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Surface Hardness of Resin Composites After Staining and Bleaching

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Clinical Relevance

Beverages, such as wine or coffee, and bleaching agents result in decreases in composite surface microhardness from baseline values.

SUMMARY

This study investigated the effect of 3 staining solutions and 3 over-the-counter tooth-bleaching systems on the microhardness of 2 dental resin composites. Forty-five specimens of Filtek Supreme and Esthet-X were randomly assigned to 3 groups. Over a 40-day test period, the specimens in each group (n=15) were immersed in 1 of the 2 staining solutions (coffee and red wine) or distilled water as the control for 3 hours a day at room temperature. The 15 specimens in each staining group were further randomly divided into 3 subgroups, and the specimens in each subgroup (n=5) were bleached using one of the bleaching agents (Night Effects, Simply White Night and Opalescence Quick). Surface hardness was measured at 24 hours after polymerization (baseline), after staining and after bleaching. Means and standard deviations were calculated, and the data were analyzed using repeated-measures analysis of variance and Duncan's Test. The microhardness of Esthet-X was significantly higher than Filtek Supreme at baseline (*p*<0.01). All specimens of both materials immersed in coffee and wine revealed a significant hardness decrease compared to baseline values (*p*<0.05). In the control group, microhardness was increased, and this increase was statistically significant for Filtek Supreme (*p*<0.05). After bleaching, there was a significant decrease in mean microhard-

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ness for all groups tested (p<0.05). No significant difference was found among bleaching agents.

INTRODUCTION

At home, dental bleaching has been widely used since its introduction in 1989 by Haywood and Heymann[1] and, over the last 5 years, dental bleaching has become more frequent, both with and without dental supervision. A number of over-the-counter (OTC) vital bleaching products are currently available to the general population, including tray-based systems, strip-based systems and paint-on systems. Among them, paint-on systems are one of the most popular options for patients, because of their relative ease of use, selective application, low cost, high percentage of success rate and safety.[2]

Current at-home whiteners are almost exclusively peroxide-based, and those with carbamide peroxide as the active ingredient dominate the market. After application, carbamide peroxide breaks down into hydrogen peroxide and urea. When hydrogen peroxide interacts with dental materials and teeth, it decomposes to form hydroxyl radical intermediates, and finally, water and oxygen. The formed hydroxyl radicals combine with the intrinsic and extrinsic stains of dental materials and teeth[3-4] and remove discolorations through oxidation.[5]

Several studies have tested the effect of bleaching agents on the properties of dental materials. In terms of microhardness, the results from those studies are contradictory.[6-12] Studies reported an increase,[7] decrease[9] or no change[6,8,10-11] in composite surface hardness after application of carbamide peroxide gels. Cooley and Burger[7] evaluated composites for changes in surface roughness, hardness and lightness after exposure to carbamide peroxide and found statistically significant increases in hardness. Nathoo and others[8] showed no significant differences in the microhardness of teeth and composite restorations tested after application of a tooth-whitening system. Bailey and Swift[9] found a slight roughening and softening of hybrid composites with carbamide peroxide. Yap and Wattanapayungkul[10] concluded that the use of 35% carbamide peroxide and 35% hydrogen peroxide did not significantly affect the microhardness of resin composite restorative materials. Similarly, García-Godoy and others[11] found that the application of bleaching agents had no effect on the surface roughness of the composites tested. In addition, there are no reports on how staining and OTC bleaching agents, including painted-on systems, affect the microhardness of dental resin composites.

This study evaluated the effect of 2 sequentially applied staining solutions, 2 over-the-counter tooth bleaching products and 1 professionally supervised bleaching system on the microhardness of 2 dental resin composites. The null hypothesis tested was that the bleaching agents used after staining had no effect on the microhardness of the composites tested.

METHODS AND MATERIALS

The 2 resin composites and 3 bleaching agents used in this study are shown in Table 1. The A2 shade of Filtek Supreme (FS) and Esthet- X (EX) were selected for the study. Forty-five disk-shaped specimens 9-mm in diameter and 2.5-mm in depth from each material (90 specimens in total) were prepared in polytetrafluoroethylene molds. The materials were handled according to the manufacturers' instructions. A nylon thread was incorporated into the specimen so that it could be suspended in the staining solutions. The mold with the composite was held between 2 glass slides, each covered with a transparent Mylar strip (Henry Schein, Melville, NY, USA), and the glass slides were gently pressed together to remove excess material. The specimens were polymerized with a conventional halogen light-curing unit (ESPE Elipar Trilight, 3M ESPE, St Paul, MN, USA) using 40-second exposure to the top and bottom surfaces, respectively. The intensity of the curing light was 500 mW/cm^2 and was monitored with a radiometer (Demetron/Kerr, Danbury, CT, USA). The distance between the light source and specimen was standardized by using a 1-mm thick glass slide. The end of the light guide was in contact with the cover glass during the light-polymerization process. After curing, all specimens were stored in distilled water for 24 hours at 37°C to assure complete polymerization. The top surfaces of all specimens were then polished with a sequential series of 3 (medium, fine and superfine) Sof-Lex disks (3M ESPE) and a slow-speed handpiece.

