentrated comonomer films. As soon as the monomers are converted to polymers, the osmotically induced water flux should cease.

Both evaporative and osmotic fluxes may result in the permeation of water along regions within the adhesive where interchain segmental mobility is increased by the hydrogen bonding of retained bound water present in the adhesive mixture. This provides a logical explanation for the predilection of normally oriented water trees along the surface of the dentin. In particular, such a theory also accounts for the observation that, in the presence of smear plugs, silver containing tracks are observed around the peritubular dentin (the channels with the least resistance) that are continuous with silver-containing interfibrillar spaces within the hybrid layers formed by mild self-etching primers, that, in turn, are continuous with the vertically-oriented water trees along the surface of the hybrid layers (Figure 4A). Theoretically, more water trees can be formed with longer periods of air drying or contact of the adhesive with dentin prior to its polymerization. On the other hand, the viscosity of the polymer matrix, cross-linking and the intensity of interchain hydrogen bonding may determine the specific morphology of the water trees created with the polymerized adhesive.

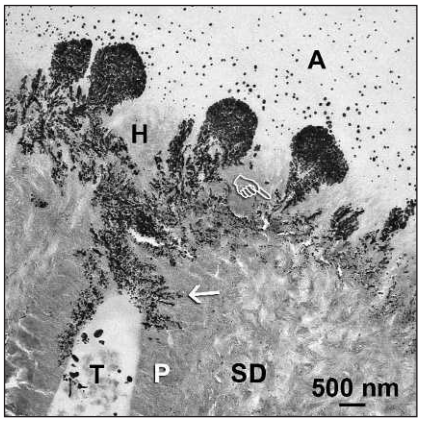

Figure 4*A. Water trees and nanoleakage are present when one-step self-etch adhesives are applied to sound dentin (SD). A. Evaporative water flux provides a logical explanation for the observation of water movement from the channels with the least resistance (arrow) in the peritubular dentin (P) into the interfibrillar spaces within the hybrid layer (pointer) that ultimately resulted in water tree formation. T: dentinal tubule.*

Indirect water flux also provides the raison d'être for the observation of reverse water trees. Hashimoto and others (2004a) recently showed that, following the detection of outward evaporative water flux after air drying of the adhesive-coated dentin, additional inward fluid flux was induced by light-activation of the adhesive. It is likely that the inward fluid flux is caused by heat generated during light curing of the adhesive. One can envisage, on a sub-second time scale, that during the light-activation process, some of the water that has reached the top of the adhesive layer cannot escape, as it is trapped by the initially better polymerized surface part of the adhesive. The heat generated by a quartz-tungsten-halogen or a second generation LED light-curing unit (Bouillaguet & others, 2005) could redirect water back into the less well-polymerized subsurface adhesive matrix, forming water tracks that are morphologically identified as reverse water trees.

Convective water flux is considerably more significant when smear plugs are removed by acid etching (Pashley, Michelich & Kehl, 1981). Many researchers have attempted to duplicate these convective water fluxes *in vitro* by bonding to dentin via perfusion at physiological pulpal pressure of about 15–20 cm water (Bouillaguet & others, 2000; Elgalaid & others, 2004; Grégoire & others, 2003; Vaysman, Rajan & Thompson, 2003; Özok & others, 2004). With simplified single-bottle adhesives, convective water movement (Vongsavan, Matthews & Matthews, 2000) has been shown to occur both *in vitro* and *in vivo* during polymerization of the adhesives (Purk & others, 2004). Water droplets trapped between the adhesive and resin composite account for the apparent incompatibility that occurs when acidic versions of these adhesives are used with slow-setting, chemical-cured resin composites. These water droplets not only reduce the bonded surface area, they may act to increase interfacial stress, which may result in the premature dislodging of the resin composites during function (Tay & others, 2003b).

In the presence of smear plugs, only slow convective water flux occurs in vital dentin (Pashley & others, 1981). Even this slow convective water flux is adequate to permit the blebbing of dentinal fluid through single-bottle type, simplified self-etching adhesives that do

not contain HEMA (Tay & others, 2003d). HEMA is miscible with water in all proportions and is often utilized as a transitional polymerizable, slightly volatile solvent for other ionic resin monomers that are sparingly soluble in water. When HEMA is absent, phase separation of the adhesive components occurs (Van Landuyt & others, 2005) on the addition of water to an adhesive in which a more volatile solvent such as ethanol has been evaporated. These phase separations probably account for the observation of discrete droplets of water within the adhesive layers of dentin bonded with the latest non-HEMA-containing, single-bottle type, one-step self-etch adhesives. Further attempts to evaporate the adhesive solvent, and light curing of the adhesive, will generate water fluxes that radiate out from these water droplets as secondary "sun-bursting" type water trees.

Water Trees Are Absent in Bonded Transparent Carious Dentin

When resin bonding is performed on sound dentin with unblocked dentinal tubules, it is difficult to resolve whether the entrapped water that causes the formation of water trees originates from water-containing self-etch adhesives or from hydrated dentin. Substantiation of the "water flux" theory of water tree formation in simplified dentin adhesives really stems from work performed with the application of these adhesives onto transparent carious dentin. It is pertinent to point out that all *in vitro* and *in vivo* studies that reported the existence of water trees were carried out using sound coronal dentin as bonding substrates. For those studies that involved the use of simplified self-etch adhesives, nanoleakage was invariably identified within even very thin hybrid layers, irrespective of the presence or absence of water trees. Ideally, nanoleakage should not occur in hybrid layers created by self-etching primers, as etching and resin infiltration occur concurrently.

Transparent carious dentin is known to heavily occlude with intratubular mineral deposits (Ogawa & others, 1983; Zheng & others, 2003). Tubular occlusion accounts for the relative impermeability of this type of bonding substrate (Tagami & others, 1992). When convective and evaporative water fluxes are eliminated by bonding to transparent carious dentin (Lee & others, 2003), any water entrapment within the adhesive should be attributed to the retention of water derived from one-step self-etch adhesives. Indeed, the use of this relatively simple experimental design has resulted in a giant step forward in our understanding of the etiology of water tree formation and in resolving the riddle as to why nanoleakage occurs in self-etch adhesives despite the simultaneously occurring processes of etching and resin infiltration.

In this experimental design, sound and carious dentin from the same tooth were bonded under dentin perfusion using an experimental 4-MET containing, single bottle-type, one-step self-etch system in which the all components were known. To prevent complications that may arise from the breakdown of the acidic monomer into non-polymerizable components (Nishiyama & others, 2004), the adhesive was supplied with the water component segregated from the resin monomers. The adhesive formulation was reconstituted immediately before application and applied to human carious teeth in which the soft, caries-infected dentin was removed with a polymer bur, leaving behind hard transparent, caries-affected dentin. The rest of the sound dentin was ground down to the same level as the transparent carious dentin, so that both bonding substrates represented deep dentin derived from the same tooth. The root of each of these teeth was sectioned 2 mm below the cementoenamel junction and the remaining crown segment was cemented to a Plexiglass slab that was penetrated by a stainless steel tube. This permitted filling of the pulp chamber with water via plastic tubing connected to a syringe barrel to deliver 20 cm of water pressure (Pashley & Depew, 1986). By keeping the syringe barrel at the level of the crown segment or raising it to 20 cm above the crown segment, a convective water flux could either be eliminated or generated. As the pulp chamber was filled with water all the time, air drying of the smear layer-covered dentin would create an evaporative water flux (Sidhu & others, 2004).

When the experimental adhesive was applied to sound dentin under the influence of evaporative water flux only, a layer of columnar-appearing water trees of almost uniform thickness could be identified above the hybrid layer (Figure 4B). In the presence of additional convective water flux, silver-filled water droplets appeared over the dentinal tubular orifices despite their occlusion by smear plugs (Figure 4C). For some sections, more than 50% of the adhesive layers were filled with water trees and droplets that were continuous with the nanoleakage identified within the hybrid layers (Figure 4D). In the design of one-step self-etch adhesives, manufacturers have probably neglected the detrimental effects of convective water flux on these highly permeable adhesives. Indeed, very few bond strength studies of one-step self-etch adhesives, in particular, the single-bottle versions, were conducted under dentin perfusion to simulate what happens in vital, sound dentin. The contribution of convective water fluxes provided the rationale for the observation of water droplets (Figure 4E) over the adhesive surface when these adhesives were applied on deep, vital dentin after crown preparations (Chersoni & others, 2004). The data obtained with the experimental adhesive have also been reproduced by the bonding of commercialized versions of single-bottle one-step self-etch adhesives under dentin perfusion and the use of a light-cured composite that was activated immediately after

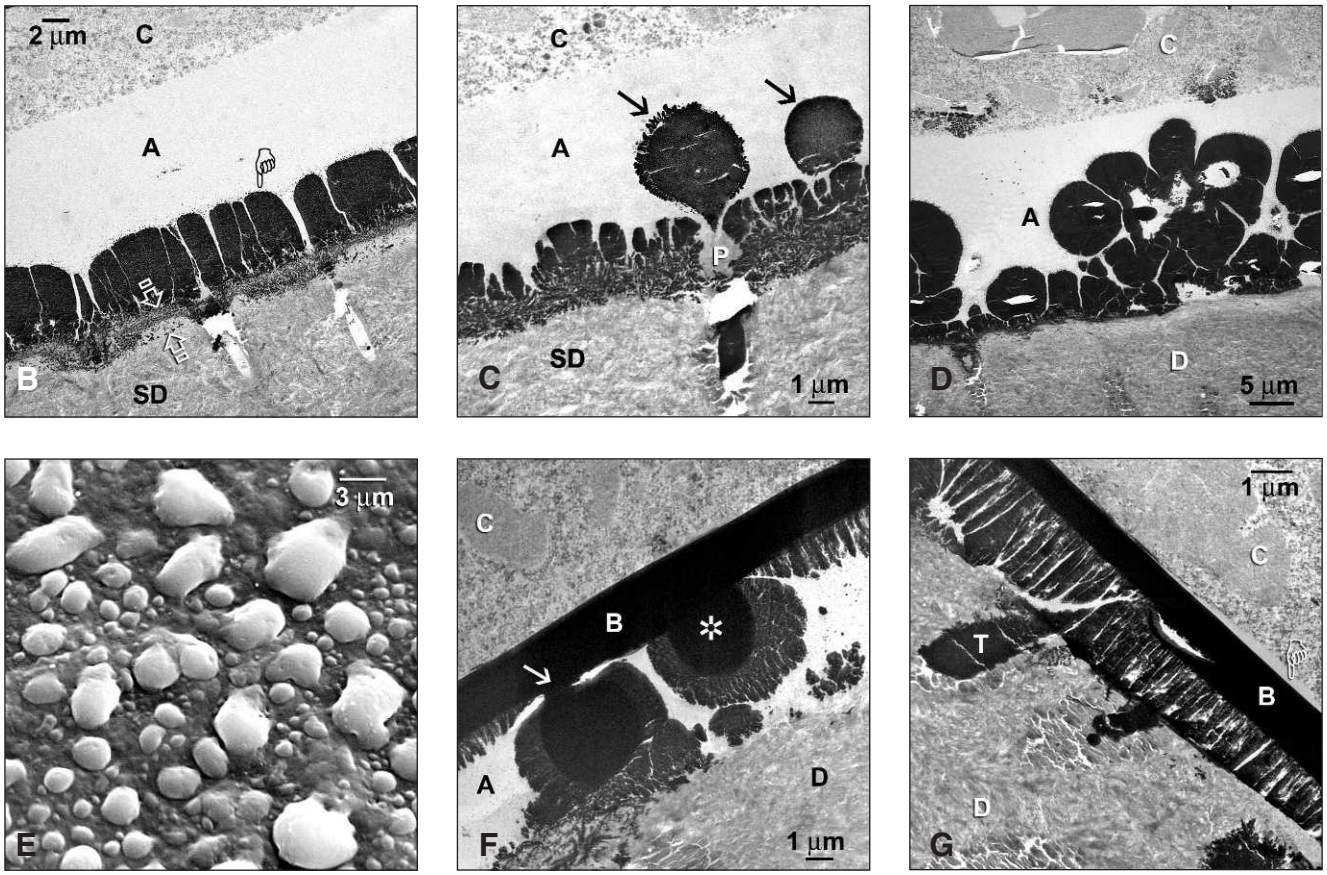

Figure 4*B. With the use of an experimental non-HEMA containing, single-bottle one-step self-etch adhesive, evaporative water flux resulted in more aggressive water tree formation (pointer) and extensive nanoleakage within the hybrid layer (open arrows). C. With the same experimental adhesive, the combination of evaporative and convective water flux resulted in the expression of water droplets (arrows) through the smear plugs (P). D. A more severe manifestation of what was depicted in Figure 4C, with silver deposits occupying more than 50% of the adhesive surface area. E. SEM micrograph showing resin replica of fluid droplets that appeared on the surface of vital sound dentin after bonding with a one-step self-etch adhesive. F. The appearance of these* in vivo *fluid droplets could be seen when the same commercial single-bottle one-step self-etch adhesive was bonded to sound dentin* in vitro *under dentin perfusion. In this micrograph, convective water fluxes resulted in the expression of water droplets (asterisks) from the dentinal tubules. These water droplets were connected (arrow) with a large water blister (B, incompletely shown) between the adhesive and the composite. G. In a different commercial single-bottle one-step self-etch adhesive that was bonded under perfusion to deep, sound dentin, through-and-through water trees that completely obscured the entire adhesive layer were connected to a water blister (B, incompletely shown). An artifactual contaction gap between the silver deposits and the composite was filled with laboratory embedding epoxy resin during subsequent laboratory processing (pointer). T: dentinal tubules.*

placement on the polymerized adhesive, with equally, if not more dramatically detrimental results (Figures 4F and 4G). These results complemented the concern raised by Özok and others (2004) regarding the pressing need for manufacturers to evaluate the behavior of one-step self-etch adhesives under dentin perfusion prior to the launching of these adhesives. In these two micrographs, the thick band of silver between the adhesive and the composite represented part of a large water droplet trapped by the light-cured composite, corresponding to the free water droplets trapped by a polyvinyl siloxane impression material seen in Figure 4E.

By contrast, both water-treeing and nanoleakage were absent from the hybrid layers of transparent, caries-affected dentin from the same tooth (Figure 5A). The hybrid layer, created in transparent dentin, was three times as thick as that observed in bonded, sound dentin. It is initially hard to rationalize why the much thicker hybrid layer in the former was devoid of nanoleakage, whereas, the much thinner hybrid layer in the former was completely occupied by nanoleakage. Such a phenomenon contradicts "the monomer diffusion concept of resin infiltration" proposed by Pashley and others (1993), in which a diffusion gradient occurs for resin monomers within a collagen network based on their molecular weight and diffusion capacity. Apparently, air drying of the solvated adhesive and bonding under physiological pulpal pressure resulted in a negligible adverse effect of water movement during bonding to transparent carious dentin. This is because both evaporative and convective water fluxes from

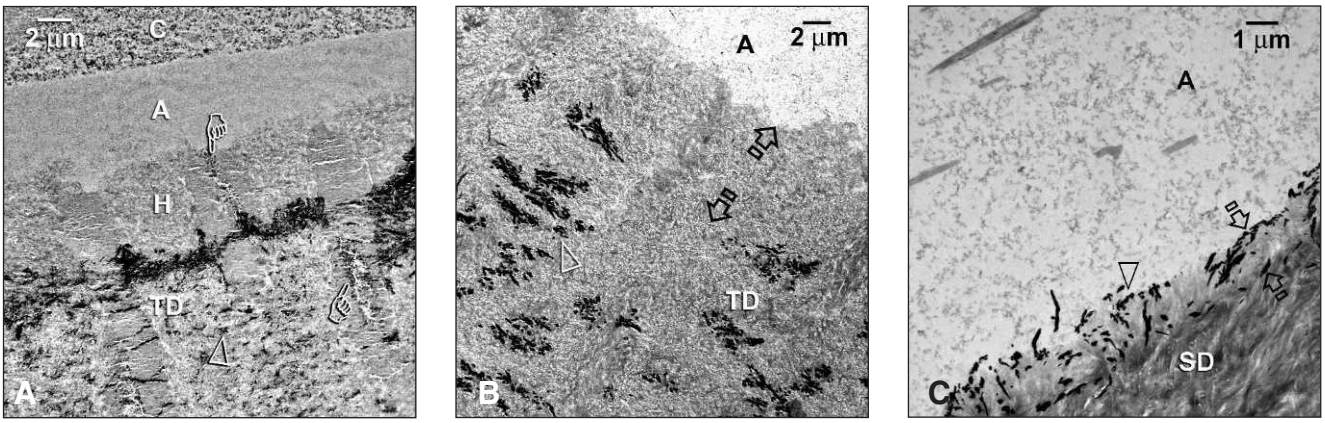

Figure 5. *Water trees and nanoleakage are absent when self-etch adhesives are applied to transparent carious dentin (TD). A. Water trees were absent and nanoleakage was minimized within the much thicker hybrid layer when the same experimental one-step self-etch adhesive was applied to transparent carious dentin. Water fluxes were minimized as the dentinal tubules were heavily occluded with mineral deposits (pointers). Severe silver deposits (open arrowhead), however, could be seen in the highly porous substrate. B. When a commercial two-step self-etch adhesive was applied to transparent carious dentin, nanoleakage was completely absent from the hybrid layer (open arrows). Islands of silver deposits within the unbonded transparent carious dentin could similarly be observed (open arrowhead). C. By contrast, nanoleakage (open arrowhead) could be seen within the thinner hybrid layer (open arrows) when the same commercial two-step self-etch adhesive containing fumed silica and NaF crystals was applied to sound dentin (SD).*

dentin were blocked by the heavily occluded dentinal tubules. This resulted in an excellent initial resin seal with the complete absence of water-rich interfacial zones above a highly porous bonding substrate that is partially depleted of its mineral content (Angker & others, 2004). Although these hybrid layers were thicker than those created in sound dentin (Yoshiyama & others, 2002), it is impossible for any adhesive to completely diffuse through a zone of partially demineralized carious dentin that may be several hundred micrometers thick (Nakajima & others, 2005). This could be appreciated by the presence of discrete islands of silver deposits directly beneath the silver-free hybrid layer and within the highly porous unbonded transparent carious dentin. At the scanning electron microscopy level, this may give the illusion that interfacial nanoleakage is present when bonding is performed on caries-affected dentin (Kubo & others, 2002).

Unlike one-step self-etch adhesives (Tay & others, 2004c), two-step self-etch adhesives (self-etching primers) rarely exhibit water-treeing prior to aging and water sorption (Donmez & others, 2005). Nevertheless, nanoleakage within the hybrid layer is invariably identified from hybrid layers created in sound dentin bonded by these adhesives (Hashimoto & others, 2004b; Reis & others, 2004). As self-etching primers may contain 30% to 50% water in their formulations, the "remnant water" theory has previously been proposed to explicate the occurrence of nanoleakage in these adhesives (Tay & others, 2002b; Li & others, 2003). Based on the authors' more recent work performed with self-etching primers on transparent carious dentin (Figure 5B) and sound dentin (Figure 5C) derived from the same teeth, nanoleakage was completely absent from the bonded interfaces of the former, despite the occurrence of islands of silver deposits within the highly porous underlying substrate. In the broadest sense, this feature resembles a much amplified version of an incompletely infiltrated hybrid layer produced by an etch-and-rinse adhesive in sound dentin. There was more nanoleakage in sound dentin bonded with self-etching primers (Figure 5C) than in caries-affected dentin (Figure 5B).

Relationship Between Nanoleakage and Water Trees

Thus, when bonding to sound dentin, the extensive nanoleakage that was seen in the hybrid layers of the latest simplified self-etch adhesives may be attributed to the combined adverse effect of evaporative, osmotic and possibly convective water fluxes that result in an outward fluid movement from both the intertubular dentin and the dentinal tubules. As the inward diffusion of acidic resin monomers demineralize intact dentin and dissolve smear layers and smear plugs, the outward, osmotically-induced water fluxes generated may dilute or even flush out the partially neutralized, but still acidic, resin monomers from the partially demineralized dentin. The presence of dilute water in partially acidic adhesive may retard the polymerization of resin within the already resin-sparse interfibrillar spaces. This is probably the mechanism responsible for the manifestation of heavily silver-impregnated hybrid layers after silver tracer immersion.

Figures 6A through 6C depict "snapshots" of what was probably a continuous series of events that link the expression of nanoleakage in self-etch adhesives with water-tree formation. Water that seeps out of normal hydrated dentin via the paths of least resistance along the smear plugs and peritubular dentin first occupies

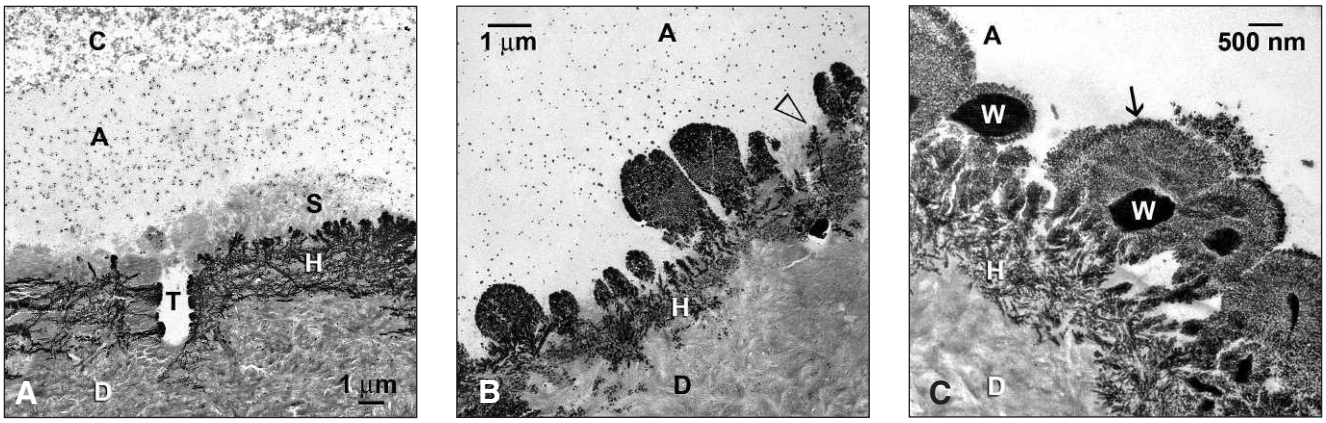

Figure 6. *"Snapshots" of what was probably a continuous series of events that link the expression of nanoleakage in a one-step self-etch adhesive with water tree formation. A. Water seeps out of normal hydrated dentin via the paths of least resistance in the peritubular dentin first and occupies the interfibrillar channels of the partially demineralized intertubular dentin beneath the smear layer (S), with the initiation of water trees along the base of the smear layer. B. The water finds its way through the smear layer and is manifested more or less vertically (arrow) as water trees within the adhesive. C. Depending on the chemical composition of the adhesive, phase separation of the bulk water may occur in the form of water droplets (W), from which secondary water trees arise (arrow).*

the interfibrillar channels of the partially demineralized intertubular dentin beneath the smear layer, with the initiation of water trees along the base of the smear layer (Figure 6A). The water finds its way through the smear layer and is manifested more or less vertically, as water trees within the adhesive polymer matrix during its gelling phase of polymerization (Figure 6B). Depending on the chemical composition of the adhesive, phase separation of the bulk water may occur in the form of water droplets, from which secondary water trees arise (Figure 6C). According to this concept, nanoleakage in self-etch adhesives, and water trees that are formed along the surface of the hybrid layer of these adhesives, have a common origin. In the case of nanoleakage within the hybrid layer, subsurface water movement occurs preferentially through pre-existing channels (interfibrillar spaces) that are determined by the arrangement of the dentin collagen network. In the case of water treeing, bulk water creates channels through the paths of least resistance within the gelling polymer matrix. These paths are likely to be hydrophilic regions within the polymer matrix in which intermolecular hydrogen bonding has been disrupted by bound residual water molecules that act as plasticizers and increase the segmental mobility of the polymer chains.

Functional Implications of Water Treeing

It has been suggested that the presence of large plasma proteins (Pashley, Galloway & Stewart, 1984) and immunoglobulins (Hahn & Overton, 1997) may reduce outward convective fluid fluxes from deep dentin by adsorbing to intratubular material that reduces the functional radii of dentinal tubules. Coagulation of these dentinal fluid proteins by glutaraldehyde and/or primer components (Bergenholtz & others, 1993; Nikaido & others, 1995) may further reduce outward convective fluid flow. Clinically, however, the use of a single-bottle, one-step self-etch adhesive containing 4-META and glutaraldehyde did not prevent dentinal fluid transudation in vital, anesthetized human crown preparations covered with the adhesive (Chersoni & others, 2004 [see Figure 4E]). Although coagulation of the plasma proteins by adhesive components may prevent large molecules, such as serum albumin, from traversing the dentinal tubules (Bergenholtz & others, 1993), this probably does not limit the movement of small molecules, such as water, through the proteinaceous hydrogel that is precipitated within the tubules and along the fine crevices that are present between the smear plugs and the walls of peritubular dentin. Both nanoleakage and water treeing are subclinical phenomena. Unlike *in vivo* convective fluid studies with the objective of collecting enough dentinal fluid for analytic purposes (Maita & others, 1991; Knutsson, Jontell & Bergenholtz, 1994), only a very small amount of fluid is necessary to produce nanoleakage and water trees, considering the thickness of the hybrid and adhesive layers.

The absence of interfacial, water-rich zones following the use of self-etch adhesives in transparent carious dentin provides reassurance of the validity of the concept of simultaneous etching and priming. Although tubular occlusion by mineral crystals prevents water treeing and nanoleakage in one-step self-etch adhesives, it is unrealistic that, clinically, one can bond only to transparent carious dentin without involving surrounding sound dentin. A likely scenario is that one may encounter a gradual transition from a strongly bonded but permeable interface to a weakly bonded (Yoshiyama & others, 2003; Yazici & others, 2004) but relatively impermeable interface (Pashley & others,

1991), as the bonding substrate shifts from sound to caries-affected dentin along the cavity floor.

At least from an *in vitro* perspective, improvements in bond strength and reduction in nanoleakage and water tree formation may be achieved when multiple coats of one-step self-etch adhesives were used on sound dentin, instead of the standard number of coats recommended by manufacturers (Hashimoto & others, 2004b; Ito & others, 2005). When one-step self-etch adhesives are applied to sound dentin, water droplets may be deposited between the adhesive-composite interface due to rapid water movement across the adhesive. When a sufficiently thick layer of adhesive is present, the phenomenon of water blistering is of minor clinical consequence, as it only occurs when a slow setting chemical-cured composite is employed or when a light-cured composite is experimentally subjected to delayed light-activation. However, this phenomenon becomes a clinical concern when only a single coat of a one-step self-etch adhesive was applied to sound dentin. (Figure 6D). As the adhesive layer is thin, direct contact of the water trees with the light-cured composite resulted in blister formation even when the composite was light activated immediately.

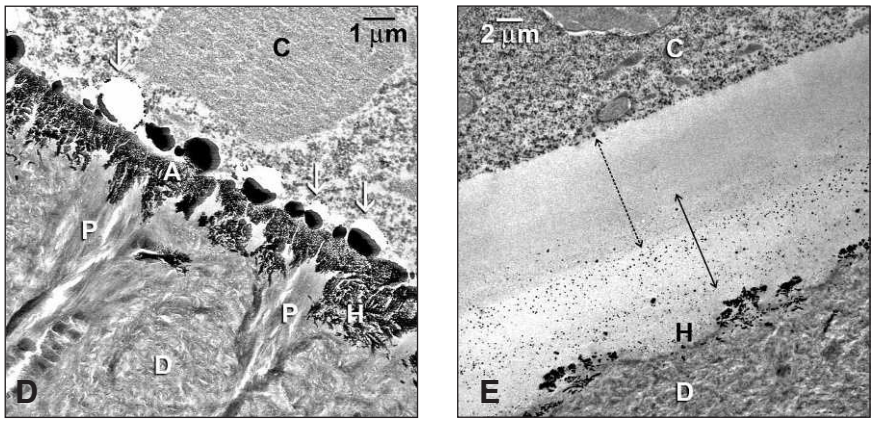

Figure 6*D. When only a single coat of a one-step self-etch adhesive was applied to sound dentin, water blisters appeared between the adhesive and the light-cured composite even when the composite was light-cured immediately. This resulted in substantial weakening of the adhesive-composite interface. E. One-step self-etch adhesive can be made less permeable by placing a layer of less hydrophilic bonding resin (solid line) on top of the adhesive (dotted line).*

One-step self-etch adhesives can be rendered less permeable by executing the rationale behind the use of two-step self-etch adhesives—by treating the one-step self-etch adhesive as a primer and covering it with a less hydrophilic resin coating such as those that are employed in conventional etch-and-rinse adhesives (Carvalho & others, 2004). This extra step converts one-step into two-step self-etch adhesives (King & others, 2005) and render them less permeable to water movement (Figure 6E). However, that strategy circumvents the original claims of simpler, faster application and improved user friendliness, which are employed as marketing strategies for these simplified adhesives.

Water Sorption and Degradation of Adhesive Bonds

Water is regarded by many to be an ubiquitous agent in the degradation of adhesive bonds (Kinloch, 1987; Dickstein & others, 1991; Nguyen, Byrd & Bentz, 1995). Bond degradation is preceded by water sorption into a polymer matrix. Water sorption is enhanced by the presence of hydrophilic and ionic resin monomers (Tanaka & others, 1999), which, in turn, facilitate ion movement within a polymerized resin matrix, The supramolecular structure of glassy polymers is normally not in equilibrium. A non-equilibrium structural state may be preserved for an unlimited period of time if the segmental mobility of macromolecules is "frozen," as exemplified by storing a piece of polymerized adhesive in a dry state below its glass transition temperature (T_g). Such a state is altered on water sorption, as the bound water acts as a plasticizer, reduces the T_g of the polymer, increases the segmental mobility of the polymer chains and "defreezes" the physical process of structural relaxation (Zaikov, Iordanskii & Markin, 1988). Water diffusion in glassy polymer matrices can be divided into three categories (Alfrey, Gurnee & Lloyd, 1996):

- Case I or Fickian diffusion, where the speed of water diffusion is much slower than the speed of polymer segmental relaxation.
- Case II diffusion, where the speed of water diffusion is much faster than with the speed of polymer segmental relaxation.
- Anomalous diffusion or Non-Fickian diffusion, which appears when the speed of water diffusion and polymer segmental relaxation are comparable.

Most water sorption studies with methacrylate resins were performed using neat, non-porous resin monomer blends that were polymerized optimally in the absence of water (Venz & Dickens, 1991; Patel & others, 2001; Hunter & others, 2003; Sideridou, Tserki & Papanastasiou, 2003; Unemori & others, 2003; Mortier & others, 2004). Unlike water transport in non-porous films that is solely controlled by diffusion, water movement may additionally occur through pores, voids and defects in porous films of the polymerized resin matrix of adhesives with hydrophilic and ionic resin monomers. Water transport in porous media is more complicated and can be contributed to simultaneously by a) diffusion, that is driven by a gradient in water concentration, as described by Fick's laws; b) saturated viscous flow, that is driven by a difference in osmotic or

hydrostatic pressure, as described by Darcy's law (Hilfer, 1996) and c) capillary transport, that is driven by gradients in suction stress (Martys & Ferraris, 1997). The latter two components, transport through microcracks and capillarity, have been referred to as damage-dependent mechanisms that enhance water penetration into a polymer or fiber-reinforced composite (Marom, 1985). Thus, a higher permeability and more rapid water movement may be expected in porous films, such as those created by bonding solvated one-step self-etch adhesives on dentin. The water channels present in these resin-dentin bonds may speed up the transport of water and serve as sites for the accumulation of water. It has been shown that moisture can also cause structural damage by inducing micro-cavities or crazing in polymeric materials (Apicella & others, 1979; Diamant, Marom & Broutman, 1981), and this damage can further accelerate moisture diffusion (Brewis, Comyn & Tegg, 1980). Unfortunately, little information is available on the properties of porous dentin adhesive films and how the incorporation of water channels may influence their degradation characteristics; interpretations are often based on bulk properties derived from non-porous neat resins. It is possible that the hydrolytic degradation and leaching of the hydrolyzed components may be expedited in porous dentin adhesive coatings, with the associated increase in permeability creating a vicious cycle that increases the deterioration of mechanical properties by manifold when compared with non-porous coatings produced from the same resin mixtures. These questions may be answered in the future by comparing the impedance properties of porous *vs* non-porous coatings using electrochemical impedance spectroscopy (Senkevich, 2000; Pradelle-Plasse & others, 2004; Davies, Rich & Drzal, 2004).

Although absorbed fluids may plasticize and induce relaxation in the adhesive polymer and swell the adhesive, leading to a loss of mechanical properties, degradation of the interface is the primary reason for failure of many adhesive joints (Kinloch, 1979). The diffusion of water into adhesive joints created with an epoxy adhesive was studied by comparing the calculated diffusion rates between non-bonded adhesive specimens and bonded adhesive joints (Zanni-Deffarges & Shanahan, 1995). The authors observed that the diffusion coefficient of the adhesive joint was greater than that of the bulk adhesive and concluded that the diffusion rate at the interface was greater than in the bulk adhesive. Nguyen and others (1997) and Linossier and others (1999) have also compared the diffusion rates of water between bulk specimens and adhesive joints using Fourier transform infrared spectroscopy in the multiple internal reflection mode (FTIR-MIR). They detected significant water diffusion at the interface for poorly adhered adhesive systems where adhesion forces are governed by secondary interactions such as hydrogen bonding and van der Waals forces. Vine, Cawley and Kinloch (2001) studied the moisture uptake of an epoxy bonded to aluminum adherends with various surface treatments. They observed faster diffusion in three-layer sandwich specimens than predicted, based on mass-uptake experiments performed on diffusion in bulk specimens. They attributed this behavior to the presence of micro-cavities in the adhesive layer and similarly suggested that diffusion at the interface is possibly faster than in bulk.

The studies mentioned above were all conducted on epoxy adhesives bonded to non-degrading metallic adherends. Substrate corrosion had not been taken into account in these studies. Similar to substrate corrosion that accompanies water sorption in some adhesive joints (Stevenson, 1987), bonding to dentin is complicated by degradation of the collagenous components within the hybrid layer, an issue that will be addressed below. Nevertheless, the same principle derived from adhesive joints created with epoxy resins may be extrapolated to thin dentin adhesive films that are bonded to dentin. The degradation of dentin adhesive joints can occur in the absence of water trees, but it is likely that degradation will be expedited in the presence of additional water channels, given the more rapid water sorption and leaching that may occur in their presence. As water trees are predominantly identified from the interface between simplified adhesives and the dentin hybrid layer, it is not unreasonable to expect that such an interface is the most susceptible to degradation (Figure 7A) and that interfacial adhesive failure is the predominant failure mode after aging in an aqueous environment (Bowditch, 1996). Indeed, a recent study reported premature bond failures in almost all specimens bonded with a one-step self-etch adhesive after 15 months of water storage (Armstrong & others, 2003). The unfavorable *in vitro* results associated with this adhesive were also supported by *in vivo* results obtained in a clinical trial (Brackett, Covey & St Germain, 2002).

Post-aging Water Treeing

So far, the discussion has been limited to water trees that were formed immediately during bonding to dentin. As water treeing was hypothesized to be one of the mechanisms that contribute to resin-dentin bond degradation (Tay & Pashley, 2003b), the question remains whether water trees can arise in interfaces that are initially free of water trees during the aging of adhesive interfaces in water. Using an experimental single-bottle etch-and-rinse adhesive, Tay and others (2003c) demonstrated that both the reticular and spot-type of nanoleakage patterns increased in adhesive interfaces after one year of aging in artificial saliva. In particular, interfacial changes occurred between the adhesive and hybrid layer that the authors attributed

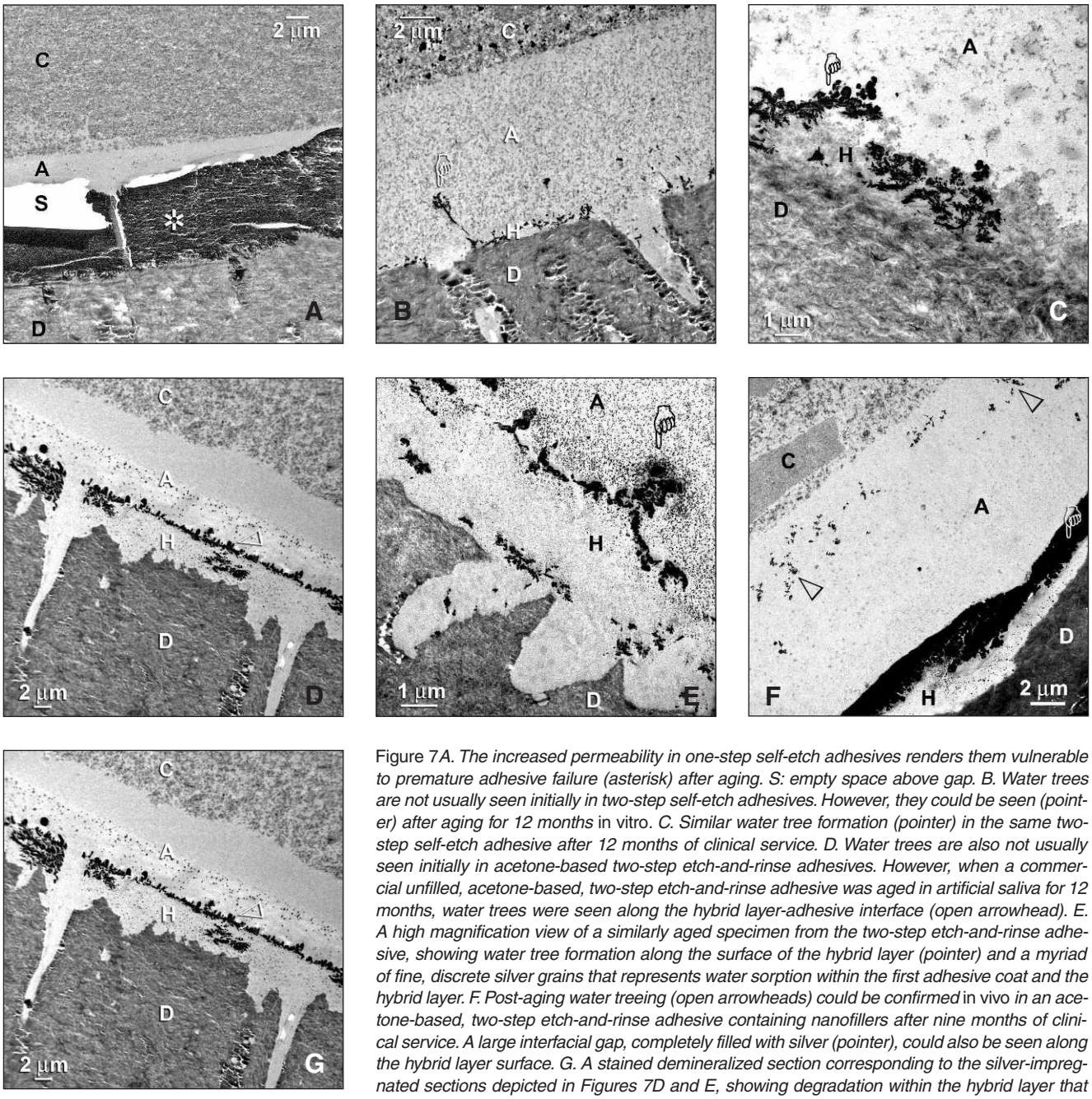

Figure 7*A. The increased permeability in one-step self-etch adhesives renders them vulnerable to premature adhesive failure (asterisk) after aging. S: empty space above gap. B. Water trees are not usually seen initially in two-step self-etch adhesives. However, they could be seen (pointer) after aging for 12 months* in vitro. *C. Similar water tree formation (pointer) in the same two-step self-etch adhesive after 12 months of clinical service. D. Water trees are also not usually seen initially in acetone-based two-step etch-and-rinse adhesives. However, when a commercial unfilled, acetone-based, two-step etch-and-rinse adhesive was aged in artificial saliva for 12 months, water trees were seen along the hybrid layer-adhesive interface (open arrowhead). E. A high magnification view of a similarly aged specimen from the two-step etch-and-rinse adhesive, showing water tree formation along the surface of the hybrid layer (pointer) and a myriad of fine, discrete silver grains that represents water sorption within the first adhesive coat and the hybrid layer. F. Post-aging water treeing (open arrowheads) could be confirmed* in vivo *in an acetone-based, two-step etch-and-rinse adhesive containing nanofillers after nine months of clinical service. A large interfacial gap, completely filled with silver (pointer), could also be seen along the hybrid layer surface. G. A stained demineralized section corresponding to the silver-impregnated sections depicted in Figures 7D and E, showing degradation within the hybrid layer that was manifested as non-stainable patches (arrows).*

as evidence of post-aging water-tree formation. Such a hypothesis has since been confirmed both *in vitro* (Figure 7B) and *in vivo* (Figure 7C) in human teeth bonded with two-step self-etch adhesives that do not demonstrate initial water-tree formation (Donmez & others, 2005). Even more convincing data on post-aging water treeing than that previously reported by Tay and others (2003c) has since been collected by the authors, using an unfilled, two-step etch-and-rinse adhesive (Figure 7D). In this group of specimens, it is significant to point out that only the adhesive coat adjacent to the bonded dentin demonstrated silver uptake (Figure 7E), while the second coat was not affected. The results could be duplicated *in vivo* with the use of a two-step etch-and-rinse adhesive containing nanofillers after nine months of clinical service (Figure 7F). In this adhesive, only one adhesive coat was applied, and post-aging water-tree formation was apparent on the top part of the adhesive layer, while those that were present along the surface of the hybrid layer were probably obscured by the thick layer of silver that represented frank gap formation. An important lesson that may be learned from these results is that, contrary to the recommendations of some manu-

facturers, simplified adhesives are best utilized in more than one coat (Hashimoto & others, 2004c), with the first coat functioning as a dentin primer, and the second coat as a coupling resin. Even so, substantial post-aging water treeing could be identified along the junction of the hybrid layer and the first adhesive coat.

As mentioned earlier, the degradation of resin-dentin bonds is more complex than non-biological adhesive joints in that hydrolytic degradation of the collagenous component occurs simultaneously, as resin leaches out of these adhesive joints (Hashimoto & others, 2003a,b). Figure 7G represents a stained, demineralized section from the same tooth as Figures 7D and 7E after one year of water storage. In this stained section, water treeing and nanoleakage could not be seen, as the section was not previously subjected to silver tracer penetration, with the first adhesive coat appearing "deceptively normal" with this type of stained sections. However, the section did reveal additional important features that could not be seen in silver-impregnated, unstained, undemineralized sections—the partial disappearance of stained collagen fibrils from the hybrid layer. Such a phenomenon has been reported by De Munck and others (2003) and Armstrong and others (2004). Although not explicitly mentioned by these authors, the absence of stainable fibrillar components within the aged hybrid layers suggested the breakdown of the collagen fibrils into gelatin (Hashimoto & others, 2003b) that is still stainable as individual unraveled strands, and further into peptides that no longer take up these stains. Such a phenomenon is analogous to the appearance of clear bands in Coomassie blue-stained gelatin gels, when Western blots due to the gelatinase activity of matrix metalloproteinases (MMPs), such as MMP-2 or MMP-9 (Gendron & others, 1999; Smith & others, 2004) are used. It is now understood that the breakdown of denuded collagen fibrils within dentin hybrid layers can occur via the release and activation of endogenous matrix metalloproteinases in the demineralized layer or they can be released from the underlying mineralized dentin (Pashley & others, 2004). Using fluorescein-labeled type I collagen and gelatin, low but consistent collagenolytic and gelatinolytic activities have been recently been observed from powdered mineralized human dentin (Pashley & others, 2004).

