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Effects of Food-simulating Liquids on Surface Properties of Giomer Restoratives

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Clinical Relevance

Giomer restoratives, like other direct and indirect composites, are softened by food-simulating liquids, especially citric acid and ethanol. They are also roughened by citric acid.

SUMMARY

This study examined the effects of food-simulating liquid (FSL) on the hardness and roughness of giomer restoratives based on pre-reacted glass ionomer (PRG) technology. The materials investigated included a regular (Beautifil II [BT]) and a recently introduced injectable (Beautifil Flow Plus F00 [BF]) hybrid PRG composite. A direct hybrid composite (Filtek Z250 [ZT]) and an indirect hybrid composite (Ceramage [CM]) were used for comparison. The materials were placed into customized square molds (5 mm × 5 mm × 2.5 mm), covered with Mylar strips, and cured according to manufacturers' instructions. The materials were then conditioned in air (control), distilled water, 50% ethanol solution, and 0.02 N citric acid at 37°C for seven days. Specimens (n=6) were then subjected to hardness testing (Knoop) and surface profilometry. Data were analyzed using one-way analysis of variance and *post hoc* Scheffe test ($p<0.05$). Mean Knoop hardness values for the control group (air) ranged from 53.4 ± 3.4 (BF) to 89.5 ± 5.2 (ZT), while mean surface roughness values values ranged from 0.014 ± 0.002 (ZT) to 0.032 ± 0.001 (BT). All materials were significantly softened by FSL. The degree of soften-

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ing by the different FSLs was material dependent. The hardness of giomers was most affected by citric acid and ethanol. The smoothest surface was generally observed with the control group. Giomer restoratives were significantly roughened by citric acid.

INTRODUCTION

Resin-based composite materials are widely used in restorative dentistry. Clinically, composite restorations are exposed either intermittently or continuously to chemical agents found in saliva, food, and beverages.[1] Results of previous *in vitro* studies[2–4] have shown that food substances can significantly affect the hardness and roughness of composites. Giomers or pre-reacted glass ionomer (PRG) composites are the latest type of glass ionomer–composite hybrid materials, in which glass ionomer fillers (consisting of fluorosilicate particles pre-reacted with polyacrylic acid) are incorporated into a resin matrix. Coupling agents bond the fillers to the matrix and catalysts are added to initiate polymerization of the material. Giomers, like other dental composites, also require bonding agents to adhere to tooth structure. They are light-activated, easy to handle, and release fluoride.

The clinical performance of giomers has been evaluated in several studies.[5,6] In an eight-year trial involving Class I and II restorations, Gordan and others[5] reported no restoration failures. Significant changes were observed only for marginal adaptation at occlusal surfaces and marginal staining at proximal surfaces. In addition, no significant difference in clinical performance was observed[6] between giomer and microfilled composite restorations in Class V cavities after three years. *In vitro* studies[7,8] comparing giomers to resin-modified glass ionomer cements showed that giomers had significantly higher flexural strength. Giomers were also found to be harder than minifilled composite resins and ormocers[9] and had better polishability than did conventional glass ionomers.[10] The high-fluoride release and recharge properties of giomers minimize recurrent caries and demineralization.[11] Based on several studies,[12–16] giomers have a reported caries inhibiting effect of 14%-35% compared to non–fluoride-releasing restorative materials.

Information regarding the influence of food-simulating liquids (FSLs) on the surface hardness and roughness of giomer restoratives employing PRG technology is still not widely available in the literature. By virtue of their pre-reacted glass ionomer fillers, these materials may behave differently when compared to composites based on zirconia and other fillers.

This study investigated the effects of FSL on the surface properties of two types of giomer restoratives. It was hypothesized that the effects of FSL on giomers will differ from those of conventional composites in view of their novel PRG technology.

MATERIALS AND METHODS

The materials investigated included two giomers (Beautifil II [BT] and Beautifil Flow Plus F00 [BF], Shofu, Kyoto, Japan), a direct hybrid composite (Filtek Supreme Z250 [ZT], 3M-ESPE, St Paul, MN, USA), and an indirect hybrid composite (Ceramage [CM], Shofu). The technical profiles of the various restoratives evaluated are shown in Table 1.