Staining Process

Forty-five specimens of each composite were randomly assigned to 3 groups. The specimens in each group (n=15) were immersed in either 1 of the 2 staining solutions (coffee or red wine) or distilled water as control for 3 hours a day at room temperature over a 40-day test period. The solutions were changed daily.

The coffee group specimens were immersed in vials containing 50 ml of freshly prepared regular coffee per vial (Auroma Paramount Coffee, Pompano Beach, FL, USA); the wine group specimens were immersed in

Table 1: *Resin Composites and Bleaching Agents Tested in this Study*

Product	Code	Manufacturer
Filtek Supreme	FS	3M ESPE, St Paul, MN, USA
Esthet-X	EX	Dentsply/Calk, Milford, DE, USA
Crest Night Efffects	CNE	Procter & Gamble, Cincinnati, OH
Colgate Simply White Night	CSWN	Colgate-Palmolive Company, New York, NY, USA
Opalescence Quick	OPAL	Ultradent, South Jordan, UT, USA

vials containing 50 ml of red wine (Conchay Toro Frontera Cabernet Sauvignon 2002; Merlon, Chile) and the control group specimens were immersed in vials containing 50 ml of distilled water. The vials were sealed with parafilm (Pechiney, Menasha, WI, USA) to prevent evaporation of the staining solution. After each staining period, the specimens were gently rinsed with distilled water, air-dried and kept in distilled water at 37°C.

Bleaching Process

The bleaching agents used in the study are shown in Table 1. Among them, CNE and CSWN are OTC paint-on products, while OPAL is a bleaching system that needs to be used under professional supervision. The 15 specimens in each staining group were further randomly divided into 3 subgroups. The specimens in each subgroup (n=5) were treated with one of the bleaching agents. The bleaching agents were painted onto the specimens' top surface according to the manufacturers' instructions and remained for 8 hours per day at room temperature for 14 days to simulate the bleaching process. OPAL was tested in a protocol that far exceeds the application time determined by the manufacturer's instructions, which was no more than 2 hours per session, in order to be consistent with the other 2 paint-on products. After bleaching, the specimens were rinsed with tap water for 1 minute to remove the bleaching agent, blotted dry and kept in distilled water at 37°C.

Microhardness Testing

The specimens were blotted dry and positioned beneath the indenter of a digital microhardness tester (Vickers Hardness Testing Machine, Buehler, Lake Bluff, IL, USA). A 50 g load was applied through the indenter, with a dwell time of 20 seconds. This method depended on visualization of the surface indentations through the microscope of the testing machine. The length of the diagonal of each indentation was measured directly from the graduated eyepiece of the microscope in the testing machine. Three indentations were made at random on the top surface of each specimen, and a mean value was calculated as the microhardness for that specimen.

Microhardness was measured at 24 hours after polymerization (baseline), at the end of the staining process (40 days) and after bleaching (14 days) on the same specimen.

Statistical Analysis

Microhardness data was compared by using repeated-measures analysis of variance with 2 materials, 3 staining solutions and 3 bleaching agents and all interactions between them. Significant results were evaluated with Duncan's test ($p<0.05$)

RESULTS

Means and standard deviations of the Vickers surface microhardness of the specimens at baseline, after staining and after bleaching are shown in Tables 2 and 3. The difference between the 2 materials at baseline measurements was statistically significant ($p<0.01$). Microhardness of Esthet-X was significantly higher than Filtek Supreme at baseline. All specimens of both materials immersed in coffee and wine revealed a sig-

Table 2: *Microhardness of Esthet-X at Baseline, After Staining and After Bleaching*

	Coffee			Wine			Water		
	CNE	CSWN	OPAL	CNE	CSWN	OPAL	CNE	CSWN	OPAL
BL	61.7 (5.5)*	61.5 (3.2)	64.6 (3.0)	65.0 (3.7)	61.5 (2.8)	61.3 (1.6)	62.3 (2.2)	64.7 (3.7)	64.4 (3.2)
AS	51.4 (2.9)	50.0 (3.8)	51.0 (3.1)	50.0 (2.9)	46.5 (2.1)	46.4 (3.0)	63.5 (2.2)	64.5 (4.6)	64.7 (1.8)
AB	48.6 (2.9)	46.7 (5.0)	49.0 (4.4)	46.3 (1.9)	43.6 (1.0)	43.5 (1.0)	57.4 (2.0)	58.0 (3.0)	57.7 (1.5)