In its original context, nanoleakage was defined as the manifestation of leakage within a hybrid layer in the absence of interfacial gap formation. However, the lateral communication of bonded interfaces with the external environment (storage solution *in vitro* or saliva *in vivo*) created by post-aging interfacial water treeing may generate an additional dimension wherein leaching of hydrolytic resin and collagen components can rapidly occur. Thus, in the broadest sense, the development of post-aging water treeing along surface asperities of the resin-dentin interface may be viewed upon as an antecedent that slowly creates gap formation upon fatigue and loading stresses (Fernando, Harjoprayitno & Kinloch, 1996) and ultimate catastrophic failure of the dentin-adhesive joint.

CONCLUSION

All these examples of water treeing and their potential consequences depicted in simplified adhesives, especially the latest versions of single-bottle one-step self-etch adhesives, may be regarded as fantasy by some, or discomforting to others. Then again, they may be not—for there are antecedents, as such concerns have been well articulated in other branches of adhesive sciences such as the paint industry, photoresists and more. In the most recent review on the durability of adhesion to tooth tissues, De Munck and others (2005b) remarked that of all the classes of dentin adhesives available, the three-step etch-and-rinse adhesives remain the gold standard in terms of durability, and that only the two-step self-etch adhesives approach this gold standard. Water treeing is a morphologic phenomenon that may help us understand the initial problems associated with the bonding of simplified adhesive systems and the underlying causes of their relative lack of durability. As we approach the Golden Jubilee of enamel etching in 2005, let us double our efforts to improving adhesive dentistry to continue the legacy of the late Dr Michael Buonocore by not simply producing faster and more user-friendly adhesives, but towards improving the quality of resin bonds created in dentin.

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Clinical Research

Clinical Evaluation of a Flowable Resin Composite and Flowable Compomer for Preventive Resin Restorations

M Qin • HS Liu

Clinical Relevance

Flowable resin composite and flowable compomer can be used for preventive resin restorations. The repair should be performed immediately, in case the preventive resin restoration develops a fracture or loss.

SUMMARY

This clinical study evaluated the retention and caries protection of a flowable resin composite (Flow Line) and a flowable compomer (Dyract Flow) used in preventive resin restorations as compared to the conventional preventive resin technique which uses a resin composite (Brilliant) and a sealant (Concise). This study observed 205 permanent molars with small carious cavities less than 1.5 mm in width, which were obtained from 165 children aged 7 to 15 years. Flowable resin composite was used to treat 75 teeth, and 71 teeth were treated with flowable compomer in both cavities and caries-free fissures. For the control group, 59 teeth were treated with resin composite in cavities and sealant in caries-free fissures. The teeth were evaluated at 3, 6, 12, 18 and 24-month intervals. After three months, all 205 treated teeth were completely intact. After six months, 66 of the 71 teeth treated with flowable resin composite and 65 of the 70 teeth treated with flowable compomer were complete, compared to 57 of the 58 teeth treated with the conventional preventive resin technique. After 12 months, 60 of the 67 teeth treated with flowable resin composite and 61 of the 67 teeth treated with flowable compomer were complete, compared to 51 of the 55 teeth treated with the conventional preventive resin technique. After 18 months, 53 of the 61 teeth treated with flowable resin composite and 54 of the 62 teeth treated with flowable compomer were complete, compared to 47 of the 53 teeth treated with the conventional preventive resin technique. After 24 months, 49 of the 58 teeth treated with flowable resin composite and 45 of the 57 teeth treated with flowable compomer were complete, compared to 42 of the 52 teeth treated with the conventional preventive resin technique. There were no statistically significant differences in retention rates among all

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groups after 3, 6, 12, 18 or 24-months ($p>0.05$). One tooth treated with flowable resin composite and one tooth treated with flowable compomer developed caries after 18 and 24 months, respectively, resulting from partial loss at "caries-free fissures." Five teeth developed caries in the conventional preventive resin group; one after 12 months, two after 18 months and one after 24 months, due to loss at cavities. The final caries occurred after 24 months, resulting from partial loss at "caries-free fissures." The differences in caries development among the three groups were not statistically significant ($p>0.05$). This study suggested that flowable resin composite and flowable compomer could be used for preventive resin restorations. Meanwhile, a vigilant recall should be followed-up due to the risk of failure for flowable materials in "caries-free" fissures. The repair should be performed immediately, in case the preventive resin restoration develops a fracture or loss.

INTRODUCTION

The preventive extension of cavities in the treatment of occlusal caries for permanent molars in children has been in use for some time. According to preventative extension, all pits and fissures are eradicated with a bur when the tooth is prepared in order to ease the placement of amalgam. This means that some non-carious tooth structure is sacrificed during placement. Fortunately, a better understanding of the caries process and remineralization have catalyzed the evolution in caries management from GV Black's "extension for prevention" to "minimally invasive" (Murdoch-Kinch & McLean, 2003). Simonsen (1978a) described a minimally invasive preparation and restoration, which he named the preventive resin restoration. This preparation only removed carious pits and fissures, using small burs, with tooth removal barely reaching into dentin; in some cases, only enamel was removed. The tooth was restored using an adhesive technique with a highly filled resin composite for the prepared pits and fissures, covering the remaining pits and fissures with a sealant (Simonsen, 1985). One problem with the preventive resin technique was that it required the use of two different materials.

From the time that a flowable resin composite was first described by Ibsen (1972) for use in restoring cervical erosion, flowable resin composites have been used to solve clinical problems, often in situations where no specific material had previously served. These materials have been used as the repair material for non-carious amalgam margin defects (Roberts, Charlton & Murchison, 2001), as stress-relieving gingival increments in Class II restorations (Malmstrom & others, 2002; Estafan, Estafan & Leinfelder, 2000), as pit and fissure sealants (Autio-Gold, 2002) or to bond together chipped teeth for a long-term temporary emergency (Small, 1996).

Flowable materials are a modification of restorative resins; thus, they tend to contain lower filler content and more of a resin matrix. Flowable composites are easier to place and more self-adaptable compared to conventional restorative resin composites. These flowable materials have a viscosity that allows them to be used for minimally invasive preparations and also as a sealant for the untouched part of the occlusal surface. When flowable resin composites were first introduced, they appeared to be a one-dimensional restorative material used in preventive resin restorations. Flowable compomers are polyacid-modified resin composites that possess the characteristics of both flowable composites and glass ionomers. Flowable compomers claim to improve the adhesive and fluoride-releasing properties of conventional glass ionomer cements, while also retaining the esthetic properties of conventional glass ionomer cements. As preventive resin restorations have evolved, flowable resin composites and flowable compomers have become a logical choice for restoring these lesions.

This clinical study evaluated the retention and caries protection of a flowable resin composite (Flow Line, Heraeus Kulzer GmbH, Wehrhein, Germany) and a flowable compomer (Dyract Flow, Dentsply Inc, Milford, DE, USA) for preventive resin restorations compared to the conventional preventive resin technique (Simonsen 1978b).

METHODS AND MATERIALS

The subjects were recruited from patients seeking routine dental care at the Department of Pediatric Dentistry, Stomatological Hospital, Peking University between 2000 and 2001. The subjects selected for this study included 165 children, 79 boys and 86 girls, aged 7 to 15 years, with an average age of 10 years and 5 months with 205 permanent molars containing small carious lesions. The procedures and potential discomforts, risks and benefits were explained to the parents, and their informed consent was obtained.

The width of the small carious cavities was limited to 1.5 mm, controlled by a small round bur (No 2 round bur, IOS #010, Thomas, FFDM Pneumat, Bourges Cedex, France), with no limitation on depth. The restoration preparation only removed the carious lesions using small burs (No 1/2 round bar, ISO #006; No 1 round bur, ISO #008 and No 2 round bur ISO #010, Thomas, FFDM Pneumat). The carious cavities extended no further than the medium third of the dentin. The cavities were recorded on a "sketch map" of occlusion surfaces in order to be re-checked easily during the follow-up examination. The teeth were etched for 30 seconds with a 20% phosphoric acid etchant (Heraeus

Table 1: *Number of Restoration Reviewed and Recall Rate Percentages*

	Baseline	Three Months	Six Months	12-Months	18-Months	24-Months
Flowable Composite Resin Group	75	75(100%)	71(94.7%)	67(89.3%)	61(81.3%)	58(77.3%)
Flowable Compomer Group	71	71(100%)	70(98.6%)	67(94.4%)	62(87.3%)	57(80.3%)
Conventional PRR Group	59	59(100%)	58(98.3%)	55(93.2%)	53(89.8%)	52(88.1%)
Total Teeth	205	205(100%)	199(97.1%)	189(92.2%)	176(85.9%)	167(81.5%)

Table 2: *Distribution of Retention Rates and Recurrent Caries Rates*

Groups	Flowable Composite Resin Group					Flowable Compomer Group					Conventional PRR Group				
Score	0	1	2	3	4	0	1	2	3	4	0	1	2	3	4
3-month	75 100%					71 100%					59 100%				
6-month	66 93.0%	5 7.0%				65 92.9%	5 7.1%				57 98.3%		1 1.6%		
12-month	60 89.9%	7 10.1%				61 91.0%	6 9.0%				51 92.7%		3 5.5%		1 1.8%
18-month	53 86.9%	7 11.6%		1 1.5%		54 87.1%	8 12.9%				47 88.7%	1 1.9%	3 5.7%		2 3.8%
24-month	49 84.5%	7 12.1%	2 3.4%			45 78.9%	8 14.0%	3 5.3%	1 1.8%		42 80.8%	3 5.8%	5 9.6%	1 1.9%	1 1.9%

0 - intact restoration
1 - complete restoration at the cavity with fracture or loss at the caries-free fissure, but no caries development
2 - incomplete restoration at the cavity but no caries development
3 - complete restoration at the cavity with a fracture or loss at the caries-free fissure with caries development
4 - incomplete restoration at the cavity with caries development

Kulzer GmbH, Wehrhein, Germany), then rinsed for 20 seconds with an air-water spray and dried, leaving the dentin slightly moist. In the conventional preventive resin group, the cavities were treated with resin adhesive (Single Bond Adhesive, 3M Dental Products, St Paul, MN, USA) and polymerized for 20 seconds using a curing light (Spectrum, Dentsply Inc, Milford, DE, USA) with an output intensity $\geq 420 mW/cm^2$. The preparations were then restored with a micro-hybrid resin composite (Brilliant, Coltène/Whaledent Inc, Altstatten, Switzerland) and polymerized for 40 seconds. The remaining caries-free pits and fissures were re-dried for a few seconds, until the surface was chalky white and covered with a sealant (Concise, 3M Dental Products) (Figure 2). In the flowable resin composite and flowable compomer groups, all pits and fissures were coated with Single Bond adhesive and polymerized for 20 seconds. Following the coating with adhesive, the flowable resin composite (Flow Line) and flowable compomer (Dyract Flow) were dispensed carefully, avoiding air bubbles by using the needle provided by the manufacturer, and polymerized for 40 seconds (Figure 1). The occlusion was adjusted in the three groups. Careful moisture control was maintained by using accepted cotton-roll-isolation procedures and a chair-side assistant.

Two hundred and five teeth from 165 children were treated at baseline. After three months, 165 children with 205 teeth were available for evaluation. After six months, 160 children with 199 teeth were evaluated; after 12 months, 151 children with 189 teeth were evaluated; after 18 months, 139 children with 176 teeth were evaluated and after 24 months, 130 children with 167 teeth were available for evaluation (Table 1). Further evaluations were not performed, due to the excessive dropout rate. The primary reasons for dropout included family relocation or refusal to continue in the study.

The examination for restoration retention rate and caries protection were conducted using the following criteria:

0—intact restoration.

1—complete restoration at the cavity, with fracture or loss at the caries-free fissure, but no caries development.

2—incomplete restoration at the cavity but no caries development.

3—complete restoration at the cavity, with a fracture or loss at the caries-free fissure with caries development.

4—incomplete restoration at the cavity, with caries development.

The retention, condition of the restorations and caries were evaluated with a dental explorer under visual inspection. Incomplete restorations were not reapplied between examinations until the definition of caries was met.

A contingency table and a mixed model were used for data analysis. Fisher's Exact Test was used to compare the types of materials for retention rates and caries increments.

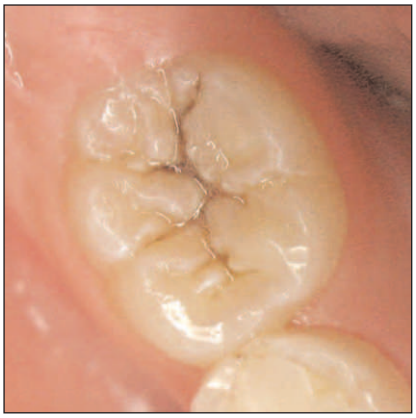

Figure 1. *The clinical process of preventive resin restoration with flowable materials.* Figure 1A: *A mandibular first permanent molar with small caries lesion in occlusal fissure.*

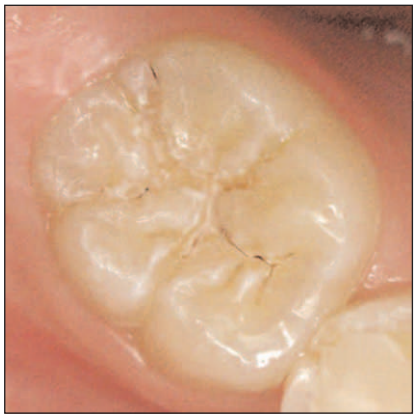

Figure 1B. *The restoration preparation only removes carious lesion without any extension into the surrounding healthy tooth structure. The width of the cavity is limited to 1.5 mm, with no limitation on length.*

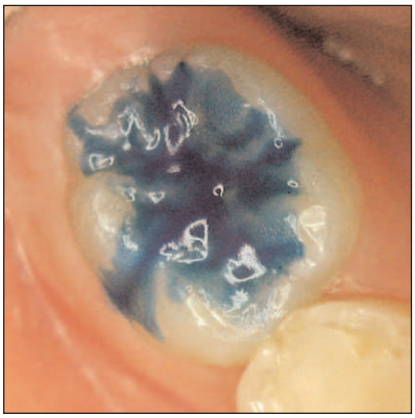

Figure 1C. *The tooth is etched for 30 seconds with 20% phosphoric acid etchant.*

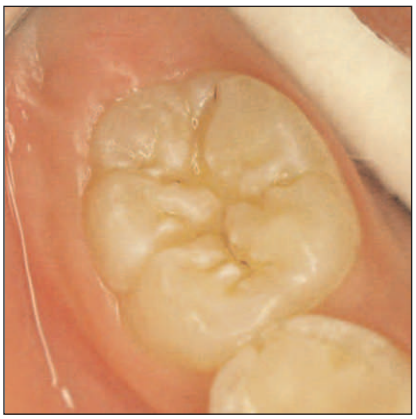

Figure 1D. *The tooth is rinsed for 20 seconds with an air-water spray and dried, leaving the dentin slightly moist.*

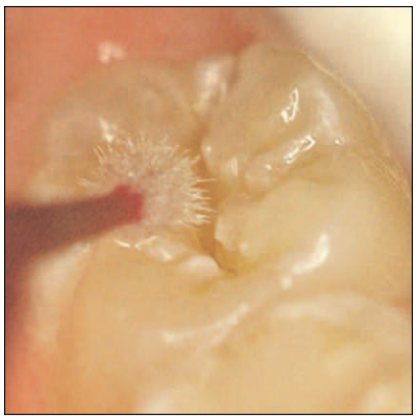

Figure 1E. *The cavity and caries-free pits and fissures are treated with Single Bond Adhesive.*

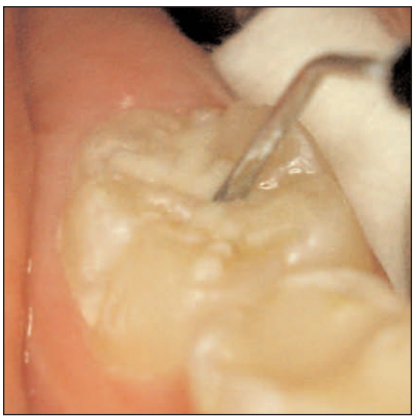

Figure 1F. *The flowable material is dispensed carefully, avoiding air bubbles by using the needle provided by the manufacturer.*

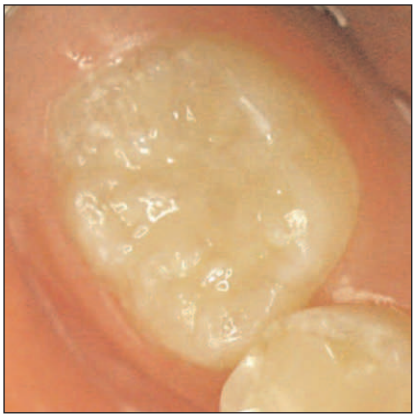

Figure 1G. *The preventive resin restoration with flowable compomer is performed on the mandibular first permanent molar.*

RESULTS

The distribution of retention and caries development after 3, 6, 12, 18 and 24 months is shown in Table 2. After three months, all 205 treated teeth were completely intact. After six months, 66 of the 71 teeth treated with flowable resin composite and 65 of the 70 teeth treated with flowable compomer were complete compared to 57 of the 58 teeth treated with the conventional preventive resin technique. After 12 months, 60 of the 67 teeth treated with flowable resin composite and 61 of the 67 teeth treated with flowable compomer were complete compared to 51 of the 55 teeth treated with the conventional preventive resin technique. After 18 months, 53 of the 61 teeth treated with flowable resin composite and 54 of the 62 teeth treated with flowable compomer were complete compared to 47 of

the 53 teeth treated with the conventional preventive resin technique. After 24 months, 49 of the 58 teeth treated with flowable resin composite and 45 of the 57 teeth treated with flowable compomer were complete compared to 42 of the 52 teeth treated with the conventional preventive resin technique. There were no statistically significant differences in retention rates among these three groups after 3, 6, 12, 18 or 24-months ($p>0.05$). One tooth treated with flowable resin composite and one tooth treated with flowable compomer developed caries after 18 and 24 months, respectively, resulting from partial loss of restorations. Five teeth developed caries in the conventional preventive resin group; one after 12 months, two after 18 months and one after 24 months, due to complete loss. The final caries developed after 24 months from partial loss of restorations. The differences in caries development among the three groups were not statistically significant ($p>0.05$).

DISCUSSION

This clinical study evaluated the retention of a flowable composite and a flowable compomer in preventive resin restorations and compared them to the conventional preventive resin technique. In the first three months, all of the restorations were intact in the three groups. Although the retention rate showed no statistically significant differences among these three groups after 6, 12, 18 or 24 months, the locations of the early loss of flowable resin composite and flowable compomer for preventive resin restorations showed different characteristics compared to the conventional preventive resin technique. The restoration fracture or loss was frequently found in "caries-free" fissures in those teeth treated with flowable materials as compared to the cavities treated with the conventional preventive resin technique.

Generally, flowable resin composite and flowable compomer are not suggested for use as restoration materials in occlusal molar caries, because of their weak resistance to abrasion and pressure. In this study, abrasion and pressure resistance were not important factors compared to retention and marginal adaptation, because the restorations were in narrow cavities within 1.5 mm width. The small, narrow preparations were the ideal size for using flowable resin composite and flowable compomer as compared to the general conventional resin composite. The flowable resin composite and compomer were easily applied with pinpoint accuracy and were self-smoothing once placed, allowing for good marginal adaptation. Restoration surface characteristics are also important when evaluating flowable materials, because a smooth surface and smooth margins contribute to plaque and caries resistance. Strassler and Goodman (2002) introduced the use of a flowable resin composite for the preventive resin restoration technique and provided a five-year follow-up case report with good clinical results. In this study, the similar technique of filling small preparations was performed with flowable resin composite and flowable compomer. After 24-months, 56 of 58 (96.55%) of the flowable resin composites and 53 of 57 (93.00%) of the flowable compomers in cavities were still complete, with no definition of caries present, compared to a completion of 45 of 52 (86.50%) for conventional composite in cavities, while four caries developed in the conventional preventive resin technique. Although flowable resin composite and flowable compomer show seemingly better retention in cavities than conventional composite, the differences were not statistically significant ($p>0.05$). It is necessary to undergo a longer-term clinical evaluation for the use of flowable materials in small carious cavities on the occlusal surface. However, clinical trials with children are difficult to perform, since follow-up depends on the parents' motivation to bring a child to the scheduled appointments. This study was concluded after 24 months due to an excessive dropout rate.

The objective of a preventive resin restoration is to fill the small carious lesion in occlusal surfaces and protect the "caries-free" fissure. In the current study, Flow Line, a flowable resin composite with medium-filled (41% volume of filler) and 0.7-mm average particle size, and Dyract Flow, a flowable compomer with a similar average particle size as Flow Line, were evaluated for preventive resin restorations. The two flowable materials did not provide a retention rate that was comparable to the unfilled sealant (Concise) in "caries-free" fissures. This is why restoration fracture or loss was found frequently in "caries-free" fissures in the teeth treated with flowable materials as compared to those cavities treated with the conventional preventive resin technique in the study.

Although flowable materials have greater microhardness values than a classical sealant, the point of using flowable materials as a sealant is because of their penetration and bond strength to fissure. Duangthip and Lussi (2003) reported an *in vitro* study which investigated the factors that could influence macroleakage, penetration ability and the formation of resin tags of different sealants. Their study indicated that classical sealants showed significantly lower microleakage than flowable composites and flowable compomers, and that agitating the etchant could improve the quality of sealing by significantly decreasing microleakage. Frankenberger and others (2002) reported an *in vitro* study that evaluated the use of flowable composites of different viscosities on bonding to enamel and dentin without the use of an intermediate bonding resin. Their study indicated that flowable composites could not bond to enamel and dentin effectively since many areas of the resin-dentin interface showed insufficient penetration of the flowable composition at the top of the hybrid

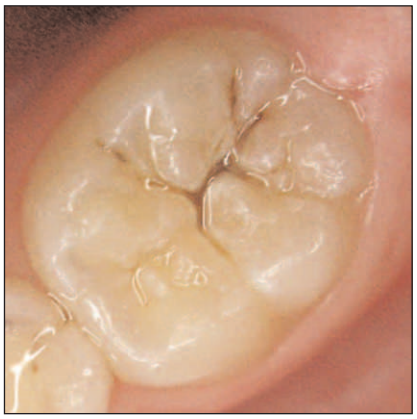

Figure 2. *The clinical process of preventive resin restoration with the conventional technique.* Figure 2A*: A mandibular first permanent molar with small caries lesion in occlusal fissure.*

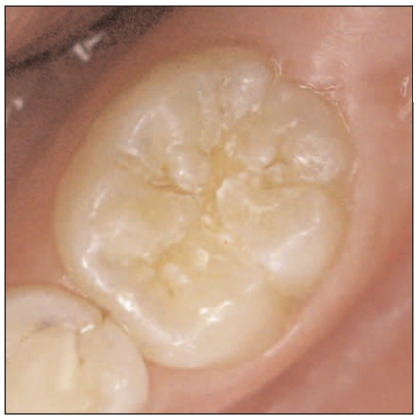

Figure 2B. *The restoration preparation only removes carious lesion without any extension into the surrounding healthy tooth structure.*

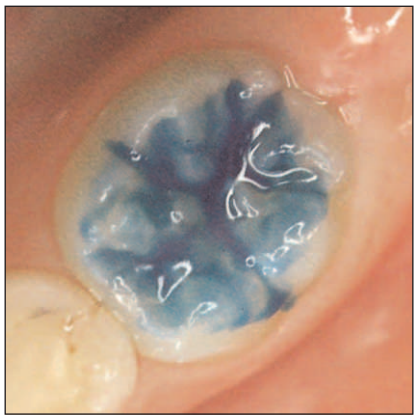

Figure 2C. *The tooth is etched for 30 seconds with 20% phosphoric acid etchant.*

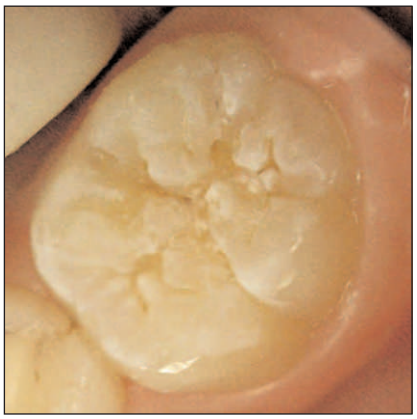

Figure 2D. *The tooth is rinsed for 20 seconds with an air-water spray and dried, leaving the dentin slightly moist.*

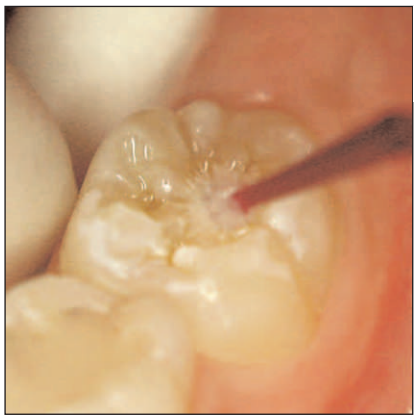

Figure 2E. *The cavity is coated with Single Bond Adhesive.*

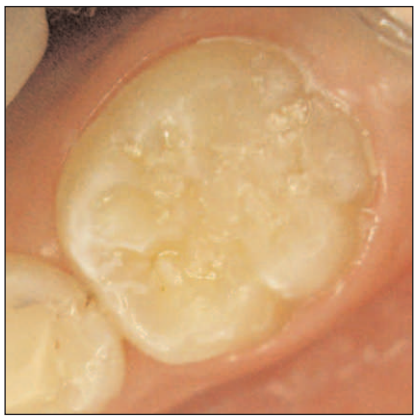

Figure 2F. *The cavity is restored with a micro-hybrid resin composite.*

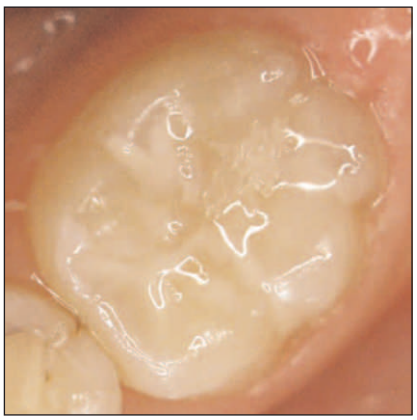

Figure 2G. *The remaining caries-free pits and fissures are re-dried for a few seconds, until the surface is chalky white, and covered with a sealant.*

layer as well as numerous tubules obstructed by filler particles. Autio-Gold (2002), in a clinical study, reported that no statistically significant differences existed in retention rate and caries increment between a medium-filled flowable resin composite (46% volume of filler) without a bonding agent and a general, unfilled sealant used to control occlusal caries on permanent molars during an 18-month clinical evaluation. A bonding agent to improve the retention rate of medium-filled materials was suggested. A clinical study by Feigal and others (2000) showed that single-bottle bonding agents improve sealant retention as compared to two-bottle bonding agents. In this study, Single Bond adhesive was used in both cavities and caries-free fissures for the two flowable materials.

The retention rates of flowable resin composite and flowable compomer in caries-free fissures were lower than for the unfilled sealant without bond in the conventional preventive resin technique. The flowable

resin composite and flowable compomer with partial loss showed some slight steps or ledges when probed. However, caries protection was present, regardless of the materials used. The early loss was believed to result from fractures caused by a failure to penetrate deep into the fissure and an adhesion failure between the flowable material and tooth enamel, rather than a wearing away of the material (Duangthip & Lussi, 2003). Mechanical preparation and air abrasion are suggested as providing better access to deeper fissures, thus enabling removal of debris and deeper sealant penetration (Feldens & others, 1994; Berry, Eakle & Summitt, 1999; Hamilton & others, 2001; Murdoch-Kinch & McLean, 2003). Further studies should evaluate the retention of a flowable resin composite and a flowable compomer in minimally invasive preparations for "caries-free" fissures.

Fluoride acts in several ways to prevent caries; it inhibits demineralization and promotes remineralization, thus, encouraging the repair or arrest of carious lesions. Compomers were developed to overcome the problems of conventional glass ionomer cement, which include early, low mechanical strengths and poor esthetics resulting from moisture sensitivity. In studies by Yap and others (2002) and Shaw, Carrick and McCabe (1998), a compomer did not have the initial fluoride "burst" effect that is associated with glass ionomer cements, and could not be replenished with fluoride. As the long-term fluoride release of compomer is questionable, it may not be as beneficial as glass ionomer cements for caries resistance. Morphis, Toumba and Lygidakis (2000) indicated that one could not expect an anti-caries advantage due to the fluoride in the product, though the addition of fluoride to sealant materials chemistry had no detrimental effect on retention. In this study, the fluoride releasing flowable resin composite (Flow Line) and flowable compomer (Dyract Flow) were evaluated for caries protection and showed similar caries resistance. Caries development resulted from the partial loss of flowable materials in "caries-free" fissures. No caries development was found in cavities in both the flowable resin composite group and flowable compomer group, because of the intact restorations. In the conventional group, four teeth developed caries due to the loss in cavities. For total caries resistance, both in "caries-free" fissures and in cavities, the flowable materials showed slightly better results than the conventional preventive resin technique due to their good retention in cavities, though the difference was not statistically significant. This study supports the earlier findings that a good retention rate could contribute to more caries resistance in preventive resin restoration treatment.

The influence of tooth isolation on retention rates has been considered in pediatric dental clinical processes. In a study by Ganss, Klimek and Gleim (1999), placement using a rubber dam resulted in significantly higher retention rates and improved sealant quality. However, some clinical studies evaluating the influence of the method of moisture control on restoration quality or adhesion revealed contradictory results (Barghi, Knight & Berry, 1991; Heringer, Almeida & Miguel, 1993; Knight & others, 1993; Smales, 1993). Many studies show that the retention rates of sealants and resin composite restorations are comparable when performed using rubber dam or cotton-roll isolation (Straffon, Dennison & Moore, 1985; Waggoner & Siegal, 1996). Autio-Gold (2002) reported the use of cotton-roll isolation and a chairside assistant in the clinical evaluation of a medium-filled flowable restorative material as a pit and fissure sealant. A similar method was used in this study. The teeth were placed in a dry field condition with cotton-roll isolation and a chairside assistant was used. No saliva contamination was observed. The etched surfaces in the three groups were made slightly moist when placing the bond adhesive. The surfaces were re-dried for a few seconds until the surface was chalky white before covering the sealant in the conventional preventive resin group.

The previous review of sealant clinical trials by Feigal (1998) showed a failure rate (judged by sealants needing repair, replacement or restoration) to be between 5% and 10% each year. In this study, one of seven losses of flowable resin composite restorations in "caries-free" fissures developed caries after 12 months, one of eight losses of flowable compomer in "caries-free" fissures developed caries after 18 months and one of three sealant losses of the conventional preventive resin technique group developed caries after six months. This study supported the earlier finding by Dennsion, Straffon and Smith (2000) that the loss of coverage of any susceptible pit and fissure leads to an immediate risk of caries attack for the uncovered area. Furthermore, the highest risk of restoration loss and cavity formation occurred in the conventional preventive resin technique group, since four of five losses in that group developed caries. This result suggested the necessity of a vigilant recall after the placement of a preventive resin restoration, because the tooth that received the treatment was highly sensitive to caries. The repair should be performed immediately, in case the preventive resin restoration develops fracture or loss.

CONCLUSIONS

Flowable resin composite and flowable compomer had similar retention in preventive resin restorations when compared to conventional composites used in the preventive resin technique. The restoration fracture or loss of flowable materials occurred frequently in "caries-free" fissures when compared to the conventional preventive resin technique in prepared lesions. Caries pro-

tection was not significantly different between the two flowable materials. Caries protection of the two flowable materials was slightly better than the conventional preventive resin technique due to its good retention in cavities, but the differences were not statistically significant.

This study suggested that flowable resin composites and flowable compomers can be used for preventive resin restorations. Meanwhile, a vigilant recall should be followed, because of the risk of failure of flowable materials in "caries-free" fissures. Repair should be performed immediately in case the preventive resin restoration develops a fracture or loss.

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A Clinical Evaluation of Bleaching Using Whitening Wraps and Strips

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Clinical Relevance

When used twice daily, Ranir Whitening Wraps were more effective in lightening teeth than Crest Whitestrips Premium when also used twice a day.

SUMMARY

This study evaluated the degree of color change of teeth and the sensitivities of teeth and gums in an *in vivo* study. Ranir Whitening Wraps (WW2) and Crest Whitestrips Premium (WP2) were used twice a day and Ranir Whitening Wraps (WW1) were used once a day. Color evaluations occurred at baseline, after five and seven-day use of bleaching agent and 14 days post-bleaching. Color change was evaluated objectively and subjectively. Sensitivity evaluations were also accomplished. Seventy-six of the 78 subjects enrolled completed the study. All three products significantly lightened teeth. WW2 lightened more than WP2 and WW1 in L*, a*, b*, E and shade guide value. WP2 lightened more than WW1 in a*, b*, E and shade guide value. There was no difference in tooth sensitivity, but WW1 and WP2 caused less gingival sensitivity than WW2. The mean age of smokers was seven years younger than non-smokers who qualified.

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INTRODUCTION

Cosmetic dentistry is a very important part of today's restorative dental practice. The appearance of teeth, health and fitness are increasingly important for patients of all ages. Cosmetic procedures have become more available for a majority of society, since standards of living have improved.

Dentistry has succeeded in reducing the frequency and severity of caries and periodontal disease, leading to the increased preservation of natural teeth throughout life. Since white teeth are believed to be associated with health and beauty, lighter colored teeth have become desirable and popular (Dunn, Murchison & Broome, 1996). Therefore, it is up to our profession to offer techniques and expertise that enables patients to safely achieve these goals. Vital tooth bleaching can be performed with a high rate of success and is a more

conservative treatment for discolored teeth compared to restorative treatment such as porcelain veneers, crowns or composite bonding (Christensen, 2002).

Today, patients have the choice of having tooth bleaching done in two ways: in-office or at-home. In-office vital tooth bleaching has been used for many years in dentistry (Barghi, 1998). It lightens teeth rapidly, but the procedure is more time-consuming for the dentist, costs more for patients and the degree of tooth whitening is usually less than that accomplished with at-home tooth whitening agents used in trays.

At-home bleaching has traditionally involved the use of trays loaded with bleaching gel. However, new products have been introduced that use a different approach (Li & others, 2003). These products are wraps and strips, impregnated with tooth whitening agent, which patients can place against the facial surfaces of their teeth.

This study evaluated the ability of bleaching wraps and strips to lighten the color of teeth using three different methods. The study was also designed to evaluate sensitivities associated with bleaching methods.

METHODS AND MATERIALS

Before participating in this bleaching study, subjects signed a consent form. The form and the research protocol were approved by the Institutional Review Board at Indiana University-Purdue University Indianapolis (IUPUI).

The following criteria was used in subject recruitment and enrollment in the study:

Inclusion Criteria

1. Have all six maxillary anterior teeth.

2. Have no maxillary anterior teeth with more than 1/6 of the labial surface of the natural tooth covered with a restoration.

3. Be willing to sign a consent form.

4. Be at least 18 years of age.

5. Be able to return for periodic examinations.

6. Have maxillary anterior teeth that are between B-56 and D-85 shades on the Trubyte Bioform Color Ordered Shade Guide (Dentsply Int, York PA, USA).

Exclusion Criteria

1. History of any medical disease that may interfere with the study or require special considerations.

2. Current or previous use of professionally applied or prescribed "in-office" or "at-home" bleaching agents.

3. Gross pathology in the oral cavity (excluding caries).

4. Pregnant or lactating women.

5. Tetracycline-stained teeth.

If the patient met the criterion, a baseline appointment was made for the subject. At that appointment color evaluation was performed using two methods: 1) subjective shade guide matching of maxillary anterior teeth with Trubyte Bioform Color Ordered Shade Guide (Dentsply Int) and 2) by using an objective color measuring device (ShadeEye, Shofu, Inc, Kyoto, Japan). The authors constructed an intraoral jig to ensure the objective color-measuring device measured the same area for each subject at all evaluation appointments.

The colorimeter measures the color of teeth based on the CIE L*a*b* color space system. This system was defined by the International Commission on Illumination (1978) and is referred to as CIELAB. The L* represents the value (lightness or darkness), a* is the measurement along the red-green axis and b* is the measurement along the yellow-blue axis. A positive a* value indicates the red direction, a negative a* value the green direction, a positive b* value the yellow direction and a negative b* value the blue direction (Matis & others, 1999). At the end of the study, total color differences or distances between two colors (ΔE) were calculated using the formula:

$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)2]^{1/2}$ (International Commission on Illumination, 1978)

A die was rolled to determine which procedure the non-smoking or smoking subjects received. If the die indicated 1 or 2, Whitestrips Premium (Procter & Gamble, Cincinnati, OH, USA) were to be used for 30 minutes, twice a day (WP2) for seven days. These strips contain 10% hydrogen peroxide gel. A 3 or 4 roll on the die indicated Whitening Wraps (Ranir Corporation, Grand Rapids, MI, USA) were to be used for 30 minutes, twice a day (WW2), for seven days. A five or six roll indicated Whitening Wraps (Ranir Corporation) were to be used for 30 minutes, once a day (WW1), for seven days. The Whitening Wraps that were evaluated contained 8% hydrogen peroxide gel. An assistant, not involved in any other part of the study, explained and demonstrated to the subjects how to use the various products according to the manufacturers recommended instructions. The subjects then left the clinic with the products in a non-identifiable bag.

The manufacturer of wraps recommends brushing immediately before applying their product, while the manufacturer of strips does not recommend brushing at that time. All subjects, however, were asked to brush at least twice daily to maintain a standardized home care regiment.

All subjects were provided a non-whitening dentifrice and a soft bristled manual toothbrush. They were also given a diary upon which they indicated on a daily basis the level of tooth and gum sensitivity that they experienced and the times during the morning and/or evening when they used the at-home wraps or strips. The active

Table 1: *Demographics of Subjects*

				Groups					
	Gender	**All**	**(Age)**	**WP/2X**	**(Age)**	**WW/2X**	**(Age)**	**WW/1X**	**(Age)**
All Subjects	Both	76	(53.4)	25	(55.1)	26	(52.1)	25	(53.0)
	Female	35	(53.7)	8	(55.3)	14	(55.8)	13	(50.4)
	Male	41	(53.1)	17	(55.0)	12	(47.8)	12	(55.8)
Non-smokers	Both	62	(54.9)	21	(56.2)	22	(53.6)	19	(54.9)
Smokers	Both	14	(46.6)	4	(49.2)	4	(43.8)	6	(47.0)

phase of the treatment lasted seven days. The subjects were asked to return to receive potassium nitrate desensitizing gel if their sensitivity was more than moderate.

The use of the strips and wraps was separated by at least three hours when required to be worn twice a day. The subjects returned in five and seven days after using the at-home bleaching strips and wraps for the same type of color evaluation conducted during the baseline evaluation. They also returned for a 14-day post-bleaching color evaluation.

Table 2: *Mean Baseline Values for the Three Groups*

Groups	**Day**	**N**	**L***	**a***	**b***	**Bioform**
WP/2X	0	25	66.12	0.31	13.85	19.07
	5	24	68.07	-0.36	11.68	13.21
	7	25	68.37	-0.51	11.04	11.73
	21	25	67.89	-0.57	11.42	12.03
WW/2X	0	26	66.55	0.45	14.08	19.06
	5	26	69.15	-0.28	11.72	11.44
	7	26	69.47	-0.44	11.17	9.04
	21	26	68.88	-0.52	11.44	9.35
WW/1X	0	25	65.93	0.34	14.60	19.93
	5	24	67.43	-0.19	12.81	14.67
	7	25	67.89	-0.29	12.69	13.48
	21	25	67.71	-0.34	12.86	14.43

RESULTS

Seventy-six of the 78 subjects enrolled completed the study. Thirty-five (46%) were female and 41 (54%) were male (Table 1). The Whitestrips Premium group had 25 subjects, Whitening Wraps 2 had 26 subjects and Whitening Wraps 1 had 25 subjects. The youngest and oldest subjects were 32 and 80, respectively. The mean age of the non-smokers was 55; the mean age of the smokers was 47 years of age. The study was open to smokers and non-smokers; however, only 18% of the subjects who qualified were smokers.

Table 3: *Mean Change for the Three Groups*

Groups	**Day**	**N**	**ΔL***	**Δa***	**Δb***	**ΔE**	**ΔBioform**
WP/2X	0	25	0.00	0.00	0.00	0.00	0.00
	5	24	1.99	-0.69	-2.16	3.27	-5.84
	7	25	2.25	-0.82	-2.81	3.95	-7.33
	21	25	1.77	-0.88	-2.43	3.37	-7.04
WW/2X	0	26	0.00	0.00	0.00	0.00	0.00
	5	26	2.61	-0.73	-2.37	3.75	-7.63
	7	26	2.93	-0.88	-2.92	4.38	-10.03
	21	26	2.33	-0.97	-2.64	3.79	-9.59
WW/1X	0	25	0.00	0.00	0.00	0.00	0.00
	5	24	1.53	-0.47	-1.49	2.38	-5.26
	7	25	1.96	-0.63	-1.90	2.97	-6.45
	21	25	1.78	-0.67	-1.73	2.82	-5.51

At baseline examinations, subjects from the WP2, WW2 and WW1 groups had mean L* values of 66.12, 66.55 and 65.93, respectively, which were not significantly different (p=0.75), a* values of 0.31, 0.45 and 0.34, respectively, which were not significantly different (p=0.71), b* values of 13.85, 14.08 and 14.60, respectively, which were not different statistically (p=0.72) and Trubyte Bioform shade values of 19.07, 19.06 and 19.93, respectively, which were not significantly different (p=0.41) (Table 2).

The color change in E and shade guide change are illustrated graphically in Figures 1 and 2, respectively. All three groups had significant mean changes from baseline to 14 days post-bleaching in ΔL*, Δa*, Δb*, ΔE and ΔTrubyte Bioform shade (p<0.001) (Table 3).

Subjects receiving the WW2 treatment had significantly different ΔL* (p=0.0208) and Δ shade guide (p=0.0079) values than did those subjects in the WP2 group. Subjects receiving the WP2 treatment had sig-

nificantly different Δa* (p=0.0029), Δb* (p=0.0022) and ΔE (p=0.0063) values than those receiving the WW1.

There were no differences among the three groups for tooth sensitivity (p=0.42). Regarding gum sensitivity, the WW2 group had significantly higher values than the WP2 (p=0.0167) and WW1 (p=0.0362) groups.

The objective color measurements indicate significantly different L* (p=0.0106) and a* (p=0.0556) change for nonsmokers, while subjective color measurement indicated significantly different Trubyte shade guide (p=0.0089) change for smokers.