The materials were placed into customized square molds (5 mm $\times$ 5 mm $\times$ 2.5 mm) and covered with Mylar strips. A glass slide was placed and pressure was applied to remove any excess material. The specimens were then light-cured according to manufacturers' instructions (Table 2). Twenty-four specimens of each material were fabricated and randomly assigned into control (air) and treatment (distilled water, 50% ethanol solution, and 0.02 N citric acid) groups in clusters of six. The specimens were conditioned in individual vials containing the different FSL at 37°C for seven days.

After conditioning, each specimen was air-dried and subjected to a 3.0-mm line scan across the center of the specimen using a surface profilometer (Surftest, Mitutoyo Corp, Tokyo, Japan) with a probe diameter of 5 μm. Surface roughness value (Ra), which is the arithmetic average of the absolute values based on the vertical deviations of the roughness profile from the mean line calculated by the computer, was recorded. Hardness testing was then carried out using a digital microhardness tester (FM Series Microhardness Tester, Future Tech, Tokyo, Japan) to attain the Knoop hardness value (KHN). A load of 500 gf with a dwell time of 15 seconds was applied to the central top surface of each specimen *via* an indentator. Statistical analysis was done using one-way analysis of variance and *post hoc* Scheffe test at a significance level of 0.05. A Pearson correlation test was also conducted to determine the relationship between the surface hardness and roughness of the individual composites.

RESULTS

The means for KHN and Ra of the four composite restoratives are shown in Tables 3 and 4. Mean KHN

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Table 1: *Chemical Composition of the Different Composite Restoratives*

Material, Lot No.	Composition	wt%	Filler Size, µm	Shade
Beautifil II, 051003	Bis-GMA (bisphenol A diglycidyl ether dimethacrylate)	7.5	0.01–4.0 0.8 (mean)	A2
	TEGDMA (triethyleneglycol dimethacrylate)	<5		
	Aluminofluoro-borosilicate glass, Al_2O_3	83.3		
	DL-camphorquinone			
Beautifil Flow Plus F00, 091013	Bis-GMA	15–25	0.01–4.0 0.8 (mean)	A2
	TEGDMA	12–14		
	Aluminofluoro-borosilicate glass, Al_2O_3	67.3		
	DL-camphorquinone			
Filtek Z250, N183958	Bis-GMA	1–10	0.01–3.5 0.6 (mean)	A2
	TEGMA	<5		
	Bis-EMA (bisphenol A polyethylene glycol diether dimethacrylate)	1–10		
	UDMA (diurethane dimethacrylate)	1–10		
	Zirconia/silica	82		
Ceramage, 071060	UDMA	Proprietary	Proprietary	A2B
	Zirconia/silica (amorphous)	73	Proprietary	

for the control group (air) ranged from 53.4 ± 3.4 (BF) to 89.5 ± 5.2 (ZT), while mean Ra values ranged from 0.014 ± 0.002 (ZT) to 0.032 ± 0.001 (BT). Results of statistical analyses are reflected in Tables 5 through 7.

The degree of softening by the different FSLs was material dependent. The greatest hardness was observed for the control group. Conditioning in ethanol generally resulted in the greatest softening. The giomers were also affected by citric acid. For the control group, ZT was significantly harder than all of the materials evaluated. No significant difference in KHN between BT and CM was noted, and BF was significantly softer than all of the other materials. When conditioned in distilled water, similar results were observed. After conditioning in citric acid, ZT and CM had comparable KHN. Both materials were significantly harder than BT and BF. KHN for CM and ZT were, again, not significantly different after conditioning in ethanol. The hardness values of CM were significantly greater than those of BT and BF, and ZT was significantly harder than that of BF.