*Means (SD)
BL: Baseline; AS: After stain; AB: After bleaching
CNE: Crest Night Effects   CSWN: Colgate Simply White Night   OPAL: Opalescence Quick

Table 3: *Microhardness of Filtek Supreme at Baseline, After Staining and After Bleaching*

	Coffee			Wine			Water		
	CNE	CSWN	OPAL	CNE	CSWN	OPAL	CNE	CSWN	OPAL
BL	58.7 (1.7)*	55.8 (1.1)	56.1 (2.0)	57.2 (1.8)	56.4 (1.8)	56.0 (2.1)	58.4 (2.1)	57.1 (1.6)	56.4 (0.9)
AS	52.5 (1.5)	50.1 (1.1)	51.4 (1.9)	52.6 (2.6)	51.3 (2.6)	51.0 (1.2)	60.4 (2.4)	61.0 (0.9)	61.2 (1.9)
AB	50.3 (1.3)	48.0 (1.6)	48.8 (2.3)	49.5 (1.6)	47.5 (1.7)	48.5 (2.4)	56.8 (3.7)	56.8 (1.4)	55.3 (2.6)

BL: Baseline; AS: After stain; AB: After bleaching
CNE: Crest Night Effects   CSWN: Colgate Simply White Night   OPAL: Opalescence Quick

nificantly lower Vickers hardness in comparison to baseline values ($p<0.05$). However, in the control group (water), microhardness values were found to be higher when compared to baseline values. This increase was statistically significant for Filtek Supreme ($p<0.05$).

In the Esthet-X group, there was a significant difference between the coffee, wine and water groups ($p<0.05$) after staining, while for the Filtek Supreme group no significant difference was found between the coffee and wine groups; the differences between the wine-water and coffee-water groups were statistically significant ($p<0.05$).

After bleaching, there was a significant decrease in mean microhardness of all groups tested, compared with BL and AS ($p<0.05$). However, no significant difference was found among bleaching agents.

DISCUSSION

Resin-based composite restorative materials undergo a series of physical changes as a result of the polymerization reaction and subsequent interaction with the wet oral environment.[13] Following polymerization, the inward movement of water molecules causes mobilization of ions within the matrix and outward movement of unreacted monomers, as well as the leaching out of ions from fillers and activators, which may cause softening of the resin matrix, reduction in stain resistance and leakage of filler elements.[14-16] Salivary enzymes, pH fluctuation, organic solvents and the ionic composition of food, beverages or saliva may influence the surface quality of dental resins.[17]

One factor affecting the surface quality of resin composites is the finishing and polishing procedure. It has been reported that the microhardness of a celluloid-strip finished composite surface was lower than the composite surface itself.[18] Finishing the composite surface with a celluloid-strip can produce the smoothest composite surface. However, the celluloid-strip finished composite surface discolors more than a polished one, probably due to its high resin concentration on the surface.[19] Therefore, in this study, as was recommended by the manufacturers, the surface of resin composite specimens was finished and polished with commonly used polishing disks.

Different immersing times were used in studies that evaluated the changes in restorative materials in various media.[20-22] Fay and others[21] immersed specimens for 120 hours continuously in different testing media and Abu-Bakr and others[22] used an immersing time of 3 hours per day for 60 days. In this study, specimens were immersed in wine and coffee for 3 hours a day over a 40-day period in order to simulate clinical conditions, since the restorative materials will unlikely be in contact with the staining solution continuously.

The 2 resin composites tested in this study showed a decrease in hardness, resulting from soaking in coffee and wine. Wine significantly reduced the microhardness of Esthet-X when compared with coffee. Filtek Supreme, after staining, showed a significant decrease in hardness values for both coffee and wine; however, no significant difference was found between them.

The difference in hardness of the materials tested after immersion in different stain solutions may be attributed to their chemical composition and the effect the liquids had on different chemical components. Experimentally, the polymer matrix has been shown to be highly susceptible to being softened by chemicals, and the extent of damage may depend somewhat on the diffusion rate, which, in turn, depends on the molecular weight of the penetrant.[23] In low pH drinks, resins show a high solubility and that solubility causes surface erosion and dissolution, which will affect the wear and hardness of the resins.[22] Previous research showed that low pH media affects the chemical erosion of the hybrid materials by acid etching the surface and leaching the matrix-forming cations.[24] As both coffee and wine have low pH, both lowered the surface microhardness of the resin composites tested.