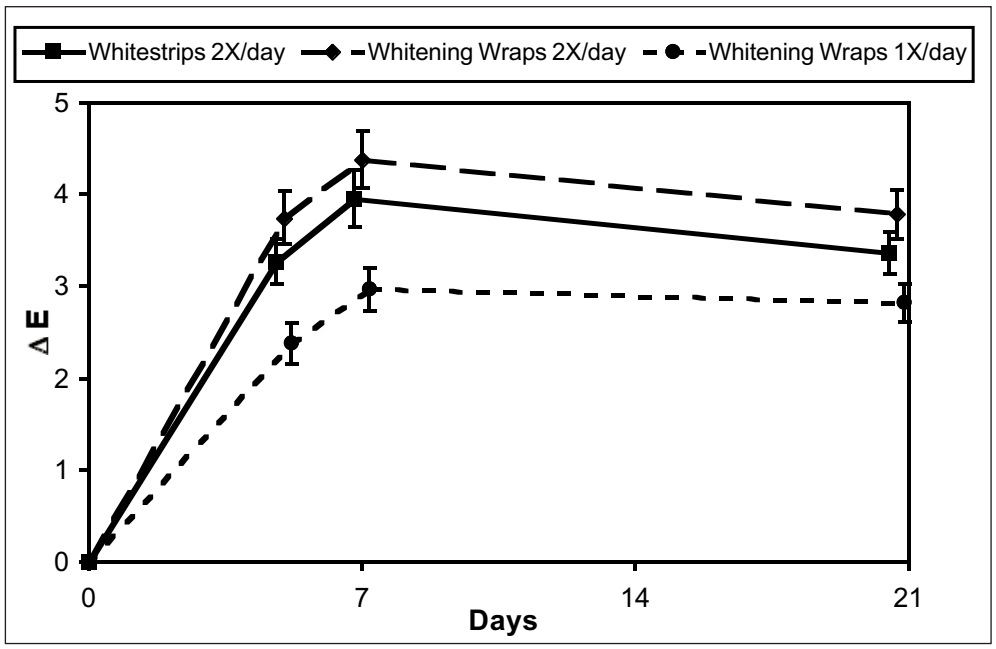

Figure 1. *Mean change in E for all teeth at 5, 7 and 21 days.*

DISCUSSION

Whitening wraps and strips were evaluated in a single blind, three-week study with 78 subjects randomized into three equal cells balanced by product used and smoking versus nonsmoking. The three cells consisted of 1) those using Whitening Wraps twice a day, 2) those using Whitening Wraps once a day and 3) those using Whitestrips Premium twice a day. Seventy-six subjects were evaluated at baseline, 5, 7 and 21 days using a colorimeter and a shade guide.

All groups had significant mean changes from baseline to 21 days for all color measurements. Some products were shown to be significantly different when comparing color change between them.

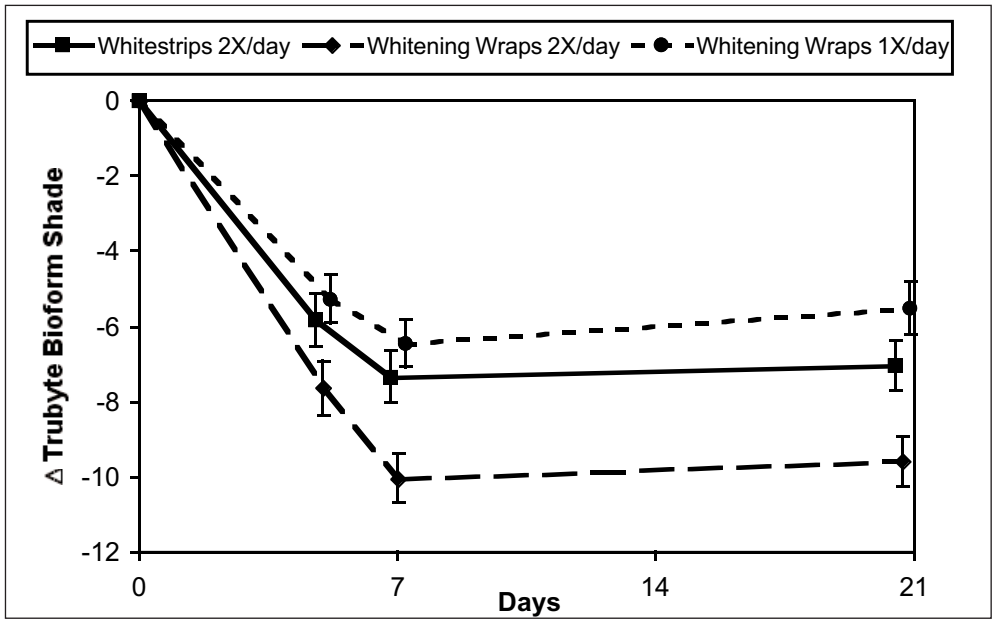

Figure 2. *Mean change in Trybyte Bioform for all teeth at 5, 7 and 21 days.*

Subjects using WW2 were significantly lighter overall in tooth whitening than those using WP2 in L* and shade guide value. Subjects using WW2 were significantly lighter overall in tooth whitening than those using WW1 in L*, a*, b*, E and shade guide values.

Subjects using WP2 were significantly lighter overall in tooth whitening than those using WW1 in a*, b*, E and shade guide values. Subjects using WP2 were significantly lighter overall in tooth whitening than those using WW1 in a*, b* and E.

There were no differences between groups for tooth sensitivities, however, subjects reported that WW2 caused significantly more gum sensitivity. Gum sensitivity seemed to occur much more frequently in subjects with shorter teeth, because placement of the rigid tray extended onto the tissues, causing rubbing of the tray on the gum tissues.

Since the strip-based tooth whitening system was introduced on the market, numerous studies have been conducted to evaluate the effectiveness of the system. Unfortunately, there have been a variety of HP concentrations used in the strips and comparisons of study data are difficult. The first over-the-counter product introduced contained 5.3% HP and was to be used twice a day for 14 days (Gerlach, Barker & Sagel, 2002). Subsequently, another over-the-counter strip was introduced containing 6.0% HP, which was to be used twice a day for seven days (Gerlach, Gibb & Sagel, 2002). A "professional strength" strip containing 6.5% HP was introduced at the same time and was to be used twice a day for 21 days (Li & others, 2003). The latest over-the-counter product is 10% HP and is intended to be used twice a day for seven days. This "professional strength" product is now 14% HP (Gerlach & Sagel, 2004; Swift & others, 2004).

This study is an attempt to inform dental practitioners about the new products, their concentration and efficacy both during use and post-bleaching. It appears that there is minimal reversal in color when wraps and strips are used. Perhaps this is because the teeth's "inherent lightness potential" (Matis, 2003) is not exceeded, therefore, no color reversal occurs.

Both smokers and non-smokers purchase over-the-counter products. The authors are not aware of any data available in the scientific literature that identifies whether smokers bleach differently than non-smokers. The mean age of smokers was seven years younger than the mean age of non-smokers. Non-smokers were measured to be significantly different in L* and a* than smokers, but smokers were measured to be significantly different with the Trubyte Bioform Color Ordered Shade Guide than non-smokers. There is no explanation for this finding.

The Whitening Wraps with 8% HP have not yet been distributed by the Ranir Corporation. It is impossible for consumers to know the concentration of active agent in tooth whitening products sold over-the-counter. Manufacturers are not required to place concentrations on the labels, only the name of the active agent. The dental profession needs to encourage manufacturers to place the concentration of active agent on labels so that patients can make an informed decision as to what concentration of whitening gel is in the product they are purchasing.

CONCLUSIONS

All products had significant tooth whitening 14 days post-bleaching.

The subjects using WW2 were significantly lighter overall in tooth whitening than those using WP2 in L* and shade guide values and improved, but not significantly so, in all other color measured parameters.

Subjects using WP2 were significantly lighter overall in tooth whitening than those using WW1 in a*, b*, E and shade guide values and improved, but not significantly so in all other color measured parameters.

There was no difference in tooth sensitivity, but WW1 and WP2 caused less gingival sensitivity than WW2.

The mean age of smokers was seven years younger than non-smokers who qualified.

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Laboratory Research

Surface Finish Produced on Three Resin Composites by New Polishing Systems

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Clinical Relevance

Diamond micro-polisher disks and rubber polishing disks produced finished surfaces of equivalent average roughness compared to aluminum oxide polishing disks.

SUMMARY

This study evaluated the surface finish of three direct resin composites polished with three different systems. Disk-shaped specimens (n=16 per material; ϕ=8.0 mm x h=2.0 mm) were formed in a stainless steel mold by packing uncured material, either a hybrid composite (Z250, 3M ESPE) or two micro-hybrid composites (Point 4, Kerr; Esthet-X, Dentsply), and light-cured from the top and the bottom surfaces with a light-emitting diode (LED) curing unit (NRG, Dentsply). After storing the specimens in deionized water at 37°C for seven days, one side of each specimen was finished through 1200-grit SiC abrasive (Buehler). Five specimens of each resin composite were randomly assigned to one of the three polishing systems (Identoflex, Kerr; Pogo, Dentsply; Sof-Lex, 3M ESPE). Manufacturers' instructions were followed during the polishing procedures. The average surface roughness (Ra) was determined by generating tracings across the polished surface of each disk using a scanning profilometer (Surfanalyzer System 5000, Federal Products Co). The results were analyzed by Kruskal-Wallis and Mann and Whitney tests ($p \leq 0.05$). The smoothest surfaces were produced with the celluloid strip (control group) on all the resin composites tested. The aluminum oxide disks (Sof-Lex) produced a statistically equivalent surface finish (Ra) on the three resin composites. The lowest mean roughness values were recorded with diamond micro-polisher disks (PoGo) on the hybrid composite (Z250). Overall, the two new polishing systems, Identoflex and PoGo, created a comparable surface finish to that produced by the Sof-Lex system on all three resin composites.

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INTRODUCTION

The use of resin composites in dentistry is now well accepted for restoring anterior (Denehy, 2000) and posterior teeth (Baratieri & Ritter, 2001; Gaengler, Hoyer & Montag, 2001). Pit-and-fissure sealants, preventive resin restorations and Class I and Class II restorations for both initial and moderate-sized lesions are all indications for the use of direct resin composites to restore posterior defects (ADA Council on Scientific Affairs, 1998). Over the years, several changes have been made in the fabrication of dental resin composites to obtain better color stability over time (Tyas, 1992), greater wear resistance (Yap, 2002) and clinically acceptable final surface smoothness of placed restorations (Setcos, Tarim & Suzuki, 1999). To achieve this last goal, manufacturers have reduced the diameter of filler particles, rendering early commercial composites with average particle sizes between 10 and 100 µm obsolete. Current restorative resin composites have an average particle size of approximately 0.4 µm for micro-hybrid composites, a bi-modal distribution of 0.04 µm and 1-4 µm particles for hybrid composites, around 0.04 µm for microfill composites and a bi-modal distribution of 0.6-1.4 µm agglomerate of particles sized between 5-20 ηm and 20 ηm free particles for nano-particulate composites (Roberson, Heymann & Swift, 2001). One of the advantages of particle size reduction is the excellent surface finish that can be achieved (Turssi & others, 2000).

Although it has been proposed that limiting the amount of finishing and polishing of resin composite restorations avoids stress at the tooth-restoration interface (Ritter, 2001), it is often necessary to use carbide burs or fine diamonds to contour the restoration and adjust the occlusion. Therefore, finishing and polishing are still important steps in the clinical procedures for resin composite restorations. Several resin composite polishing tools have been used over the years, ranging from prophylaxis and polishing pastes (Roulet & Roulet-Mehrens, 1982; Serio & others, 1988) to stones, tungsten carbide burs and fine and superfine diamond burs (Lutz, Setcos & Phillips, 1983; Jung, 1997) to abrasive disks, mounted abrasive points (Stoddard & Johnson, 1991), soft rubber cup (Jefferies, Barkmeier & Gwinnett, 1992), diamond-impregnated felt wheels (Jung, Baumstieger & Klimek, 1997), brushes with abrasive bristles (Krejci, Lutz & Boretti, 1999) to a combination of some of the instruments mentioned above (Wilson, Heath & Watts, 1990; Ashe & others, 1996; Kaplan & others, 1996; Roeder, Tate & Powers, 2000). With the ultimate goal of achieving a smooth surface of the resin composite restoration in fewer steps, it is common to see new polishing systems being introduced and, therefore, updated evaluations are necessary.

This study evaluated the surface finish of three direct resin composites polished with two new polishing systems, Identoflex and PoGo, and a well-established polishing system, Sof-Lex system. The null hypothesis was that there are no differences among the three different polishing systems on the surface finish of three resin composites.

METHODS AND MATERIALS

A hybrid and two micro-hybrid dental resin composites were tested (Table 1). Sixteen disk-shaped specimens were made of each material using a stainless steel mold (8.0-mm diameter x 2.0-mm high). Celluloid strips were placed over each surface of the uncured material to prohibit oxygen inhibition. A 2000 g load, placed for 30 seconds, was utilized to extrude the excess material. The samples were then light cured from the top and bottom surfaces, 30 seconds respectively, using a NRG (Dentsply/Caulk, Milford, DE, USA) light-emitting

Table 1: *Dental Resin Composite Materials Tested*

Resin Composites	Manufacturer	Shade	Mean Particle Size (µm)	Batch #
Hybrid (Z250)	3M ESPE Dental Products St Paul, MN, USA	A-2	0.6	20020123
Micro-hybrid (Point 4)	Kerr Corporation Orange, CA, USA	A-2	0.4	107560
Micro-hybrid (Esthet-X)	Dentsply/Caulk Milford, DE, USA	A-2	0.4	0004142

Table 2: *Polishing Systems and Use Protocols Examined in This Study*

Polishing Systems	Manufacturer	Instructions Followed
Aluminum Oxide Disks (Sof-Lex XT)*	3M ESPE Dental Products St Paul, MN, USA	Medium grit: 20 seconds, rinse and dry Fine grit: 20 seconds, rinse and dry Superfine grit: 20 seconds, rinse and dry
Rubber Polishing Disks (Identoflex)**	Kerr Corporation Orange, CA, USA	Disk gloss polisher: 1 minute, rinse and dry Disk high-gloss polisher: 30 seconds, rinse and dry
Diamond Micro-Polisher Disks (PoGo) **	Dentsply/Caulk Milford, DE, USA	Step 1: Light pressure for 1 minute, rinse and dry Step 2: Lightest pressure for 30 seconds, rinse and dry

**New disk for each sample*

***Changed after two uses*

diode (LED) curing unit. The intensity of the light was 350 mW/cm^2 and was checked before every use with a radiometer (Cure Rite, Visible Curing Light Meter, Dentsply/Caulk). Immediately after the light-curing cycle, the specimens were taken from the mold and immersed in deionized water at 37°C for seven days.

Following the storage period, one side of each sample was wet finished through 1200-grit silicon carbide abrasive paper on a rotary polisher. In this way, each sample had one side prepared to a standard surface to simulate the clinical finishing procedure. Five samples of each material were randomly assigned to each of the three polishing systems evaluated in this study (Table 2). A control group (n=3) of one specimen of each material received neither finishing nor polishing treatments after being covered with a celluloid strip and cured. Cyanoacrylate cement was used to attach the specimens to a glass slab by the side left untouched. Manufacturers' instructions were followed during the polishing procedures (Table 2) and one person performed the polishing manipulations. After each polishing procedure, resin composite disks were washed and dried before the next step to remove debris. The same slow-speed handpiece (<4000 rpm) was used for all systems.

The polished surface of each resin composite disk was subjected to a surface profile-measuring instrument (Surfanalyzer Model 5000 Profilometer, Federal Products Co, Providence, RI, USA). The average surface roughness (R_a) was the major parameter reported. By definition, R_a is the arithmetic average height of roughness component irregularities from the mean line measured within the sampling length (Setcos & others, 1999). Three successive measurements were made on each sample and the readings were taken at 120-degree intervals. The roughness value was recorded as the average of these three readings and the mean values for R_a were determined for each group (n=5/group).

The results were subjected to Kruskal-Wallis and Mann and Whitney tests. The tests were performed at a 5% ($p \leq 0.05$) level of significance.

RESULTS

The mean values and standard deviations of surface roughness (R_a, µm) for each combination of resin composites and polishing systems and the control group are given in Table 3. The smoothest surfaces occurred for resin composite specimens cured against a celluloid strip (control group). The Kruskal-Wallis test revealed no significant difference among resin composite materials (p=0.558). The aluminum oxide disks (Sof-Lex) produced statistically equivalent surface finish (mean R_a) on each of the three resin composites (p=0.230). No significant difference in method of polishing existed with the micro-hybrid Esthet-X (p>0.05). The lowest roughness value, 0.51 ± 0.15 µm, was recorded with the diamond micro-polisher disks (PoGo) on the hybrid composite Z250. The rubber polishing disks, Identoflex, were significantly more effective on the micro-hybrid Point 4 ($p \leq 0.05$) than the other two resin composites.

Table 3: *Average Roughness Values (R_a, µm) and Standard Deviations for the Three Resin Composite Materials and Three Polishing Systems Tested*

	Roughness (R_a) (µm ± SD)			
Resin Composites	Celluloid Strip	Sof-Lex	Identoflex	PoGo
Hybrid (Z250)	0.47 ± 0.29^{a,c}	1.12 ± 0.27^{b}	1.53 ± 1.70^{b,c}	0.51 ± 0.15^{a}
Micro-hybrid (Point 4)	0.30 ± 0.00^{a}	1.49 ± 0.60^{b}	0.62 ± 0.58^{a}	1.69 ± 0.70^{b}
Micro-hybrid (Esthet-X)	0.33 ± 0.06^{a}	1.47 ± 1.05^{b}	1.87 ± 1.66^{b}	1.39 ± 0.67^{b}

*Values with same superscript are not significantly different.

DISCUSSION

Many studies have demonstrated that the smoothest surface on resin composite restorations is achieved using a clear matrix in contact with the composite surface during polymerization (Ozgunaltay, Yazici & Gorucu, 2003; Wilson & others, 1990). This study confirms that finding. Also, no significant difference in surface roughness was shown among unfinished materials (celluloid matrix surface). Of course, the clinical environment does not always allow the dentist to place a celluloid strip on the last layer of a resin composite restoration before final polymerization. Even if it was possible, further contouring and finishing are usually required. Furthermore, the smooth surface produced by the celluloid strip in contact with the resin composite contains a resin-rich layer on the top, which needs to be removed by polishing to avoid accelerated clinical wear (Roberson & others, 2001).

Aluminum oxide disks (Sof-Lex) are used to polish plane resin composite surfaces (Hondrum & Fernandez, 1997), and they are particularly convenient for refining embrasure forms of posterior and anterior resin composite restorations. Obtaining an adequate polished surface with this type of polishing system has been demonstrated over the years (Wilson & others, 1990; Jung, Voit & Klimek, 2003). New commercial polishing products aim toward reducing steps and manipulations. The effectiveness of restoration finishing and polishing procedures depends on the careful use of successively finer abrasive materials to eliminate larger scratches or defects, replacing them with smaller ones (Roberson & others, 2001). It is important to test the new polishing systems to confirm their ability to produce the same quality of resin com-

posite surface finish. The results of this study indicate that the aluminum oxide disks (Sof-Lex) produced statistically equivalent surface finish (R_a) on each of the three resin composites, with mean values ranging from 1.12 ± 0.27 µm to 1.47 ± 1.05 µm. The gloss and high-gloss rubber polishing disks (Identoflex) created a comparable surface finish to that produced by the aluminum oxide disks. The same results were obtained with the diamond micro-polishing disks (PoGo). With this system, only one disk is used to finish and polish the composite surface. The difference between the two procedures is the amount of pressure applied while the disk is in contact with the resin composite surface. It has been reported that the motion employed during finishing and polishing can have a significant effect on the quality of the finished restoration (Fruits, Miranda & Coury, 1996). The use of two different pressures combined with the effectiveness of the diamond-particle coating on the disks may explain why, with only one polishing disk and fewer polishing steps, the surface finish was similar to the aluminum oxide disks.

In this study, the average roughness values ranged from 0.51 µm to 1.87 µm. It is reported that surface roughness in the range of 0.2 to 0.6 µm is achievable using submicron polishing pastes on materials that include submicron filler particles (Roberson & others, 2001). The specimens in this study were finished without the use of polishing pastes. Therefore, it may partially explain why the mean surface roughness values were higher than the range expected (0.2 to 0.6 µm). However, a study by Kaplan and others (1996) indicated that R_a values less than 10 µm are clinically undetectable and, hence, any system that produces a surface roughness less than 10 µm is acceptable. With roughness values closer to 1 µm than to 10 µm, the polished specimens in this study showed acceptable surface finish. Further studies are needed to evaluate the real effect of these polishing systems under *in vivo* conditions.

CONCLUSIONS

The null hypothesis was rejected, because polishing the hybrid (Z250) resin composite with micro-polisher disks (PoGo) resulted in significantly less surface roughness than aluminum oxide (Sof-Lex) and rubber polishing disks (Identoflex). Moreover, the rubber polishing disks produced significantly less surface roughness than the two other polishing systems on the micro-hybrid composite Point 4. However, all three polishing systems created a comparable surface finish on the micro-hybrid composite Esthet-X.

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Influence of Vision on the Evaluation of Marginal Discrepancies in Restorations

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Clinical Relevance

This paper confirms the importance of the dental explorer in diagnosing marginal discrepancies in restorations, but highlights the need to develop alternatives to the explorer to distinguish between steps and gaps along cavosurface margins.

SUMMARY

This study investigated the influence of visual inspection in the detection and discrimination between principle types of marginal discrepancies in restorations. Using devices simulating vertical steps, horizontal gaps and the combination of a vertical step and horizontal gap at the margin of a restoration, and explorers with four different tip diameters, 10 experienced dental faculty members were asked to identify discrepancies and the boundary between Alpha (excellent) and Bravo (clinically acceptable) marginal adaptation ratings under three different visual conditions—with and without visual inspection and visual inspection aided with binocular loupes. A significant correlation was found to exist between explorer tip diameter and the Alpha/Bravo boundary for horizontal gaps, but not for vertical steps. There was no significant difference in the detection of the Alpha/Bravo boundary for the three visual conditions. It was concluded that visual inspection aided and unaided with loupes had no significant effect on the evaluation of simulated marginal discrepancies. These findings highlight the importance of the traditional dental explorer, in the absence of a more discriminatory devices in the assessment of marginal discrepancies in restorations.

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INTRODUCTION

A recent study by Hayashi and Wilson (2003) lends support to the widely held view that restorations with detectable marginal discrepancies are likely to fail sooner than restorations with ideal marginal adaptation. To evaluate a restoration for marginal discrepancies, most clinicians aim to examine the restored tooth clean, dry and well illuminated, and use visual assessment and a dental explorer to complete the task.

Regarding the diameter (sharpness) of the tip of the dental explorer best suited to evaluations in clinical trials, Hayashi and others (2004) have found that the use

of an explorer with a tip diameter of 500 µm or greater may be indicated to reliably distinguish between Alpha and Bravo ratings. When seeking to discriminate the different types of marginal discrepancies in Alpha rated restorations, the use of an explorer with a tip diameter of less than 500 µm is indicated. Hayashi and others (2005) concluded that explorers with a tip diameter within the range of 120 µm to 500 µm may be found to have no significant effect on the detection of step discrepancies, both alone and in combination with a gap, but to have a significant effect on the detection of gap-only discrepancies.

The extent to which visual assessment is critical to the evaluation of marginal discrepancies has not been investigated. It may be considered that the more effective the visualization of a restoration and its margins, the more reliable the evaluation of marginal discrepancies is likely to be; hence, recommendations to use magnification aids, for example, loupes in the evaluation of restorations (L'Estrange & others, 1991; Whitehead & Wilson, 1992).

This study was undertaken to test the hypothesis that visual inspection, unaided and aided with loupes, has a significant effect on evaluators' ability to detect and discriminate among the different types of marginal discrepancies, using a range of dental explorers of known tip diameter. This study forms part of a series of investigations to establish a reliable, reproducible method to evaluate marginal discrepancies in restorations.

METHODS AND MATERIALS

Devices for simulating marginal discrepancies were developed from the device described by Rappold, Ripps and Ireland (1992). In this study, three devices simulating a vertical step, a horizontal gap and the combination of both a vertical step and a horizontal gap of increasing size were constructed from precisely milled metal plates attached to a metal base (Figure 1). The plates were positioned next to each other so that the vertical and/or horizontal distance between them increased from 0 µm at the origin to 270 µm. The edges of the plates were sharp right angles, with discrepancies between the plates increasing continuously at the rate of 2 µm per mm. The size of a discrepancy in microns at any point along the plates could be determined by measuring the distance (mm) from the origin and doubling the value.

Dental explorers of four different tip diameters, 170 µm, 220 µm, 350 µm and 500 µm, were produced especially for this study (DENTSPLY Ash Instruments, Plymouth, UK). The general features of the explorers, such as the design of the handle and shank, were identical and similar to that for existing instruments of a similar pattern.

Using the explorers of four different tip diameters in a standardized manner, 10 experienced dental faculty members in Operative Dentistry were asked to identify the minimum detectable discrepancies and boundary between Alpha (excellent margin, replacement unnecessary) and Bravo (clinically acceptable margin, replacement questionable) ratings, according to modified USPHS criteria (Raskin & others, 1999) in each of the three metal plate devices.

The measurements were conducted under three different sets of conditions to test the influence of vision in evaluating marginal discrepancies. The first approach, which simulated ordinary conditions for the assessment of marginal evaluation in clinical trials, involved the use of the standardized explorers and unaided visual inspection. The second approach involved use of the explorers alone, that is, without visual inspection. For this approach the assessors were blindfolded and prevented from having sight of the metal plate devices—the probe being directed to the device to be assessed by the principle investigator. The third approach involved use of the explorers and visual inspection aided with binocular loupes (SurgiTel, General Scientific Co, Ann Arbor, MI, USA) with a 2x

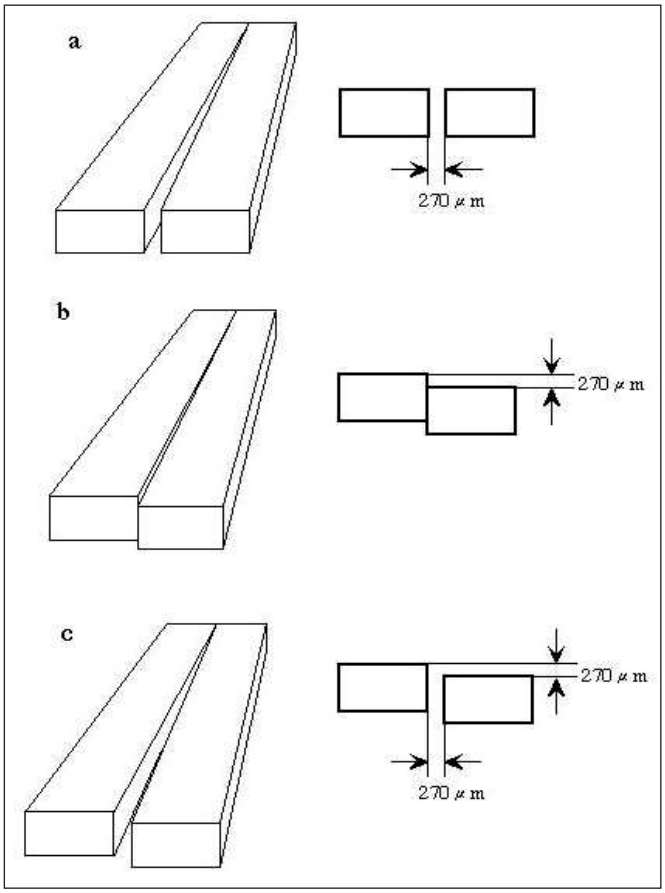

Figure 1: *Design of the three devices: a) device simulating a horizontal gap, b) device simulating a vertical step, c) device simulating the combination of a horizontal gap and a vertical step.*

magnification. The second and third approaches were conducted at intervals of at least three weeks. In all cases, the assessors were allowed to familiarize themselves with the devices, probes and assessment conditions prior to completing any assessments.

Each assessor evaluated the discrepancies without knowledge of the type of device or sharpness of the explorer used. Twelve combinations of the three devices and four explorers were tested. Five measurements were made for each of the 12 combinations of device and explorer, with three types of visualization by each assessor and mean values for each assessor recorded. After excluding the maximum and minimum values in each set of data, the mean of the remaining eight values was then taken to be the minimum detectable discrepancy or the mean boundary value between Alpha and Bravo ratings.

The values obtained with the four different explorers under the different conditions were compared by means of the ANOVA and Scheffe's *F* tests, with a 95% level of confidence (StatView Macintosh v4.5, SAS Institute Inc, Cary, NC, USA).

RESULTS

The findings, with respect to the minimum detectable discrepancies, are summarized in Table 1. For all three visual conditions, no correlations were found between the tip diameter of the explorers and the minimum detectable discrepancies, irrespective of the nature of the discrepancy.

Significantly smaller horizontal gaps and combined horizontal and vertical discrepancies could be detected when using loupes, compared to unaided visual inspection. In contrast, there were no significant differences with respect to the minimum vertical discrepancies detected under the three visual conditions.

Table 1: *Minimum Discrepancies Detected by the Standardized Explorers of Different Tip Diameter Under Different Visual Conditions*

Explore tip diameter (μm)	Discrepancy (μm) Horizontal	Combination	Vertical
With visual inspection			
170	43 ± 14	39 ± 5	29 ± 9
220	58 ± 22	43 ± 6	36 ± 10
350	53 ± 25	42 ± 7	40 ± 14
500	58 ± 30	44 ± 9	42 ± 14
Without visual inspection			
170	31 ± 15	34 ± 8	28 ± 12
220	40 ± 17	38 ± 8	33 ± 10
350	37 ± 14	35 ± 14	29 ± 9
500	39 ± 23	34 ± 17	35 ± 15
With binocular loupes (2x)			
170	14 ± 10	22 ± 12	25 ± 12
220	23 ± 11	27 ± 11	28 ± 10
350	19 ± 12	25 ± 12	23 ± 10
500	24 ± 18	30 ± 14	30 ± 15

Mean±sd, unit: μm, n=8

Table 2: *Boundary Between Alpha and Bravo Marginal Adaptation Ratings as Detected by the Standardized Explorers of Different Tip Diameter Under Different Visial Conditions*

Explore tip diameter (μm)	Discrepancy (μm) Horizontal	Combination	Vertical
With visual inspection			
170	96 ± 19	57 ± 11	61 ± 13
220	116 ± 18	62 ± 8	58 ± 11
350	132 ± 9	70 ± 13	70 ± 16
500	151 ± 5	80 ± 9	75 ± 12
Without visual inspection			
170	86 ± 20	63 ± 14	67 ± 15
220	102 ± 19	66 ± 13	72 ± 17
350	108 ± 21	77 ± 8	76 ± 15
500	144 ± 28	83 ± 9	83 ± 14
With binocular loupes (2x)			
170	85 ± 23	65 ± 25	67 ± 13
220	109 ± 15	68 ± 11	72 ± 13
350	139 ± 27	81 ± 9	78 ± 9
500	161 ± 34	79 ± 16	81 ± 17

Values connected with lines showed significant differences by means of ANOVA and the Scheffe's F test at a 95% level of confidence.

Mean±SD, n=8

The findings, with respect to the boundaries between the Alpha and Bravo ratings, are summarized in Table 2. A positive correlation was found between the tip diameter of the explorers and the values of the boundaries between the Alpha and Bravo ratings in relation to the horizontal gap. No strong correlations were found to exist between the sharpness of the explorer tip and the detection of the vertical step and combined vertical step and horizontal gap in all three approaches to assessment.

No significant differences were found in the data for the boundary between the Alpha and Bravo ratings, irrespective of the method of visualization and combination of device and explorer.

DISCUSSION

This study, which forms part of a series of investigations to establish a reliable, reproducible method to evaluate marginal discrepancies in restorations, was undertaken to test the hypothesis that visual inspection, unaided and aided with loupes has a significant effect on evaluators' ability to detect and discriminate between different types of marginal discrepancies. As in a previous study (Hayashi & others, 2005), dental explorers with a tip diameter ranging from 120 µm to 500 µm were investigated.

The findings of this study confirm the findings of Hayashi and others (2005) in that they demonstrate that the diameter of the tip of a dental explorer, within the range investigated, has no significant effect on the detection of a step discrepancy, both alone and in combination with a gap, but has a significant effect on the detection of a gap alone. In addition, these findings indicate that visual assessment, both aided with loupes and unaided, does not influence the evaluation of marginal discrepancies or the determination of Alpha and Bravo ratings. Together, these findings highlight the importance of the dental explorer in the evaluation of marginal discrepancies in restorations. As suggested in a previous publication (Hayashi & others, 2005), explorers of different tip diameter are required for different purposes. The findings in this study lend support to the view that none of the explorers included in this series of investigations is capable of discriminating between the different types of discrepancies, as they occur clinically.

Further studies are clearly indicated. These studies will include the research and development of possible alternatives to the dental explorer for the diagnosis of the presence and, in particular, the nature of marginal discrepancies in restorations. Such work is considered to be of considerable importance to the effective application of modern approaches to restorative dentistry. For example, it is considered important to be able to distinguish between step discrepancies and gaps when considering various options for a minimally invasive repair or refurbishment of a restoration with less than ideal marginal adaptation.

In the meantime, the findings of this study could be considered to raise questions about the value of magnifying aids in the evaluation of the margins of restorations. On the contrary, it is suggested that the findings demonstrate that the use of loupes does not adversely influence the evaluation of marginal discrepancies and, as such, should be considered to facilitate the assessment of restorations, notably given the associated benefits of loupes (L'Estrange & others, 1991; Whitehead & Wilson, 1992).

CONCLUSIONS

The results of this study indicate that visual inspection, aided and unaided with loupes, had no significant effect on the evaluation of marginal discrepancies. The findings highlight the importance of the traditional dental explorer in the absence of a more discriminatory device in the assessment of marginal discrepancies in restorations.

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Effect of Polymerization Modes and Resin Composite on the Temperature Rise of Human Dentin of Different Thicknesses: An *In Vitro* Study

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Clinical Relevance

The intrapulpal temperature rise of human dentin depends on the energy released by the light curing unit and is dependent on the presence of resin composite and dentin thickness.

SUMMARY

This *in vitro* study evaluated the effect of different polymerization modes and the presence of resin composite on the temperature rise (TR) in human dentin of different thicknesses. For this purpose, 90 specimens were assigned to 30 groups (n=3): five polymerization modes (1-conventional; 2-soft-start; 3-high intensity; 4-ramp cure: progressive and high intensity; 5-high intensity with the tip of the light cure at a distance of 1.3 cm for 10 seconds and the tip leaned in the sample); two levels of resin composite presence (absence or presence of resin composite) and three dentin thicknesses (1, 2, 3 mm). During polymerization, temperature was measured by a digital laser thermometer (CMSS2000-SL/SKF). Three-way ANOVA and Tukey tests were performed. There were statistical differences in TR among polymerization modes, presence of resin composite and dentin thicknesses. Within the limits of this study, it can be concluded that 1) conventional and high intensity polymerization modes presented lower TR means, and it was statistically different from soft start, distanced tip and ramp curing polymerization modes; 2) the presence of resin composite showed a statistically significant reduction TR means and 3) the thicker the dentin, the less the temperature rise.

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INTRODUCTION

Light-activated resin composite restorative material has revolutionized clinical dentistry (Rueggeberg,

Caughman & Curtis Jr, 1994). A photoinitiator molecule of resin composite is activated by absorbing a photon. The absorbed energy of the photon is used to change the molecular structure, forming a radical. This radical can attach to a monomer and, in addiction, will activate the monomer to attach to another monomer, resulting in a polymer network (Althoff & Hartung, 2000). The conversion of the monomer to a polymer network is accompanied by a closer packing of the molecules, causing contraction in the composite (Friedl & others, 2000; Feilzer, de Gee & Davidson, 1990). The temperature of the composite and, consequently, of the surrounding structures, increases during polymerization, and, together with the energy released by the light source, can generate heat on the tooth's dentin and pulp tissue (Hansen & Asmussen, 1993; Hannig & Bott, 1999; Althoff & Hartung, 2000).

The dental pulp is a low-compliance system which does not respond well to increased temperature (Goodis & others, 1990). Zach and Cohen (1965) studied the effects of heat on pulp tissue. They concluded that even a minimal intrapulpal temperature rise (5.5°C) can cause irreversible pulpitis in 15% of the teeth tested in *Macaca Rhesus* monkeys. When the intrapulpal temperature rose to 11.1°C, 60% of the pulp of the teeth tested became necrotic. This study was carried out on intact teeth, whose pulp responds better than teeth with caries, that were submitted to cavity preparations (Hansen & Asmussen, 1993; Murray & others, 2002).

Cavities with a reduced remaining dentin thickness, because of the extent of disease progression and treatment regime, have a reduced number of odontoblasts, impairing their ability to secrete reactionary dentin in response to gross injuries of the dentin matrix (Murray & others, 2002). Thus, the pulp of a tooth submitted to cavity preparation becomes more sensitive to other damaging agents, such as heat from a light source, compared to the pulp of an intact tooth. Nevertheless, intrapulpal temperature rises may damage the pulp and should be avoided as much as possible (Hansen & Asmussen, 1993).

For filling those cavities, in general, restorative materials that require curing (polymerization) by photo activation are used. Resin composites, adhesive systems, hybrid materials and resin cements are examples of materials that use the blue light of light-curing units to initiate polymerization reaction (Hansen & Asmussen, 1993; Hannig & Bott, 1999). Adequate polymerization is a crucial factor in obtaining optimal physical performance of these materials (Knezevic & others, 2001). There are several light-curing units with different intensities and photo activation modes available in dentistry. These photo activation modes have been studied, for the purpose of reducing curing time and controlling the stress caused by polymerization shrinkage without decreasing the physical properties of the resin composite.

This *in vitro* study evaluated the effect of five different polymerization modes on the temperature rise in human dentin of three different thicknesses to evaluate the effect of the presence of resin composite on dentin temperature rise and to evaluate the relation between dentin thickness and temperature rise.

METHODS AND MATERIALS

A week after extraction, three sound, human third molars were cleaned, polished and examined in order to eliminate those with cavities. A cylindrical specimen of deep dentin was obtained from each tooth. The crown of each tooth was set in an acrylic plaque that was fixed in a precision slow speed water cooled diamond saw (Imptech PC10, Equilam Lab Equip, Diadema-SP Brazil) with two parallel disks distanced 1, 2 or 3 mm from each other and perpendicular to the buccal surface of each of the three teeth. The disks were positioned at the crown of each tooth, 3 mm above the cemento-enamel junction. Each tooth was cut in the mesial-distal direction, resulting in a cylindrical specimen of deep dentin. The test design used to quantify the temperature rise of the specimens was adapted from Loney and Price (2001).

Each of the three specimens of dentin of different thicknesses was used during polymerization of the resin composite samples according to the 30 experimental groups (n=3) (Figure 1): three dentin sample thicknesses (1 mm, 2 mm and 3 mm); five polymerization modes (1-conventional; 2-soft-start; 3-high intensity; 4-ramp cure: progressive and high intensity; 5-high intensity with the tip of the light cure at a distance of 1.3 cm for 10 seconds, with the tip leaned in the sample) and levels of two resin composite presence (absence or presence of resin composite). Polymerization time was performed in accordance with manufacturers' recommendations.

Polymerization of the resin composite samples was randomly performed according to their experimental groups. For groups with resin composite between the dentin and light source, plastic rings 5.0-mm in internal diameter and 2.0-mm in depth were filled with P60 resin composite (3M ESPE, St Paul, MN, USA), held between two glass slabs separated by mylar strips, then pressed with a 50N load. After 20 seconds, the load, slabs and matrix strips were removed, the matrix was positioned (Figure 2) and polymerized with one of the polymerization modes tested in this study (Table 1). For the groups without resin composite, the matrix was positioned in the same way but was empty, and the light curing unit was turned on.

Between the light guide and thermometer, there was a Mylar strip, the matrix with or without resin composite and a cylinder of dentin (1-, 2- or 3-mm thick). A thermometer was positioned at a distance of 1 mm from

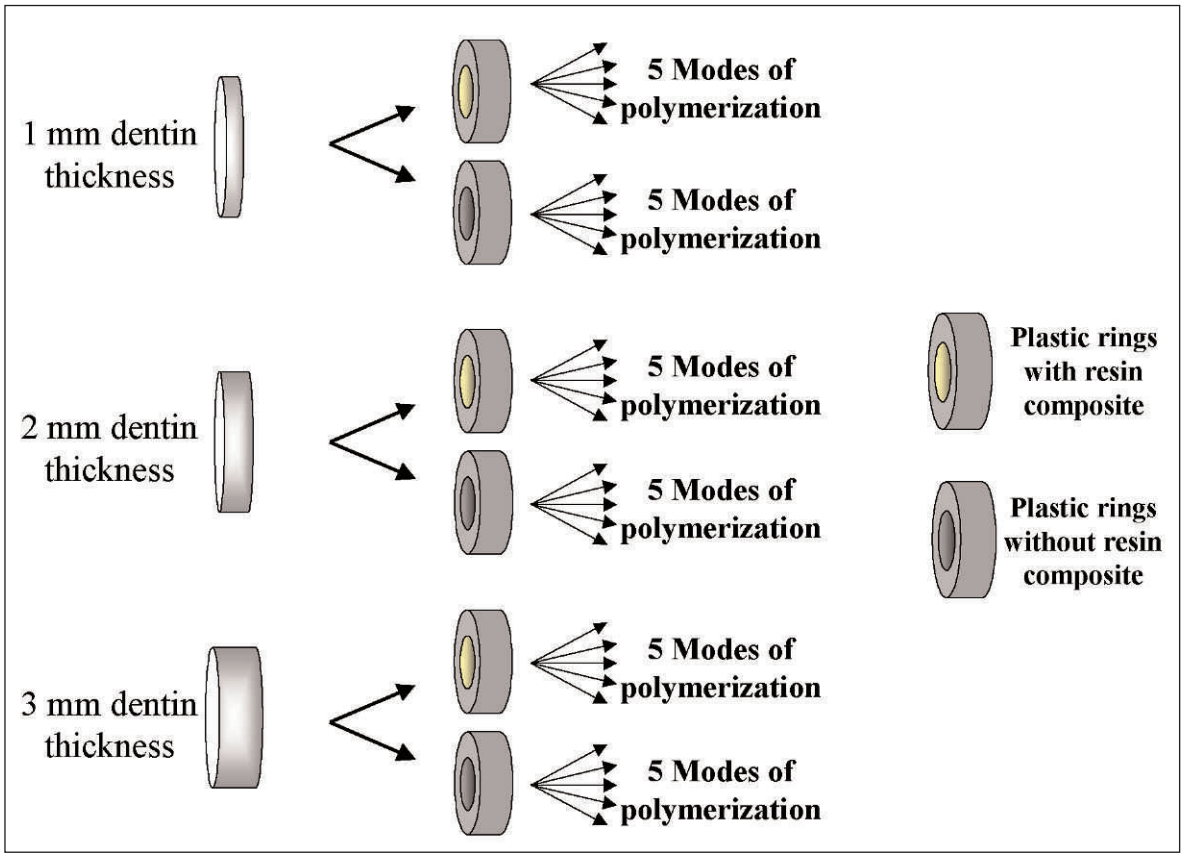

Figure 1: *Diagrammatic representation of the experimental groups.*

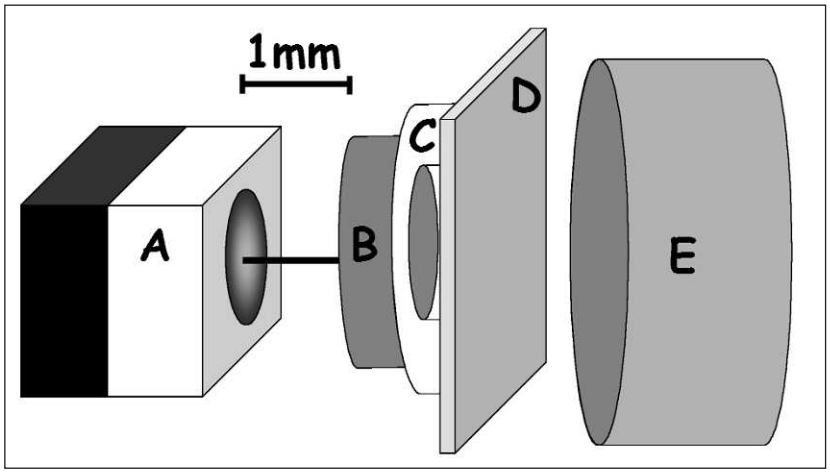

Figure 2. *Thermometer position, in relation to dentin specimen, plastic matrix and light curing unit tip. A) Digital laser thermometer; B) Dentin specimen; C) Plastic matrix; D) Mylar strip; E-Light curing unit tip.*

the dentin (Figure 2). For all measurements, only one cylinder of each thickness of dentin was used. These dentin specimens were kept in a physiological saline solution and air dried immediately prior to the test.