Table 2: *Manufacturers' Curing Instructions*

Materials	Mode	Curing Unit	Curing Time, s
Beautifil II	Light cure	Elipar S10 (3M-ESPE)	20
Beautifil Flow Plus F00	Light cure	Elipar S10 (3M-ESPE)	20
Filtek Z250	Light cure	Elipar S10 (3M-ESPE)	20
Ceramage	Oven cure	Solidlite (Shofu)	240

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Table 3: *Mean Knoop Hardness (KHN) for the Composites in the Different Food-simulating Liquids (FSLs)[a]*

Materials	Air	Distilled Water	Citric Acid	Ethanol
Beautifil II	77.6 (2.4)	63.8 (2.0)	41.4 (3.0)	42.0 (1.4)
Beautifil Flow Plus F00	53.4 (3.4)	40.5 (2.3)	37.4 (4.3)	32.6 (4.3)
Filtek Z250	89.5 (5.2)	71.7 (4.3)	74.9 (1.8)	50.7 (7.6)
Ceramage	75.4 (8.3)	59.3 (3.4)	69.9 (1.6)	56.3 (9.6)

[a] Standard deviations (SDs) in parentheses.

Table 4: *Mean Surface Roughness (Ra) for the Composites in the Different Food-simulating Liquids (FSLs)[a]*

Materials	Air	Distilled Water	Citric Acid	Ethanol
Beautifil II	0.032 (0.001)	0.025 (0.002)	0.083 (0.002)	0.033 (0.004)
Beautifil Flow Plus F00	0.020 (0.006)	0.040 (0.004)	0.057 (0.003)	0.041 (0.005)
Filtek Z250	0.014 (0.002)	0.017 (0.002)	0.017 (0.003)	0.022 (0.002)
Ceramage	0.025 (0.012)	0.027 (0.003)	0.029 (0.003)	0.031 (0.005)

[a] Standard deviations (SDs) in parentheses.

Regardless of conditioning medium, ZT had the smoothest surface among the materials evaluated. After conditioning in air and citric acid, BT was significantly rougher than ZT. BT also had higher Ra values than BF and CM after exposure to citric acid. After conditioning in distilled water and ethanol, BF was significantly rougher than the other materials. Significant negative correlations between hardness and roughness were observed for all composites except for CM. The correlation for ZT was strong.

Table 5: *Comparison of Mean Knoop Hardness (KHN) of the Composites in Different Food-simulating Liquids (FSLs)*

FSL	Materials
Air	ZT > BT, CM > BF
Distilled water	ZT > BT, CM > BF
Citric acid	ZT, CM > BT, BF
Ethanol	CM > BT, BF ZT > BF

Abbreviations: BT, Beautifil II; BF, Beautifil Flow Plus F00; ZT, Filtek Z250; CM, Ceramage.

DISCUSSION

Hardness is defined as the resistance of a material to permanent indentation.[17] Studies[18,19] have linked low hardness values to inferior surface wear resistance. Worn and roughened surfaces may be plaque retentive, allowing bacterial flora to flourish, leading to increased caries risk and periodontal inflammation.[20] The liquids used to condition the materials in this study are among those recommended in guidelines from the US Food and Drug Administration to be used as food simulators.[21,22] Aqueous ethanol-water solution simulates alcoholic liquids and is also the medium of choice for accelerated ageing of composite restorations, as its solubility parameter is comparable to that of bisphenol A diglycidyl ether dimethacrylate (bis-GMA).[2,23] The latter is one of the most commonly utilized resin monomers in composite resins. Citric acid (0.02 N) stimulates acid in foodstuffs such as vegetables, fruits, candy, and syrup, as well as certain beverages. Distilled water simulates the wet oral environment, while air serves

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Table 6: *Comparison of Mean Surface Roughness (Ra) of the Composites in Different Food-simulating Liquids (FSLs)*

FSL	Materials
Air	BT > ZT
Distilled water	BF > CM, BT > ZT
Citric acid	BT > BF > CM > ZT
Ethanol	BF > BT, CM > ZT

Abbreviations: BT, Beautifil II; BF, Beautifil Flow Plus F00; ZT, Filtek Z250; CM, Ceramage.

Table 7: *Comparison of Mean Knoop Hardness (KHN) of the Composites in Different Food-simulating Liquids (FSLs)*

Materials	FSLs
Beautifil II	Air > distilled water > ethanol, citric acid
Beautifil Flow Plus F00	Air > distilled water > ethanol, air > citric acid
Filtek Z250	Air > citric acid, distilled water > ethanol
Ceramage	Air, citric acid > ethanol, air > distilled water

as the control medium. As the most significant changes in hardness have reportedly[3] occurred during the first week of exposure to FSL, a seven-day conditioning period was selected for this investigation.

