Effect of alcoholic beverages on surface roughness and microhardness of dental composites

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The aim of this study was to evaluate the microhardness and surface roughness of composite resins immersed in alcoholic beverages. Three composite resins were used: Durafill (Heraeus Kulzer), Z250 (3M-ESPE) and Z350 XT (3M-ESPE). The initial surface roughness and microhardness were measured. The samples were divided into four groups ($n=30$): G1-artificial saliva; G2-beer; G3-vodka; G4-whisky. The samples were immersed in the beverages 3× a day for 15 min and 30 days. The surface roughness and microhardness assays were repeated after immersion period. The data were statistically analyzed by two-way ANOVA and Tukey-HSD test ($p<0.05$). Surface roughness increased for all composite resins immersed in beer and whisky. Microhardness of all groups decreased after immersion in alcoholic beverages. The effect of these beverages on dental composites is depended upon the chemical composition, immersion time, alcohol content and pH of solutions.

Keywords: Composite resin, Alcoholic beverages, Surface roughness, Microhardness

INTRODUCTION

The use of resin-based dental restorative materials has increased because of their good aesthetic, easy handling, and ability to establish a bond to dental hard tissue¹. The long-term clinical performance of restorations depends on the type of restorative material and how the degradation procedure may affect this material changing its mechanical properties such as wear resistance, bond strength, integrity of the interface between tooth and restoration, esthetic quality, damage and decrease hardness and roughness^{1,2}.

Dental composites are compounds by an organic polymerizable matrix, inorganic fillers, borosilicate, silica, and a silane-coupling agent^{2,3}. The organic phase (resin phase) of composite resins is constituted by monomers such as Bis-GMA (bisphenol A diglycidyl dimethacrylate) and/or UDMA (urethane dimethacrylate), co-monomers such as TEGDMA (triethylene glycol dimethacrylate) and/or Bis-EMA (ethoxylated bisphenol A glycol dimethacrylate), additives, initiator (canphoroquinone), co-initiator (dimethyl-aminobenzoic acid-ester), a polymerization inhibitor, and a photostabilizer (benzophenone). The inorganic phase is composed by fillers of different types and particle sizes¹.

There are different types of polymer degradation such as thermal, mechanical, and passive hydrolysis. This process is associated with composite resin erosion

that designates the material losses. Dental erosion is a risk factor for oral health, accordingly with patient lifestyle and eating habits⁴. The wear phenomenon is caused by several problems as abrasion, corrosion, and fatigue⁵. The dental erosion has intrinsic or extrinsic etiology⁵. Extrinsic factors include, in most cases, the consumption of acidic foods, alcoholic beverages, soft, and energy drinks⁶. Intrinsic factors include chronic gastrointestinal disorders, anorexia and bulimia, where the regurgitation and persistent act of vomiting are common reducing pH in the oral environment⁷. The effects of such factors in restorative materials are softening, surface roughness increases, and tooth erosion, making them more susceptible to wear^{8,9}. Even with the technological development and the significant improvement in physical and mechanical properties of the dental composites, the material degradation is still a problem¹⁰.

The increased consumption of alcoholic beverages has raised questions about these drinks' erosive potential on the dental hard tissues and clinical performance of restorative materials. The alcoholic beverages can promote erosion on surface of the composite resin¹¹. The alcohol present in these beverage diffuses into the resin plasticizing the polymer matrix reducing the mechanical properties such as hardness, wear strength, and roughness, directly acting on the filler-resin matrix interface⁶.

The roughness of tooth structure and restorative

materials caused by erosion affects the retention of microorganisms. So the rougher surface allows the fast colonization by microorganisms and maturation of the biofilm, increasing the risk of the development of dental caries and periodontal disease as well as the susceptibility to staining of the restoration¹²⁾.

Thus, the aim of this study was to analyze the influence of alcoholic beverages on the microhardness and surface roughness of different composite resins.

MATERIALS AND METHODS

Specimen preparation

The composite resins evaluated in the current study and their compositions are shown in Table 1. All the materials were of shade A2.

One hundred and twenty samples were prepared for each composite resin and beverage tested ($n=30$) using a silicone matrix (6 mm in diameter and 2 mm in thickness). The dental composites were inserted into elastomer molds in a single increment. The specimens were covered with a polyester matrix strip (Quimidrol, Curitiba, PR, Brazil) and a rigid glass microscope slide. Finger pressure was applied on the slide to extrude the excess material. The material was light cured for 40 s using a light emitting diode (LED, 1,200 mW/cm², Radii Plus, SDI, Victoria, Australia). The irradiance value was measured by radiometer (Demetron, Kerr, Sybron

Dental, Orange, CA, USA).