During polymerization, the temperature was measured by a digital laser thermometer (CMSS2000-SL/SKF, San Diego, CA, USA). The temperatures were recorded for all polymerization times of the specimens. The temperature was measured from the moment the light curing unit was turned on until the exact moment it was turned off. All measurements were performed in a room with controlled temperature (23.8°C) and humidity (58 ± 2%).

RESULTS

The means of temperature rise (TR) is presented in Table 2 and Figure 3. Three-way ANOVA and Tukey tests were performed (p=0.001). Power analysis (>0.99) was also performed and it supports a number of three specimens (n=3) in this study.

There were statistical differences in TR among the three factors studied (resin composite presence, polymerization modes and dentin thickness) and a double interaction between polymerization modes and dentin thickness. The highest TR occurred in those groups tested without resin composite with a dentin thickness of 1 mm. All groups tested without resin composite showed the highest TR

means, which was statistically different from those groups tested with resin composite ($p<0.001$).

Groups with a dentin thickness of 1 mm exhibited the highest TR means, which was significantly different from the 2-mm and 3-mm groups. Groups with 3-mm dentin thickness showed the lowest TR means and were significantly different from the 2-mm dentin thickness groups ($p<0.001$). With regard to polymerization modes, conventional polymerization showed the lowest TR means under almost all conditions, just as soft start showed the highest.

DISCUSSION

The polymerization modes of this study were tested at different intensities and for the time recommended by the light source manufacturers. The results of this study showed that the soft start polymerization mode showed the highest temperature changes in dentin specimens, irrespective of dentin thickness. The temperature rise of the distanced tip polymerization mode did not show statistical differences compared to the soft start mode for 0-, 1- and 3-mm dentin thicknesses. According to Loney and Price (2001), the difference in energy produced by the light curing units is an important factor for different temperature rises in different polymerization modes. For both polymerization modes, the energy produced is higher than that of the other modes of polymerization. For soft start polymerization, the energy produced was 19.6 J/cm^2 (20 seconds

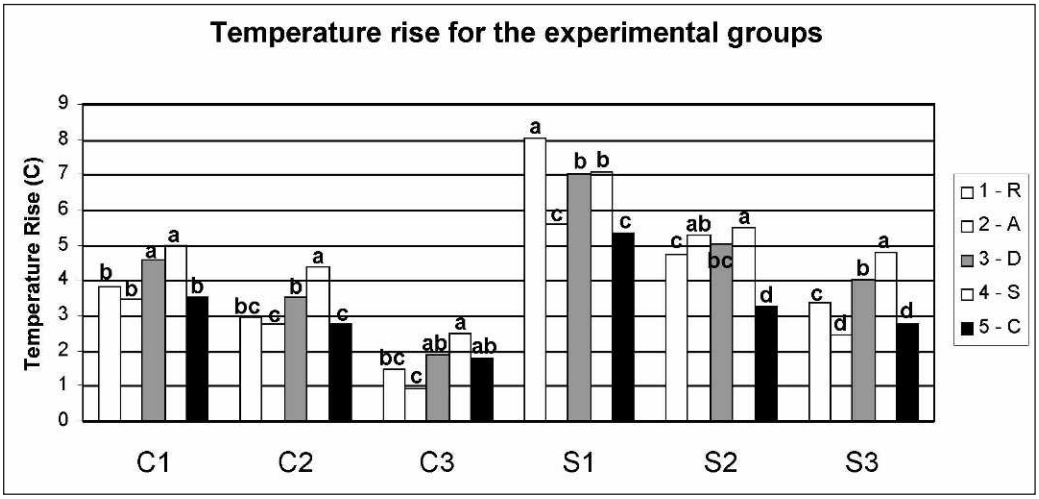

Figure 3. *Results of temperature rise (°C) for the experimental groups. R- ramp curing; A– high intensity; D- distanced tip; S- soft start; C- conventional; C1, C2, C3- groups with resin composite and dentin thickness of 1, 2 and 3 mm, respectively; S1, S2, S3—groups without resin composite and dentin thickness of 1, 2 and 3 mm, respectively. Mean values with the same letter were not statistically different (p<0.001) (same lower case letter were not statistically different for comparison with the same dentin thickness groups) for the same group with or without resin composite.*

Table 1: *Experimental Polymerization Modes (Degulux Soft Start Curing Light Device, Degussa, Hüls AG, Hanau, Germany; Demetron, Sds Kerr, Danbury, CT, USA; XL 3000, 3M ESPE, Grafenau, Germany)*

Experimental Groups	Light Curing Intensity	Light Curing Unit
Soft start mode	320mW/cm^2 - 20 seconds + 660mW/cm^2 - 20 seconds	Soft Start - Degussa
Distanced tip mode	380mW/cm^2 - 10 seconds + 128mW/cm^2 - 10 seconds	Optilux 501 C - Demetron
Ramp curing mode	0mW/cm^2 until 128mW/cm^2 - 10 seconds	Optilux 501 C - Demetron
High intensity mode	128mW/cm^2 - 10 seconds	Optilux 501 C - Demetron
Conventinal mode	580mW/cm^2 - 20 seconds	XL 3000 - 3M

Table 2: *Results of Temperature Rise Means (°C) for Experimental Groups (SD–standard deviation)*

	Light Curing Mode	Dentin Thickness (±SD) 1 mm	2 mm	3 mm
Without Resin Composite	Soft Start	7.07 (± 0.058)	5.50 (± 0.173)	4.77 (± 0.058)
	Distanced Tip	7.03 (± 0.208)	5.03 (± 0.208)	4.00 (± 0.265)
	Ramp Curing	8.03 (± 0.153)	4.73 (± 0.058)	3.37 (± 0.058)
	High Intensity	5.60 (± 0.100)	5.27 (± 0.578)	2.43 (± 0.208)
	Conventional	5.33 (± 0.058)	3.27 (± 0.058)	2.73 (± 0.153)
With Resin Composite	Soft Start	5.00 (± 0.300)	4.37 (± 0.208)	2.47 (± 0.503)
	Distanced Tip	4.60 (± 0.300)	3.53 (± 0.379)	1.90 (± 0.300)
	Ramp Curing	3.83 (± 0.208)	2.93 (± 0.289)	1.50 (± 0.265)
	High Intensity	3.43 (± 0.231)	2.73 (± 0.252)	0.90 (± 0.200)
	Conventional	3.50 (± 0.346)	2.73 (± 0.321)	1.80 (± 0.173)

@ 320 mW/cm² + 20 seconds @ 660 mW/cm²), and for the distanced tip polymerization mode, the energy was 15.6 J/cm² (10 seconds @ 380 mW/cm² + 10 seconds @ 1280 mW/cm²).

The conventional polymerization mode showed the lowest means of temperature rise for the 0-, 1- and 2-mm dentin thicknesses. This polymerization mode produced lower energy than was produced by other polymerization modes–11.6 J/cm² (20 seconds @ 560mW/cm²). The high intensity polymerization mode did not differ statistically from the conventional mode for the 1- and 2-mm thicknesses, and the energy produced by this polymerization mode was 12.8 J/cm² (10 seconds @ 1280 mW/cm²). Although the energy produced by the ramp curing mode was approximately 15.6 J/cm² (0mW/cm² until 1280mW/cm²–10 seconds + 1280mW/cm²–10 seconds), this mode did not differ from the high intensity mode in any of the dentin thicknesses and from soft start in the 1- and 2-mm dentin thicknesses.

The energy produced by the polymerization mode was as dependent on polymerization time as on intensity of the light. However, in some clinical situations, the use of the soft start polymerization mode (Mehl, Hickel & Kunzelmann, 1997; Burgess & others, 1999; Friedl & others, 2000), ramp curing or distancing the light source from the resin composite surface (Dennison & others, 2000; Aguiar, Ajudarte & Lovadino, 2002) is essential for decreasing stress during polymerization (Friedl & others, 2000), improving the marginal sealing of the restoration and reducing microleakage (Aguiar & others, 2002). However, these techniques require long cure times and, consequently, increase the energy produced, raising the temperature of the resin composite and surrounding dentin. In deep cavities, these techniques should be used with caution or glass ionomer cement could be used on the pulp cavity floor to avoid excessively heating the pulp (Hansen & Asmussen, 1993).

Another factor involved in temperature rise is the exothermic reaction of the resin composite curing reaction. However, for the three dentin thickness studied, in all light curing modes, the means of groups without resin composite were higher and statistically different from the means of the groups with resin composite. Although the setting reaction of the resin composite produces heat (Lloyd, 1984; Hansen & Asmussen, 1993), the decisive factor for temperature rise during light-activated polymerization of resin composites is the energy absorbed during irradiation (Hannig & Bott, 1999; Shortall & Harrington, 1998).

The exothermic resin composite polymerization process is a secondary factor in temperature rise (Hannig & Bott, 1999) and is dependent on the energy that arrives in the deepest part of the increment (Shortall & Harrington, 1998). Thus, the exothermic reaction depends on the intensity of the light source and filling material thickness. In this study, the increment was 2 mm. According to Rueggeberg and others (1994), as light passes through the bulk of the restorative material, the intensity is greatly decreased, thus decreasing curing potential. On the other hand, clinicians should be prudent when they light cure the adhesive. Perhaps, due to the fact that this material is not very thick, the temperature could, as shown by the results of this study, increase in those groups that were light cured without resin composite.

The last factor determining temperature rise considered in this study was the thickness of the residual dentin. For all light curing modes, the temperature was higher for those samples with 1-mm thick dentin. Prati and others (1999) demonstrated that the intensity of the light is highly reduced by dentin and enamel. The results of this study show the importance of dentin as natural pulp protection. In their *in vivo* study, Camps and others (2000) described the remaining dentin thickness as an extremely important factor for avoiding pulpal reactions. In this study, the thicker the dentin, the less the temperature increased, due the low heat conductivity of dentin (Tjan & Dunn, 1988).

In vivo studies must been done to confirm the relation of composite polymerization and pulpal reactions in deep cavities. It is difficult to establish a direct relation to an *in vitro* and *in vivo* result; however, *in vitro* studies may guide one to conclusions that can be later confirmed *in vivo*, then improve clinical activity. This study, as with Loney and Price's study (2001), represents a clinical scenario where the light-curing tip touched a clear Mylar matrix that was in direct contact with the resin composite. This resin composite was also in contact with the dentin specimen. In clinical situations, this situation is unlikely to occur, but in some cases, such as Classes IV and V, it is possible. In Class I, II and III, the light curing tip is far from the resin composite and the intensity of the light that hits the first increment, in contact with the dentin, is lower (Prati & others, 1999). On the other hand, the Mylar matrix placed between the resin composite and light tip in this study caused an attenuation of the light source energy (Loney & Price, 2001). Nonetheless, Hussey, Biagioni and Lamey (1995), in an *in vivo* study, demonstrated the resin composite temperature rise could increase up to 12°C.

CONCLUSIONS

Within the limits of this study, it can be concluded that:

1. The presence of resin composite reduced the temperature rise;
2. Conventional and high intensity polymerization modes showed lower means of tempera-

ture rise than soft start, distanced tip and ramp curing polymerization modes;

3. Dentin thickness is inversely proportional to temperature rise.

Acknowledgement

The Ethical Committee in Research, protocol n.11/2003, at Piracicaba School of Dentistry, Campinas State University, SP, Brazil, approved this investigation.

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The Effect of 10% Carbamide Peroxide, Carbopol and/or Glycerin on Enamel and Dentin Microhardness

RT Basting • AL Rodrigues, Jr • MC Serra

Clinical Relevance

Changes in enamel and dentin microhardness may be related not only to carbamide peroxide, but also to the presence of other components in bleaching agents, such as carbopol and glycerin. Carbopol and its associations may cause alterations in microhardness compared to Opalescence. None of the treatment agents or associations evaluated was inert for dental microhardness, although glycerin seemed to affect enamel and dentin to a lesser degree.

SUMMARY

This study evaluated the effects of 10% carbamide peroxide, carbopol and glycerin and their associations on microhardness over time on enamel and dentin. Eight treatment agents were evaluated: a commercial bleaching agent containing 10% carbamide peroxide (Opalescence 10% Ultradent), 10% carbamide peroxide, carbopol, glycerin, 10% carbamide peroxide + carbopol, 10% carbamide peroxide + glycerin, carbopol + glycerin and 10% carbamide peroxide + carbopol + glycerin. Three hundred and twenty human dental fragments, 80 sound enamel fragments (SE), 80 demineralized enamel fragments (DE), 80 sound dentin fragments (SD) and 80 demineralized dentin (DD) fragments, were exposed to the treatment agents (n=10). These agents were applied onto the surface of the fragments eight hours a day for 42 days. After eight hours, they were washed from the dental fragment surfaces after five back-and-forth movements with a soft bristle toothbrush under distilled and deionized running water. During the remaining time (16 hours per day), the fragments were kept in individual vials in artificial saliva. After the 42-day treatment period, the specimens were kept individually in artificial saliva for 14 days. Knoop microhardness measurements were performed at baseline, after eight hours, and 7, 14, 21, 28, 35 and 42 days, and 7 and 14 days post-treatment (corresponding to 49 and 56 days after the initial treatment agent applications). The non-parametric Kruskal-Wallis analysis showed significant differences among the agents at each time interval, except at baseline for sound and demineralized enamel and dentin. For SE, SD and DD, there was a decrease in microhardness values during treatment with all agents. There was a tendency towards lower microhardness values after treatment with carbopol and its associa-

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tions for sound tissues. DD showed low microhardness values during and after treatment with CP and its associations. For DE, there was an increase in microhardness values during treatment with all agents and in the post-treatment phase. The baseline microhardness values were not recovered during the 14-day post-treatment phase. Opalescence 10%, carbamide peroxide, carbopol, glycerin and their associations may change the microhardness of sound and demineralized dental tissues, even in the presence of artificial saliva.

INTRODUCTION

Bleaching procedures with 10% carbamide peroxide agents have been used as a simple and effective technique for the removal of intrinsic and extrinsic stains (Haywood, 1994, 2000). The clinical protocol employs a bleaching agent in a tray for two to eight hours during the day or night for two to six weeks of treatment (Haywood & Heymann, 1989; Haywood, 2000; Ritter & others, 2002).

Ten percent carbamide peroxide seems to be effective and safe (Curtis & others, 1996; Ritter & others, 2002) and has the American Dental Association acceptance seal for some brands (Haywood, 1993; Haywood & Robinson, 1997). The addition of carbopol and glycerin as thickening agents improves adherence of the bleaching agent to the surface of dental structure, allowing for a prolonged time for the release of carbamide peroxide (Haywood, 1994; McCracken & Haywood, 1996).

Because the bleaching of vital teeth involves direct contact of the whitening agent with the outer surface of enamel and dentin in areas of defects, abfraction or abrasion lesions, exposed root surfaces and marginal areas between teeth and restorations, many studies have evaluated the potential effects of these agents on superficial micromorphology, changes in mineral content and microhardness. Scanning electron microscopic evaluations have reported porosities and erosion on enamel (Ben-Amar & others, 1995; Bitter, 1998; Bitter & Sanders, 1993; Ernst, Marroquin & Willershausen-Zonnchen, 1996; Flaitz & Hicks, 1996; Josey & others, 1996; Shannon & others, 1993; Smidt, Weller & Roman, 1998; Zalkind & others, 1996) and dentin (Zalkind & others, 1996). *In vitro* studies have also reported some alterations in mineral content and both enamel and dentin microhardness after exposure to 10% carbamide peroxide (Attin & others, 1997; Basting, Rodrigues Jr & Serra, 2003; Freitas & others, 2002; de Oliveira & others, 2003; McCracken & Haywood, 1995, 1996; Pécora & others, 1994; Rodrigues & others, 2001; Rotstein & others, 1996; Smidt & others, 1998; Seghi & Denry, 1992).

Changes in enamel and dentin microhardness may be related not only to the acidic pH of the bleaching agents, which is responsible for a prolonged storage time of the product, but also to the presence of other components in commercial bleaching agent products. McCracken and Haywood (1995) verified a significant decrease in microhardness in the outer 25.0 µm of enamel surface after treatment with a product containing carbopol. Basting and others (2003) also reported a significant decrease in enamel surface microhardness when using a placebo agent with carbopol and glycerin with a neutral pH, even in the presence of artificial saliva. Freitas and others (2002) showed the same behavior for this product in dentin. However, no *in vitro* studies evaluated the effects of bleaching agents on demineralized dental tissues. Bleaching agents have possibly been applied to active carious lesions in enamel and dentin.

In an *in situ* study, Basting, Rodrigues Jr and Serra (2001) observed significant differences in enamel microhardness after treatment with 10% carbamide peroxide bleaching agent and a placebo containing carbopol and glycerin. The sound and demineralized enamel submitted to the 10% carbamide peroxide bleaching agent showed significantly lower microhardness values than that submitted to a placebo agent. However, no differences were found between the sound and demineralized dentin treated with bleaching or placebo agents, but slightly higher microhardness values for dentin exposed to a bleaching product.

However, the isolated effects of carbopol, glycerin and 10% carbamide peroxide, and even the combined effects of those components on the microhardness of sound and demineralized enamel and dentin tissues, are also unknown.

This study evaluated *in vitro* the effects of 10% carbamide peroxide, carbopol, glycerin and their associations on the microhardness of sound and demineralized enamel and dentin tissues and compared their values with those of a 10% carbamide peroxide commercial bleaching product at different time intervals.

METHODS AND MATERIALS

Experimental Design

The factors under study were:

Treatment Agents (eight levels): Opalescence 10% Ultradent, 10% carbamide peroxide; carbopol, glycerin, 10% carbamide peroxide + carbopol, 10% carbamide peroxide + glycerin, carbopol + glycerin, and 10% carbamide peroxide + carbopol + glycerin (Table 1).

Time (nine levels): baseline, 8 hours, and 7, 14, 21, 28, 35 and 42 days of treatment, and 7 and 14 days post-treatment period (corresponding to 49 and 56 days after the beginning of the bleaching treatment).

The experimental units consisted of 320 dental slabs: 80 sound enamel slabs; 80 demineralized enamel slabs;

80 sound dentin slabs and 80 demineralized dentin slabs. Ten dental fragments of each dental tissue (n=10) were randomly and evenly assigned to the eight different treatment agents. The effects of the different treatment agents on enamel were not compared to dentin, neither were the effects of sound tissues compared to the demineralized ones.

Three repeated measurements of Knoop microhardness were taken from the surface of each specimen at each time interval.

Dental Fragments Preparation

This study had the approval of the FORP/USP Ethical Committee Guidelines in accordance with the National Health Council (Conselho Nacional de Saúde, 2003). Seventy-seven non-erupted third molars were used. Immediately after extraction for reasons other than the experiment, the teeth were kept in 0.1% thymol. They were sectioned with double-faced diamond discs (KG Sorensen, Barueri, SP, Brazil) at a low motor speed (Kavo do Brasil, Joinville, SC, Brazil), dividing the root from the coronary portion to obtain 320 dental slabs with 3 mm x 3 mm x 2 mm (160 enamel slabs and 160 dentin slabs). In the root, the apical third was discarded and only the cervical region was used. Care was taken not to leave the dental fragments dehydrated for long periods. Those slabs that presented stains or cracks after observation under stereomicroscope loupe (Meiji Techno EMZ Series, Saitama, Japan) at 30x were discarded.

The dental fragments were embedded individually in a self-curing polyester resin in a polyvinylchloride ring mold 2.0-cm in diameter, with the external surface of the enamel or dentin exposed. The molds were removed and the external surfaces of the dental fragments were leveled by a water-cooled mechanical grinder (Maxgrind/Solotest, São Paulo, SP, Brazil). These procedures were conducted to form parallel planar surfaces for the Knoop microhardness tests. For the enamel surfaces, aluminum oxide discs of 400, 600 and 1000 grit were used sequentially (Carborundum/3M do Brasil Ltda, Sumaré, SP, Brazil) with water cooling. Polishing was performed using polishing cloths (Top, Gold and Ram, Arotec Ind e Com Ltda, Cotia, SP, Brazil) and diamond pastes of 6, 3, 1 and 1/4 µm (Arotec Ind e Com Ltda) with mineral oil cooling (Red lubricant, Arotec Ind e Com Ltda). For the dentin fragments, only aluminum oxide discs were used in a sequential granulation of 600, 1000 and 1200 grit (Carborundum/3M do Brasil Ltda, Sumaré, SP, Brazil) with water cooling. Between each sequential disc, the dental fragments were immersed in an ultrasonic distillated water bath for 10 minutes.

Dental Slabs Preparation

To obtain 80 demineralized enamel slabs and 80 demineralized dentin slabs, caries-like lesions were generated by a dynamic model of demineralization and remineralization cycles similar to the model proposed by Featherstone and others (1986) and modified by Delbem and Cury (2002).

The enamel and dentin fragments were submitted to cycles of de-remineralization. The group that made up the sound group of each dental tissue was not submitted to the de-remineralization cycles; instead, the specimens were stored in a humid environment.

Specification of the Treatment Agents

The treatment agents are presented at Table 1 according to composition and manufacturer.

A 10% carbamide peroxide commercial bleaching agent (Opalescence 10% Ultradent, South Jordan, UT, USA) was used as a control as it is accepted as safe and effective by the American Dental Association (ADA). It contains 10% carbamide peroxide and amounts of carbopol and glycerin not specified by the manufacturer. The flavor tested was "regular."

The treatment agents evaluated include 10% carbamide peroxide (CP), carbopol (C) and glycerin (G). Their associations were also tested: 10% carbamide peroxide + carbopol (CP + C), 10% carbamide peroxide + glycerin (CP + G), carbopol + glycerin (C + G) and 10% carbamide peroxide + carbopol + glycerin (CP + C + G).

Table 1: *Composition and Manufacturer of Each Treatment Agent*

Treatment Agents	Composition	Manufacturer
Opalescence 10% (OPA)	10% carbamide peroxide; carbopol; glycerin; flavoring*	Ultradent Products Inc, South Jordan, UT, USA
10% carbamide peroxide (CP)	10% carbamide peroxide	Proderma – Pharmacy, Piracicaba, SP, Brazil
Carbopol (C)	Carbopol	Proderma – Pharmacy, Piracicaba, SP, Brazil
Glycerin (G)	Glycerin	Proderma – Pharmacy, Piracicaba, SP, Brazil
10% carbamide peroxide + carbopol (CP + C)	10% carbamide peroxide + carbopol	Mixed formula, Proderma – Pharmacy, Piracicaba, SP, Brazil
10% carbamide peroxide + (CP + G)	10% carbamide peroxide + glycerin	Mixed formula, Proderma – Pharmacy, Piracicaba, SP, Brazil
Carbopol + glycerin (C + G)	Carbopol + glycerin	Mixed formula, Proderma – Pharmacy, Piracicaba, SP, Brazil
10% carbamide peroxide + carbopol + glycerin (CP + C + G)	10% carbamide peroxide + carbopol + glycerin	Mixed formula, Proderma – Pharmacy, Piracicaba, SP, Brazil

**The manufacturer does not indicate the percentage of each component.*

These products were freshly obtained and/or prepared in a dispensing pharmacy. The consistency of the association of carbamide peroxide + carbopol + glycerin and carbopol + glycerin was similar to the commercial product.

Application of Treatment Agents

Prior to treatment, an individual tray for each specimen was manufactured from a 1.0-mm thick flexible ethyl vinyl acetate polymer (Bio-Art Equipamentos Odontológicos Ltda, São Carlos, SP, Brazil) placed in a vacuum-forming machine (P7, Bio-Art Equipamentos Odontológicos Ltda).

Both the sound and demineralized enamel and dentin fragments were exposed to the treatment agents eight hours a day for 42 days. A syringe was used to apply 0.02 mL of each agent to each specimen. The specimens were individually covered with a tray and soaked in individual closed vials with 13.5 mL of artificial saliva (pH 7.0) at 37°C ± 2°C.

After eight hours, the treated specimens were taken out of the storage media and the trays removed. The treatment agents were removed from the dental fragment surfaces by making five back-and-forth movements with a soft bristle toothbrush (Oral B 35/Gillette do Brasil Ltda, Manaus AM, Brazil) to remove the viscous film that formed on the fragment surfaces, which was then washed under distilled and deionized running water for five seconds.

During the remaining daily time (16 hours per day), the fragments were kept in individual vials with 13.5 mL of artificial saliva (pH 7.0) at 37.0°C ± 2.0°C. The artificial saliva was changed every two days and consisted of a remineralization solution proposed by Featherstone and others (1986) and modified by Delbem and Cury (2002). It contained calcium hydroxide, phosphoric acid, potassium chloride, buffering agent and deionized and distilled water.

This cycle was repeated for 42 days, corresponding to the maximum clinical period for a bleaching treatment of six weeks as recommended by Haywood and Heymann (1989).

Post-treatment Phase

After the 42-day treatment period, the specimens were kept in their individual vials with 13.5 mL of artificial saliva (pH 7.0) at 37.0°C ± 2.0°C during 14 days. The artificial saliva was also changed every two days. Thus, the possible remineralizing effects of the artificial saliva on the microhardness of sound and demineralized dental fragments could be evaluated.

Microhardness Testing

Microhardness measurements were taken before initial exposure to the treatment agents (baseline) after 8 hours and 7, 14, 21, 28, 35 and 42 days, and 7 and 14 days post-treatment (corresponding to 49 and 56 days after initial application of the treatment agents). A Knoop indenter was used; the long axis of the diamond was kept parallel to the dentinal surface in a microhardness testing machine (Future Tech, FM-1e, Tokyo, Japan). Three microhardness measurements were taken on each specimen at different time intervals. A load of 25.0 grams was used for the enamel fragments and a load of 10.0 grams was used for the dentin for five seconds.

Statistical Analysis

Knoop microhardness responses were statistically evaluated by Kruskal-Wallis test, followed by pair-wise multiple comparison (Conover, 1971), according to dental slab tissue/type (enamel or dentin, sound or demineralized). Means were obtained after triplicates were averaged. The response value at each time was then subtracted from its respective baseline mean, yielding the ultimate response value.

RESULTS

Statistical analysis showed significant differences among the agents at each time interval, except at baseline, for sound and demineralized enamel and dentin.

Table 2: *Means and the Results of Pair-wise Comparisons of the Knoop Microhardness Difference Values for Sound Enamel Slabs*

Time (hours)	Treatment Agents							
	OPA	CP	C	G	CP + C	CP + G	C + G	C + G + CP
8	-71.4 d	-32.6 g	-200.8 a	-34.1 f	-205.3 a	-73.8 e	-113.1 b	-84.6 c
168	-103.2 b	-50.4 d	-297.8 a	-83.9 c	-303.3 a	-140.3 b	-306.5 a	-87.9 c
336	-83.8 c	-26.1 c	-314.0 a	-71.2 d	-310.0 a	-83.0 c	-302.6 a	-89.2 b
504	-106.9 c	-29.8 d	-317.7 a	-44.8 d	-311.7 b	-108.9 c	-325.3 ab	-95.1 c
672	-140.4 b	-14.3 e	-316.7 a	-54.6 d	-310.7 a	-106.6 c	-324.7 a	-97.4 c
840	-107.8 c	-4.9 f	-318.4 a	-56.5 e	-309.0 ab	-70.7 d	-305.0 b	-107.7 c
1008	-120.3 b	-7.0 e	-317.9 a	-27.5 d	-305.4 a	-99.7 c	-313.4 a	-109.6 b
1176	-90.2 b	18.9 d	-301.7 a	-27.1 c	-308.8 a	-60.2 c	-301.0 a	-94.8 b
1344	-76.7 c	1.5 ed	-298.4 a	-52.7 e	-298.7 a	-44.3 d	-303.4 a	-82.0 b

**Equal letters horizontally indicate mean values that are not significantly different.*

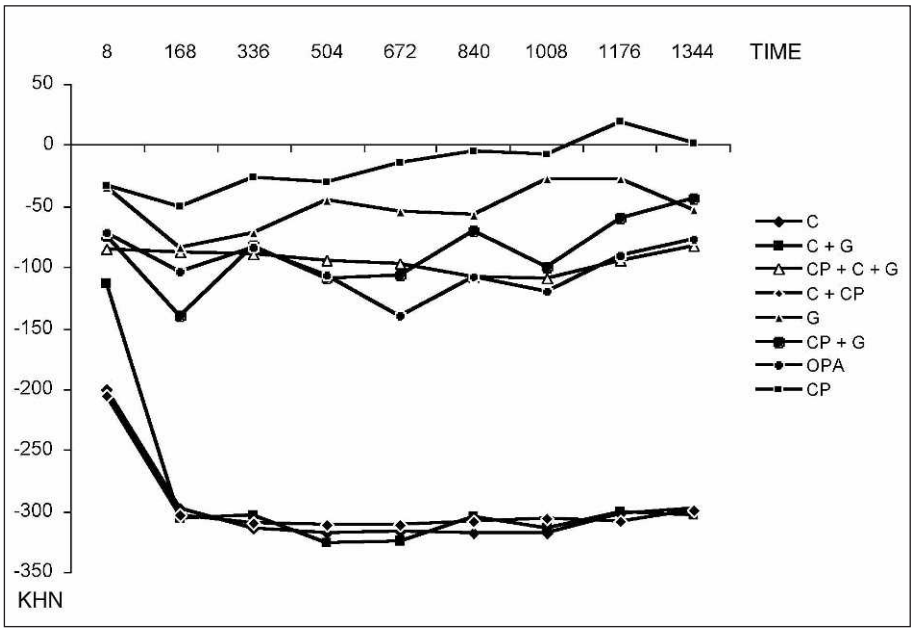

Figure 1: *Linear diagram of the means of Knoop Microhardness Number (KHN) differences for each treatment agent over time for sound enamel slabs.*

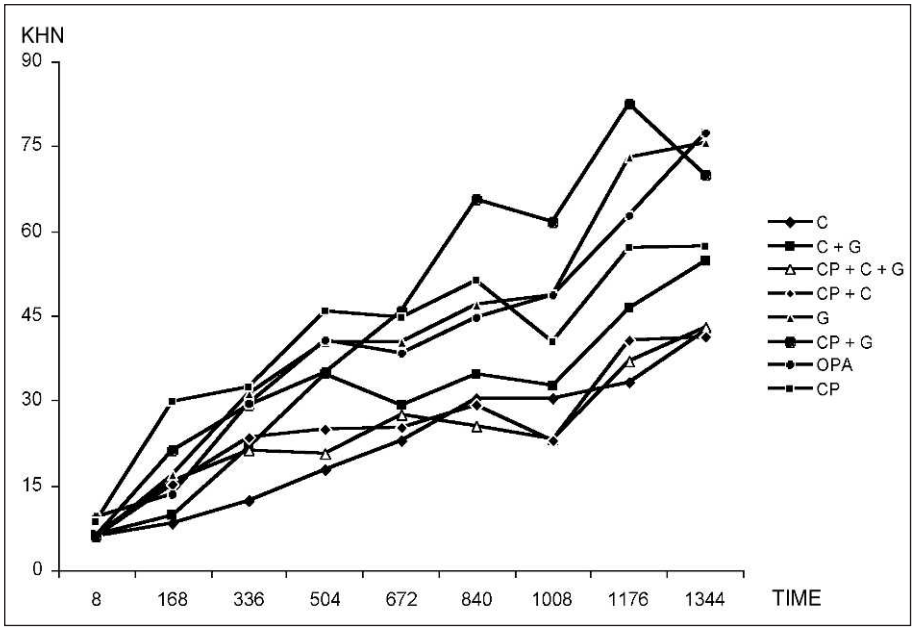

Figure 2: *Linear diagram of the means of Knoop Microhardness Number (KHN) differences for each treatment agent over time for demineralized enamel slabs.*

Table 2 and Figure 1 show the means and results of the pair-wise multiple comparisons for the sound enamel fragments. There was a decrease in enamel microhardness values over time for all agents evaluated. Lower microhardness values were obtained after treatment with C, C + G and CP + C, even eight hours after application and during the post-treatment period. An increase in microhardness values above baseline was observed when using CP during the post-treatment phase.

For the demineralized enamel fragments, there was an increase in microhardness values during treatment with all the agents and in the post-treatment phase (Table 3 and Figure 2).

There was a decrease in microhardness values for sound dentin fragments after treatment with all agents; however, lower values were obtained with the use of OPA, C, CP + C, CP + G and CP + C + G (Table 4 and Figure 3).

The demineralized dentin fragments showed a decrease in microhardness values during the treatment period for all agents, mainly after the application of CP, CP + G, C + G and CP + C + G. However, these fragments showed an increase in microhardness values during the post-treatment phase, which was not observed for the other dental tissues (Table 5 and Figure 4).

DISCUSSION

Although research has been conducted to evaluate the effects of bleaching agents on enamel and dentin (Attin & others, 1997; Ben-Amar & others, 1995; Bitter, 1998; Bitter & Sanders, 1993; Ernst & others, 1996; Flaitz & Hicks, 1996; Josey & others, 1996; McCracken & Haywood, 1995, 1996; Nathoo, Chmielewski & Kirkup, 1994; Pécora & others, 1994; Rodrigues & others, 2001; Rotstein & others, 1996; Seghi & Denry, 1992; Shannon & others, 1993; Smidt & others, 1998; Zalkind & others, 1996), it does not consider the isolated effects of each component of these products, which may adversely affect dental hard tissues. Different brands of 10% carbamide peroxide bleaching agents present different effects on enamel and dentin, and this variation may be related to the composition of each product (Basting & others, 2003; McCracken & Haywood, 1996).

The chemistry of carbamide peroxide bleaching agents is based on its ability to generate free radicals, which are highly reactive. The free radicals of hydrogen peroxide are non-specific, extremely unstable and can potentially react not only with the pigmented carbon rings, but also with other organic molecules to achieve stability (Goldstein & Garber, 1995). Thus, changes in the chemical or morphological structure of a tooth must

be of concern when using bleaching techniques as a treatment for whitening teeth. Although some studies have reported no significant changes in dental microhardness when using short-term regimens of carbamide peroxide (Nathoo & others, 1994; Potocnik, Kosec & Gaspersic, 2000; Seghi & Denry, 1992; Shannon & others, 1993), others observed a decrease in enamel and dentin microhardness when using these bleaching agents for two weeks or more, even with the use of artificial saliva or fluoride solutions (Attin & others, 1997; Basting & others, 2003; Freitas & others, 2002; McCracken & Haywood, 1995; de Oliveira & others, 2003; Rodrigues & others, 2001; Smidt & others, 1998).

In this study, a decrease in microhardness for sound enamel and dentin was found even after eight hours of treatment with all agents. Although the remineralizing effect of saliva was expected during the 16-hours of immersion in artificial saliva, a slow, continuing decrease and maintenance of low values of enamel and dentin microhardness was observed throughout the experimental phase.

Some of the thickening agents in saliva substitutes generally use carbopol, carboxymethylcellulose or other polymers (Christersson, Lindh & Arnebrant, 2000; van der Reijden & others, 1997). In this study, the artificial saliva used was supersaturated in minerals and no salivary proteins were considered (Featherstone & others, 1986), but its remineralization effect was observed during the post-treatment period.

Polymers used as thickening agents for saliva substitutes largely inhibited further demineralization, except carbopol, which causes demineralization, especially in a remineralization solution. Carbopol completely inhibited hydroxyapatite crystal growth because of its high calcium-binding capacity (van der Reijden & others, 1997). Carbopol was not added as an ingredient to the artificial saliva used in this study, but it was evaluated alone or in combination with glycerin and carbamide peroxide. A decrease in microhardness values for sound enamel and dentin during treatment with almost all agents containing carbopol was observed, showing a continuing demineralization of enamel and dentin at neutral pH. In a microhardness evaluation comparing the effects of two 10% carbamide peroxide bleaching agents with and without carbopol on enamel, McCracken and Haywood (1995) showed a significant decrease in microhardness in the outer 25 µm of the enamel surface after treatment with the product containing carbopol. This difference was related not only to the pH

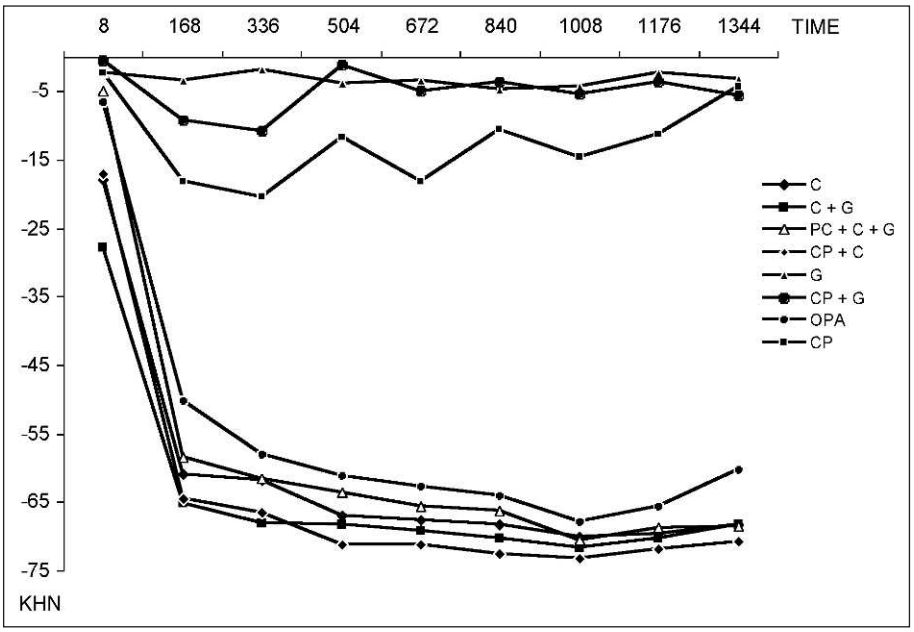

Figure 3: *Linear diagram of the means of Knoop Microhardness Number (KHN) differences for each treatment agent over time for sound dentin slabs.*

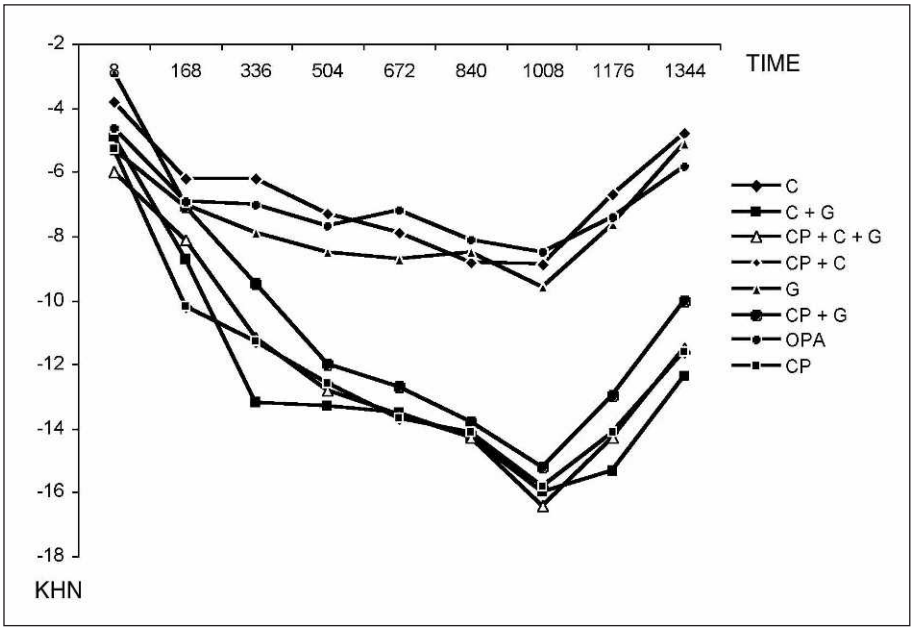

Figure 4: *Linear diagram of the means of Knoop Microhardness Number (KHN) differences for each treatment agent over time for demineralized dentin slabs.*

Table 3: *Means and the Results of Pair-wise Comparisons of the Knoop Microhardness Difference Values for Demineralized Enamel Slabs*

Time (hours)	Treatment Agents							
	OPA	CP	C	G	CP + C	CP + G	C + G	C + G + CP
8	9.4 d	8.5 d	6.1 ab	5.8 a	6.0 ab	5.9 c	6.3 abc	6.4 bc
168	13.5 c	29.7 h	8.2 a	17.0 f	15.2 d	21.3 g	9.7 b	15.7 e
336	29.5 d	32.3 d	12.4 a	31.2 d	23.5 c	29.2 d	21.8 c	21.2 b
504	40.7 f	46.0 f	17.9 a	40.4 e	24.8 b	35.0 d	34.8 c	20.7 a
672	38.4 c	44.7 d	23.0 a	40.3 c	25.2 a	45.9 e	29.1 b	27.6 a
840	44.8 d	51.4 d	30.4 b	47.0 d	29.3 a	65.7 e	34.8 c	25.4 a
1008	48.6 e	40.5 d	30.3 b	48.7 e	22.9 a	61.5 f	32.8 c	23.3 a
1176	62.8 d	57.1 c	33.2 a	73.2 d	40.8 a	82.6 e	46.4 b	36.9 a
1344	77.5 f	57.3 d	42.5 b	75.8 e	41.2 a	69.9 e	54.7 c	43.1 b

**Equal letters horizontally indicate mean values that are not significantly different.*

Table 4: *Means and the Results of Pair-wise Comparisons of the Knoop Microhardness Difference Values for Sound Dentin Fragments*

Time (hours)	Treatment Agents							
	OPA	CP	C	G	CP + C	CP + G	C + G	C + G + CP
8	-6.6 c	-2.2 e	-18.0 b	-2.3 e	-17.0 ab	-0.5 e	-27.8 a	-4.9 d
168	-50.1 e	-18.1 f	-60.8 c	-3.3 h	-64.4 b	-9.2 g	-65.1 a	-58.4 d
336	-58.0 e	-20.4 f	-61.9 c	-1.9 h	-66.4 b	-10.7 g	-68.0 a	-61.5 d
504	-61.2 e	-11.7 f	-67.0 c	-3.8 g	-71.3 a	-1.1 g	-68.2 b	-63.6 d
672	-62.7 d	-18.1 e	-67.5 c	-3.3 f	-71.3 a	-5.0 f	-69.1 b	-65.6 c
840	-64.0 e	-10.6 f	-68.3 c	-4.8 g	-72.6 a	-3.6 g	-70.3 b	-66.2 d
1008	-67.9 c	-14.6 d	-70.0 b	-4.2 e	-73.2 a	-5.3 e	-71.6 a	-70.5 a
1176	-65.6 d	-11.3 e	-69.7 c	-2.3 f	-71.9 a	-3.6 f	-70.2 ab	-68.8 b
1344	-60.3 c	-4.3 de	-68.3 b	-3.1 e	-70.7 a	-5.7 d	-68.3 ab	-68.4 ab

**Equal letters horizontally indicate mean values that are not significantly different.*

Table 5: *Means and the Results of Pair-wise Comparisons of the Knoop Microhardness Difference Values for Demineralized Dentin Fragments*

Time (hours)	Treatment Agents							
	OPA	CP	C	G	CP + C	CP + G	C + G	C + G + CP
8	-4.6 b	-5.3 c	-5.3 e	-2.9 d	-3.8 a	-5.3 cd	-4.9 b	-6.0 b
168	-6.9 de	-10.2 e	-10.2 g	-7.0 f	-6.2 c	-7.1 d	-8.7 a	-8.1 b
336	-7.0 d	-11.3 f	-11.3 h	-7.9 g	-6.2 c	-9.5 e	-13.2 b	-11.2 a
504	-7.7 c	-12.6 d	-12.6 f	-8.5 e	-7.3 b	-12.0 d	-13.3 b	-12.8 a
672	-7.2 b	-13.7 d	-13.7 e	-8.7 d	-7.9 a	-12.7 c	-13.5 a	-13.6 a
840	-8.1 c	-14.1 d	-14.1 e	-8.5 d	-8.8 a	-13.8 d	-14.3 b	-14.3 ab
1008	-8.5 c	-15.8 e	-15.8 f	-9.6 e	-8.9 a	-15.2 d	-16.0 b	-16.4 a
1176	-7.4 c	-14.1 de	-14.1 f	-7.6 e	-6.7 b	-13.0 d	-15.3 b	-14.3 a
1344	-5.8 b	-11.6 c	-11.6 f	-5.1 c	-4.8 a	-10.0 d	-12.4 a	-11.5 a

**Equal letters horizontally indicate mean values that are not significantly different.*

level of the products, but also to the presence of carbopol. Probably, the neutralizing effect of saliva in the mouth and the combination of carbopol with other components of bleaching agents may reduce its negative effect on dental microhardness, although other formulations may be developed for reducing the hazardous effects of this product on dental mineral content.