The results of this study corroborate other studies with food-simulating liquid that suggested some food substances, for example, alcoholic beverages, cause softening and accelerated wear of resin composites.[20,23,25-27] As reported by the manufacturer, Esthet-X is a microhybrid composite containing BisGMA, BisEMA and TEGDMA and 0.6-0.8 µm sized fillers, and Filtek Supreme is a nano-composite with a primary 20 nm silica filler and loosely bonded cluster zirconia/silica particle size range from 0.6 to 1.4 µm. Filtek's resin matrix is composed of BisGMA, UDMA, BisEMA and TEGDMA. It is known that both BisGMA- and UDMA-based polymers are susceptible to chemical softening by alcohol.[25] In this study, when compared with coffee, wine significantly reduced the hardness of Esthet-X. However, no difference was found between coffee and wine in the Filtek Supreme group. Although Kao[25] reported that resins containing UDMA are more susceptible to softening by alcohol, Yap and others[26] reported that Esthet-X showed a greater decrease than the resin composite containing UDMA, and they concluded that Bis-GMA-based composites were susceptible to the softening effects of food-simulating liquids, which is similar to the findings in this study.

Resin composites have been shown to harden post-polymerization.[13-14] This may be the reason for the increase in hardness shown in the group where composite materials were suspended in water.

Changes in the chemical and morphological structure of restorations must be of concern when bleaching is used as a whitening treatment. Although some studies revealed that dental whitening agents could change the surface texture of restorations,[28-29] others reported that there were no significant changes in resins.[9,11,30] Bailey and Swift[9] demonstrated that bleaching agents containing 10% carbamide peroxide caused only slight changes to the surface of microfilled resin composites. Turker and Biskin[30] found that surface roughness of the microfilled composite was not affected by different carbamide peroxide concentrations. Also, García-Godoy and others[11] found similar results. Contrary to these findings, Çehreli and others[31] and Whitehead and others[32] showed that resin composites were significantly affected by bleaching agents. These contrary findings might be due to several factors, such as composition of the materials, concentration of bleaching agents and the methodology used in different studies. In this study, after bleaching, there was a significant decrease in mean microhardness values for all groups tested; however, no significant differences were found among bleaching agents.

The majority of home bleaching agents are 10% or 15% carbamide peroxide; however, some products use hydrogen peroxide. Both Crest Night Effects and Colgate Simply White Clear Whitening Gel are OTC tooth-whitening products where the whitening gel is painted onto the teeth with a brush. Crest Night Effects contains 16% carbamide peroxide and Colgate Simply White Clear Whitening Gel contains 18% carbamide peroxide. Opalescence Quick is a highly viscous 35.5% carbamide peroxide "waiting room" whitener. In this study, the composite specimens were not continuously exposed to bleaching products all day long; instead, they were exposed for only 8 hours a day for 14 days as recommended by the manufacturers' of the OTC products. Opalescence Quick was tested in a protocol that far exceeds the application time in order to test bleaching agents at a standardized time and to be consistent with the other 2 paint-on products tested. It appears that OPAL, with prolonged contact with the specimens, had a similar effect on hardness compared to the OTC products.

The bleaching agents tested in this study appeared to have a softening effect on both microhybrid and nano resin composites though there was no significant difference when exposed to different concentrations of carbamide peroxide (35%, 18% and 16%). These results are similar to other studies where a microhybrid and microfill resin composite was exposed to 10% and 6% carbamide peroxide.[9,30] The significant reduction in microhardness in the resin composites tested was expected, since microhybrid and nano resin composites contain a high concentration of resinous matrix to be oxidized by hydrogen peroxide.[6,9]

Previous studies reported that the microhardness of resin composites remained unchanged[8-11] or even increased[6-7] when materials were exposed to carbamide peroxide at different concentrations; however, the materials tested were different and, in none of those studies, were resin composites sequentially stressed under staining solutions and whitening procedures.

In this study, both the staining solutions and bleaching systems tested affected the hardness of the microhybrid and nano resin composite tested. Water was used as a control in the staining stage, so that the specimens in this group were mainly influenced by bleaching agent. After bleaching, their microhardness was higher than specimens from the coffee or wine groups. Thus, there were probably additive effects of staining solutions and tooth bleaching agents on the hardness of the specimens.

However, additional *in vitro* studies evaluating the effects of saliva and controlled clinical trials are necessary to determine any clinical implication

CONCLUSIONS

1. Staining solutions, such as coffee and wine, decrease the microhardness of microhybrid resin composites (Esthet-X) and nano resin composites (Filtek Supreme).
2. There was a statistically significant decrease in the surface microhardness of both resin composites, because of exposure to 2 over-the-counter (Crest Night Effects and Colgate Simply White Clear Whitening Gel) and 1 "waiting room" (Opalescence Quick) bleaching system after staining.
3. There were no significant differences in microhardness among the bleaching agents for any of the restorative materials.

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