After storage in distilled water at 37°C for 24 h the specimens were subjected to finishing and polishing system (Diamond Pro, FGM, Joinville, SC, Brazil and SofLex, 3M-ESPE, St. Paul, MN, USA). Then, the samples were ultrasonically cleaned in distilled water for 10 min.

Specimens cycling

The samples of each composite resin were randomly divided into four groups: (1) artificial saliva; (2) beer; (3) vodka; and (4) whisky. The composition of these solutions are described in Table 2. The assay was performed maintaining the samples immersed for 15 min, three times a day, for 30 days period¹³⁾. During cycling each sample was individually stored in eppendorf containing 1.5 mL of artificial saliva while it was not immersed in the alcoholic beverages. The pH value of each alcoholic beverage was determined using a pH meter (Orion 420, Beverly, MA, USA). Each sample was immersed in 10 mL of alcoholic beverage, according to ISO 4049:2009¹⁴⁾.

Microhardness evaluation

The microhardness analysis was performed on the surface of the composite resins using an indenter (HMV-2, Shimadzu, Kyoto, Japan). For each specimen five indentations were performed under a load of 50 g for 15 s. The Knoop Hardness number (KHN, kg/mm²) for

Table 1 Dental composites used in this study

Material	Monomers*	Filler*	Manufacturer	Batch Number
Filtek Z350 XT (nanofilled)	Bis-GMA, UDMA, TEGDMA, Bis-EMA, initiator	Zirconia, silica (5–20 nm) -72.5 wt.%, 55.6 vol.%	3M-ESPE, St. Paul, MN, USA	N248065BR
Filtek Z250 (microhybrid)	Bis-GMA, Bis-EMA, UDMA, initiator	Zirconia, silica (0.01–3.5 µm) -78 wt.%, 66 vol.%	3M-ESPE	N287540BR
Durafill (microfilled)	UDMA, Bis-GMA, TEGDMA, initiator	Silicon dioxide (0.02–0.07 µm), prepolymerized filler (10–20 µm) -50.5 wt.%, 40 vol.%	Heraeus Kulzer, South Bend, IN, USA	010215

*Information provided by the manufacturers.

Table 2 Solutions tested

Solution	Composition*	ABV (%) [‡]	pH	Manufacturer
Artificial saliva	Sodium carboxymethylcellulose, potassium chloride, di-potassium phosphate, sodium chloride, calcium chloride, magnesium chloride and aspartame.	0	7.1	Pharmacêutico, Maceió, AL, Brazil
Heineken® (beer)	Water, malt, hops and yeast.	5	4.1	Heneiken, Araraquara, SP, Brazil
Absolut® (vodka)	Water and alcohol.	40	7.0	Absolut Company, Sweden
Red Label® (whisky)	Grain, malt whiskies, flavours and citrus oils.	40	3.76	Johnnie Walker, Diageo, SP, Brazil

*Information provided by the manufacturers. [‡]Alcohol by volume (ABV).

each specimen was recorded as the average of the five readings^{13,15}.

Surface roughness evaluation

The analysis of the surface roughness was performed by an Atomic Force Microscope (AFM, SPM-9500J3, Shimadzu) equipped with a scanner of maximum ranges 125 μm in the horizontal and vertical directions (x and y axes) and up to 8 μm in the orthogonal direction (z axis) respectively, and an optical microscope (0.8–5 $\times$ magnification, Kyowa Optical, Kanagawa, Japan) to locate the zones of interest by monitoring the sample using a TV screen (Victor TM-A14s). All figures were acquired in contact mode by Si_3N_4 probe (200 μ) with a constant force of 0.15 Nm^{-1} and resonance frequency at 1 Hz.

The roughness value (Ra) was determined using the arithmetic average determined as average deviation of the midline section¹⁶.

Statistical analysis

The Shapiro-Wilk test confirm the normal distribution of the roughness and microhardness data, therefore, parametric tests were used for the inferential analysis. Surface roughness and microhardness values were tested for significant differences ($p < 0.05$) using two-way analysis of variance (ANOVA) with repeated measurement, Tukey's honest significant difference

(HSD) for multiple comparisons. This analysis was repeated seven times for each item (microhardness and surface roughness) with the following comparisons: 'Artificial Saliva $\times$ 3 composites', 'Beer $\times$ 3 composites', 'Vodka $\times$ 3 composites', 'Whisky $\times$ 3 composites', 'Z350 $\times$ 4 solutions', 'Z250 $\times$ 4 solutions' and 'Durafill $\times$ 4 solutions'. The paired *t*-test was used for intragroup comparisons regarding between the baseline and after 30 days of the bavarege challenge. All the statistical tests used in this study were applied at a significant level of 5%.