Although carbamide peroxide was thought to significantly change microhardness values for sound dental tissues due to the release of hydrogen peroxide and urea, this agent and its association with glycerin showed slight decreases compared to other agents evaluated, probably due to the rise in the hydrogen ion concentration (pH) of the solution (Haywood & Heymann,

1989). Urea is capable of penetrating into enamel and affecting not only the surface, but also the interprismatic regions of enamel. The increase in enamel permeability may cause structural changes (Arends & others, 1984; Goldberg & others, 1983) due to the dissociation of H-bonds between the CO and NH groups (Goldberg & others, 1983). Urea denatures proteins and causes conformational changes, although the increased porosity of the outer enamel surface shown by Hegedüs and others (1999) may be caused mainly by nascent oxygen when released in the inner structure. When using 10% carbamide peroxide on sound dental tissues for seven days, Zalkind and others (1996) showed moderate morphological changes in the dentin surface, but none in enamel. Rotstein and others (1996) also showed an increase in the calcium levels of enamel following treatment with 10% carbamide peroxide, although there was a decrease in the calcium/phosphorus ratio and potassium levels of dentin. Changes in the levels of these minerals may indicate damage to the organic component of the matrix, especially in dentin, due to the higher organic concentration.

Glycerin also presented slight decreases in microhardness for sound enamel and dentin, similar to the effect of carbamide peroxide. It could act as an adsorbed layer barrier to artificial saliva and to a remineralizing effect.

For demineralized enamel fragments, treatment with all agents and daily immersion in artificial saliva contributed to a remineralization process shown as an increase in microhardness values. However, microhardness decreases were observed for demineralized dentin. Haywood and Robinson (1997) have advocated the use of carbamide peroxide bleaching agents for initial caries lesions, mainly root caries, as the caries progression is retarded or stopped during bleaching. For demineralized dentin, the effect is a high decrease in microhardness values that could increase the depth of lesion formation, and bleaching should not be indicated as a common procedure. Although the post-treatment period seems to allow for an increase in microhardness values, probably due to a mineral deposition on dentin through a remineralization process, immersion of the fragments in artificial saliva did not provide recovery of the baseline values.

The demineralizing effects of agents, other than carbamide peroxide contained in bleaching agents, may play a role. As a general trend, 10% carbamide peroxide, carbopol, glycerin and their association seem to decrease sound enamel and dentin microhardness and demineralized dentin. Carbopol and its associations cause severe alterations in microhardness compared to Opalescence, which is a commercial brand available on the market. None of the agents evaluated were inert for dental microhardness, although glycerin seemed to affect enamel and dentin to a lesser degree. Thus, these results may be an advice warning to manufacturers to re-formulate the composition of some bleaching products or provide a better agent that does not cause enamel or dentin demineralization. The damage to sound and demineralized enamel and dentin in this experiment does not, however, necessarily imply demineralization *in vivo*, but should be kept in mind in further research.

CONCLUSIONS

Ten percent carbamide peroxide, carbopol, glycerin and their association decreased sound enamel and dentin microhardness and demineralized dentin. Carbopol and its associations caused alterations in microhardness, although glycerin seemed to affect enamel and dentin to a lesser degree.

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Surface Roughness of Different Dental Materials Before and After Simulated Toothbrushing *In Vitro*

SD Heintze • M Forjanic

Clinical Relevance

Brushing with toothpaste roughened the surface of most composite materials though, in general, hybrid composites were more prone to surface alterations than microfilled composites. Dental ceramics and amalgam, on the other hand, showed no increase in surface roughness after toothbrushing. On the contrary, the roughness of some ceramic materials even decreased, which was comparable to dentin and enamel.

SUMMARY

This study measured the effect of toothbrushing with a slurry of toothpaste on different dental materials that have been optimally polished. Specimens (n=8) of 21 dental materials, 16 resin composites, 1 amalgam and 4 ceramic materials, were subjected to 36,000 cycles (approximately five hours) of circular toothbrushing with a force of 1.7 N and a slurry of toothpaste (RDA 75) in a device for simulated toothbrushing. The unpolished enamel and dentin of extracted anterior teeth were used as a control. The mean roughness (Ra) was measured with an optical sensor (FRT MicroProf) before and after toothbrushing. To compare the roughness of the different materials, ANOVA with a post hoc Tukey B test was applied ($p<0.05$). Among the resin composites, the hybrid composites showed the greatest increase in mean roughness, while the microfilled composites and the compomer Compoglass F demonstrated the lowest increase. No statistically significant difference in roughness was found before and after simulated toothbrushing for the Amalcap amalgam, Esthet-X resin composite, TPH Spectrum resin composite, d.SIGN ceramic and the experimental ceramic. The other ceramic materials and dentin and enamel specimens showed a statistically significant decrease in mean roughness after simulated toothbrushing. Glazed Empress demonstrated a statistically significant higher initial roughness than polished Empress. For resin composites, no correlation was found between the mean particle size and mean roughness after simulated toothbrushing.

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INTRODUCTION

The surface texture of dental materials has a major influence on plaque accumulation, wear and discoloration of restorations which may eventually impair their aesthetic appearance, although other factors such as shade, shape and contour of the restoration contribute to the aesthetics, as well. Furthermore, a smooth surface adds to the patient's comfort, as even a

change in surface roughness on the order of 0.3 µm can be detected by the tip of the patient's tongue (Jones, Billington & Pearson, 2004). While ceramic materials are considered inert, meaning they do not change during wear in the oral cavity, composite materials suffer degradation due to mechanical and/or chemical interaction with the oral environment. In addition to intrinsic roughness caused by the food bolus, contact with the mucosal membrane, toothbrushing using a brush and paste plays an important role in the alteration of the surface roughness of restorative materials. Increasing roughness is correlated with increased deposition of plaque (Bollen, Lambrechts & Quirynen, 1997) and roughness is a determining factor for staining (Reis & others, 2003). *In vivo* studies on the threshold surface roughness for bacterial plaque retention showed that a mean roughness above 0.2 µm was related to a substantial increase in bacterial retention (Quirynen & others, 1996). On the other hand, increased surface roughness accelerates the wear of dental materials. For enamel-enamel contact areas, a mean roughness of 0.64 µm has been found on replicas from longitudinal clinical studies (Willems & others, 1991).

With regard to the effect of toothbrushing procedures on dental materials and dental hard tissues, most studies investigate wear after toothbrushing. The roughness of dental materials is mostly evaluated in combination with prophylactic pastes and different polishing agents (Bollen & others, 1997; Roulet & Roulet-Mehrens, 1982). In 1991, Willems and others (1991) reported on surface roughness after toothbrush simulation of 70 composite materials that were available at that time; more than 70% of those materials had a mean roughness after toothbrushing of more than 0.2 µm and 40% of them had a mean roughness of more than 0.64 µm. However, from these 70 composites only seven are still on the market today. Unfortunately, this study did not include an evaluation of roughness before simulated toothbrushing and therefore made it impossible to assess the difference (increase or decrease) in roughness caused by simulated toothbrushing. Little information is available on the influence of the toothbrush and toothpaste on the surface texture of current dental materials, including ceramic materials. Recent studies evaluated the influence of simulated toothbrushing on composite materials after polishing with different polishing systems (Neme & others, 2002) or the effect of toothbrushing on the roughness and wear of seven composites for indirect restorations and one ceramic material (Tanoue, Matsumura & Atsuta, 2000).

This study measured the roughness of current dental materials (composite, amalgam, ceramic) before and after toothbrushing with a toothpaste slurry in a device for simulated toothbrushing and compared the results with those of dental hard tissues.

METHODS AND MATERIALS

Twenty-one dental materials were selected: 16 resin composites, 4 ceramic materials and 1 amalgam (Table 1). Additionally, untreated human enamel and dentin were analyzed. Furthermore, the effect of glazing was investigated in a single ceramic material.

Specimen Preparation

Round specimens with a diameter of 10 mm and a thickness of 6 mm were produced from all dental materials by means of a cylindro-conical metal mold. Eight specimens were produced from each material. The resin composites and amalgam were placed directly into the mold with a plane condenser. The resin composites were light cured for 2x40 seconds with Astralis 10 (Ivoclar Vivadent, Schaan, Liechtenstein) at a light intensity of 1200mW/cm^2 and the composite material was compressed before curing, using a pressing device, and 1.5 bar to reduce voids in the material. The Amalcap Plus amalgam (Ivoclar Vivadent) was prepared in a mixer (Silamat S5, Ivoclar Vivadent) for 10 seconds. For the Empress pressable ceramic (leucite ceramic) and the experimental ceramic (lithium disilicate), wax forms were made with the same metal molds and pressed according to the manufacturer's instructions. The fluorapatite materials d.SIGN and Eris were directly processed in a PMMA mold with a distance holder and fired in a furnace (Progamat P90, Ivoclar Vivadent) at 940°C. The underside of all specimens were acid-etched with hydrofluoric acid (Ceramic Etching Gel, Ivoclar Vivadent) for 60 seconds, rinsed with water, dried and silanized with Monobond-S (Ivoclar Vivadent) for 60 seconds. All specimens were luted to sandblasted SEM holders with the Dual Cement (Ivoclar Vivadent) dual-curing resin composite and polymerized with Astralis 10 for 20 seconds. After dry storage at 37°C for 24 hours, the surface was polished with 1200 grit, 2500 grit and 4000 grit SiC abrasive paper under running water using a polishing device (Phoenix 4000, Wirtz-Buehler, Düsseldorf, Germany); additionally, a non-agglomerating suspension of a 0.05 µm particle size (Masterprep, Buehler, Lake Bluff, IL, USA) was used in conjunction with the 4000 grit abrasive paper (Buehler). With regard to the natural tooth substrates, caries and restoration-free lower incisors were used. The teeth were cleaned for 30 seconds with rotating nylon brushes (KerrHawe, Bioggio, Switzerland) at 10,000 rpm using a slurry of pumice. The labial enamel of the incisors was measured to evaluate the effect of simulated toothbrushing on the enamel, while the root dentin of the second group of incisors was used to assess the impact on dentin.

Simulated Toothbrushing

For simulated toothbrushing, a slurry of toothpaste was prepared by mixing 150 g of Signal Anticaries

toothpaste (Lever, Switzerland) with a relative dentin abrasiveness (RDA) of 75 and 75 ml of distilled water in a magnetic mixer for 10 minutes. The toothpaste slurry was inserted into the reservoir of the device for simulated toothbrushing (Willytec, Gräfelfing, Germany). During simulation, the slurry was constantly replaced in the specimen box by means of a pump. For simulation, plane brush heads with rounded bristles (ECOlogic, Trisa, Triengen, Switzerland) were used with a force of 1.7 N and a total of 36,000 strokes of circular toothbrushing movements, which corresponds to approximately five hours of toothbrushing. After each test series, the brush heads and toothpaste slurry were replaced. Before the roughness was measured, the specimens were cleaned under running water.

Measurement of Roughness

Before and after simulated toothbrushing, the roughness was measured with an optical sensor (FRT MicroProf, Fries Research & Technology GmbH, Bergisch-Gladbach, Germany). With a special optical sensor (CWL), the specimen was illuminated by focused white light. An internal optical device, whose focal length has a strong wavelength dependency, splits the white light into different colors (corresponding to different wavelengths). A miniature spectrometer detects the color of the light reflected by the specimen and determines the vertical position on the specimen surface by means of an internal calibration table. The xy-position is measured with rotary encoders. The device enables measurements with a 10-nm resolution in height and 1-2 µm lateral, with a maximum measuring frequency of 1,000 Hz. The FRT MicroProf automatically scans up to 16 specimens with the help of a software script and automatically measures all typical roughness parameters and stores the results in an Excel table. The 1 mm^2 measurement area was situated at the center of the test specimen. For clarity, only the results of the mean surface roughness Ra are presented.

Statistical Analysis

The SPSS software package (11.01, SPSS Inc, Chicago, IL, USA) was used for the statistical analysis. The results suggested unequal variances between the different materials, which was confirmed by the Levene-Test. An estimation of a suitable data transformation revealed that the logarithmic procedure was the most appropriate transformation method for reducing the heterogeneity of the variances of the materials. All comparative statistical analyses for surface roughness before and after toothbrush simulation have been performed with the lg-transformed data. For the values of the percentage of differences in surface roughness before and after toothbrush simulation, the data were more homogenous and a data transformation was not carried out. To compare the mean roughness before and after simulated toothbrushing, a paired parametric *t*-test for dependent specimens was applied on the lg-transformed data. To differentiate between materials before and after toothbrushing, ANOVA with a post hoc Tukey B test was used. The significance level was set at $p<0.05$. To evaluate the correlation between the mean particle size and roughness after simulated toothbrushing, a regression analysis was performed.

RESULTS

For the sake of comparison with other studies and for better illustration, instead of the lg-transformed data, the mean value and standard deviation of surface roughness was presented in Table 2. However, comparative statistical analyses are based on the logarithmic transformed data.

Table 1: *List of Products Used in this Study, Manufacturer and Batch Number; Composition and Mean Particle Size as Reported by the Manufacturer*

Material	Manufacturer	Batch #	
Amalgam			
Amalcap Plus	Ivoclar Vivadent	E47308	
Ceramic			
d.SIGN	Ivoclar Vivadent	D52027	
Empress	Ivoclar Vivadent	E33919	
Eris	Ivoclar Vivadent	E26908	
Experimental lithium di-silicate ceramic	Ivoclar Vivadent	E79133	
Resin Composite			**MeanParticle Size (µm)**
Adoro	Ivoclar Vivadent	E16057	0.4
Compoglass F	Ivoclar Vivadent	E40043	1.0
Esthet-X Enamel	Dentsply	0008000259	0.9
Four Seasons Enamel	Ivoclar Vivadent	F454485	0.6
Heliomolar HB	Ivoclar Vivadent	D54268	0.1
Heliomolar RO	Ivoclar Vivadent	C39042	0.1
Herculite XRV	KerrHawe	9-1300	0.6
InTen-S	Ivoclar Vivadent	E39025	1.0
Miris Enamel	Coltène	KE347	0.6
Point 4	KerrHawe	208372	0.4
SureFil	Dentsply	981208	1.0
Tetric Ceram	Ivoclar Vivadent	E70886	0.7
Tetric Ceram HB	Ivoclar Vivadent	E43092	0.7
Tetric Flow	Ivoclar Vivadent	E24065	0.7
TPH Spectrum	Dentsply	0206001800	1.4
Z100	3M ESPE	2RX	0.7

Roughness Before Simulated Toothbrushing

Except for the experimental ceramic material, no statistically significant difference in the mean roughness (Ra) was found for the polished materials (Table 2). Enamel, dentin and glazed Empress showed a statistically significant higher mean roughness.

Roughness After Simulated Toothbrushing

Both the dental hard tissues and ceramic materials Empress and d.SIGN exhibited a decrease in mean roughness after simulated toothbrushing; the roughness of enamel and dentin decreased by approximately 40%, Empress by approximately 40-50% and d.SIGN by approximately 18% (Table 2). The difference, however, was only statistically significant for Empress and the hard tissues. The increase in the mean roughness of the other two ceramic materials was only statistically significant for Eris but not for the experimental ceramic. The roughness of the amalgam remained practically unchanged. The surface roughness of most composite specimens doubled or even tripled after simulated toothbrushing. Heliomolar HB, TPH Spectrum, Point 4, Esthet-X and Compoglass F exhibited the lowest mean roughness after simulated toothbrushing, while Herculite XRV, Tetric Ceram, Tetric Flow and Tetric Ceram HB demonstrated the greatest roughness (Figure 1). With regard to the mean percentage of roughness increase, Heliomolar HB and TPH Spectrum showed the lowest increase, Four Seasons, Miris and Herculite XRV the highest. The increase in mean roughness, however, was not statistically significant for Esthet-X and TPH Spectrum.

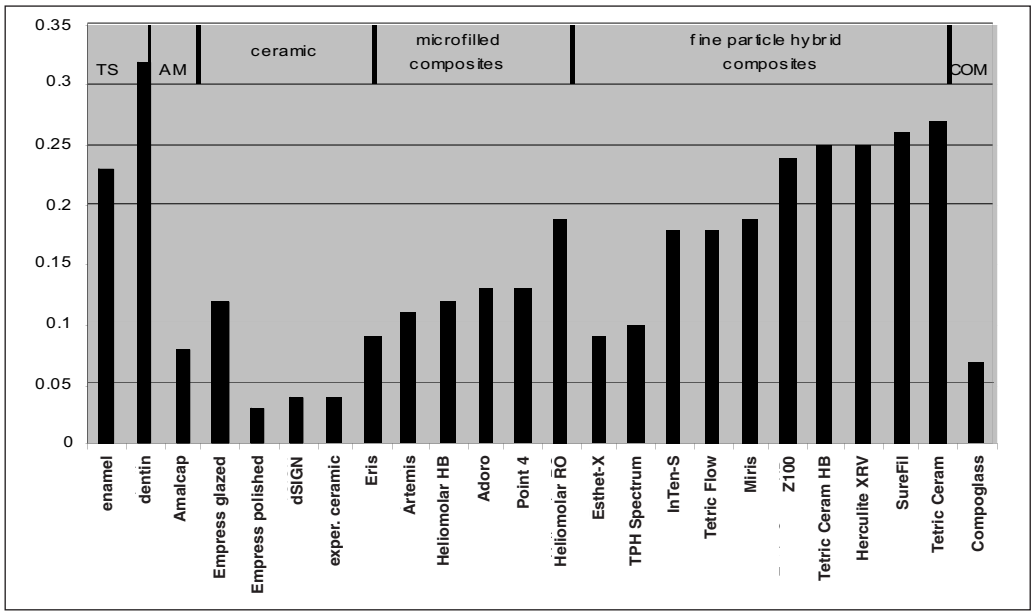

Figure 1: *Mean roughness Ra (µm) after simulated toothbrushing, differentiated according to material classes. Standard deviation and statistics see Table 2.*
TS = tooth substrate, AM = Amalgam, COM = compomer

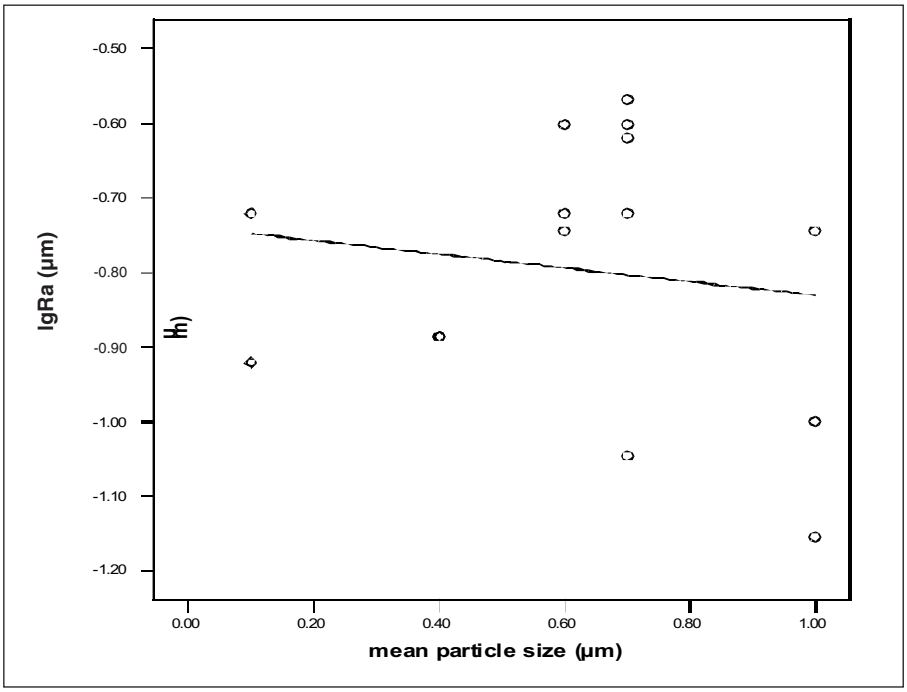

Figure 2: *Scatter diagram of the relationship between mean particle size and mean lg-transformed roughness Ra of 16 resin composites after simulated toothbrushing. Regression curve: y=-0.74-0.092x, R^2=0.02 (p=0.6)*

No correlation was found between the mean particle size and roughness after simulated toothbrushing in a regression analysis including all 16 resin composites (Figure 2). The regression curve indicates that there is no linear relationship between particle size and surface roughness.

DISCUSSION

In the oral cavity, the surfaces of restorative materials are subjected to a variety of factors that may alter the

Table 2: *Mean Roughness (Ra) Before and After Simulated Toothbrushing (STB), Difference in Percentage, Probability of Paired* t*-test Before and After STB*

		Ra (µm) Before STB	**Ra (µm) After STB**	**Difference in % Before and After STB (SD)**	**Probability Paired *t*-test Before and After STB**
Material	N	Mean (SD)	Mean (SD)	Mean (SD)	
Adoro	8	0.06(0.02) ab	0.13(0.02) cdefg	166(117) cdefg	0.002
Amalcap	8	0.09(0.02) b	0.08(0.04) bcd	3(57) abc	0.694
Compoglass F	8	0.05(0.01) ab	0.07(0.00) b	149(25) abcd	0.001
d.SIGN	8	0.06(0.03) ab	0.04(0.01) a	-18(43) ab	0.134
Dentin, human	8	0.50(0.05) d	0.32(0.10) i	-35(25) a	0.029
Empress polished	8	0.05(0.02) ab	0.03(0.01) a	-42(23) a	0.006
Empress glazed	8	0.30(0.21) c	0.12(0.10) bcd	-55(25) a	0.001
Enamel, human	8	0.41(0.05) cd	0.23(0.11) ghi	-45(26) a	0.010
Eris	8	0.05(0.02) ab	0.09(0.03) bcd	106(110) abcdef	0.022
Esthet-X	8	0.06(0.06) ab	0.09(0.05) bc	101(149) abcde	0.194
Experimental ceramic	8	0.03(0.01) a	0.04(0.00) a	26(37) abc	0.120
Four Seasons	8	0.07(0.01) b	0.11(0.03) bcde	72(57) abc	0.004
Heliomolar HB	8	0.09(0.02) b	0.12(0.01) cdef	47(46) abcd	0.008
Heliomolar RO	8	0.11(0.08) b	0.19(0.04) fghi	147(126) bcdef	0.017
Herculite XRV	8	0.06(0.03) ab	0.25(0.02) hi	425(165) g	0.000
InTen-S	8	0.07(0.01) b	0.18(0.07) efgh	150(91) bcde	0.001
Miris	8	0.05(0.03) ab	0.18(0.03) efgh	329(178) fg	0.000
Point 4	8	0.05(0.01) ab	0.13(0.03) defg	149(64) bcdef	0.000
SureFil	8	0.07(0.01) b	0.26(0.07) hi	241(64) ef	0.000
Tetric Ceram	8	0.08(0.01) b	0.27(0.03) hi	245(62) ef	0.000
Tetric Ceram HB	8	0.10(0.04) b	0.24(0.01) hi	205(147) def	0.000
Tetric Flow	8	0.08(0.02) b	0.25(0.10) hi	205(114) def	0.000
TPH Spectrum	8	0.08(0.05) b	0.10(0.04) bcd	71(123) abcd	0.169
Z100	8	0.07(0.01) b	0.19(0.03) fghi	168(74) cdef	0.000

*Statistics ANOVA post hoc Tukey B (*p*<0.05): Same letter means that the material is in the same statistical subgroup.*

The comparative analysis for surface roughness before and after toothbrush simulation as well as the paired t*-test is based on lg-transformed data.*

quality of the surface. Among other factors, oral hygiene procedures play a significant role. While toothbrushing with a fluoridated toothpaste helps decrease the incidence of caries (Marinho & others, 2003), the frequent use of prophylactic home procedures may have side effects, such as roughening of the surface of restorative materials and dental hard tissues, thereby enhancing bacterial growth. As far as direct restorations are concerned, this is especially important for those restorations which cover the labial or buccal aspects of the restored tooth, such as Class IV or Class V fillings, while the proximal parts of Class II fillings are less affected by toothbrushing procedures. The occlusal parts are more influenced by the interaction with the antagonist and food items during chewing than by toothbrushing. In indirect restorations such as crowns and bridges, the whole buccal or oral part is subjected to toothbrush/toothpaste interaction. During toothbrushing, the toothpaste is quickly diluted by saliva; in experiments *in vitro*, this effect is simulated by diluting the toothpaste with distilled water. However, the special properties of saliva, which contains specific proteins and ions that may diminish the roughening effect of the toothbrush, cannot be simulated. Besides toothbrushing, several other interventional procedures, such as the finishing and polishing of dental restorations, the pressing of materials against metal or acrylic matrices and professional tooth cleaning devices, influence the surface roughness, which is dependent on the material and the kind of intervention as a literature review has concluded (Bollen & others, 1997).

In terms of the parameters for simulated toothbrushing, those parameters that have some clinically proven relevance were chosen. For this study, a force of 170 g, 36,000 strokes of circular movements and an optimal polishing procedure was defined. With regard to the force applied by human beings during toothbrushing, a clinical study of 94 subjects revealed a mean toothbrushing force of 330 g, with a range from 140 g to 720 g; plaque removal, however, was not influenced by the toothbrushing force (Van der Weijden & others, 1998). Similar forces were detected in other studies (Volpenhein & others, 1994). The chosen force is therefore at the lower end of the clinically relevant forces. However, it is in line with the technical specification of ISO on wear testing by toothbrushing, which defines a force between 0.5 and 2.5 N (ISO, 1999). Other studies used 350 g and 20,000 strokes (Tanoue & others, 2000), 200 g and 60,000 strokes (Neme & others, 2002), 500 g and 20,000 strokes (Cho, Yi & Heo, 2002) or 200 g and 30,000 cycles (dos Santos & others, 2003). A comprehensive study evaluating 70 composites applied a force of 150 g, and the test ran for three hours without counting the number of strokes (Willems & others, 1991). It may be assumed that higher forces result in a higher absolute roughness. There is no evidence that the ranking of materials is affected by the application of higher forces during simulated toothbrushing. In this study, applying 36,000 strokes took about five hours, which may correspond with the amount of toothbrushing carried out over three years, if it is assumed that a pair of premolars or molars is brushed twice a day for 10 seconds on average. Videotaped recordings of 31 patients and their toothbrushing habits revealed a mean stroke length of 1 cm per stroke, a brushing rate of 15 cm/second for circular toothbrushing (Volpenhein & others, 1994). Furthermore, the patients spent 32 seconds on average toothbrushing posterior segments, which would correspond to about eight seconds for each posterior segment.

The rationale for using 4000 grit was to produce an optimally polished surface that had a comparable initial roughness for the materials to better assess the material's intrinsic roughness. This is in contrast to the technical specification on wear by toothbrushing, which recommends polishing with 1000 grit silicon carbide paper (ISO, 1999). Studies that systematically analyzed the influence of varying initial roughness values, forces and numbers of brushing cycles on the roughness of the tested materials were not found. The relative dentin abrasiveness (RDA) of the toothpaste is another variable that influences the surface roughness of the materials. An *in vitro* study showed that the higher the RDA of the toothpaste, the higher both the surface roughness and wear of the dental materials (McCabe & others, 2002); this was true for resin composites (Z250, Filtek A110), a resin modified glass ionomer cement or compomer (Dyract AP), a glass ionomer cement (Ketac Fil) and enamel; Dyract AP showed the highest wear and roughness. In this study, the Compoglass F compomer exhibited a roughness that was comparable to the microfilled composites after simulated toothbrushing.

The standard deviations of the mean roughness varied widely between materials, and the coefficient of variation (CV) was between 10% (experimental ceramic and Compoglass F) and 77% for glazed ceramic after toothbrushing. The logarithmic transformation of the raw data reduced the variability, for example, for glazed ceramic from 77% to 28%. The glazing step is done manually, which eventually results in a non-standardizable surface texture. The higher variation of the roughness values of dentin (CV=31 %, lg-transformed 30%) and enamel (CV=48%, lg-transformed 32%) may be explained by the impossibility to standardize biological substrates. There was a tendency for materials with higher surface roughness to have a lower coefficient of variation than materials with a lower surface roughness, which is in line with other studies (Willems & others, 1991). The difference in scattering of the results may be explained by 1) the random distribution of roughness lines caused by the toothbrush, 2) the

restricted measuring area of 1 mm^2 in the center of the specimen and 3) the possibility of voids in the material that influence the measurement of surface roughness.

With regard to the performance of the different materials, it can be concluded that the hybrid composites showed the highest increase in mean roughness of all composites, while the microfilled composites and the Compoglass F compomer demonstrated the lowest increase (Figure 1). For Amalcap amalgam, Esthet-X and TPH Spectrum resin composites, d.SIGN and the experimental ceramic, no statistically significant difference in roughness before or after simulated toothbrushing was found. The other ceramic materials, as well as the dentin and enamel specimens, showed a statistically significant decrease in mean roughness after simulated toothbrushing. Glazed Empress demonstrated a statistically significant higher initial roughness than polished Empress, which is in line with other studies (Bollen & others, 1997). In terms of the resin composites, similar results were found in other studies, although the absolute values cannot directly be compared (Cho & others, 2002; dos Santos & others, 2003; Kanter, Koski & Martin, 1982; Tanoue & others, 2000; Willems & others, 1991). The differences in the results of other studies may be explained by different toothpaste slurries, forces of the toothbrush, number of toothbrushing cycles, hardness and stiffness of toothbrush bristles, differences in preparation of the specimens and quantification of the mean roughness. When comparing the results of this study on current composites with those of a study by Willems and others that used similar toothbrushing parameters on composites that were available 10-15 years ago, one may conclude that the intrinsic roughness of current composites is much lower than the older ones (Willems & others, 1991). While about 70% of the older composites had a mean surface roughness of more than 0.2 µm, this was true for only about 20% of the composites evaluated in this study.

In composite materials, the gradual removal of filler particles leads to an increase in material roughness. The effect is less pronounced in microfilled resins than in hybrid resins. However, the mean particle size alone is obviously not a reliable predictor for the performance of resin composites if these materials are subjected to a device for simulated toothbrushing, as has been shown in this study and which is in line with a study comparing the mean surface roughness of 69 composites and the measured mean particle size (Willems & others, 1992). In this study, those composites with a particle size greater than 1 µm produced lower roughness values than those with a particle size between 0.6 µm and 1 µm. In most composites, distribution of the filler particles does not correspond to a normal distribution but rather to a skewed one. Therefore, the mean particle size does not give valid information about the amount of small, medium and large particles. This, however, may affect the resin's resistance to wear-induced attrition and toothbrushing, as do the shape of the filler particles and the distance between them, the composition of the resin matrix, the chemical link between inorganic fillers and the resin matrix and the conversion rate after polymerization (Roulet, 1987).

What is the clinical significance of the surface roughness of restorative materials? The answer has to be split into two parts: first, the influence on the aesthetic appearance of the restoration (discoloration and wear), which can be recognized by both the dentist and patient, has to be investigated and, second, the biological consequences regarding periodontal health, especially the occurrence of gingivitis, and the development of secondary caries have to be examined.

Various factors, including toothbrushing, contribute to the disintegration of dental materials. Prospective clinical trials which compare ceramic with composite inlays and ceramic veneers with composite veneers confirm that the aesthetic appearance of ceramic is significantly more durable than composite (Hickel & Manhart, 2001; Krämer, Lohbauer & Frankenberger, 2000; Meijering & others, 1998; Peumans & others, 2000). The surface of a composite disintegrates over time, which leads to roughness, pitting, staining and increased wear. A comparative study of ceramic and composite veneers revealed higher patient satisfaction after two years of service in terms of the aesthetic appearance of ceramic veneers compared to composite veneers (Meijering & others, 1997). Aesthetic appearance can be improved by repolishing the surface. However, a systematic evaluation of such maintenance procedures has not yet been performed in relation to the type of composite, duration, frequency and patient and dentist satisfaction. Such procedures involve additional chairtime and lead to a loss of material. On the basis of their experience, clinicians assume that microfilled composites demonstrate better polishability, stability of surface texture and gloss than hybrid composites. However, no randomized controlled clinical trials are available that objectively appraise the aesthetic appearance of different composites over time; most clinical trials focus on the overall survival of the restorations, which is more limited by fractures and marginal disintegration than by aesthetics. On the other hand, a retrospective trial which evaluated the reasons for replacement of the composite fillings revealed unaesthetic appearance to be the second most prevalent reason for the replacement of fillings, after secondary caries (Wilson, Burke & Mjör, 1997). However, not only surface texture but also the shape and shade strongly contribute to impaired aesthetics. Another approach to maintaining surface texture is the application of a monomer containing sealant that penetrates into surface irregularities. An *in vitro* investigation showed a

lower increase in the roughness of sealed surfaces than in unsealed surfaces in some composites after simulated toothbrushing (dos Santos & others, 2003). Again, there is no evidence from clinical trials that this procedure has sustainable clinical relevance.

With regard to the attrition of composite materials, tests *in vitro* with chewing simulators suggest an increased attrition rate of materials with larger particles compared to those with small particles (Heintze, Zappini & Rousson, 2005). Hybrid composites and macro composites tend to get rough during attrition wear simulation, resulting in increased friction between the stylus and the material, thus accelerating the wear of the material and the antagonist. Therefore, materials with high intrinsic roughness should exhibit more attrition wear *in vivo*. Roughness evaluations of the occlusal contacts of 11 molar replicas taken from seven subjects of unreported age and selection criteria concluded that the mean surface roughness between enamel-enamel contact areas to be 0.64 µm (Willems & others, 1991). This value was regarded as a "biological" intrinsic roughness of dental enamel with which restorative materials should be compared. The authors tested 70 composites that were available around 1990 with a toothbrush simulation device and grouped them according to their mean roughness. In this study, however, the correlation between the intrinsic value of composites and the wear *in vivo* has not been proven to date. Intrinsic roughness is only one material variable among many others that may influence wear *in vivo*.

With regard to the impact of surface roughness on gingival health, clinical studies have shown that rough surfaces increase plaque formation and reduce the cleaning efficiency of oral hygiene procedures (van Dijken, Sjöström & Wing, 1987). A clinical study on titanium implant abutments revealed that the critical threshold value for bacterial retention is a mean roughness of 0.2 µm (Quirynen & others, 1996); surface free energy apparently plays a secondary role for bacterial colonization (Quirynen & Bollen, 1995). However, whether or not this holds true for other materials or dental hard tissues has not been systematically investigated in clinical trials. The dentin and enamel specimens showed a mean roughness well above the critical threshold before toothbrushing, which decreased substantially after toothbrushing but was still above this value. It seems questionable whether the proposed threshold value is valid for all materials and organic substrates in the mouth; furthermore, patient-related factors may interact with bacterial colonization. In terms of dental materials, a prospective trial on Class V fillings with resin-modified glass ionomer cements over two years revealed periods of decrease and increase of surface roughness which were material-dependent but which levelled out at the two-year recall (Sidhu, Sherriff & Watson, 1997). The mean surface roughness was measured on replicas and was calculated to be between 9 and 20 µm, which seem to be questionable values, since studies *in vitro* showed that this type of material exhibits a mean roughness between 0.3 and 1.5 µm (Bollen & others, 1997). In this study, Herculite XRV, SureFil and the materials of the Tetric family showed a mean roughness above 0.2 µm. This, however, does not mean that teeth restored with these materials are more prone to secondary caries or gingivitis. There is no evidence from clinical studies that restorations with these materials are more closely linked to gingival inflammation. With regard to longevity, Tetric Ceram and Herculite XRV produced similar results compared to other materials (Hickel & Manhart, 2001). However, these two materials and SureFil showed an increase of nearly 0.2 µm after toothbrushing, which can be detected by the majority of patients with the tip of their tongue, as has been recently shown in a clinical trial with different roughened surfaces (Jones & others, 2004). Again, clinical trials did not report an increased discomfort in patients who have received restorations with these materials; obviously, there are effects in the oral cavity that counteract the roughening caused by toothbrushing and other procedures.

In a clinical trial that involved a split-mouth design, 17 volunteers treated with subgingivally placed Class II restorations with a resin composite, a resin modified glass ionomer cement and a polyacid-modified resin material, refrained from oral hygiene for seven days (van Dijken & Sjöström, 1998). In the subsequent examination, no statistically significant difference between the materials was found with regard to gingival crevicular fluid and gingival sulcus bleeding, although SEM replicas revealed a significant difference in roughness between the materials. However, unrestored control sites showed statistically significant lower crevicular fluid. An important confounder, which has to be taken into account, is the presence of proximal overhangs that may be found in at least 25% of restored tooth surfaces and 33% of adult patients (Brunsvold & Lane, 1990). Overhangs are related to increased bone loss, attachment loss and gingival inflammation compared to restored teeth without excess (Lang, Kiel & Anderhalden, 1983). Furthermore, subgingivally placed restorations are more likely to have gingival inflammation and attachment loss than supragingivally-placed restorations (Schätzle & others, 2001). Another confounder is the influence of possible toxic products of resin composites on soft tissue; this topic, however, is not well studied (Nadarajah, Neiders & Cohen, 1997). With regard to the occurrence of secondary caries, clinical studies showed a higher amount of caries-inducing bacteria, such as *mutans streptococci* on resin composites, compared to unrestored enamel and amalgam (Heintze & Twetman, 2002). Randomized controlled

clinical trials revealed a higher longevity of amalgam fillings compared to resin composites (Chadwick, Dummer & Dunstan, 1999). There is, however, no conclusive evidence that the difference is due to the occurrence of secondary caries. One of the great shortcomings of clinical trials that evaluate restorative procedures is the lack of evidence-based international evaluation criteria. Probably, the problem of secondary caries is not truly assessed. In clinically visible areas, marginal discoloration is often related to secondary caries. Marginal discoloration, however, is no predictor of secondary caries, neither for its development, nor for its presence (Mjör & Toffenetti, 2000). Thus, secondary caries is overestimated in clinically visible areas, whereas in cervical gingival areas of Class II fillings, that is, an area that is difficult to access for clinical inspection, secondary caries is often not detected, leading to an underestimation. It has been shown that secondary caries most often starts from that site (Mjör, 1998). The proportion of over- and underestimation, however, is not clear.

In addition to biological consequences, the current evidence of the clinical significance of surface roughness of dental materials is insufficient. Resin composites are more prone to disintegration with subsequent roughness and discoloration than metal alloys and ceramic materials. Furthermore, the disintegrated particles are swallowed by the patient, which may have systemic medical implications in the long run.

CONCLUSIONS

Simulated toothbrushing with a slurry of toothpaste and distilled water roughens the optimally polished surface of most resin composites but not amalgam, ceramic materials and dental hard tissues. Hybrid composites demonstrated a rougher surface than microfilled composites or compomer. The variables that have an influence on the surface of dental materials during simulated toothbrushing (number of cycles, force, specimen preparation, slurry) should be systematically investigated to better weigh the influencing factors of simulated toothbrushing on the surface of dental materials. Furthermore, clinical research is needed to evaluate the clinical significance of the *in vitro* findings. As durable smooth surfaces contribute to both a favorable aesthetic appearance of dental restorations and prevention of secondary caries, periodontal disease and wear, dental manufacturers should strive to develop restorative materials that are not significantly altered by normal oral hygiene measures.

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Evaluation of the Adhesion of Fiber Posts to Intraradicular Dentin

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Clinical Relevance

When luting fiber posts to intraradicular dentin, a total-etch resin cement shows greater bonding potential than a self-etch and a self-adhesive resin cement.

SUMMARY

The interfacial strength and ultrastructure of a total-etch, self-etch and self-adhesive resin cement used to lute endodontic glass fiber posts (FRC Postec, Ivoclar-Vivadent) was assessed with the "thin-slice" push-out test and transmission electron microscopy (TEM). The tested adhesive cements were Variolink II (Ivoclar-Vivadent), Panavia 21 (Kuraray Co) and RelyX Unicem (3M ESPE). In each group, seven posted roots were used for push-out tests and two were processed for TEM observations. The interfacial strength achieved by Variolink II (10.18±2.89 MPa) was significantly higher than Panavia (5.04±2.81 MPa) and RelyX Unicem (5.01±2.63 MPa), which were comparable to each other. TEM micrographs of the interface between Variolink II and intraradicular dentin revealed that the smear layer was totally removed and an 8-10 micron thick hybrid layer was formed. In the other group specimens, the smear layer was not completely dissolved and smear plugs were retained. Gaps were present between the hybridized complex and the adhesive layer in the Panavia 21 specimens and between the smear layer and underlying root dentin in the RelyX Unicem specimens. Interfacial strengths and microscopic findings were in agreement and indicated that the bonding potential of the total-etch resin cement was greater. The acidic-resin monomers responsible for substrate conditioning in Panavia 21 and RelyX Unicem appeared unable to effectively remove the thick smear layer created on root dentin during post space preparation.