Oxygen inhibits the surface polymerization of composite resins. The depth of inhibition in atmospheric air ranges between 25 μm and 105 μm and varies between composites.[24] The materials were adapted against a Mylar strip to minimize the oxygen inhibition layer[24] and were cured according to manufacturers' instructions. This method produced a consistent smooth surface across all specimens.[25] The latter ensures accurate hardness readings and prevents discrepancies associated with finishing/polishing procedures. The unpolished surface is, however, matrix-rich and may result in a greater degree of softening. It is therefore less characteristic of the bulk material.[26,27] As the materials were not subject to any mechanical forces, any observed changes in hardness and surface roughness can be attributed to exposure to the FSL.

BF is marketed as a flowable hybrid composite to be used as a restorative, base, and liner. "Flowability" is mainly achieved by its lower filler loading of approximately 67.3% weight. The filler loading values of the other materials investigated were higher and ranged from 73% to 83.3% weight. A general trend is observed between increased filler loading and improved hardness, compressive strength, and stiffness.[28,29] BF had the lowest hardness values in the control medium and was significantly softer than ZT and CM for all test mediums. The lower filler fraction in BF could have played a significant role in its lower hardness as compared to the other composites. The higher hardness values of ZT and CM can also be attributed to the use of zirconia-silicate fillers over alumino-fluoro-borosilicate glass fillers in the giomers. In addition, CM was oven-cured (Solidlite, Shofu) under heat and high-voltage (600-W) light.[30,31]

The giomers investigated were found to be significantly degraded by citric acid. This may be attributed to the greater susceptibility of fluorosilicate glass fillers to degradation by weak acids.[32] The giomer materials and ZT contain bis-GMA as part of their resin matrix. Ethanol has a solubility index similar to that of bis-GMA,[2,23] which enhances its disintegration. In addition, triethyleneglycol dimethacrylate (TEGDMA) has been shown[33] to have the greatest amount of liquid sorption in 50% ethanol-water solution when compared to bis-GMA, bisphenol A polyethylene glycol diether dimethacrylate, and diurethane dimethacrylate.

Liquid uptake will leach unreacted components from the resin matrix, causing a reduction in mechanical properties. Diffusion of solvent into the resin network interferes with bonding, separates the chains, and interrupts the arrangement of the polymer chains in the compound, causing significant reduction of the material's physical properties.[33] BF, which contained the highest percentage weight of bis-GMA and TEGDMA, was observed to be most affected by ethanol as compared to the other three materials. Distilled water exerts a similar degradative effect through liquid sorption and dissolution of the resin matrix.[33]

Roughness parameters are dependent on several factors, such as filler particle size, percentage of surface area occupied by filler particles, hardness, degree of polymer conversion to resin matrix, and filler-matrix interaction.[34] For all FSLs, the Ra

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values of the giomer composites were significantly higher than that of ZT. The greater surface roughness corresponded to the larger average particle sizes (0.8 μm) of BT and BF, as compared to the smaller average particle sizes (0.6 μm) found in ZT. The fillers exposed on the surface of giomers after surface degradation by the FSL are consequently coarser, leading to a rougher surface profile. All roughness values of the materials were, however, below the threshold surface roughness for bacterial retention (Ra=0.2 μm).[20] The values may have little bearing in a clinical setting, as an initial unpolished surface is uncommon. The negative correlation between roughness and hardness was significant for all materials except CM. It was strong for ZT (r=−0.746). The surface roughness of giomers was therefore weakly associated with their surface hardness. A softer surface corresponded to a rougher giomer surface.

CONCLUSIONS

Within the limitations of this *in vitro* study,

1. Giomers, like other direct and indirect composites, were degraded by FSLs.
2. Hardness of giomers was significantly affected by citric acid and ethanol.
3. Roughness of giomers was significantly affected by citric acid.
4. With the exception of the indirect composite, significant and negative correlations were observed between hardness and roughness.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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