RESULTS

Results for the microhardness analysis are shown in Table 3. Before and after 30 days of immersion in different solutions, Z250 and Z350 XT composite resins showed significantly higher microhardness than Durafill composite resin ($p < 0.05$). Moreover, after the immersion in whisky the Z350 XT showed higher microhardness than Z250 ($p < 0.05$). Microhardness of all groups significantly decreased after immersion in the tested beverages ($p < 0.05$).

Whisky and vodka produced greater reduction in microhardness than artificial saliva and beer for Z250 and Z350XT ($p < 0.05$). For 30 days of immersion in whisky Durafill showed lower microhardness than other beverages ($p < 0.05$). There was no difference between beer and vodka in microhardness for Durafill ($p > 0.05$).

Table 3 Means (SD) for microhardness (KHN)

	Artificial Saliva		Beer		Vodka		Whisky	
	Initial	30 days	Initial	30 days	Initial	30 days	Initial	30 days
Z350 XT	118.40 (9.94) aA	101.40 (6.88) aB	114.40 (5.92) aA	102.39 (5.43) aB	112.00 (7.25) aA	93.44 (6.32) aC	119.60 (10.11) aA	91.22 (3.66) aC
Z250	123.70 (11.60) aA	98.77 (10.03) aB	117.60 (4.94) aA	101.47 (8.79) aB	121.10 (10.11) aA	87.68 (8.09) aC	112.83 (7.81) aA	79.87 (2.76) bC
Durafill	39.45 (4.94) bA	32.02 (3.56) bB	37.72 (2.55) bA	24.55 (3.06) bC	36.03 (2.76) bA	22.59 (1.99) bC	37.01 (2.85) bA	18.79 (1.48) cD

Means followed by distinct capital letters in the same line, and small letters in the same column, are significantly different ($p < 0.05$).

Table 4 Means (SD) for surface roughness (nm)

	Artificial Saliva		Beer		Vodka		Whisky	
	Initial	30 days	Initial	30 days	Initial	30 days	Initial	30 days
Z350 XT	402.27 (184.26) aC	462.47 (175.77) aC	419.80 (89.38) aC	644.23 (89.09) aA	505.10 (135.36) aC	585.65 (158.43) aB	409.50 (135.93) aC	664.17 (184.59) bA
Z250	411.35 (74.30) aB	493.04 (79.42) aB	527.93 (173.20) aB	645.45 (202.13) aA	472.29 (182.48) aB	538.59 (168.17) aB	359.60 (76.79) aB	614.01 (140.26) bA
Durafill	375.34 (106.91) aB	424.54 (117.39) aB	428.60 (122.21) aB	731.43 (134.27) aA	406.35 (129.49) aB	465.79 (158.81) aB	349.41 (106.02) aB	830.10 (148.86) aA

Means followed by distinct capital letters in the same line, and small letters in the same column, are significantly different ($p < 0.05$).

Fig. 1 Initial and 30 days of immersion in vodka of the Z350 XT dental composite.

Fig. 2 Initial and 30 days of immersion in beer of the Z250 dental composite.

Fig. 3 Initial and 30 days of immersion in whisky of the Durafill dental composite.

Table 4 shows that surface roughness in the initial and 30 days periods showed no significant differences between the composite resins immersed in the solutions evaluated. All dental composites showed significantly higher surface roughness for 30 days period of immersion in beer and whisky ($p < 0.05$), except for Z250 and Durafill immersed in vodka ($p > 0.05$). For 30 days of immersion in whisky, Durafill showed significantly higher surface roughness than Z250 and Z350 XT ($p < 0.05$).

The figures of the composite resins immersed in

beer, vodka, and whisky obtained by AFM (Figs. 1–3) showed after immersion of 30 days period loss of resin matrix and protrusion of filler particles or voids.

DISCUSSION

The influences of chemical and thermal factors are related to the clinical success and long-term performance of dental composite restorations. Chemical factors play a fundamental role in the degradation process of the

composite resins surface¹⁷. The influence of the chemical degradation reflects in a change of the surface roughness and microhardness¹⁸.

The current results indicated changes on the surface of different dental composites with reduced microhardness when exposed for 30 days in alcoholic beverages. The results are in agreement with those presented by other studies¹⁹, which reported reduction of hardness after immersion in alcoholic beverages. Moreover, in the control group immersed in artificial saliva, the results showed that the composite resins evaluated (Durafill, Z250 and Z350 XT) showed lower surface microhardness values for 30 days.

The 30 days period of immersion evaluated was enough to produce significant differences in surface roughness and microhardness. Groups immersed in alcoholic beverages were able to cause chemical degradation in the polymer of all dental composites tested. For 30 days of immersion in beer Durafill showed significant lower hardness than artificial saliva. But this situation did not occur with composite resins Z250 and Z350XT. These results are in agreement with other studies²⁰ that showed no differences between beer and water after 14 days of immersion.