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INTRODUCTION

Fiber posts bonded to root canal dentin via resin cements are now routinely employed for the restoration of endodontically treated teeth. The similarity in elastic moduli of the fiber post, resin cement, core material and dentin (Boschian, Pest & others, 2002) was perceived to be advantageous in improving the performance of these restorations, as compared with cast metal post and core restorations (Asmussen, Peutzfeldt & Heitmann, 1999; Albuquerque Rde & others, 2003). Although the occurrence of root fractures, the most frequent cause of failure with metallic posts is rare with the use of fiber posts (Akkayan & Gulmez, 2002); recent clinical trials indicate that fiber post restorations may fail via dislodging of the bonded posts (Dallari & Rovatti, 1996; Fredriksson & others, 1998; Ferrari, Vichi & García-Godoy, 2000b; Monticelli & others, 2003). Previous bond strength and morphologic studies have shown that bonding to root canals may be influenced by endodontic procedures prior to post cementation (Nikaido & others, 1999; Morris & others, 2001; Ngoh & others, 2001; Erdemir & others, 2004), the variability of intraradicular dentin (Ferrari & others, 2000; Mannocci & others, 2001; Serafino & others, 2004), the compatibility of resin cements with dentin adhesives (Dong & others, 2003; Pfeifer, Shih & Braga, 2003; Carvalho & others, 2004) and the cement film thickness (Alster & others, 1997; Hagge, Wong & Lindemuth, 2002; De Jager, Pallav & Feilzer, 2004).

The coupling of resin-based cements traditionally requires the adjunctive use of dentin adhesives that are either total-etch or self-etch in nature (Mak & others, 2002). Total-etch resin cements utilize phosphoric acid-etching that completely dissolves the smear layer and creates a zone of partially demineralized dentin. Upon rinsing the acid conditioners, adhesive primers and resins are applied to the demineralized dentin to achieve micromechanical bonding. Conversely, self-etch resin cements utilize adhesives containing increased concentrations of acidic resin monomers to simultaneously demineralize and infiltrate the smear layer-covered dentin. A further reduction in working steps has been accomplished with the recent introduction of a self-adhesive resin cement (RelyX Unicem, 3M ESPE, St Paul, MN, USA) that does not require any pre-treatment of tooth substrates. Apart from the marketing data supplied by the manufacturer, little information is available on the efficacy of the new self-adhesive resin cement for the luting of fiber posts to intraradicular dentin.

Bonding to intraradicular dentin presents challenges to clinicians due to the radically different bonding conditions that are present (Ferrari & others, 2000). For example, the highly unfavorable cavity configuration factors within the dowel spaces warrant the use of self-cured, slow-setting resin cements for more effective dissipation of polymerization shrinkage stresses (Bouillaguet & others, 2003). Although microtensile bond strength studies are available on the use of resin cements in root canals, the dowel spaces in these studies were only filled with resin cements, (Ngoh & others, 2001; Foxton & others, 2003), were ground flat in order to gain unrestricted access to canal walls during the bonding procedure (Gaston & others, 2001; Sahafi & others, 2003) or were bonded with custom-made posts fabricated out of pre-polymerized hybrid resin composites (Bouillaguet & others, 2003). While these studies provide significant contributions to the adhesive mechanisms of different types of resin cements to root dentin, they are more akin to simplified modeling approaches and do not reflect the dislocation resistance of bonded posts in intact dowel spaces that involve multiple interfaces. Reliability of the microtensile test in assessing the bonding of fiber posts to intact dowel spaces was challenged in a recent study (Goracci & others, 2004), with frequent observation of premature bond failures when specimens were prepared for bond testing using either the trimming or non-trimming technique. The propensity of these premature bond failures was thought to be caused by pre-existing interfacial gaps and/or the superimposition of in-service stresses generated during specimen preparation for microtensile bond testing, upon the potentially destructive macroscopic (Type I) residual stress fields (Withers & Bhadeshia, 2001) present in the bonded canals.

Similar to the retention of implant attachments in bone (Berzins & Summer, 1999), the resistance to dislocation of fiber posts bonded to intact root canals with resin- or glass-ionomer-based cements may be considered a net sum of micromechanical interlocking, chemical bonding and sliding friction (Dhert & Jansen, 1999). For this reason, pull-out and push-out test results have been more successfully employed as indicators of the interfacial strengths of bonded fiber posts in root canals (Boschian Pest & others, 2002; Kurtz & others, 2003; Prisco & others, 2003). Similar to the microtensile bond test, an additional advantage to using the "thin slice" push-out test (Kallas & others, 1992; Chandra & Ananth, 1995) is that multiple specimens may be retrieved from a bonded root canal.

This study utilized a "thin-slice" push-out test and transmission electron microscopy (TEM) to examine the interfacial strength and ultrastructure of a total-etch, self-etch and self-adhesive resin cement that were employed for the bonding of glass fiber posts to intact root canals. The null hypothesis tested was that there was no difference in the efficacy of the three resin

cements for bonding of glass fiber posts to intraradicular dentin.

METHODS AND MATERIALS

Twenty-seven single-rooted teeth extracted for periodontal reasons were used in the study. The crown portion of each tooth was removed with a water-cooled diamond saw (Isomet, Buehler, Lake Bluff, IL, USA), and the root was endodontically instrumented at a working length of 1 mm from the apex using a #35 master apical file. A step-back preparation technique was followed using stainless-steel K-files (Union Broach, New York, NY, USA), Gates-Glidden drills #2 to #4 (Union Broach) and 2.5% sodium hypochlorite irrigation. For canal obturation, thermoplasticized, injectable guttapercha (Obtura, Texceed Corp, Costa Mesa, CA, USA) and a resin sealer (AH-26, DeTrey, Zurich, Switzerland) were employed. In each root-treated tooth, a 9-mm deep dowel space was prepared with low-speed drills provided by the post manufacturer, and a 1.3-mm diameter translucent glass fiber post (FRC Postec, Ivoclar-Vivadent, Schaan, Liechtenstein) was tried-in and cut to the adequate length. After sectioning to the appropriate length, the glass fiber post was cleaned in ethanol, silanized with Monobond-S (Ivoclar-Vivadent) and left to air-dry before coating with adhesive or resin cement.

The teeth were randomly divided into three groups of nine specimens each. For each group, a different resin-based luting agent was utilized for fiber post cementation. The tested materials were Excite DSC/Variolink II (Ivoclar-Vivadent, Schaan, Liechtestein, Group I), ED Primer/Panavia 21 (Kuraray Medical Inc, Tokyo, Japan, Group II) and RelyX Unicem (Group III). Variolink II is a dual-cured cement that requires phosphoric acid for substrate conditioning and the application of self-activated Excite DSC as a coupling dentin adhesive. Panavia 21 is an auto-cured resin cement that is used in combination with the proprietary one-step self-etching primer (ED primer). RelyX Unicem is a dual-cured cement that the manufacturer claims to be self-adhesive in nature and does not require pretreatment of the tooth substrates. The components of the three resin cements are shown in Table 1. The materials were handled according to the manufacturers' instructions (Table 2).

After storage in water for 24 hours at room temperature, seven roots from each group were randomly selected for evaluation of push-out strength, and the remaining two roots were used for TEM examination.

Push-out Strength Evaluation

The portion of each root that contained the bonded fiber post was sectioned into five to six 1-mm thick serial slices with the Isomet saw under water cooling (Figure 1). Seven bonded roots were used for each group, resulting in 36 to 37 slices per group for push-out strength evaluation.

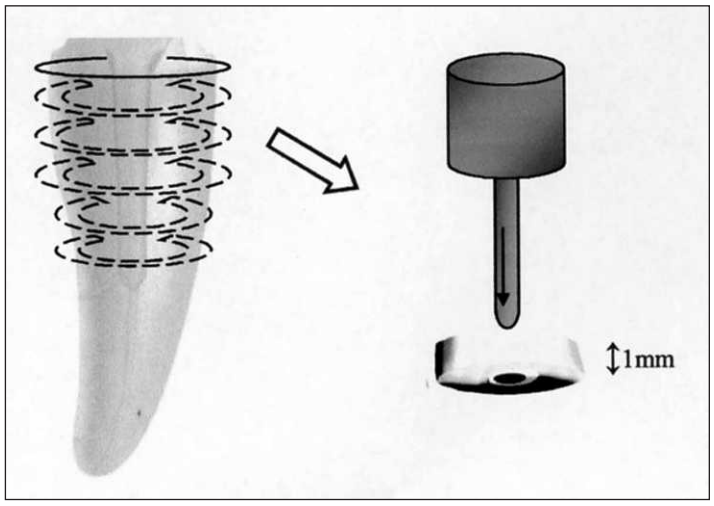

Figure 1. *A schematic representation of the preparation of thin root slices containing bonded fiber post and the setup for the push-out test.*

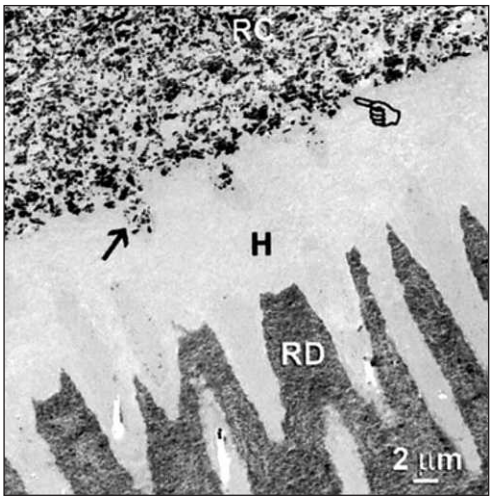

Figure 2. *An unstained, undemineralized TEM micrograph of the interface between root dentin and Excite DSC/Variolink II (bar = 2 µm). A completely demineralized, 6-8 µm-thick hybrid layer (H) was created after the root dentin (RD) was etched with phosphoric acid. The dentinal tubular orifices are visible on the surface of the acid-etched dentin (arrow). A thin layer of unfilled adhesive could be recognized (pointer) and some of the resin cement (RC) was found within some of the dentinal tubules (arrow).*

The thickness of each slice was individually measured by means of a digital caliper, then firmly fixed with cyanoacrylate glue to a loading fixture. A compressive load was applied on the apical aspect of the slice via a universal testing machine (Controls SPA, Milano, Italy) equipped with a 1-mm diameter cylindrical plunger (Figure 1). The plunger was positioned so that it only contacted the bonded post on loading,

Table 1: *Components of the Resin Cements Tested in This Study*

Group I	Group II	Group III	
Excite DSC	**ED Primer**	**RelyX Unicem**	
HEMA, TEGDMA, phosphoric acid acrylate, silicon dioxide, initiators, stabilizers, alcohol	HEMA, MDP, 5-NMSA, sodium benzene sulfinate N, N-diethanol p-toluidine, water	*Powder*	*Liquid*
		Glass fillers, silica, calcium hydroxide, self-cure initiators, pigments	Methacrylated phosphoric esters, dimethacrylates, acetate, stabilizers, self-cure initiators, light-cure initiators
VariolinkII	**Panavia 21**		
Dimethacrylates, silicon dioxide, self-cure initiators, light-cure initiators	Glass and silica powder, sodium fluoride, bis-phenol A polyethoxy dimethacylate, 10 MDP, hydrophilic and hydrophobic dimethacylates, self-cure initiators		

Table 2: *Manufacturers' Instructions for the Handling of Resin Cements*

Excite DSC/Variolink II
-Etch with 37% phosphoric acid gel for 15 seconds, rinse with water from an endodontic syringe, remove excess water with paper points.
-Apply Excite DSC in four to five layers by means of the self-activating microbrush. Remove the excess adhesive with paper points.
-Mix base and catalyst of Variolink II, carry the cement into the root canal with a lentulo drill.
-Insert the post and light-cure the cement for 20 seconds through the post (Optilux 501 Kerr/Demetron, Orange, CA, USA, 750 mW/cm^2).
ED Primer/Panavia 21
-Mix equal amounts of ED Primer liquids A and B. Apply the mix inside the canal with a brush and leave it undisturbed for 60 seconds. Remove excess adhesive with paper points, dry with a gentle air flow.
-Mix equal amounts of base and catalyst for 20 seconds, apply the cement onto the post with a brush.
-Insert the post and let the cement cure without any interference.
RelyX Unicem
-Dry the canal with paper points and a gentle blow of air.
-Mix powder and liquid by triturating the activated capsule.
-Apply the cement onto the post surface.
-Insert the post and let the cement cure initially without any interference, followed by light curing for 20 seconds through the post.

introducing shear stresses along the bonded interfaces. The loading force was exerted in an apical-coronal direction, in order to move the post towards the larger part of the root slice. Loading was performed at a speed of 0.5-mm/minute until failure, as manifested by extrusion of the post segment from the root slice. This was further confirmed by the appearance of a sharp drop along the load/time curve recorded by the testing machine.

The interfacial strength (MPa) was computed by dividing the load at debonding by the area (A) of the bonded interface. The latter was calculated through the formula $A=2\pi rh$ where r represents the post radius and h the thickness of the slice in mm.

Statistical Analysis

The interfacial strength data were first verified by the Kolmogorov-Smirnof test for their normal distribution, then, using regression analysis to ensure that the root of origin was not a significant factor for differences in strength measurements. A one-way analysis of variance was subsequently performed to assess the significance of the differences in interfacial strength among three resin cements, followed by the Tukey test for post-hoc comparisons. In all the analyses, the level of significance was set at the 95% probability level.

TEM Examination

A root slice was retrieved from the center portion of the bonded fiber post of each of the two remaining teeth in each experimental group. The root slices were fixed initially in Karnovsky's fixative, post-fixed with osmium tetroxide, dehydrated in an ascending ethanol series and processed for epoxy resin embedding according to the protocol reported by Tay, Moulding and Pashley (1999) for undemineralized TEM specimen preparation. After complete polymerization of the laboratory epoxy resin (TAAB 812, TAAB Laboratories, Aldermaston, UK) at 60°C for 48 hours, the bulk of the fiber post was carefully removed from the resin blocks with a 30-flute tungsten carbide dental bur under an endodontic microscope (OPMI pico, Carl Zeiss, Oberkochen, Germany). Blocks 2x2 mm containing resin cement-root dentin interfaces were sectioned from the root slice and further embedded in epoxy resin for ultra-microtomy. Undemineralized sections 90-120-nm thick were prepared and collected on carbon- and formvar-coated single slot copper grids (Electron Microscopy

Table 3: *"Thin Slice" Push-out Test Results*

Resin Cement Systems	Number of Specimens Tested	Push-out Strength (MPa)*
Excite DSC/Variolink II	37	10.18 ± 2.89[A]
ED Primer/Panavia 21	36	5.04 ± 2.81[B]
RelyX Unicem	37	5.01 ± 2.63[B]

**Values are means ± standard deviations. Groups with the same upper case superscripts are not significantly different (p>0.05).*

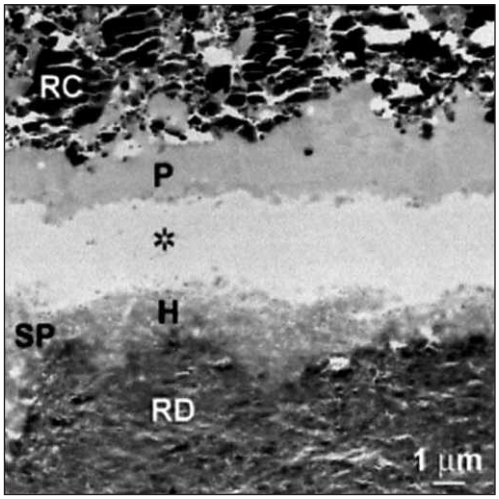

Figure 3. *A representative TEM micrograph of the interface between the root dentin and ED Primer/ Panavia 21. A 1 µm-thickness of partially demineralized hybridized dentin (H) was present, together with some hybridized smear layer remnants. This hybridized complex was separated from the unfilled primer (P) and resin cement (RC), and the space (asterisk) was infiltrated with laboratory epoxy resin. Smear plugs (SP) were present in the dentinal tubules. RD: root dentin.*

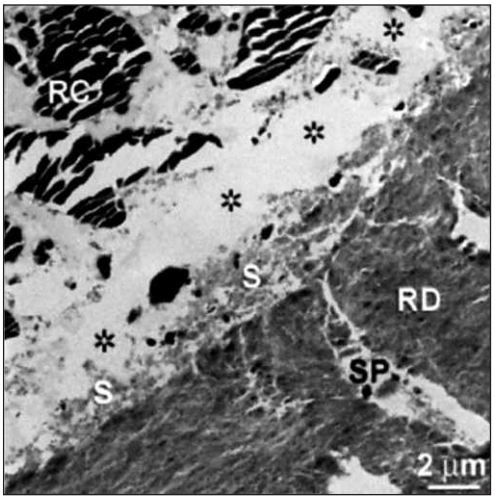

Figure 4. *A representative TEM micrograph of the interface between the root dentin and RelyX Unicem. A 3-4 µm-thick, loosely organized, highly mineralized smear layer (S) was present that was partially separated from the underlying unetched, intact root dentin (RD). The self-adhesive resin cement (RC) was predominantly attached to the smear layer and only partially delaminated from the latter. The weak link in this resin cement was more likely to be caused by its inability to etch through a clinically relevant thick smear layer. Smear plugs (SP) were also evident within the dentinal tubules. Spaces between the root dentin, smear layer and resin cement (asterisks) were infiltrated by the laboratory embedding epoxy resin.*

Sciences, Fort Washington, PA, USA) and examined without further staining, using a TEM (Philips EM208S, Eindhoven, The Netherlands) operated at 80 kV.

RESULTS

Push-out Strengths

The results of the push-out strength measurements are represented in Table 3. The interfacial strengths to root dentin achieved by Excite DSC/Variolink II (Group I) were significantly higher ($p<0.05$) than those obtained for ED Primer/Panavia 21 (Group II) and RelyX Unicem (Group III). There was no difference between the interfacial strengths in Groups II and III ($p>0.05$).

TEM Observations

The resin-dentin interface in the Excite DSC/Variolink II specimens revealed complete dissolution of the smear layer and formation of an 8-10 µm-thick hybrid layer in which the collagen matrix was completely demineralized by the phosphoric acid etchant (Figure 2). There was no separation of the hybridized dentin from the resin cement.

When ED Primer/ Panavia 21 was used for fiber post luting, the root dentin smear layer was almost completely dissolved and smear plugs were retained within the tubular orifices. A 1-1.5 µm thick hybridized complex could be seen, consisting of the some smear layer remnants and an underlying partially demineralized dentin matrix (Figure 3). Gaps existed between the unfilled primer layer and the surface of the hybridized dentin.

Conversely, RelyX Unicem did not dissolve or etch through the 3-4 µm-thick smear layer created in the intraradicular dentin (Figure 4). The smear layer remained heavily mineralized and no hybrid layer was seen in the intact root dentin. Although the self-adhesive cement appeared to adhere to the smear layer, separation occurred between the smear layer and the underlying root dentin.

DISCUSSION

In light of the push-out test results, the null hypothesis, that there is no difference in the efficacy of the three resin cements for bonding of glass fiber posts to intraradicular dentin, has to be rejected. Indeed, the system Excite DSC/Variolink II demonstrated a significantly greater bonding potential than the other two materials (Table 3). As the three resin cement systems contain proprietary ternary catalytic systems, making them optimally compatible with acidic resin monomers (Suh & others, 2003), the differences observed cannot be attributed to the incompatibility between resin cements and dentin adhesives.

On the contrary, the TEM findings of this study suggest that the differences among the adhesive systems may be partially attributed to the ability of the dentin adhesives or self-adhesive resin cements to etch

through clinically relevant thick smear layers (Ogata & others, 2002). Most of the *in vitro* studies on mild self-etching adhesive products performed by manufacturers are conducted by polishing dentin with 600-grit silicon carbide papers that produced thin smear layers less than 1 μm thick (Tay & others, 2000; Chan & others, 2003). It has been shown that self-etch adhesives of different aggressiveness respond variably to smear layers created by different bur types (Ogata & others, 2001). In this study, the intraradicular dentin was prepared using slow speed stainless steel drills, creating dentin smear layers that are 3-4 μm thick (Figure 4). The use of phosphoric acid etching completely dissolved such a thick smear layer (Figure 2), while the use of the milder self-etching ED primer partially dissolved the smear layer (Figure 3). Nevertheless, in both resin cement systems, the complementary adhesives were able to etch into the underlying dentin to create micromechanical retention with the intact bonding substrates.With regard to RelyX Unicem, although this initially anhydrous resin cement system may bond to the smear layer via the mechanisms of water generation and subsequent water recycling proposed by the manufacturer, the weak link in this system lies in its lack of genuine hybridization of the intact bonding substrates. This is clearly illustrated by the failure between the smear layer and the unetched intraradicular dentin in Figure 4.

Although ED primer was able to etch through the smear layer and create a thin zone of partially demineralized, hybridized dentin, this self-etching primer is a one-step self-etch adhesive that is designed exclusively for resin cement. Like all one-step self-etch adhesives currently available in the market that behave as permeable membranes after polymerization (Tay & others, 2002; Chersoni & others, 2004), crown dentin treated with the proprietary ED primer was also highly permeable to water movement (Mak & others, 2002; Carvalho & others, 2004). Using silver nitrate as a tracer, mushroom-shaped water blisters have been previously observed between the crown dentin and ED primer (Carvalho & others, 2004). These water blisters may act as stress raisers that result in premature delamination of the primer layer from the hybridized dentin complex. As vital teeth and endodontically treated teeth do not differ significantly in their moisture content (Papa, Cain & Messer, 1994), the effect of adhesive permeability is also applicable to bonding within root canals. Although positive pulpal pressure is absent in endodontically-treated teeth, an increase in radicular permeability may follow reduction in root dentin thickness and the removal of sealers that penetrated the dentinal tubules during the preparation of post-spaces for cementation of endodontic posts (Fogel, Marshall & Pashley, 1988; Guignes, Faure & Maurette, 1996). By taking impressions of the intraradicular dentin from dowel spaces that were bonded with simplified dentin adhesives in human patients, water droplets were detected along the surface of the polymerized adhesives (Chersoni & others, unpublished results). Such a scenario may be responsible for the weak push-out strengths recorded for this simplified, self-etch resin cement system.

The "thin slice" push-out test, adapted from its widespread use for the testing of ceramic matrix composites (CMCs), metal matrix composites (MMCs) and intermetallic matrix composites (IMCs), is emerging as a practical tool for evaluating the interfacial shear behavior of the attachment of fiber posts to intact root canals. The latest studies in this field have highlighted the important contribution of sliding friction to the interfacial strength in composite materials (Lin, Geubelle & Sottos, 2001; Chandra & Ghonem, 2001; Chai & May, 2001; Kalton & others, 1998). By plotting the load/displacement curves of reinforcing fibers slowly pushed-out or pulled-out of the embedding matrix, it has been shown that friction between the newly debonded interfaces plays a major role in delaying the final failure of the specimen, thus significantly increasing the load carrying capability of the composite material (Lin & others, 2001). When a compressive load is applied on top of a fiber, friction occurs between the debonded portion of the fiber and the facing matrix; whereas, shear stress continues to develop at the front of the propagating crack. From complete debonding to extrusion, only friction opposes fiber dislocation (Lin & others, 2001; Chandra & Ghonem, 2001).

The described progress can be assumed to occur in the push-out test of an endodontic post. The retentive strength of a bonded post can be considered to be the combined result of micromechanical interlocking, chemical bonding and sliding friction. Thus, interpreting the results derived from a record of the maximum load during a push-out test as "bond strength," as it has commonly been referred to in the dental literature (Boschian Pest & others, 2002; Kurtz & others, 2003; Vallittu & Kurunmaki, 2003), has to be viewed with reservation. In this study, the authors prefer to address these results as push-out strengths.

In this experimental setting, the fiber post surface was silanized in order to enhance the post-cement bond. In addition, in a previous study where the bond strengths of several cements at the post-cement and cement-dentin interfaces were assessed separately, all the tested luting materials achieved a stronger adhesion to the post than to root dentin (Boschian Pest & others, 2002). Furthermore, in previous microtensile bond strength tests on composite overlays luted to coronal dentin with Panavia F (Mak & others, 2002), a microscopic analysis of the fractured specimens revealed that the most frequent failure mode was adhesive along the cement-dentin interface. Based on

these premises, failure of the bonded posts in this study can be expected to occur at the cement-dentin interface and be accompanied by the development of friction between the cement-coated post, possibly pictured as a "macro-fiber," and its embedding matrix, the root canal.

As the TEM results demonstrated the existence of gaps in the interfaces of the self-etching resin cement system ED primer/Panavia 21 (Figure 3) and the self-adhesive cement RelyX Unicem (Figure 4), it is speculated that, in this study, the push-out strengths obtained for these two resin cements were predominantly contributed by sliding friction. Such a speculation appears to be supported by the very low and highly inconsistent microtensile bond strength results obtained for the bonding of fiber posts via resin cements inside intact dowel spaces (Bouillaguet & others, 2003; Goracci & others, 2004). The authors are currently testing this hypothesis by performing comparative thin-slice push-out tests for total-etch and self-etch resin cement systems in the presence and absence of dentin adhesive applications.

The potential clinical implication from this study is that resistance to dislocation of fiber posts from root canals via the use of mild self-etch or self-adhesive resin cement systems may have very little to do with the actual bonding ability of these systems and may be largely contributed to by friction within the dowel spaces. Under such a premise, it would be clinically significant to compare the resistance to the dislocation of fiber posts luted with conventional zinc phosphate cements or resin-modified glass-ionomer cements, or posts constructed out of bonded amalgams, with the timely and much advertised resin composite cement systems. Ongoing work is also underway in the authors' laboratories to evaluate the long-term aging of resin cement systems for the luting of fiber posts with the use of the thin slice push-out test.

CONCLUSIONS

Interfacial strengths and ultrastructural findings concurrently demonstrated a greater bonding potential of the total-etch resin cement investigated. Conversely, the acidic resin monomers responsible for substrate conditioning in Panavia 21 and RelyX Unicem appeared to be less effective in etching through the thick smear layer created on root dentin during post space preparation. This may have accounted for the significantly lower retentive strength recorded by posts luted with these materials.

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Inhibitory Activity of Glass-ionomer Cements on Cariogenic Bacteria

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Clinical Relevance

This study demonstrated that glass-ionomer materials, especially the resin-modified glass-ionomer cement Vitrebond, regardless of the curing activation mode used, presented a striking inhibitory effect on cariogenic bacteria.

SUMMARY

This study evaluated the antibacterial activity of the glass-ionomer cements Vitrebond (3M ESPE), Ketac Molar (3M ESPE) and Fuji IX (GC America) against *S mutans*, *S sobrinus*, *L acidophilus* and *A viscosus*, using the agar diffusion test. Inocula were obtained by the seed of indicators cultures in BHI broth incubated at 37°C for 24 hours. Base layers containing 15 mL of BHI agar and 300 µL of each bacteria suspension were prepared in Petri dishes. Six wells measuring 4 mm in diameter were made in each plate and completely filled with one of the testing materials. A 0.2% chlorhexidine solution applied in round filter papers was used as control. Tests were performed 12 times for each material and bacteria strain. After incubation of the plates at 37°C for 24 hours, the zones of bacterial growth inhibition around the wells were measured. Overall, the results showed the following sequence of antibacterial activity: Vitrebond (despite the activation mode) > 0.2% chlorhexidine > Ketac Molar > Fuji IX, according to Kruskal-Wallis and Mann-Whitney statistical tests.

This study confirmed significant antibacterial activity for two conventional glass-ionomers and one resin-modified glass-ionomer material. The resin-modified glass-ionomer cement Vitrebond, regardless of the activation mode, presented the best antibacterial activity against *S mutans* and *S sobrinus*. The antibacterial activity against *A viscosus* for Vitrebond was similar to 0.2% chlorhexidine, while light activation reduced its antibacterial activity against *L acidophilus*.

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INTRODUCTION

Undesirable events, such as secondary caries (Bergenholtz & others, 1982; Brännström, 1984) and pulp injury (DeSchepper, White & von der Lehr, 1989a), are frequently associated with the presence of residual bacteria after carious dentin removal and the ingress of new microorganisms in the tooth-restoration interface as a result of microleakage. Dental materials, especially those applied in direct contact with the contaminated substrate, should have appropriate antibacterial

activity in order to prevent residual bacteria from continuing their metabolic activity, in addition to impairing new bacteria from reaching the tooth-restoration interface.

Incomplete removal of caries has been advocated in techniques such as indirect pulp capping and alternative restorative treatment (ART). In these procedures, glass-ionomer cements are used, in direct contact with residual carious tissue, to promote the rehardening of the tissue and to reduce the viability of residual bacteria (Botelho, 2003), thus preventing the occurrence of secondary caries (Donly & Ingram, 1997). Besides antibacterial activity, such materials should also have good biocompatibility with the dentin-pulp complex (de Souza Costa & others, 2003a,b).

Glass ionomer cements are able to inhibit the *in vitro* growth of some oral bacteria species (DeSchepper & others, 1989a; DeSchepper, Thrasher & Thurmond, 1989b; Scherer, Lippman & Kaim, 1989; Loyola-Rodriguez, García-Godoy & Lindquist, 1994; Meiers & Miller, 1996; Herrera & others, 2000), because of their initial low pH and/or capacity for fluoride release (McComb & Ericson, 1987; DeSchepper & others, 1989a). The same antibacterial inhibitory effect has been demonstrated for a new generation of materials known as resin-modified glass ionomer cements (RMGICs). These glass ionomers have better mechanical properties due to the incorporation of hydrophilic resin monomers, conventionally 2-hydroxyethyl dimethacrylate (HEMA) and better adhesion to dentin (Mathis & Ferracane, 1989) without losing the benefits of conventional glass ionomer cements, such as fluoride release (Meiers & Miller, 1996; Friedl & others, 1997; Herrera & others, 2000). RMGICs are referred to in the literature as having enhanced antibacterial properties compared to conventional glass-ionomer cements (Meiers & Miller, 1996; Herrera & others, 2000).

Therefore, the primary objective of this *in vitro* study was to evaluate the antibacterial activity of two conventional glass ionomer cements recommended for alternative restorative treatments (ART) and one RMGIC against selected cariogenic bacteria, *S mutans*, *S sobrinus, L acidophilus* and *A viscosus*. The secondary objective was to compare the antibacterial activity of the three materials against the same cariogenic bacteria.

METHODS AND MATERIALS

The materials used in this study are shown in Table 1. The antibacterial activity of each material was evaluated against *Streptoccocus mutans* (ATCC #25175), *Streptoccocus sobrinus* (ATCC # 27607), *Lactobacillus acidophilus* (ATCC # IAL-523) and *Actinomyces viscosus* (T14V # IAL.5) using the agar plate diffusion test. Indicator strains were grown in brain heart infusion broth (BHI, Difco Laboratories, Detroit, MI, USA) for 48 hours at 37°C, according to the physiological characteristics of each microorganism. The resultant bacteria were again placed in 5 mL BHI for 24 hours at 37°C to form a suspension (inoculum).

In each sterilized Petri dish (20x100 mm), a base layer containing 15 mL of BHI agar mixed with 300 µL of each inoculum, was prepared. After solidification of the culture medium, six wells measuring 4 mm in diameter were made in each plate and completely filled with one of the testing materials listed in Table 1. Twelve wells were filled with each material and bacteria strain. All the materials were handled under aseptic conditions according to the manufacturer's instructions. Two mixes of Vitrebond were prepared. The first was allowed to self-cure and the second was light activated (Vitrebond–LC) for 30 seconds with a visible light (Optilux 500, Demetron Research Co, Danbury, CT, USA, 450 mW/cm^2). One microliter of aqueous 0.2% chlorhexidine digluconate was applied on sterile filter paper discs (n=12), also 4-mm in diameter, which acted as a control. The plates were kept for two hours at room temperature for diffusion of the materials, then incubated at 37°C for 24 hours.

Zones of bacterial growth inhibition were recorded in millimeters (mm) using a digital caliper (Mitutoyo, SP, Brazil). Measurements were taken at the greatest distance between two points at the outer limit of the inhibition halo formed around the well. This measurement

Table 1: *Materials Used in This Study*

Materials	Classification	Composition	Batch #
Vitrebond (3M ESPE, St Paul, MN, USA)	Resin-modified glass ionomer	Powder: (Zn)fluoroaluminosilicate glass Liquid: copolymer of acrylic and maleic acid, HEMA, photo-activator, water	20020314
Ketac Molar (3M ESPE, St Paul, MN, USA)	Conventional glass-ionomer	Powder: Al-Ca-La fluorosilicate glass, 5% copolymer acid (acrylic and maleic acid) Liquid: polyalkenoic acid, tartaric acid, water	L/119169 P/119862
GC Fuji IX (GC, Tokyo, Japan)	Conventional glass-ionomer	Powder: alumino-flurosilicate glass Liquid: water, polyacrylic acid, polybasic carboxylic acid	0107041

was repeated three times and the mean was computed for each well. Statistical analysis was conducted using Kruskall-Wallis non-parametric tests to compare the inhibition zones of the materials against each bacteria strain at a significance level of 5%. Complementary Mann-Whitney tests (Wilcoxon rank-sum tests) were performed to identify the differences by pairing the materials.

RESULTS

Table 2 and Figure 1 show the mean values and standard deviations of the inhibition zones for each material according to the bacteria strain. All glass-ionomer cements produced diffusion zones that were not considered as representing antibacterial activity. Antibacterial activity was only considered to have occurred when a true inhibition zone was present, whether or not it was associated with the diffusion zone. Among the tested materials, Vitrebond showed the greatest inhibitory effect against *S mutans* and *S sobrinus*, regardless of the activation mode, followed by chlorhexidine (control group). Light activation of this RMGIC reduced its antibacterial activity against *L acidophilus*. Against *A viscosus*, both Vitrebond and chlorhexidine produced statistically similar inhibition zones, followed by Ketac Molar. This latter material showed good antibacterial activity against *L acidophilus* and *A viscosus*, after Vitrebond and chlorhexidine. Fuji IX, chlorhexidine and Ketac Molar produced statistically similar inhibition zones against *S mutans* and *S sobrinus*.

DISCUSSION

The agar plate diffusion test is an accepted method to initially discriminating antibacterial activity among materials, although some limitations should be considered. Without further tests, for example, it cannot be determined whether the data obtained from a specific material reflected bactericidal or just bacteriostatic effects. Also, it is extremely difficult to compare bacterial inhibition data with this technique, even for the same material, because of the variables involved (Tobias, Browne & Wilson, 1985), such as the type of agar and the number of microorganisms inoculated.

The results of this study indicated that all evaluated glass ionomer cements showed antibacterial activity, inhibiting the selected cariogenic bacteria growth. This antibacterial property has been related to either initial setting low pH, fluoride release or other chemical components present in the powder of these materials (DeSchepper & others, 1989b; Scherer & others, 1989; Loyola-Rodriguez & others, 1994).

Besides enhancing remineralization in the cyclic demineralization-remineralization caries process, fluoride can act on cariogenic microorganisms by altering their physiological status. The three main microorganism-growth inhibitory mechanisms of fluoride are direct binding of F-/HF to enzymes and other bacterial proteins, the binding of metal F complexes and action as a transmembrane proton carrier (Marquis, Clock &

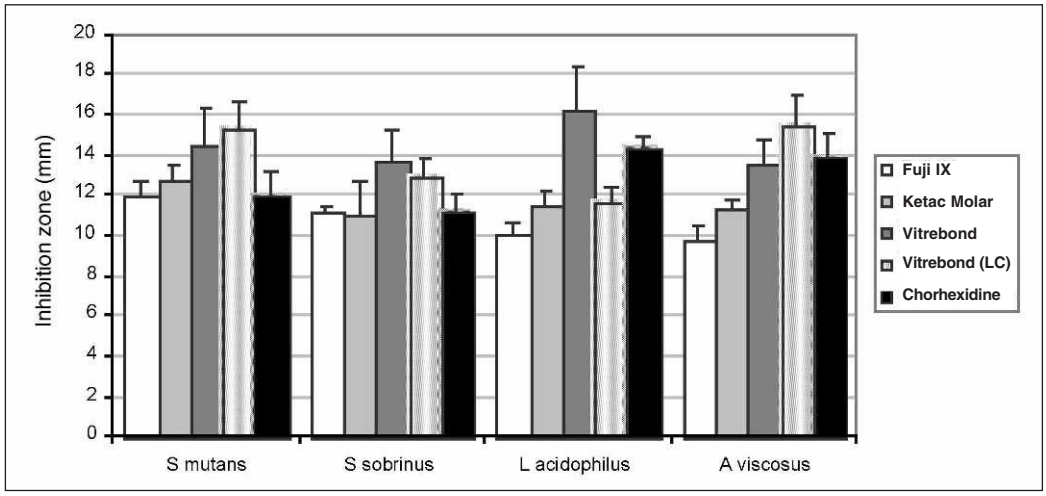

Figure 1. *Mean inhibition zone for each material according to bacteria strain (error bar = SD).*

Table 2: *Mean and Standard Deviation of the Inhibition Zones (mm) Evaluated on Inoculated Agar Plates after 24 Hours [n=12]*

	Materials				
Bacteria	Fuji IX	Ketac Molar	Vitrebond	Vitrebond (LC*)	Chlorhexidine
S mutans	11.9 ± 0.8[a**]	12.7 ± 0.8[b]	14.5 ± 1.8[c]	15.3 ± 1.4[c]	12.4 ± 1.2[a,b]
S sobrinus	11.2 ± 0.2[a]	11.0 ± 1.7[a]	13.7 ± 1.5[b]	12.9 ± 0.9[b]	11.2 ± 0.9[a]
L acidophilus	10.1 ± 0.6[a]	11.5 ± 0.7[b]	16.2 ± 2.2[c]	11.6 ± 0.7[b]	14.3 ± 0.6[e]
A viscosus	9.8 ± 0.6[a]	11.3 ± 0.5[b]	13.5 ± 1.3[c]	15.5 ± 1.5[c]	13.9 ± 1.2[c]

*light-cured
**For the same bacteria strain, means followed by the same letter are not different according to the Mann Whitney test (p>.05).

Mota-Meira, 2003). High concentrations of fluoride ranging from 0.16 to 0.3 mol/L can inhibit bacteria growth (Bowden, 1990).

Fluoride release from Vitrebond is higher than the conventional glass ionomers Ketac Molar and Fuji IX (Vermeersch, Leloup & Vreven, 2001). This finding could be justified by the fact that the powder/liquid ratio affects the rate of fluoride release (Forsten, 1995). A lower ratio results in increased solubility and fluoride liberation. In this study, Vitrebond was hand-mixed at a powder/liquid ratio of 1.4/1.0 by weight, while the ratio for Ketac Molar and Fuji IX were 3.6/1.0 and 3.5/1.0, respectively. The higher powder/liquid ratio improves the mechanical properties of conventional glass-ionomers for their use in posterior restorations.

In this study, RMGIC Vitrebond produced the greatest inhibition zones against *S mutans* and *S sobrinus*, regardless of the activation mode. Several other studies also demonstrated the same remarkable antibacterial activity for Vitrebond (DeSchepper & others, 1989a; Loyola-Rodriguez & others, 1994; Donly & Ingram, 1997; Herrera & others, 2000; Estrela & others, 2000). Kozai and others (2000) observed a strong association between fluoride release (about 6.93mg/L on the first day) and antibacterial activity for this particular material. Such activity is still observed even after the initial setting, where the pH is lower (Loyola-Rodriguez & others, 1994), suggesting that fluoride release is its primary antibacterial mechanism.

The main composition-related difference between conventional and resin-modified glass-ionomer cements is the addition of hydrophilic monomers to the liquid of the latter. As these monomers are conventionally water-soluble hydroxyethyl methacrylate (HEMA), it is tempting to also attribute the better antibacterial activity observed for Vitrebond to the presence of this component, as suggested by Coogan and Creaven (1993) and Benderli and others (1997). In fact, it has been shown that besides fluoride, RMGICs release several other components, including hydrophilic HEMA (Geursten, Spahl & Leyhausen, 1998). However, it has been demonstrated that HEMA has no antibacterial effect on cariogenic bacteria such as *S mutans, S sobrinus* and *L acidophilus* (Schmalz, Ergucu & Hiller, 2004).

Other chemical components, such as zinc sulfate, improve the antibacterial activity of glass ionomer cements. The addition of 5% $ZnSO_4$ to ionomer products significantly increases the inhibition of *S mutans* growth and fluoride release without interfering with the mechanical properties of these products (Osinaga & others, 2003). Zinc is one of the components present in the glass particles of Vitrebond, which could account for its higher antibacterial activity.

When Vitrebond is light-cured, an exothermic reaction is produced, increasing the temperature by nearly 20°C (Bourke, Walls & McCabe, 1992). Such a temperature change could be significant, but the antibacterial activity does not seem to be influenced by this factor, since similar bacterial growth inhibitory effects have also been shown for the chemical mode of activation of Vitrebond (Coogan & Creaven, 1993). In this study, the activation mode did not influence the inhibitory activity of Vitrebond, except against *L acidophilus*.

The marked antibacterial activity of Vitrebond could indicate high levels of toxic agents (Coogan & Creaven, 1993). When evaluated in cell culture, RMGICs showed a pronounced cytotoxic effect comparable to that of a restorative resinous material (de Souza Costa & others, 2003b). Conversely, pulp response to the application of Vitrebond in deep cavities performed in human teeth was comparable to a hard setting calcium hydroxide (de Souza Costa & others, 2003a).

Both conventional glass ionomers evaluated in this study were developed to have improved mechanical properties by increasing the powder/liquid ratio. The low solubility presented by these materials could explain their inferior antibacterial activity observed in this study when compared to RMGICs, which is probably related to lower fluoride release. The water solubility of Ketac Molar is decreased by a factor of five compared to a standard filling glass ionomer cement (Guggenberger, May & Stefan, 1998).

Ketac Molar and Fuji IX exhibited moderate antibacterial activity, demonstrating less inhibition than Vitrebond and 0.2% chlorhexidine. Boeckh and others (2002) evaluated the effects of some restorative materials on *S mutans* growth in liquid culture assays and observed that Ketac Molar exhibited significant growth inhibition effects when compared to other restorative materials. In this study, except for *S sobrinus*, Ketac Molar presented better antibacterial activity than Fuji IX. For this latter material, in a study by Botelho (2003), no measurable antibacterial activity was observed against *S mutans* and *L acidophilus*, suggesting the incorporation of antibacterial agents such as chlorhexidine into the cement.

CONCLUSIONS

This study confirmed significant antibacterial activity for two conventional and one resin-modified glass-ionomer cement. Among the glass ionomer materials, Vitrebond presented the best antibacterial activity against all cariogenic bacteria tested. This was true regardless of the activation mode employed, except against *L acidophilus*. For this particular bacteria strain, light activation of Vitrebond reduced its antibacterial activity to the level of Ketac Molar, which was still considered satisfactory.

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Influence of Light Activation on the Volumetric Change of Core Foundation Resins

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Clinical Relevance

The data suggests that the volumetric shrinkage of core foundation resins can be influenced by the intensity and duration of curing light. Care should be taken in the method of polymerization when using core foundation systems for endodontically treated teeth.