Even with pH of 7, the vodka reduced the surface hardness of the dental composites evaluated for 30 days of immersion. This result may be explained by the alcoholic content of the vodka (40% alcohol by volume-ABV) that penetrate into the organic matrix causing hydrolysis. This process expand the spaces between the linear chains of the polymers and loss of chemical bond between filler particles and the resin matrix. Filler particles dislodge from the outer surface of the material²¹ causing surface roughness and decreasing hardness. This situation causes plasticizer effect of the organic matrix and consequent degradation¹⁷. The degradation of the resin-filler interface and inorganic fillers may also play a role in the reduction of surface hardness²².

The difference of hardness in the composite resins immersed in different beverages is also associated to the chemical composition²³. The dental composites Z250 and Z350 XT have in organic matrix BisEMA monomer with high molecular weight and it is more resistant to degradation due to removal of terminals OH-groups, which are susceptible to absorption and solubility²⁴. The composite resin Durafill has the highest percentage of organic matrix, based on UDMA, and a lower percentage of inorganic filler, which may explain the lowest values of surface hardness. Moreover, UDMA, TEGDMA, and Bis-GMA are monomers highly susceptible to absorption and solubility in contact with alcohol, causing softening and degradation of the organic matrix^{17,20,25}. In the presence of organic acids the degradation is more evident in whisky group (40% ABV and pH 3.76) that showed be the beverage most aggressive, providing lowest surface microhardness values for all composite resins evaluated. The loss of hardness may contribute to deterioration of the composite resin restorations, including loss of anatomical form and discoloration²⁶. Furthermore, chemical softening may have a negative

effect on wear and abrasion decreasing the life span of these restorations⁸.

The surface roughness analysis was performed by AFM that has been suggested as a accurate method to determine the quality of surface composites^{11,15}. The surface roughness of composite resins evaluated (Durafill, Z250 and Z350 XT) increased significantly in 30 days of samples immersion in beer and whisky, except for Durafill and Z250 immersed in vodka.

The pH is a very important factor to determine the erosive potential of a solution²⁷. When immersed in artificial saliva, all tested dental composites showed no difference between initial and 30 days period. The results of this study are in agreement with other studies²⁸, which reported no statistically significant difference in the surface roughness values of composite resins between initial and 180 days of immersion periods in distilled water. Low pH and alcohol may affect the surface integrity of composite resins. Absorption of alcohol molecules contained in beverages into the resin matrix could result in softening of composite resins surface. This explains the change in surface roughness of the dental composites immersed in alcoholic beverages²⁹. Although the vodka showed a pH of 7, similar to artificial saliva (neutral), the Z350 XT dental resin showed increased of the surface roughness after immersion in vodka. In surface analysis using AFM it was found that the surface of the composite resin Z350 XT stored in vodka (Fig. 1) showed exposed filler particles after chemical degradation process, creating voids and leaving the dental composite surface more irregular.

The composite resin Z350 XT has a combination of zirconia and silica nanoparticles. Furthermore, these particles form nanoclusters (0.6 to 1.4 μm). These nanoclusters act like a single filler. Despite the neutral pH, the high ABV may have been diffused into resin matrix providing hydrolysis between silane and filler removing these nanoclusters. According to manufacturer the composite resin Z350 XT has a higher percentage of diluent monomer (TEGDMA). This monomer shows poor wear resistance in contact with alcohol or acid solutions³⁰.

Durafill showed the largest values of surface roughness when immersed in beer and whisky. This may be explained by chemical composition of this material with lower percentage of filler. Thus it is more susceptible to surface degradation due the low pH and the high presence of alcohol in whisky. The chemical degradation acts also on resin matrix due to the heterogeneous polymerization process of the methacrylate-based monomers²⁸.

Moreover, the low pH of a solution causes damage to the mechanical performance of composite resins. In this study, the alcoholic beverages were changed three times daily. Thus, the pH of the solutions were kept and their potential to promote degradation³¹. However, different results were found in other studies³², where it was observed that the composite resin Filtek Supreme, which can be compared structurally to Z350 XT, showed no change on surface roughness when immersed in

lactate buffered solution (pH 2.7) and water.

The surface roughness and chemical degradation are dependent of the composite resin composition³³. However, the effect of the alcoholic beverages in oral environment can be different, which may promote different effects on composite resins evaluated. The influence of other agents of daily diet and oral hygiene can also interfere on the action of these alcoholic beverages.

CONCLUSION

Based on the methodology employed it was concluded that all solutions reduced the surface hardness of the dental composites after immersion for 30 days, whisky was the most aggressive beverage, and beer and whisky produced higher changes on surface roughness of the dental composites evaluated.

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