SUMMARY

A core foundation system is frequently used in endodontically treated teeth that suffer excessive loss of the coronal portion of their structure. The volumetric shrinkage of core foundation resins may create marginal gaps that influence the bonding ability and longevity of a restored tooth. Little is known about how activation conditions of resin core foundation resin pastes affect their volumetric shrinkage. This study evaluated the influence of light intensity and light activation duration on volumetric shrinkage of direct core foundation resins. Two dual- and one light-activated core foundation resin pastes were employed. The material was placed in a Teflon mold 4 mm in diameter and 2 mm in height and extruded into a water filled dilatometer. The specimens were then light activated and the change in height of the meniscus of water was recorded using a charged-coupled device camera. The average volumetric shrinkage of the core foundation resins after 180 seconds ranged from 1.53% to 2.63%. For all materials tested, there was a tendency for increased volumetric shrinkage with increased light activation time and intensity. The results of this study indicate that the volumetric change of core foundation resins is influenced by the time and intensity of light activation.

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INTRODUCTION

The recent development of direct core foundation systems enable dentists to restore non-vital teeth by only replacing the tooth structure that was lost due to endodontic treatments (Leonard, Gutmann & Guo, 1996; Mannocci, Innocenti & Ferrari, 1998; Mannocci & others, 1999; Ferrari & others, 2000; O'Keefe & Powers, 2001). Wettability of the conditioned adherent surface with adhesives of core foundation system is important in the bonding of core foundation resins, whether the bond to tooth is created through chemical, micromechanical interlocking or a combination of these mechanisms (Vichi & others, 2002). Proper infiltration and retention of the employed adhesive system is required, because bonding to the root canal surface is always challenged by polymerization shrinkage of the core foundation resins (Heydecke, Butz & Strub, 2001). Selecting an appropriate adhesive system in combination with a core foundation resin for this purpose is a determining factor for success of the treatment (Duncan & Pameijer, 1998; Stockton, 1999).

The volumetric shrinkage of core foundation resins may create marginal gaps that influence the bonding ability and longevity of a restored tooth. For restorative resin composites, it is reported that water sorption and subsequent swelling might lead to partial compensation of the initial volumetric shrinkage and setting stress that begin immediately with the start of light activation (Feilzer & others, 1995; Attin & others, 1995; Kim, Hirano & Hirasawa, 1998). For core foundation resins, it may be difficult to absorb enough water from the oral environment to compensate for polymerization contraction. Once a gap is created, it will remain as a flaw to initiate failure of the restored tooth. The composition of the core foundation resin and polymerization activation method serve as factors that can influence shrinkage characteristics during polymerization. Since the direct core foundation resins have to flow into narrow spaces in the root canal, filler contents of the resin pastes are relatively low in order to facilitate handling properties. Therefore, they may exhibit more volumetric shrinkage than the hybrid type resin composites for fillings. The geometry of the root canal is also related to contraction stress induced by polymerization reaction. According to the concept of the C-factor (Feilzer, de Gee & Davidson, 1987), resin pastes that flowed into the root canal may be the worst case for contraction stress development. For dual- and light-activated resins, proper light activation is needed for a relatively high degree of polymerization to achieve good mechanical properties and bonding to the canal surface.

This study evaluated the effect of light intensity and exposure duration on the volumetric shrinkage of several core foundation resins. The null hypothesis to be tested was that there was no difference in volumetric shrinkage of direct core foundation resins with regards to light activation intensity or time.

METHODS AND MATERIALS

Core Foundation Resins and Visible Light Activation Unit

Two dual-activated and a light-activated core foundation resin were used. Their composition filler contents, lot numbers and manufacturers are listed in Table 1.

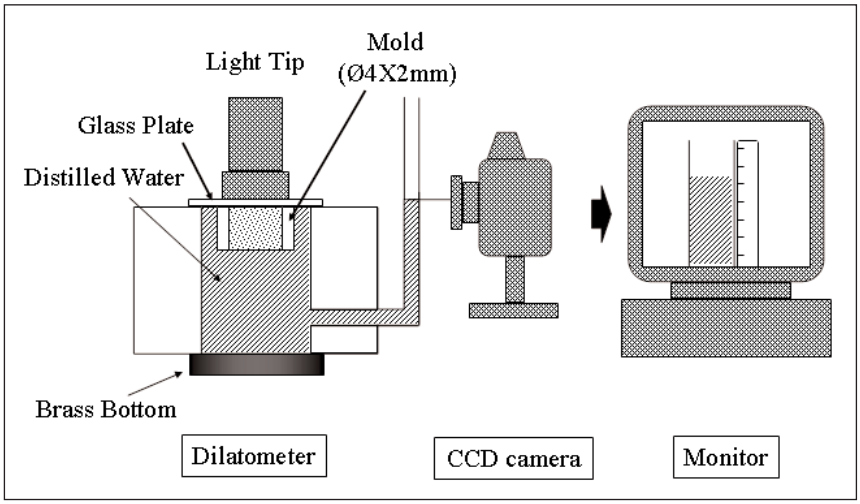

Figure 1. *Schematic diagram of the volumetric change recording system.*

Table 1: *Core Foundation Systems Used*

Core Foundation Resin	Component	Filler Contents#	Lot #	Manufacturer
Clearfil DC Core*	Bis-GMA, dimethacrylate, filler, photo/chemical initiator	82 wt%	C: 0176 U: 0169	Kuraray Medical, Tokyo, Japan
UniFil Core*	UDMA, dimethacrylate filler, fluoroaluminosilicate glass photo/chemical initiator	79 wt%	0201291	GC Corp, Tokyo, Japan
Clearfil Photo Core**	Bis-GMA, dimethacrylate filler, photo initiator	83 wt%	01586A	Kuraray Medical, Tokyo, Japan

**: Dual-polymerized, **: Light-polymerized*
#: Manufacturer's information

The input voltage of the activation unit, Optilux 400 (Demetron/Kerr, Danbury, CT, USA), was connected to a variable transformer to control light intensity. The light intensity was measured with a curing radiometer (Model 100, Demetron/Kerr) and adjusted to 200, 400 and 600 mW/cm^2.

Measurement of Volumetric Shrinkage

The test apparatus consisted of a dilatometer tube containing a capillary of uniform diameter and a length of approximately 130 mm attached to a density bottle of volume 25 cm^3 by means of a ground glass joint as shown in Figure 1 (Miyazaki & others, 1991). The capillary diameter was 0.5 mm and was graduated in divisions of 0.1 mm. The density bottle was filled with distilled water. During the test, the temperature of the liquid was controlled by maintaining the density bottle on a thermostatically controlled plate (TA-740, Cooltron, Dentoronics Co, Tokyo, Japan). The bottom part of the density bottle was made of brass to keep the temperature at 23 ± 1°C. The work was done in a constant temperature room at 23 ± 1°C, 50 ± 5% relative humidity and was illuminated with only a small red light.

Resin pastes were placed in a Teflon mold, 4.0 mm in diameter and 2.0 mm in height, on a 0.5 mm-thick cover glass (Matsunami Glass, Tokyo, Japan). The specimen and cover glass were placed on a rubber O-ring attached to the specimen window of the density bottle (the free surface of the restorative material extruded into the water in the density bottle) and was then covered tightly with a lid that had a 15-mm hole. The light tip of the activation unit was placed into the hole in the lid (Figure 2). Light activation of the specimen was initiated immediately after the dilatometer was equilibrated. The change in height of the meniscus of water was recorded using a CCD camera (DS-505, Nikon Corp, Tokyo, Japan) and replayed on a VRC (CT-1450, Hitachi Corp, Tokyo, Japan) from the start of light activation. The volumetric shrinkage of the specimen (ΔV) was calculated from the change in water meniscus (Δh) as follows:

$$\Delta V = 0.25\pi \bullet \Delta h \bullet d^2$$

where "d" is the diameter of the capillary. The percentage of volumetric change was calculated as $\Delta V/V \times 100$, where V is the original specimen volume.

For recording the thermal expansion of the liquid and the material itself due to irradiation heating (the peak temperature was around 80°C at the end of the light guide tip), the polymerized material was placed in the dilatometer and light activated in the same manner. The rise in the height of the liquid in the dilatometer was recorded, and this data was then subtracted from the raw data at each respective recording time in order to obtain the actual polymerization shrinkage.

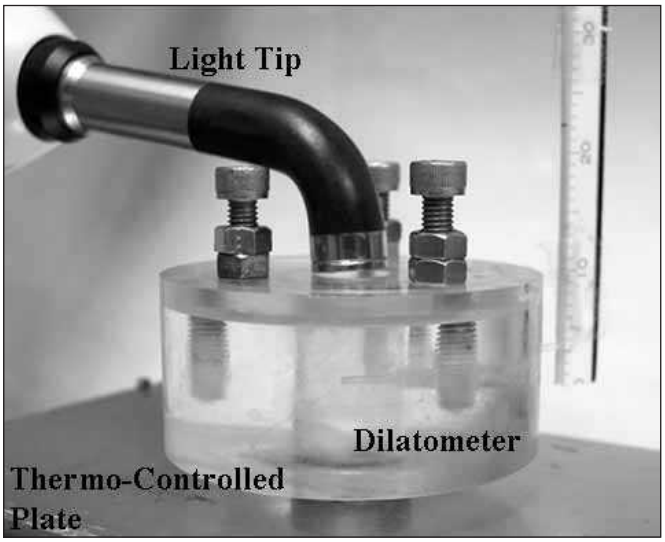

Figure 2. *Modified dilatometer placed on the thermostatically controlled plate, in operation. The light tip of Optilux 400 is also shown.*

The experiments were designed to study the influence of the following factors on volumetric shrinkage.

(1) Effect of light activation time: The light activation times were 20, 30 (control) and 60 seconds (light intensity; 400 mW/cm^2).

(2) Effect of light intensity: The light intensity was adjusted at 200, 400 (control) and 600 mW/cm^2 and measured with the radiometer (light activation time; 30 seconds).

Statistical Analysis

The sample size for each experimental condition was five. The mean and standard deviation of the total volumetric shrinkage at the end of light activation and after 180 seconds were calculated. The data were subjected to two-way ANOVA followed by Tukey HSD test at a p-value of 0.05 to make comparisons among the materials using a computer statistics package (Sigma Stat Ver 2.03, SPSS Inc, Chicago, IL, USA).

RESULTS

Volumetric Shrinkage

The results of the volumetric change during 180 seconds for each material activated under the control condition (30 seconds light activation with light intensity of 400 mW/cm^2) are shown in Figure 3. Volumetric shrinkage began shortly after the start of light exposure and continued after stopping light exposure. The average volumetric shrinkage of the core foundation resin pastes after 180 seconds ranged from 1.53% to 2.63%. No significant differences were found in the total shrinkage after 180 seconds for the core foundation resins used.

The effects of light activation time on total volumetric shrinkage of core foundation resins are shown

in Table 2 and Figure 4. For all the materials tested, the volumetric shrinkage increased with prolonged light activation time, and the influence of this variable was significant for the materials tested.

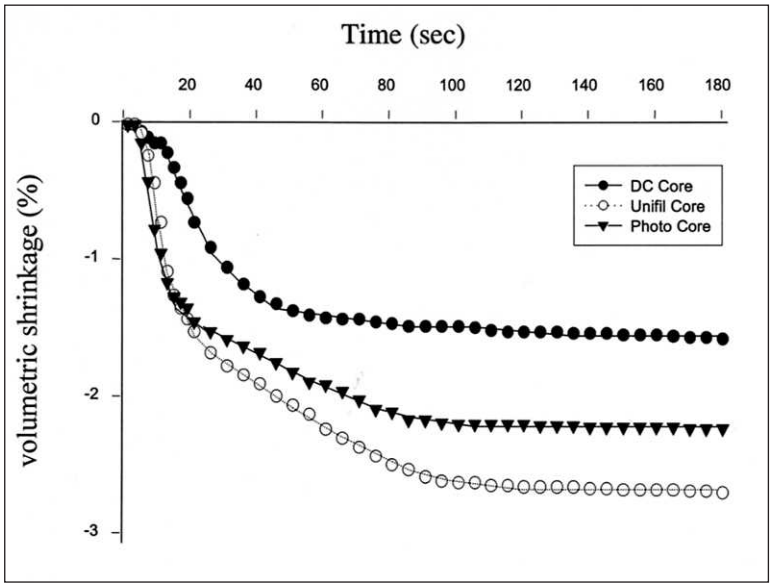

Figure 3. *Volumetric changes in the three-core foundation resins during 180 seconds.*

The effects of light intensity on the volumetric shrinkage of core foundation resins are shown in Table 3 and Figure 5. For all materials tested, the volumetric shrinkage increased with increased light activation intensity, and the influence of this variable was significant for the materials tested. Multiple comparisons showed that, in general, the groups were significantly different from each other.

DISCUSSION

Different methods of measuring polymerization shrinkage of restorative materials have been investigated. A test method based on the drop of water level in a pyconometer containing a material exhibiting polymerization shrinkage has been described (de Gee, Davidson & Smith, 1981; Bandyopadhyay, 1982; Goldman, 1983). A volumetric change that occurs within the specimen will be reflected by alteration of the volume of the water column and can be detected by the water height in a capillary column. There have been some modifications to this system in an effort to increase the accuracy so that it may be employed for light-cured resin composites (Penn, 1986; Rees & Jacobsen, 1989; Lai & Johnson, 1993). Deflection disk techniques (Walls, McCabe & Murray, 1988; Watts & Cash, 1991; de Gee, Feilzer & Davidson, 1993) and He-Ne laser scanning method (Fano & others, 1997) have been introduced as non-volume dilatometric measurements. The modified dilatometer eliminates the significant attenuation of the curing light by the density bottle, because the specimen was light irradiated just through the 0.5 mm-thick cover glass (Figure 1). The measurements were conducted at 23°C following a previous study (Bandyopadhyay, 1982). In clinical situations, the material is stored at room temperature (23°C), then placed in the oral environment (37°C). This temperature discrepancy would cause a volumetric expansion. Though this situation inevitably arises in clinical practice, maintaining a constant temperature

Table 2: *Effect of Light Activation Time on Volumetric Shrinkage of Core Foundation Resins*

Material	Light Activation Time (sec)	Shrinkage at the End of Light Activation (%)	Shrinkage After 180 seconds (%)
Clearfil DC Core	10	0.22 ± 0.03	1.36 ± 0.08
	30	1.02 ± 0.08	1.53 ± 0.08
	60	1.85 ± 0.08	2.00 ± 0.17
UniFil Core	10	0.69 ± 0.02	2.11 ± 0.11
	30	1.66 ± 0.18	2.63 ± 0.09
	60	2.02 ± 0.15	2.77 ± 0.13
Clearfil Photo Core	10	0.90 ± 0.05	1.64 ± 0.16
	30	1.50 ± 0.12	2.17 ± 0.15
	60	1.78 ± 0.10	2.33 ± 0.16

mean ± SD, n=5
Values connected by lines indicate no significant differences among the group (p<0.05).

Table 3: *Effect of Light Activation Intensity on Volumetric Shrinkage of Core Foundation Resins*

Material	Light Intensity (mW/cm^2)	Shrinkage at the End of Light Activation (%)	Shrinkage After 180 seconds (%)
Clearfil DC Core	200	0.39 ± 0.02	1.03 ± 0.11
	400	1.02 ± 0.08	1.53 ± 0.08
	600	1.28 ± 0.13	2.05 ± 0.18
UniFil Core	200	1.20 ± 0.12	1.47 ± 0.10
	400	1.66 ± 0.18	2.63 ± 0.09
	600	2.13 ± 0.13	2.83 ± 0.19
Clearfil Photo Core	200	0.75 ± 0.08	1.33 ± 0.18
	400	1.50 ± 0.12	2.17 ± 0.15
	600	1.69 ± 0.17	2.20 ± 0.08

mean ± SD, n=5
Values connected by lines indicate no significant differences among the group (p<>0.05).

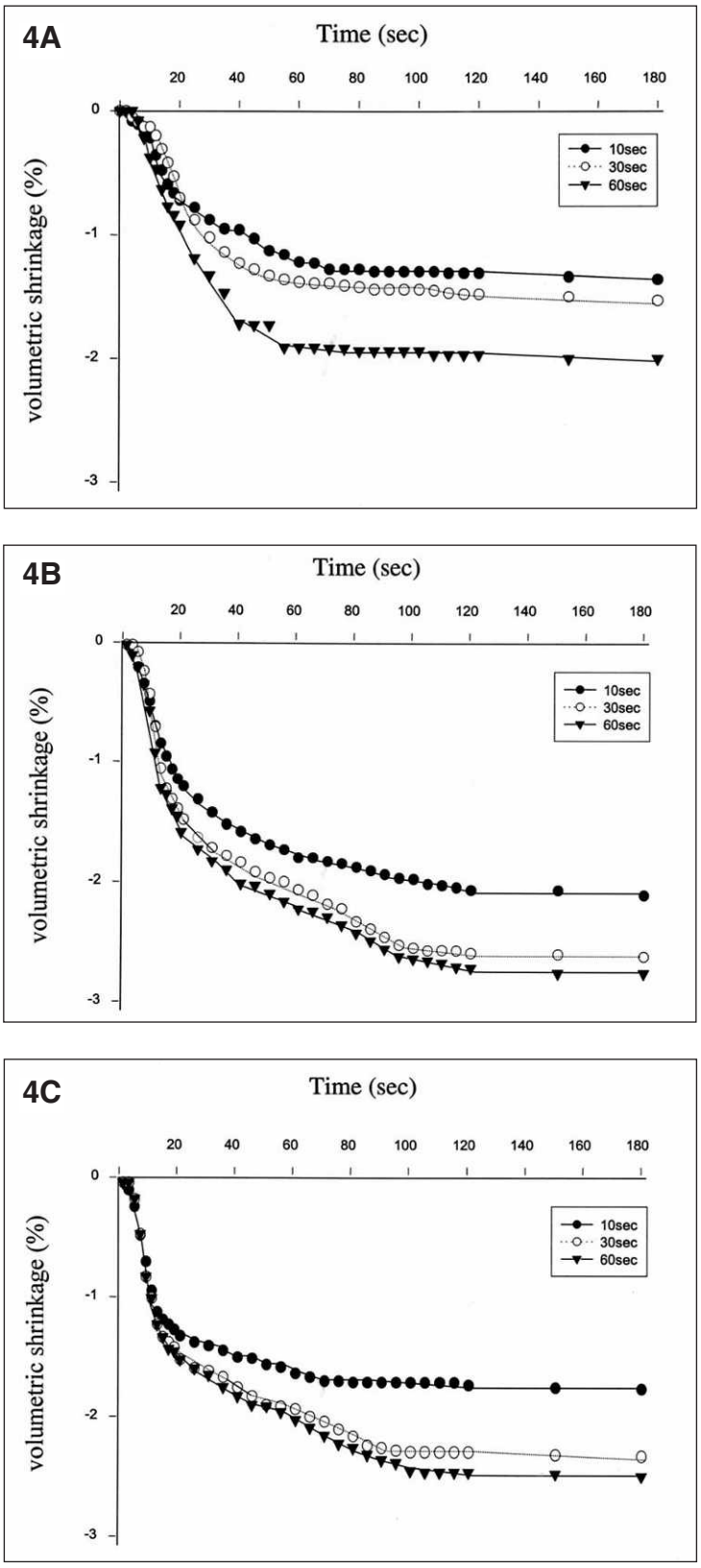

Figure 4. *Effect of light activation time on volumetric shrinkage of core foundation resins. A: Clearfil DC Core, B: UniFil Core, C: Clearfil Photo Core.*

environment for the dilato-meter during the test is critical to the success of the measurement (Lai & Johnson, 1993). In this system, this is achieved by maintaining room temperature and by placing the dilatometer on a thermo-controlled plate.

From the results of this study, the experimental null hypothesis was not confirmed. The volumetric shrinkage of the core foundation resins used in this study was influenced by the light activation conditions of the resin pastes. The volumetric shrinkage of a resin paste depends on many factors, such as filler loading, filler type and filler size. Other factors that contribute to the shrinkage are the monomer systems and initiating systems, because they determine the structure of the material (Soh & Yap, 2004). Most light-cured materials use camphorquinone as a photoinitiator (Taira & others, 1988; Hayakawa, Kikutake & Nemoto, 1999), and an adequate intensity of visible light at wavelengths around 470 nm is required to initiate polymerization (Nomoto, 1997; Nomoto & Hirasawa, 1999). Light of the appropriate wavelength is absorbed by the photosensitizer, which then reacts in its excited state with an amine-reducing agent to produce reactive free radicals (Ikemura, Kouro & Endo, 1996; Nyunt & Imai, 1996). The transmission of light through the material and the composition of the photoinitiator both influence the mechanical properties of light-cured materials (Cook, 1992). The intensity of light passing through a material is controlled by absorption and by the scattering of its components (Arikawa & others, 1998; Arikawa & others, 2000). The rate of polymerization is reduced at lower light intensities, which results in a lower shrinkage rate.

An activation unit with high light intensity is recommended based on mechanical properties (Yoshida & Greener, 1994; Miyazaki & others, 1995). On the other hand, the possible negative influence of higher light intensity on stress development must be considered. As polymerization shrinkage increases with increasing light intensity, adverse effects on the marginal adaptation of resin fillings may occur. Uno and Asmussen (1991) recommended using low light intensity to allow for the reduction of the polymerization rate and relaxation of the contraction stress in order to obtain good marginal adaptation. The optimum range of light intensity for curing light-cured materials is still controversial. Prolonged light activation time resulted in an increase in volumetric shrinkage (Table 2). This increase in shrinkage suggests that further polymerization occurred with the increased exposure duration from the time recommended by each manufacturer. Longer light activation times than those quoted by the manufacturers have been advocated to avoid poorly polymerized portions of the material. Shorter light activation times may result in

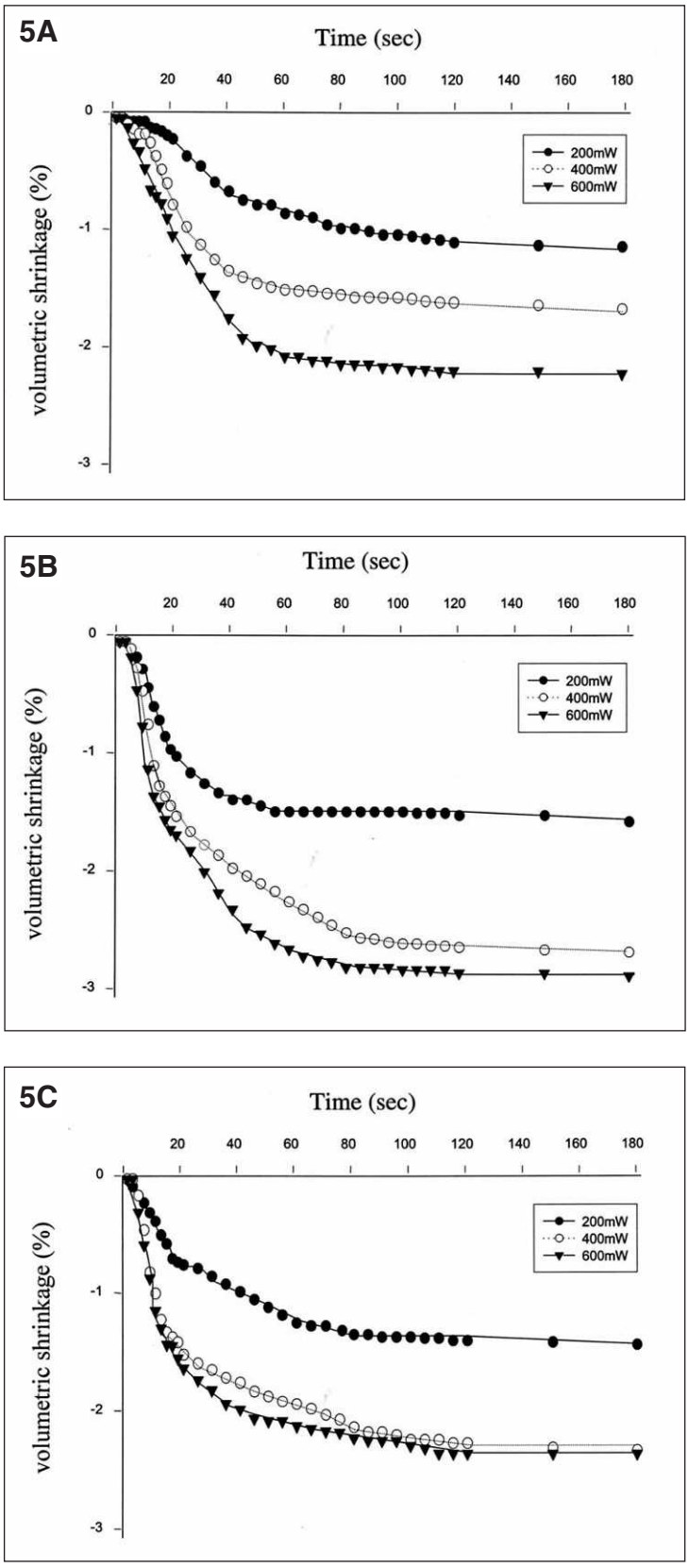

Figure 5. *Effect of light activation intensity on volumetric shrinkage of core foundation resins. A: Clearfil DC Core, B: UniFil Core, C: Clearfil Photo Core.*

some flow within the polymerized specimen as a result of its degradation in the oral environment.

Dual-activated resin pastes polymerize chemically upon mixing the two pastes and by light activation when irradiated. These resins have different polymerization characteristics due to their composition, some being more dependent on light activation than others (El-Mowafy, Rubo & El-Badrawy, 1999). Differences in polymerization shrinkage behavior observed in this study might be explained by the concentration of photo initiator. For a given resin paste, the higher the light intensity and the longer the light activation time, the higher the volumetric shrinkage. Theoretically, a high conversion rate leads to more rapid and higher shrinkage. In such a case, contraction stress induced during polymerization shrinkage would reach higher value. Considering the confined conditions for use of core foundation resins into root canals, stress relief by flow or a low elastic modulus are required. Although there is a relationship between the extent of polymerization and enhancement of physical properties (Ferracane & others, 1997), care should be taken not to infer clinical success of the core foundation from light activation methods alone.

The extent of volumetric shrinkage can create marginal gaps if the material does not sufficiently adhere to tooth structure. It should be noted that the materials might behave differently if they are bonded to cavity walls (Versluis, Tantbirojn & Douglas, 1998). Further studies are needed to determine the influence of volumetric change on the bond strength to root canal surface.

CONCLUSIONS

The results of this *in vitro* study indicated that the volumetric shrinkage of the core foundation resins was affected by light activation conditions, including resins that were dual-activated. With higher light intensity and/or longer duration of light activation, higher volumetric shrinkage was recorded.

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The Effect of Composite Type on Microhardness When Using Quartz-tungsten-halogen (QTH) or LED Lights

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Clinical Relevance

Resin composite surface microhardness is affected by the selection of different LCUs, with some LEDs providing similar performance to the QTH source. However, results vary greatly with composite brand and type (microhybrid and microfill).

SUMMARY

This study evaluates the Knoop microhardness of resin composites cured with different light-emitting diode (LED) based light curing units (LCU) or with a conventional quartz-tungsten-halogen light (QTH). Ten experimental groups with 10 specimens each were used. The specimens were prepared by placing two light-cured resin composites with similar VITA shade A2–microhybrid Filtek Z250/3M ESPE and microfill Durafil VS/Heraeus Kulzer—in a 2.0 mm-thick disc shaped mold. The specimens were polymerized for 40 seconds with the use of one QTH LCU (Optilux 501/Kerr-Demetron) and four LED LCUs: Elipar FreeLight 1 Cordless LED (3M ESPE), Ultrablue II LED with cord (DMC), Ultrablue III LED cordless (DMC) and LEC 470 I (MM Optics). Knoop microhardness was determined at the top and bottom surfaces of the specimens 24 hours following curing. Microhardness values in the microhybrid resin composite group showed no statistically significant differences when cured with LED FreeLight 1 LCU and QTH LCU ($p<0.05$). The other LED devices evaluated in the study presented lower microhardness values in both surfaces ($p<0.05$) when compared to QTH. In the microfill resin composite group, no statistically significant differences were observed among all LCUs evaluated on the bottom surfaces ($p<0.05$). However, on the top surfaces, QTH presented the highest KHN values, and the LED devices presented similar results when com-

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pared with KHN values relative to each other ($p<0.05$).

INTRODUCTION

Light-cured resin composites were introduced into the market in the 1970s. The early products were cured by UV light, while the later versions by visible light (Rueggeberg, 2002). A halogen lamp is routinely used as a dental light activation unit. These lamps produce light by incandescence, where a filament is heated and causes the excitation of atoms over a wide range of energy levels, producing a very broad spectrum of light (Fujibayashi & others, 1998). Filters are therefore needed to restrict the emitted light to the blue region of the spectrum for the polymerization of resin composites (Rueggeberg, 2002). However, halogen based light-curing units (LCU) used to polymerize dental material have several drawbacks (Yoon & others, 2002). Some examples include overheating of the incandescent lamp (Fujibayashi & others, 1998), limited effective lifetime (approximately 40-100h) of the halogen bulbs (Rueggeberg & others, 1996; Jandt & others, 2000; Stahl & others, 2000) and degradation of internal components over time (bulb, reflector, filter) due to the high operating temperatures and large quantity of heat produced during duty cycles (Mills, Jandt & Ashworth, 1999; Jandt & others, 2000). It has also been shown that many halogen LCUs do not reach the minimum power output specified by the manufacturers (Barghi, Berry & Hatton, 1994; Martin, 1998; Miyazaki & others, 1998; Mills & others, 1999; Jandt & others, 2000; Stahl & others, 2000). These shortcomings could result in inadequate curing, which could negatively affect restoration prognosis (Mills & others, 1999; Jandt & others, 2000; Stahl & others, 2000).

To overcome the problems inherent with halogen LCU, solid-state light-emitting diode (LED) technology has been proposed for curing light-activated dental materials (Mills, 1995). Rather than a hot filament, as used in halogen lamps, LED use junctions of doped semiconductor (p-n junctions) to generate blue light (Nakamura, Mukai & Sengh, 1994; Haitz, Craford & Weissman, 1995; Kurachi & others, 2001). These junctions are partially collimated by a small polymer lens positioned in front of the p-n junctions (Jandt & others, 2000; Stahl & others, 2000). LEDs operate around 470 nm, which falls conveniently within the camphorquinone absorption spectrum (Mills & others, 1999; Jandt & others, 2000; Stahl & others, 2000). Blue LEDs present spectral purity for the highly efficient curing of dental resins. Moreover, LEDs have an effective lifetime of more than 10,000 hours and do not present significant degradation of light emission over time (Haitz & others, 1995; Stahl & others, 2000).

An adequate curing of resin composites may influence the mechanical properties and clinical optimization of these materials (Ferracane, 1993; Bayne, Heymann & Swift Jr, 1994). Microhardness is a typical parameter for indicating the degree of polymerization of resin composites (Ferracane, 1985). However, adequate surface hardness does not ensure proper polymerization throughout the restoration (Asmussen, 1982). Thus, hardness analysis must also be performed on the bottom surface of the specimens, since insufficient polymerization of this area may increase the risk of bulk and marginal fracture (Quance & others, 2001). Other possible complications associated with the inadequate curing of resin composites include secondary caries and adverse tissue reactions (Shortall, Wilson & Harrington, 1995). These factors could highly contribute to an early failure of the restorative procedure (Blankenau & others, 1991; Ferracane, 1993).

Several methodologies have been proposed in order to evaluate the polymerization of light curing units. Yearn (1985) has presented a review on the three main methods for evaluating resin composite curing depth: scrape test, hardness test (Barcol, Vickers and Knoop) and degree of conversion (Multiple Internal Reflection spectroscopy and Laser Raman spectroscopy). The author concluded that the hardness test provides a convenient and efficient method for evaluating curing depth.

A composite material has been defined as a "three-dimensional" combination of at least two chemically different materials with a distinct interface separating the components (Peutzfeldt, 1997). Dental resin composites usually encompass three main components: 1) resin matrix, 2) inorganic fillers and 3) coupling agents (Peutzfeldt, 1997). The amount and size of filler particles incorporated in the resin matrix determine the type, and ultimately, the most advantageous clinical application of each composite (Wakefield & Kofford, 2001). Early materials incorporated large ground quartz particles which resulted in rough surfaces that were difficult to polish (Rueggeberg, 2002). Due to the modification of fillers to extremely small particles, microfill composites have been developed (Rueggeberg, 2002). These materials usually consist of silica particles with 0.01-0.1 μm (Wakefield & Kofford, 2001). In general, microfill composites have the advantage of high polish, which lasts, in addition to excellent esthetics (Wakefield & Kofford, 2001).

Microhybrid composites are a combination of microfill and larger filler particles. The main drawback of this group, as determined by larger particle size, is the difficulty in long-term maintenance of a high polish (Wakefield & Kofford, 2001). The advantages of these materials are strength, high percent fill (75% to 80% by weight) and a wide array of shades, opacities and translucencies that are important when considering function and esthetics in restoration (Wakefield & Kofford, 2001).

Therefore, due to the increasing use of resin composites in daily practice and the availability of different light curing devices in the dental market, this study evaluated the efficacy of polymerization of two resin composites cured with four LED and one QTH LCUs at two depths.

The research hypothesis tested was that there is no difference between the light-curing units (LED or QTH) for the Knoop microhardness of resin composites (microhybrid or microfilled).

Table 1: *Restorative Materials Used in This Study*

	Filtek Z250	Durafil VS
Manufacturer	3M ESPE Dental Products St Paul, MN, USA	Heraeus Kulzer Inc Armonk, NY, USA
Batch #	1LC	020131
Classification	Microhybrid	Microfill
Shade	A2	A2
Type of filler	Zircon/silica	Highly disperse silicon dioxide
Filler particle size (μm)	0.01-3.5	0.02-0.07
Pre-polymerized particles	---	Splinter polymer (10-20 μm)
Filler loading	60 vol%	Unknown
Photoinitiator system	Camphoroquinone	Camphoroquinone

METHODS AND MATERIALS

Specimens were prepared with microhybrid Filtek Z250 (3M ESPE Dental Products, St Paul, MN, USA) and microfill Durafil VS (Heraeus Kulzer Inc, Armonk, NY, USA) resin composites (Table 1). The four light-curing units (LCU), based on blue LED technology with a different number of LEDs and the quartz-tungsten-halogen (QTH) light LCU, are presented below:

- FreeLight 1 LED (3M ESPE Dental Products)
- Ultrablue II corded LED (DMC Equipamentos, Sao Carlos, SP, Brazil);
- Ultrablue III cordless LED (DMC Equipamentos)
- LEC 470 I (MM Optics, Sao Carlos, SP, Brazil)
- Optilux 501 (Kerr Dentistry/Demetron, Orange, CA, USA)

Specimens were prepared at random by placing a single increment of resin composite in a cylindrical metallic mold (4.0 mm in diameter and 2.0 mm in height). The mold was then positioned over a polyester strip fixed on a glass plaque. After resin insertion, a second polyester strip was placed on the top of the mold. In order to ensure a level plane on the top and bottom surfaces, a glass lamina with a 500g weight was laid on the top polyester strip for 30 seconds. Light curing was performed by touching the LCU guide on the top polyester strip for 40 seconds. The power output of the light sources was measured by the use of a radiometer attached to the Optilux 501 LCU to verify whether they were properly working. Thus, 10 experimental groups (n=10) were prepared according to the resin composite and LCU that was applied.

The specimens were stored in distilled water in a light-proof container for 24 hours and microhardness was determined by a Microhardness Tester FM (Future-Tech Corp, Tokyo, Japan). Microhardness measurements were performed by applying a 10g load for 10 seconds at five points, 1 mm apart on the specimens' top and bottom surfaces. The five measurements obtained were converted into a Knoop Hardness Number (KHN) and the average was calculated.

Statistical analysis was performed by three-way analysis of variance (ANOVA) with split-plot at a significance level of α=5%. The plots were represented by factors LCU and resin composite, and sub-plots were represented by factor surface (top and bottom). Tukey test was applied when significant differences were detected by ANOVA (α=5%).

RESULTS

The results showed statistically significant differences between the LCUs (p=0.00001), resin composites (p=0.00001) and depths (p=0.00001) evaluated. It also verified a significant interaction among LCU vs resin composites vs depths (p=0.00148).

Mean hardness values and standard deviations (SDs) at top and bottom depths for each resin composite and the LCUs are shown in Table 2. Analyzing the bottom surface of the microhybrid resin composite group, the FreeLight 1 LED LCU was the only device that presented no statistically significant difference when compared with QTH LCU (LED-53.1 ± 6.5^{A}, QTH LCU-57.7 ± 4.2^{A}). In addition, there was no statistically significant difference between Ultrablue II corded LED LCU (46.9 ± 5.4^{C}) and Ultrablue III cordless LED LCU (48.45 ± 2.5^{C}). Also, LEC 470 I LED LCU presented the lowest values of KHN (40.4 ± 11.5^{D}). However, for the microfill group, no statistically significant difference was found among all evaluated LCUs.

On the top surface of the microhybrid group, FreeLight 1 LED LCU also presented no statistically significant difference when compared to QTH LCU (LED-57.1 ± 3.7^{A}, QTH LCU-60.1 ± 6.5^{A}). In addition, no statistical difference was observed between Ultrablue II corded LED (51.2 ± 5.2^{B}) and Ultrablue III cordless LED LCU (53.21 ± 4.8^{B}). In the microfill group, QTH LCU presented the highest values of KHN.

Comparing Knoop hardness values at the top and bottom surfaces, it was verified that the top surface of the microhybrid resin presented the highest KHN

values. However, when the microhybrid resin was cured by QTH lamp, no statistically significant difference could be noted between the two surfaces. As for the microfill group, no statistically significant difference was observed between the two surfaces for all LED devices. Nevertheless, KHN values for QTH LCU on the bottom surface were lower (16.46 ± 1.1[b]) than the top surface values (25.1 ± 2.4[a]).

DISCUSSION

By analyzing the data obtained in this study, it could be verified that the research hypotheses tested in this study were rejected.

The microhybrid resin composite presented the highest KHN values (Table 2) regardless of the light source (QTH or LEDs) or surface (top or bottom). The size and content of filling particles in the organic matrix of microhybrid resin composites might have accounted for the highest KHN values reported (Ruyter & Oysæd, 1982; Pilo & Cardash, 1992; Rueggeberg & others, 1993).

The polymerization of microhybrid resin composite with FreeLight 1 LED LCU resulted in similar microhardness values when compared to QTH LCU (Table 2). Although LED LCU presents a 32% smaller light intensity (282 mW/cm^2) than QTH LCU (866 mW/cm^2), an adequate polymerization of microhybrid could be verified in the current investigation. A possible explanation for these results may be the fact that LED LCUs present a specific pattern of light emission, which is similar to the absorption spectrum of the camphorquinone photoinitiator of resin composites (Mills & others, 1999; Jandt & others, 2000; Stahl & others, 2000). This spectral purity allows total usage of the emitted light by LED, which does not happen with QTH (Whitters, Girkin & Carey, 1999; Althoff & Hartung, 2000). Hofmann, Hugo and Klaiber (2002) also evaluated the microhardness of Filtek Z250 microhybrid resin cured with QTH (800mW/cm^2 for 40 seconds) and LED (320mW/cm^2 for 40 seconds). These authors observed no statistically significant differences between these two light sources, which is in agreement with the results of this study.

The remaining LED-based LCUs evaluated in this study showed lower hardness values when compared with QTH LCU (Table 2). Some authors report that LED-based LCUs have a narrow spectral range, with a peak around 470 nm, which matches the optimum absorption wavelength for the activation of the camphorquinone photoinitiator (Jandt & others, 2000; Uhl & others, 2004a; Uhl, Sigusch & Jandt, 2004b; Bennett & Watts, 2004). This study did not evaluate the wavelength of the LCUs. However, it was verified that both Ultrablue LED LCUs presented low power output of 130 mW/cm^2 (15% of QTH intensity) and LEC 470I LED LCU presented 91 mW/cm^2 (14% of the QTH power output). Thus, the low light intensity presented by both Ultrablue LED LCUs and LEC 470I LED LCU could explain these results. Other studies have also shown that LED LCUs with relatively low irradiance sold on the market may result in insufficiently cured composites and, therefore, inferior mechanical properties of the restorations (Hofmann & others, 2002; Uhl & others, 2002; Mills, Uhl & Jandt, 2002).

An alternative way to improve LED-based LCUs curing effectiveness could be to increase the light exposure time during polymerization (Leonard & others, 2002; Kurachi & others, 2001). However, this could result in a longer clinical restorative procedure. Thus, the use of argon laser and plasma arc based LCUs could be suggested, since these devices are capable of curing dental composites in a shorter period of time (Vargas, Cobb & Schmidt, 1998; Hasegawa & others, 2001). Recently, new LEDs, so-called second generation LEDs, were

Table 2: *Mean Hardness Values and Standard Deviations (SD) at Top and Bottom Surfaces for Each Resin Composite and LCUs Evaluated by the Tukey's Test (p<0.05)*

Resin Composites	LCUs	Top Surface		Bottom Surface		Surface Hardness Ratio
		Mean	SD	Mean	SD	
Microhybrid	FreeLight 1	57.1 ^{AB,a}	3.7	53.1 ^{AB,b}	6.5	0.92
	Ultrablue II Corded	51.2 ^{BC,a}		46.9 ^{C,b}	5.4	0.91
	Ultrablue III Cordless	53.2 ^{BC,a}	4.8	48.5 ^{BC,b}	2.5	0.91
	LEC 470 I	49.7 ^{C,a}	11.0	40.4 ^{D,b}	11.9	0.81
	Optilux 501	60.1 ^{A,a}	6.5	57.7 ^{A,a}	4.2	0.95
Microfill	FreeLight 1	16.4 ^{B,a}	1.5	14.9 ^{A,a}	1.2	0.90
	Ultrablue II Corded	14.9 ^{B,a}	0.8	12.8 ^{A,a}	1.2	0.85
	Ultrablue III Cordless	14.9 ^{B,a}	1.4	13.4 ^{A,a}	1.2	0.89
	LEC 470 I	14.1 ^{B,a}	1.6	12.3 ^{A,a}	1.7	0.87
	Optilux 501	25.1 ^{A,a}	2.4	16.4 ^{A,b}	1.1	0.65

Statistically significant differences are expressed by upper case letters in columns and by lower case letters in rows (p<0.05). n=10 specimens per experimental condition.

introduced to the market and will be used in several LED LCUs. These single InGaN LEDs consist of multiple emitters on the same substrate. The new prototype achieved a higher irradiance and depth of cure than the QTH LCU, which indicates that the new LED may lead to enhanced mechanical properties of composites (Uhl & others, 2004). On the other hand, this might lead to higher temperatures within the restoration (Uhl, Mills & Jandt, 2003), because of its high power output.

Comparing the microhardness performed on the top and bottom surfaces, the microhybrid resin showed a hardness reduction on the opposite surface from the light exposure (bottom surface) for all LED LCUs evaluated (Table 2). These results indicate that the curing degree decreases as a function of depth, as demonstrated previously by other studies (Hansen & Asmussen, 1993; Kurachi & others, 2001). However, when the QTH device was evaluated, no statistically significant difference was found between these two surfaces. According to Leonard and others (2001), the minimum required irradiance of a halogen lamp for proper polymerization of a 2.0 mm-thick microhybrid resin composite is 300 mW/cm^2. However, the halogen light Optilux 501 used in this study has a high irradiance level (866 mW/cm^2), which could explain the previous results.

The QTH resulted in the greatest hardness mean value (25.17) on the top surfaces of the microfill resin group. This could be due to the high intensity of the halogen light emitted on this surface (866 mW/cm^2). However, when evaluating the bottom surfaces, no statistically significant differences were found among the groups cured with LED and QTH. It is thought that microfill resin based composites are more difficult to cure, because their small filler particles cause light to scatter, decreasing the effectiveness of the curing light (Nomoto, Uchida & Hirasawa, 1994; Kawaguchi, Fukushima & Miyazaki, 1994; Leonard & others, 2001). This could explain the poor results for hardness at the bottom surfaces of the microfill resin, regardless of the light source.

Yap and Severante (2001) and Yap, Soh and Siow (2002) suggested a bottom/top surface hardness ratio to verify the efficiency of cure in deep surfaces when compared to the surfaces located closest to the light sources. Theoretically, the hardness of the cured resin composite at the bottom surface has to present 80% (0.80) of the hardness at the top surface. In this study, for the microhybrid resin composite, it was observed that all LCUs tested reached ratios of more than 0.80; however, for the microfill resin composite, the surface hardness ratio bottom/top presented a value of 0.65, which was lower than that considered the minimum, when the QTH unit was used (Table 2). This lower value can suggest that this QTH device was not efficient, though it is important to observe the highest microhardness values were observed at the top surface for this type of unit, which contributed to reducing this ratio.

According to the methodology proposed by this investigation, it can be concluded that the microhardness of resin composites vary according to the type of resin (mycrohybrid and microfill), the light curing unit and the depth at which hardness is measured. However, further investigation is necessary to verify the performance of LED based LCUs in cavities deeper than 2.0 mm, such as clinical in-depth Class II cavities.

CONCLUSIONS

Within the limitations of this study, the authors conclude:

1. For the microhybrid composite, Freelight 1 was the only LED light providing equivalent hardness values as those from a conventional QTH light.
2. For the microfill composite, all LED lights provided similar performance, while the QTH provided the hardest surfaces.
3. The microhardness of resin composites vary according to the type of resin (mycrohybrid and microfill), the light curing unit and the depth at which hardness is measured.

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Two-year Color Changes of Light-cured Composites: Influence of Different Light-curing Units

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Clinical Relevance

Curing composites using conventional halogen curing units, high intensity halogen curing units and LED units might reduce color changes of the material when compared to PAC units.

SUMMARY

This study determined color changes in a composite cured with various types of curing units after two years. A hybrid (Clearfil AP-X) composite was cured with a conventional halogen, a high intensity halogen, a plasma arc and a light emitting diode unit. The specimens were stored in light-proof boxes after the curing procedure to avoid further exposure to light and stored in 37°C in 100% humidity. Colorimetric values of the specimens immediately after curing and after two years were measured using a colorimeter. The CIE 1976 *L*a*b* color system was used to determine color differences. Differences from baseline were calculated as ΔE^*_{ab}. Data were analyzed with two-way analysis of variance ($p<0.05$). The ΔE^*_{ab} values varied significantly, depending on the curing unit used. The specimens cured with a plasma arc curing unit induced significantly higher color changes than any other specimen and the color differences were also visually appreciable by the non-skilled operator (ΔE^*_{ab} >2.5). The specimens cured with a high intensity halogen curing unit produced the lowest color change; however, there were no statistically significant differences among the color changes of specimens cured with conventional halogen, high intensity halogen and the light emitting diode unit, and the color changes were not clinically relevant (ΔE^*_{ab} <2.5). The results of this study suggest that composite materials undergo measurable changes due to curing unit exposure. The specimens cured with a plasma arc light showed the highest color changes as compared to specimens cured with other curing units.

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INTRODUCTION

The appearance of composite restorations changes over time. External stain accumulation, marginal leakage, secondary caries and internal discoloration can make a restoration visually unacceptable. In addition to secondary caries, discoloration is one of the main reasons for the removal of composite fillings (Mjör & Toffenetti, 1992). Water accumulation, changes in chemical compounds necessary for photo-polymerization, photo-oxidation and other processes have been thought to be

responsible for internal color changes (Seghi, Gritz & Kim, 1990; Ferracane, Moser & Greener, 1985).

Until recently, light emitted from a halogen light bulb has been used to cure composites. These types of curing units usually operate at light intensities between 400 and 800 mW/cm^2 and cure composite filling material within 40 seconds. Halogen bulbs produce light when electric energy heats a small tungsten filament to high temperatures. Despite their common use in dentistry, halogen bulbs have several disadvantages. The basic principle of light conversion by this technique is claimed to be inefficient, as the light power output is less than 1% of the consumed electrical power. In addition, the halogen bulbs have a limited effective lifetime of approximately 100 hours due to degradation of bulb components from the high amount of heat generated (Ferracane & others, 1985).

The plasma arc curing (PAC) unit is designed for the high-intensity curing of direct composite restorations and may be a time saving alternative to conventional halogen lights (Stahl & others, 2000). As stated by the manufacturer, highly filled and pigmented composite materials can be cured in 10 seconds, and more transparent materials can be cured within five seconds (Usumez, Buyukyılmaz & Karaman, 2003). Solid-state light emitting diode (LED) technology was proposed in 1995 for the polymerization of light-cured dental materials to overcome the shortcomings of halogen visible light-curing units (Oesterle, Newman & Shellhart, 2001). LEDs use junctions of doped semiconductors to generate light instead of the hot filaments used in halogen bulbs (Nakamura, Mukai & Senoh, 1994). LEDs have a lifetime of more than 10,000 hours and undergo little degradation of output over this time. LEDs require no filters to produce blue light, are resistant to shock and vibration and take little power to operate (Oesterle & others, 2001). LEDs' longer life span and more consistent light output compared with halogen bulb technology show promise for dental applications (Dunn & Taloumis, 2002).

Depth of cure is a very important concept in direct composite application (Rasetto, Driscoll & von Fraunhofer, 2001). However, adequate curing is a crucial factor in obtaining optimal physical properties and achieving satisfying clinical performance of a composite material. Inadequate curing diminishes the physical properties of composite; changes in strength, stiffness, water sorption and color stability might be expected (Pires & others, 1993).

This study investigated color changes in a composite cured with selected curing units: conventional halogen (20 and 40 seconds), high intensity halogen (20 and 40 seconds), PAC unit (5 and 10 seconds) and LED unit (20 and 40 seconds) after two years. The hypothesis tested assumed that there is no difference in color change in composites cured with these four curing units after two years.

METHODS AND MATERIALS

The composite used in this study was Clearfil AP-X (color A3) (Kuraray, Osaka, Japan) hybrid light-cured composite. A 10 mm-diameter hole was made in a 2 mm-thick Teflon plate and was filled with composite. The Teflon plate was sandwiched between 1 mm-thick glass plates, then placed on a white sheet. The glass plates flattened the composite and protected it from the oxygen inhibition zone.

The composite was exposed to light through the upper plate with a conventional halogen unit (Hilux 550, Express Dental Products, Canada) for 20 or 40 seconds, 20 or 40 seconds with a high intensity halogen unit (Optilux 501, Kerr, USA), 5 or 10 seconds with a PAC unit (Power PAC, ADT, USA) and 20 or 40 seconds with an LED unit (Elipar FreeLight, 3M ESPE, St Paul, MN, USA) (Table 1). The plates were reversed, so that the lower side of the plate was on the top. The composite was once again irradiated for the same irradiation times. Five specimens each were prepared for the eight groups. No polishing techniques were used to avoid modification of the surfaces, which could have influenced the results. The specimens were stored in light-proof boxes after the curing procedure and initial color measurement to avoid further exposure to light and stored in 37°C in 100% humidity.

The colorimetric values of the specimens immediately after curing and after two years were measured with the Colorimeter (Minolta Chroma meter, CR-300, Minolta Inc, Osaka, Japan). Color measurement was

Table 1: *Visible Light Curing Units Studied*

Brand	Type of Unit	Output of Light Tip (mW/cm^2)	Diameter of the Tip (mm)	Applied Polymerization Time(s)	Manufacturer
Hilux	Conventional Halogen	450	10	20, 40	Express Dental Products, Toronto, Canada
Optilux 501	High Intensity Halogen	810	8	20, 40	Kerr, Danbury, CT, USA
Power PAC	PAC	1190	6.5	5, 10	ADT, San Carlos, CA, USA
Elipar Freelight	LED	380	8	20, 40	3M ESPE, St Paul, MN, USA

Table 2: *Chromatical Values Immediately After Curing (Value 1) and After Two Years (Value 2)*

Curing Unit	L* Value 1	L* Value 2	a* Value 1	a* Value 2	b* Value 1	b* Value 2	ΔE
Conventional Halogen							
20 seconds	64.30 (0.71)	62.73 (0.33)	1.66 (0.22)	2.35 (0.46)	20.17 (0.65)	21.71 (0.83)	2.37 (0.49)
40 seconds	62.49 (0.44)	61.68 (0.31)	1.59 (0.09)	1.73 (0.16)	17.62 (0.55)	18.57 (1.21)	1.69 (1.14)
High Intensity Halogen							
20 seconds	63.04 (0.29)	62.13 (0.21)	1.84 (0.10)	2.16 (0.17)	16.86 (0.50)	17.98 (0.86)	1.63 (0.70)
40 seconds	62.49 (0.44)	62.14 (0.20)	1.46 (0.49)	1.51 (0.72)	17.38 (1.72)	17.79 (1.55)	0.86 (0.53)
PAC							
5 seconds	65.61 (0.88)	63.35 (0.48)	1.91 (0.19)	3.44 (0.40)	20.45 (0.29)	22.35 (0.82)	3.44 (1.15)
10 seconds	64.94 (0.55)	62.51 (0.43)	1.48 (0.24)	2.51 (0.25)	19.41 (0.64)	21.83 (1.12)	3.73 (0.86)
LED							
20 seconds	64.24 (0.15)	62.57 (0.22)	1.52 (0.19)	2.37 (0.11)	18.65 (0.95)	19.97 (0.63)	2.38 (0.43)
40 seconds	63.76 (0.59)	62.42 (0.41)	1.77 (0.24)	2.11 (0.41)	17.76 (0.46)	18.34 (0.51)	1.69 (0.80)

Table 3: *Pooled* ΔE^*_{ab} *Values of Composite Cured with Various Curing Units*

	Mean ± SD	Tukey Grouping*
Conventional Halogen	2.0 ± 0.9	A
High Intensity Halogen	1.2 ± 0.7	A
PAC	3.6 ± 0.9	B
LED	2.0 ± 0.7	A

Mean ± SD: standard deviation.
**Groups with different letters are statistically significantly different.*

performed on consecutive tests on central parts of the specimens. The specimens were positioned at the same place on different occasions to assure consistency of the repeated measurements.

All specimens were chromatically measured three times and the average values were calculated. Then, each of five specimens of the same group was averaged and the color difference obtained from the average color parameters. The CIE *Lab* color system was used to determine color differences (Inokoshi & others, 1996). The total color difference, ΔE^*_{ab} between two color stimuli, each given in terms of L^*,a^*,b^*, is calculated from (Commission Internationale de L'Eclairage, 1986):

$$\Delta E^*_{ab}=[(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2]^{1/2}$$

The literature is not in agreement with respect to the limitations of the human eye in terms of appreciating differences in color, considering that this limit differs from individual to individual (as it is a combination of eye characteristics and skill from the operator) (Douglas & Brewer, 1998; Kuehni & Marcus, 1979; Ruyter, Nilner & Möller, 1987; Johnston & Kao, 1989). The ΔE^*_{ab} value of 2.5 presented a borderline value recognizable by all people in a color test (Hosoya, 1999).

The data were entered into a spreadsheet (Excel, Version 4.0, Microsoft, Seattle, WA, USA) for calculation of the descriptive statistics. The obtained data were analyzed by two-way ANOVA, then Tukey HSD tests (SPSS/PC, Vers 10.0; SPSS, Chicago, IL, USA) for comparisons among groups at the 0.05 level of significance.

RESULTS

Colorimetric values of the composite cured with four test curing units immediately after curing and two years are given in Table 2.

Different curing time data with the same curing unit was pooled, as no statistically significant effect of curing time was observed on colorimetric values with ANOVA (p=.42). Pooled ΔE^*_{ab} values of the composite cured with the four test curing units are given in Table 3.

The ΔE^*_{ab} values varied significantly depending on the curing unit used (ANOVA, $p<0.001$). The specimens cured with the PAC unit (3.6±0.9) induced significantly higher color changes than any other specimens (Tukey, $p<0.05$). The specimens cured with the high intensity halogen curing unit produced the lowest color change (1.2±0.7); however, there were no statistically significant differences among the color changes of specimens cured with conventional halogen (2.0±0.9), high intensity halogen and the LED unit (2.0±0.7) (Tukey, $p>0.05$).

For PAC unit cured specimens, color differences were also visually appreciable for the non-skilled operator ($\Delta E^*_{ab}>2.5$). The color changes of specimens cured with other curing units (conventional halogen, high intensity halogen and LED unit) were not clinically relevant ($\Delta E^*_{ab}< 2.5$).

In all curing time groups, the L^* values showed a tendency to decrease as time elapsed and the colors of all specimens darkened. The a^* values showed the tendency to increase as time elapsed, which implies an

increase in red color factor. The *b** values showed a tendency to increase as time elapsed, which implies an increase in yellow color factor.

DISCUSSION

This *in vitro* study measured the color changes of specimens cured with each of four commercially available curing units after two years. The results of this study do not support the hypothesis that there is no difference in color change in a composite cured with these four curing units. There were significant differences in color changes within groups. Under the conditions of this study, the highest color changes were recorded from the specimens cured with the PAC unit. There were no statistically significant differences among the other specimens cured with other curing units.

Several laboratory tests have been proposed in order to simulate and accelerate the discoloration that occurs under clinical conditions over a relatively long time (Powers, Fan & Raptis, 1980; Davis, Friedl & Powers, 1994). For this study, the samples were stored for two years in 37°C in 100% humidity, then measured for color determination. No polishing techniques were used, because the oxygen inhibited zone might exist and positively influence resin discoloration.

A white color plate, used for the background color in this study, was specially made to substitute for the lining material, so that the chromatical values backed by a white colored plate could be considered as the colors of resin composites filled on the lining material in the oral cavity.

A study by Marais and others (1997) has suggested that power density (irradiance) does not have an effect on the conversion of composite resin at depths beyond 2 mm, and because of this, 2-mm thickness was used in this study. The samples were cured from both sides, effectively reducing the thickness of resin being cured to 1 mm to achieve maximum conversion.

Previous investigators reported that the color changes of composites were caused by the following factors: the chemical activator (Ferracane & others, 1985), resin initiator and inhibitor (Peutzfeldt & Asmussen, 1990), activator progress (Fukuda, 1986), polymer quality, Bis-GMA of monomer, type of filler (Peutzfeldt & Asmussen, 1990), oxidation of unreacted carbon-carbon double bonds (Ferracane & others, 1985) and heat and water (Morikawa, Yonekawa & Shino, 1989).

A high degree of cure provides color stability as well as hardness and strength to the material (Peutzfeldt, Sahafi & Asmussen, 2000). Thus, a reduction in the remaining double bonds to the lowest possible level is considered a desirable feature of a curing system. The degree of cure of a given composite is influenced by energy density (Peutzfeldt & others, 2000). For composites, a light intensity of more than 400mW/cm^2 is generally recommended (Jung & others, 2001).

It is conceivable that oxidation reactions of unreacted C=C double bonds produce colored peroxide products (Ferracane & others, 1985). In this investigation, the initiation of cure by different curing units was evaluated by recording the color changes over two years.

Various types of light sources were used in this study. One type is LED. LEDs are being aggressively marketed; however, independent research has not yet verified the potential of this technology to replace halogen visible light-curing units (Dunn & Taloumis, 2002). Optimal cure times for LEDs and their ability to cure all resins are still unknown (Clinical Research Associates, 2001). A number of studies have confirmed the potential of LED technology for the light activation of dental materials. Fujibayashi, Ishimaru and Kohno (1996) detected no differences in composite hardness and depth of cure between an LED and a halogen light and obtained a deeper cure of 470 nm wavelength with the LED compared to the halogen unit at 10, 20, 40 and 60 seconds (Fujibayashi, Ishimaru & Kohno, 1996; Fujibayashi & others, 1998). Mills compared a light source containing 25 LEDs with a halogen unit adjusted to an irradiance of 300 mW/cm^2 (Mills, Jandt & Ashworth, 1999). The LED unit cured composite specimens to a significantly greater depth than the halogen unit when tested at 40 and 60 seconds (Mills & others, 1999). The LED unit used in this study had 19 LEDs.

Mean power densities of the light curing units used in this study are presented in Table 1. The halogen-based light-curing unit had a higher mean power density than the LED-curing unit. However, color changes of specimens cured with the LED were not statistically different from the halogen-based curing light. Fujibayashi and others (1998) demonstrated that the quality of light curing is not exclusively due to light intensity: the narrow absorption peak of the initiator system must also be taken into account. This makes the emitted spectrum an important determinant of a curing light's performance. The absorption curve of camphorquinone extends between 360 and 520 nm, with its maximum at 465 nm. It has been shown that, within this range, the optimal emission bandwidth of the light source lies between 450 and 490 nm (Nomoto, 1997). In conventional curing devices, a major portion of the photons is emitted outside the optimal spectrum range for light curing. These photons cannot be absorbed by camphorquinone or only with reduced probability. In contrast, 95% of the emission spectrum of blue LEDs is situated between 440 and 500 nm. Furthermore, the emission maximum of the blue LEDs used in this study is approximately 465 nm, which is almost identical to the absorption peak of camphorquinone. At clinically realistic irradiances, mod-

estly greater depth of cure was found when composites were cured with an LED lamp compared to using a halogen lamp, despite the former having a measured output of approximately 70% of the latter (276 versus 388 mW/cm^2 when measured between 410 and 500 nm) (Mills & others, 1999). Knezevic and others (2001) demonstrated only a minor increase in conversion degree values when 66 times stronger halogen curing units were compared to an LED with a minimal intensity of 12 mW/cm^2. This finding also supports the importance of considering the emission spectra of curing lamps relative to the absorption spectrum of camphorquinone when assessing the quality of light curing.

The PAC unit has filters that narrow the spectrum of visible light to a band centered on the 470-nm wavelength for activating part of a dual catalyst of the camphorquinone (Sfondrini & others, 2001). A high-energy, high-pressure ionized gas in the presence an electrical current is used to create a curing unit strong enough to increase the curing rate of composites and resin-modified glass ionomers (Sfondrini & others, 2001). It was pointed out that both universal hardness and depth of cure after conventional polymerization were significantly higher than PAC (Jung & others, 2001). Caughman and Rueggeberg (2002) found that three seconds of light activation with a PAC is insufficient to produce maximum polymerization. In this investigation, statistically significant differences in ΔE^*_{ab} values were observed between specimens cured with other curing units and a PAC unit. This implies that one gets a lower degree of conversion when utilizing a PAC unit, which may indicate that the rather narrow band of wavelengths emitted by PAC curing units is outside the range of maximum sensitivity of the camphorquinone of this composite material or the curing time is not sufficient.

It is generally thought that, according to the time elapsed, the color of composite changes to a dark color and yellowing occurs (Hosoya, 1999). In this study, specimens cured with different curing units revealed a color shift to darker yellow (positive Δb^*) and to darker red (positive Δa^*) after two years of storage in water. All specimens became darker during the investigation (negative ΔL^*).

In this study, a slightly chalky, white discoloration on the composite disks was observed by the naked eye, especially in the PAC unit cured group. In this study, composite specimens were stored at 37°C in 100% humidity and color changes were measured. In the oral cavity, the composite surface roughly changes by mastication and the other factors, which are included in food deposited on the rough composite surface. In addition, the influence of heat caused by hot drinks or hot food must be considered (Um & Ruyter, 1991). It is possible that composite discoloration in the oral cavity might be greater than the results of this study.

CONCLUSIONS

Within the limitations of this *in vitro* study, it was concluded that the composite specimens cured with the PAC unit showed significantly higher color changes compared to the conventional halogen curing unit, high intensity halogen curing unit and LED unit.

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Effects of pH on the Microhardness of Resin-based Restorative Materials

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Clinical Relevance

In patients who consume large quantities of acidic beverages and food substances, compomers and giomers should be used with caution, especially in "stress-bearing" areas.

SUMMARY

This study determined the effect of pH on the microhardness of commonly used resin-based restorative materials which included a resin composite (Esthet-X, Dentsply), a new generation compomer (Dyract Extra, Dentsply) and a giomer (Beautifil, Shofu). Fifty-four specimens (3-mm wide x 3-mm long x 2-mm deep) were made for each material. The specimens were divided into six equal groups and conditioned in the following solutions at 37°C for one week: 0.3% citric acid at pH 2.5, sodium hydroxide—buffered citric acid at pH 3, 4, 5, 6 and 7. After conditioning, the specimens were subjected to hardness testing using a digital microhardness tester (load 500gf; dwell time 15 seconds). Data was analyzed using one-way ANOVA and Scheffe's test at a significance level of 0.05. The effects of pH on the microhardness of resin-based restoratives were material dependent. The compomer and giomer materials were more affected by acids of low pH than the composite material that was evaluated.

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INTRODUCTION

The role of acids in causing dental erosion, where there is irreversible loss of dental hard tissue by a chemical process without the involvement of microorganisms, has long been established (Eccles, 1979). The acids may be derived from intrinsic sources in patients with recurrent vomiting due to anorexia, bulimia or gastroesophageal reflux (Meurman & ten Cate, 1996) or extrinsic sources derived from the environment, medications, lifestyle and diet (Zero, 1996). It was reported that in 2002, Americans consumed nearly 53 gallons of soft drinks per person per year (National Soft Drink Association, 2002) and there is an increasing trend towards increased consumption of fruit drinks in children (Park & others, 2002). These drinks are acidic and their over-consumption has been linked to tooth surface loss from erosion (Milosevic, Lennon & Fear, 1997). Acidulants, which are added to soft drinks for a pleasant taste, act as a preservative. Some soft drinks contain phosphoric and citric acids, while others use malic or tartaric acids.

Just like tooth surfaces, dental restorations may also be adversely affected by exposure to either intrinsic or extrinsic acids. Low acidic pH may cause erosion of materials and leaching of the principle matrix forming substances (Yip, Peng & Smales, 2001; Abu-Bakr & others, 2000; Lee & others, 1998). Abu-Bakr and others (2000) found SEM changes in surface texture when materials are immersed in low pH soft drinks (orange juice and cola). SEM revealed that the surface of resin composite was slightly affected, whereas the surfaces of the compomer and RM-GIC showed a rough surface with voids and protruding glass particles. They also found that compomers are softer than composites but harder than resin-modified glass ionomer cements when immersed in acidic media. The exact clinical significance of the microhardness of materials, though, is difficult to define. However, it has been suggested that materials with lower surface hardness suffer more abrasive wear, and a recent *in vitro* study has proven a strong relationship between hardness and wear of materials (Say & others, 2003). The effects of acids on surface microhardness of the new generation compomer and the pre-reacted glass ionomer composite (giomer), which is claimed to have better physical properties, have not been reported. This study determined the effect of pH on the microhardness of resin-based restorative materials.

METHODS AND MATERIALS

The resin-based restorative materials used in this study are listed in Table 1. These include a mini-filled composite resin (Esthet-X), a compomer (Dyract Extra) and a giomer (Beautifil). The materials were placed in the rectangular recesses (3-mm long x 3-mm wide x 2-mm deep) of customized acrylic molds and covered with acetate strips (Hawe Neos Dental, Bioggio, Switzerland). A glass slide was placed over the mold and pressure was applied to extrude excess material. All the materials used were shade A2 and light polymerized for 40 seconds through the glass slide using a Polylux 2 curing light (KaVo Dental, 88400 Biberach, Germany). The mean light intensity of the light source (410 ± 5 mW/cm^2) was determined with a radiometer (Cure Rite, EFOS Inc, Ontario, Canada) before starting the experiment. Immediately after polymerization, the acetate strips were discarded and the specimens stored in deionized distilled water at pH 7 for two weeks at 37˚C. Fifty-four specimens were made for each material.

The specimens were the randomly divided into six groups of nine specimens and conditioned in solutions of different pH levels (pH 2.5, 3, 4, 5, 6 & 7) at 37˚C for one week; 0.3% citric acid, which had a pH of 2.5, was used to produce the different solutions for conditioning the materials. The pH of the citric acid was adjusted to 3, 4, 5, 6 and 7 by titration with a 1M sodium hydroxide solution. A sample size of 9 was adopted based on previous studies (Yap, Low & Ong, 2000; Yap & Mok, 2002). The pH of the solutions was measured using a pH meter (MP 220, Mettler-Toledo GmbH, CH-8603 Schwerzenbach, Germany). After one week, the samples were rinsed lightly in water and gently dabbed dry with absorbent paper before being subjected to hardness testing.

The specimens were positioned centrally beneath the indenter of a digital microhardness tester (FM7,

Table 1: *Technical Profiles of the Restorative Materials Evaluated in This Study*

Material	Category	Resins	Fillers	Filler Size (μm)	Filler Content (% volume)	Batch #
Esthet-X Dentsply–De Trey, Konstanz, Germany	Composite	BisGMA – adduct BisEMA TEGDMA	Bariumaluminoflu-oroborosilicate glass Nano-sized silicon dioxide	0.6 – 0.8 0.02	60	020607
Dyract Extra Dentsply–De Trey, Konstanz, Germany	Compomer	BisEMA UDMA TCB TMPTMA TEGDMA	Strontium-alumino-sodium-fluoro-phosphor-silicate glass	0.8	45	0303000639
Beautifil Shofu Inc, Kyoto, Japan	Giomer	BisGMA TEGDMA	S-PRG based on fluoroboro-aluminosilicate glass	0.01 - 5	66	110262

BisGMA = Bisphenol-A-glycidyl methacrylate

TEGDMA = Triethylene glycol dimethacrylate

BisGMA adduct = Adduct of 2,2-Bis[4-2-hydroxy-3methacryloyloxypropoxy-phenyl]propane with hexamethylene diisocyanate

BisEMA = Ethoxylated bisphenol-A-glycidyl methacrylate

UDMA = Urethane dimethacrylate

TCB = Carboxylic acid modified dimethacrylate

TMPTMA = Trimethylolpropane trimethacrylate

Future-Tech Corp, Tokyo, Japan). A 500g load was applied through the indenter with a dwell time of 15 seconds. The Knoop Hardness Number (KHN) corresponding to each indentation was computed using the formula KHN=14.2 (F/d^2), where F is the test load in kgf and d is the longer diagonal length of an indentation in millimeters. All statistical analysis was carried out at a significance level of 0.05. One-way ANOVA and Scheffe's post-hoc tests were used for inter-solution and inter-material comparisons. The percentage change in microhardness of a material was determined using the formula DH/H x 100%, where DH is the difference in mean KHN between the test and control pH (pH 7) and H is the mean KHN at control pH 7.

RESULTS

The mean microhardness values (KHN) for the different materials at various pH levels are shown in Table 2. The results of statistical analysis are reflected in Table 3. The percentage change in microhardness of the materials is reflected in Table 4 and Figure 1.

The effect of pH on microhardness was material dependent. Microhardness values ranged from 15.19 to 40.94 KHN and 47.02 to 53.48 KHN for pH 2.5 and 7, respectively. Significant differences in microhardness values were observed between pH levels. Specimens of Esthet-X conditioned in acids of pH 2.5 and 3 were significantly softer than those conditioned in pH 4, 5, 6 and 7. Dyract Extra specimens conditioned at pH 5 were significantly harder than that conditioned in pH 2.5, 3 and 4 but significantly softer than those stored at pH 6 and 7. Beautifil specimens conditioned at pH 4 and 5 were significantly harder than at pH 2.5 but significantly softer than pH 7. The material also had significantly higher microhardness values when conditioned in acids of pH 6 and 7 than in pH 2.5 and 3.

The materials exhibited different degrees of softening at different pH levels. All the materials had decreased microhardness when conditioned in acids compared to control pH 7. Esthet-X showed a small percentage decrease in microhardness of 0.7–1.7% at pH 6 to pH 4 and a greater degradation of 20.6% and 22.1% at pH 3 and pH 2.5, respectively. Dyract Extra showed

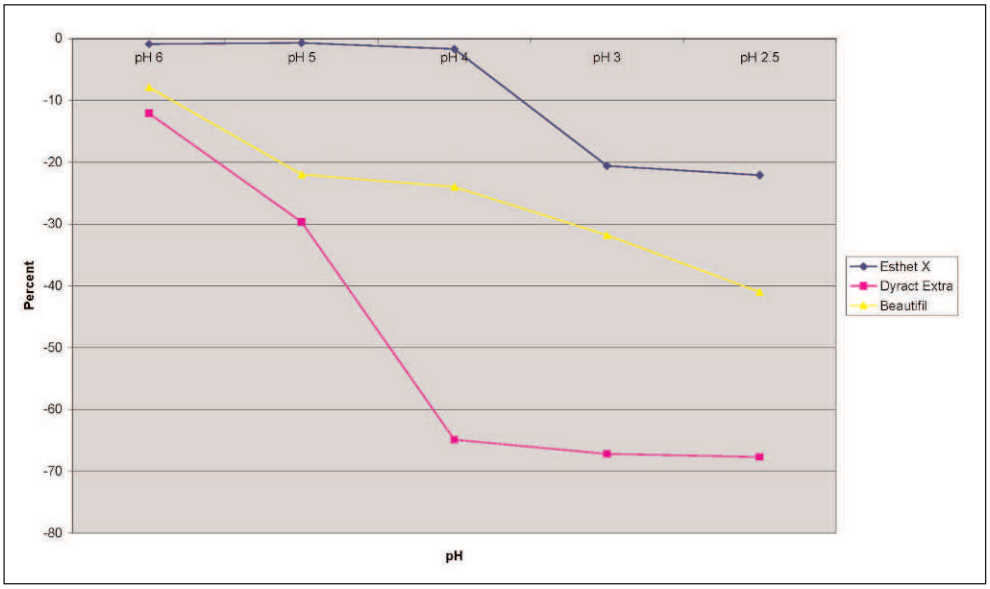

Figure 1. *Percentage of change in microhardness at different pH levels.*

Table 2: *Mean Hardness at Different pH (standard deviation in parentheses)*

pH	Esthet-X	Dyract Extra	Beautifil
pH 2.5	40.94 (2.08)	15.19 (3.36)	31.56 (5.15)
pH 3	41.77 (1.95)	15.42 (1.81)	36.47 (6.46)
pH 4	51.70 (1.06)	16.52 (4.40)	40.66 (7.75)
pH 5	52.24 (2.10)	33.08 (2.31)	41.71 (2.66)
pH 6	52.10 (1.73)	41.33 (2.21)	49.24 (3.59)
pH 7	52.58 (1.52)	47.02 (1.17)	53.48 (1.50)

Table 3: *Results of Statistical Analysis*

	Comparison Between pH
Materials	**Differences**
Esthet-X	pH 2.5, pH 3< pH 4, pH 5, pH 6, pH 7
Dyract Extra	pH 2.5, pH 3, pH 4 < pH 5 < pH 6 < pH 7
Beautifil	pH 2.5 < pH4, pH 5 < pH 7
	pH2.5, pH 3 < pH 6, pH 7

< indicates statistically significant difference. Results of one-way ANOVA/Scheffe's test (p<0.05)

Table 4: *Percentage Change in Microhardness (negative sign denotes reduction in microhardness compared to control)*

pH	Esthet-X	Dyract Extra	Beautifil
pH 2.5	-22.1%	-67.7%	-41.0%
pH 3	-20.6%	-67.2%	-31.8%
pH 4	-1.7%	-64.9%	-24.0%
pH 5	-0.7%	-29.7%	-22.0%
pH 6	-0.9%	-12.1%	-7.9%

a marked decrease in microhardness when compared to the control. This ranges from 12.1% to 67.7% at pH 6 and pH 2.5, respectively. The giomer Beautifil behaved somewhere between the composite and compomer, with the decrease in microhardness values ranging between 7.9% and 41% at pH 6 and pH 2.5, respectively.

DISCUSSION

The specimens were stored in deionized distilled water at pH 7 for two weeks at 37˚C to allow for post-irradiation hardening of the composites (Wan, Yap & Hastings, 1999; Yap, 1997; Leung, Adishian & Fan, 1985) and stabilization of the acid-base reaction between fluorosilicate glass and ionized polyacrylic acid in compomers (Eliades, Kakaboura & Palaghias, 1998). Citric acid was chosen as the erosive medium, as it is one of the most common acids found in fruit juices and drinks and is often added as a preservative to drinks and foodstuff. Citric acid is also commonly used in *in vitro* studies (Barbour & others, 2003; Eisenburger, Addy & Rossbach, 2003; Vanuspong, Eisenburger & Addy, 2002). The different pH levels investigated reflect the levels of dietary acid that the restorative materials would be subjected to intra-orally, for example, apples pH 3.3–4, grapefruit pH 3–3.75, lemon juice pH 2–2.6 (US Food and Drug Administration, 2003). Compomers or polyacid-modified composites are materials that contain either or both of the essential components of glass ionomer cement but at levels insufficient to promote the acid-base cure reaction in the dark (McLean, Nicholson & Wilson, 1994). Dyract Extra, a new generation compomer, which is claimed to have advanced physical properties suitable for use in both anterior and posterior restorations, was used in this study. The Giomers (Beautifil) are a new class of glass ionomer containing restorative material that contains both essential components of glass ionomer cements. They cannot be classified as compomers, as the acid-base reaction has already occurred and, therefore, a new term "pre-reacted glass ionomer (PRG) composite" is used to describe the material.

The effect of pH on microhardness was material dependent, with the new generation compomer (Dyract Extra) exhibiting lower microhardness values than the composite and giomer at various pH levels. The microhardness of a giomer (Beautifil) was found to closely approximate that of resin composites at pH 7 and 6 but degraded in acids at levels between the compomer and composite. For all the materials tested, the highest microhardness value was observed when the materials were conditioned in a medium with neutral pH 7, which served as the control. It appears that all the materials degraded after conditioning in acids. The critical pH at which degradation occurs varies among the three types of resin-based restorations. The composite Esthet X showed a high percentage reduction in microhardness in acids at pH 3; whereas, the compomer (Dyract Extra) and giomer (Beautifil) exhibited marked degradation from pH 5. The percentage change in hardness from pH 5 to 2.5 was gradual for the giomer (Figure 1). For the compomer, a marked decrease in hardness was observed for pH 5 and 4. The change in hardness was minimal from pH 4 to 2.5.

All the materials tested can be considered biphasic, with one phase embedded in the other. Previous studies have demonstrated that there is a positive correlation between hardness and filler content of resin-based materials; that is, the higher the filler content, the higher the hardness values (Boyer, Chalkley & Chan, 1982; Chung, 1990). Therefore, a material's hardness value will be affected if the bond between the filler and the polymer matrix is broken (Say & others, 2003). Geurtsen, Leyhausen and García-Godoy (1999) suggested that in acidic solutions, compomers disintegrate predominantly due to hydrolytic degradation of the matrix-filler interface, with eventual loss of the filler particles. Another possibility is that the unreacted fluoroaluminosilicate glass ionomer fillers are easily attacked by the conditioning acid, with its resultant loss from the surface matrix. In the giomer, the pre-reacted glass ionomer fillers are better able to resist acid degradation before finally being lost from the matrix. This may be due to the polygel matrix surrounding the glass fillers. Furthermore, since compomers generally contain a more organic matrix than composites, they have increased susceptibility to water absorption, which causes surface disintegration in the aqueous environment (Geurtsen & others, 1999). This is also likely in the case of the giomer, resulting in a decrease in microhardness seen in low pH conditions as compared to the composite material.

The hardness of resin-based materials has also been associated with the degree of polymerization of the matrix (Asmussen, 1982). Resin composites have been reported to reduce surface hardness after being soaked in organic acids due to the softening of Bisphenol-A-glycidyl methacrylate (Bis-GMA)-based polymers (Asmussen, 1984), which could be caused by leaching of the diluent agents such as Triethylene glycol dimethacylate (TEGDMA) (Lee & others, 1998). That study also found that Bis-GMA resins leached less moieties than Bis-GMA/Urethane dimethacylate (UDMA)-based resins when conditioned in organic acids. This explains the better surface microhardness of Esthet-X and Beautifil, as they are both Bis-GMA/TEGDMA based, while Dyract Extra is not.

Hardness is defined as the resistance of a material to indentation (O'Brien, 1997) and is related to the material's strength and rigidity (Anusavice, 1996). Any chemical softening of the restorative materials when subjected to conditioning in acids of low pH would have implications on their clinical durability in stress

bearing areas. The softened material would be lost as a result of *in vivo* wear, thus exposing a fresh surface to further attack (Chadwick & others, 1990). Therefore, there needs to be careful selection of a restorative material in patients with high exposure to extrinsic acids in the form of dietary acids and intrinsic acids for those with a history of gastric reflux or eating disorders.

The result of this study suggests that there is a critical pH unique to individual resin-based materials at which the surface microhardness was significantly affected, which requires further investigation.

CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusions can be made:

- The effect of pH on surface microhardness of resin-based restoratives was material dependent.
- At all pH levels, the ranking of percentage decrease in hardness was as follows: Composite < Giomer < Compomer
- For each material there appears to be a critical pH at which chemical degradation occurs.
- Compomers and giomers should be used with caution in patients who consume large amounts of acid substances.

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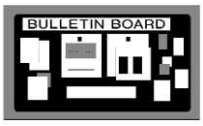
Departments

Classifieds: Faculty Positions

The Ohio State University College of Dentistry
Section of Restorative and Prosthetic Dentistry

The Ohio State University College of Dentistry, Section of Restorative and Prosthetic Dentistry, Columbus, Ohio, is seeking applicants for a full-time tenure track position at the assistant/associate professor level to assume a leadership role in Operative Dentistry teaching. Excellent collaborative research opportunities are available with active Dental Materials and Clinical Research teams. Duties will begin immediately and include pre-clinical and clinical teaching and research. Preference will be given to those with advanced training in operative teaching or general dentistry. Applicants must be eligible for licensure in Ohio. An intramural practice opportunity is available for up to one day per week. Salary and rank are set commensurate with the candidate's qualifications and experience. The Ohio State University is an Equal Opportunity Affirmative Action Employer. Qualified women, minorities, Vietnam-era Veterans, disabled veterans and individuals with disabilities are encouraged to apply. Send supporting documents to Dr Stephen F Rosenstiel, Chair, Section of Restorative and Prosthetic Dentistry, Postle Hall, 305 W 12th Avenue, PO Box 182357, Columbus, Ohio 43218-2357; phone: (614) 292-0941; e-mail: rosenstiel.1@osu.edu

Announcements



Ralph Phillips Student Research Award
2006

Applications are invited for the 2006 Ralph Phillips Student Research Award, sponsored by the Founders Fund of the Academy of Operative Dentistry. The Award is open to students at all levels wishing to undertake novel research relevant to contemporary operative dentistry.

The application should take the form of a protocol outlining the background, aims and hypothesis of the proposed research, the methodology to be employed, the anticipated work schedule and the expected impact of the work on the clinical practice of operative dentistry. The protocol should not exceed three double-spaced, typewritten pages and a budget sheet, including details for payment of the award. The budget for the proposed research may not exceed $6,000.

The recipient of the award will be required to present a table clinic reporting the findings of the research supported by the Award at the 2007 annual meeting of the Academy of Operative Dentistry to be held in Chicago, 22-23 February, 2007. Additional funds not to exceed $1,000 will be made available to offset the cost of attending the meeting.

A research mentor, who is a member of the Academy of Operative Dentistry, should be named and be a co-signatory to the application.

If the research supported by the Award leads to a research report intended for publication, it is expected that the report be submitted in the first instance to *Operative Dentistry*.

Applications for the award must be submitted electronically to Dr Nairn Wilson (nairn.wilson@kcl.ac.uk), Chairman of the Research Committee of the Academy of Operative Dentistry no later than 31st December 2005.

The award recipient will be announced during the 2006 Annual Meeting of the Academy of Operative Dentistry, which will be held in Chicago, 23-24 February, 2006.

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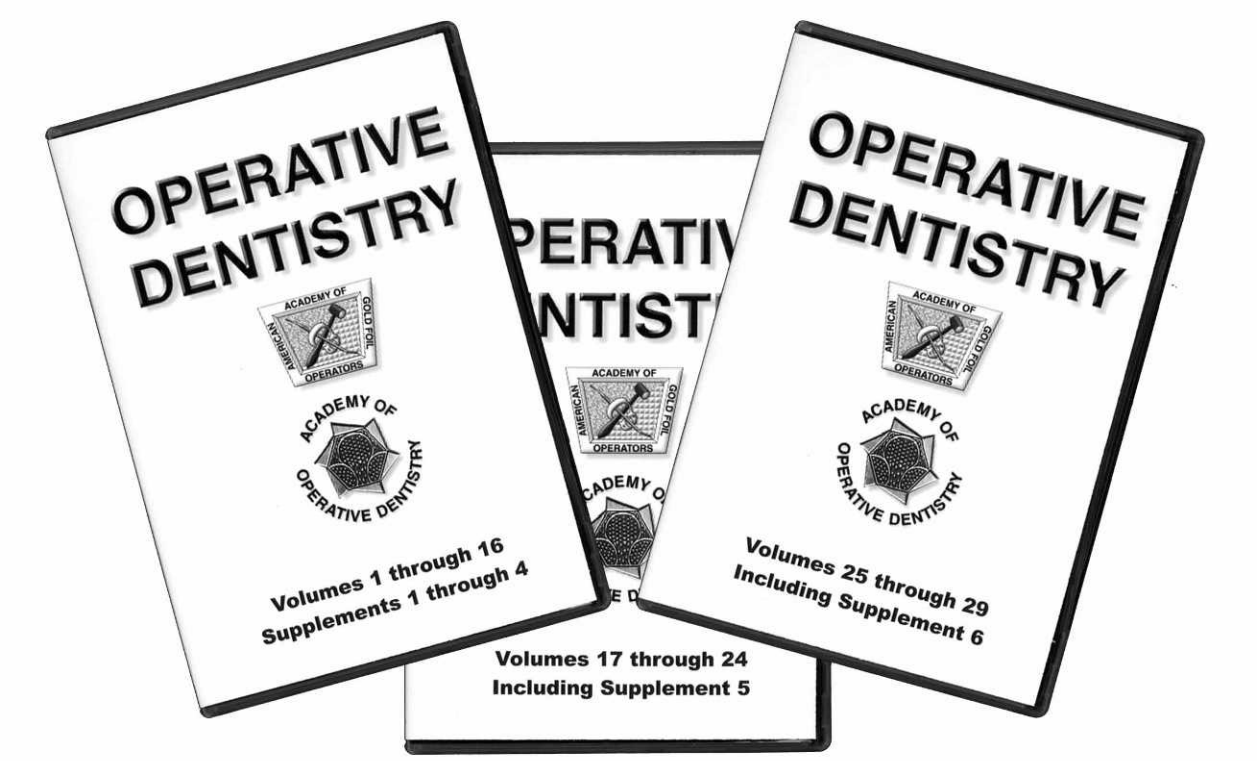
NOTE: As of May 1, 2005, all papers must be submitted electronically.



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