ORIGINAL ARTICLE

Microhardness evaluation of silorane and methacrylate composites submitted to erosion and abrasion processes

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Abstract

Objective: The aim of this study was to evaluate the Knoop hardness number (KHN) of methacrylate (MC) and silorane (SC) composites after being submitted to erosion and abrasion processes.
Material and methods: Forty samples were made with each composite: MC and SC. The samples were divided into eight groups ($n = 10$) according to the type of composite (G1–G4, MC; G5–G8, SC) and the beverages involved in the erosion process (G1 and G5 – Control (C), without erosion, with abrasion; G2 and G6 – Orange Juice (OJ), abrasion; G3 and G7 – Smirnoff Ice® (SI), abrasion; G4 and G8 – Gatorade® (GA), abrasion). The KHN test was performed 24 h after the last cycle of erosion/abrasion.
Results: The MC groups showed smaller KHN values for the SI group ($p < 0.05$) when compared to the Control and OJ groups; however, for the SC groups, no differences were found ($p > 0.05$).
Conclusion: Methacrylate composite when submitted to acidic beverages erosive challenge combined with abrasive process might alter its surface microhardness. However, the beverages used in the present study were not able to interfere in silorane composite surface microhardness.

Introduction

Methacrylate composites (MC) were first used in dentistry in the 1960s by Bowen, and today, they are considered an excellent aesthetic restorative material. However, one of the greatest concerns related to methacrylate composite relates to the shrinkage stress on cavity walls by the monomeric reduction during the polymerization process.[1] Manufacturers have created another type of monomers, known as low-shrinkage, in an attempt to reduce this problem.[2] The chemical constitution of the silorane composite (SC) is siloxane, of high hydrophobicity and extremely resistant oxirane. The oxirane is constituted by ring-shaped cationic molecules, and during the polymerization process, the oxirane cationic rings open, which compensates the monomer volumetric contraction.[3]

However, other aspects related to the silorane composite are presented in the literature. Among these is the controversial discussion about silorane not being subject to oxygen interference during the polymerization process.[4] The discussion becomes relevant when this composite is compared to methacrylates. In the latter material, the oxygen reacts with the free radicals that participate in the polymerization, leading to a superficial sub-polymerized layer.[5]

The sub-polymerized composite resin becomes less resistant to any variation in the complex oral environment. In this context, it is important to consider the increase in acid beverage consumption in recent years, relative to the erosive process in the buccal cavity, which affects not only the dental structure but also the dental materials for rehabilitation.

Erosion is one of the most common causes of dental structure loss, resulting in a rounding of the cusps that then needs to be successfully rehabilitated.[6] However, acid activity on the composites promotes micro degradation of the resin matrix, which combined with toothbrushing abrasion and the mechanical chewing forces, could release the inorganic particles of the composite.[7] Clinically, this phenomenon can lead to a decrease in microhardness and the deterioration of the organic part of the resin, damaging the restoration.[8] Considering that the silorane composite is a new material for clinical parameters, this study proposed to evaluate the surface microhardness of both MC and SC after erosion and abrasion processes.

Material and methods

The experimental design consisted of two study factors: the type of composite (two levels) – Z250 and P90;

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erosion/abrasion process (four levels) – immersion in artificial saliva at pH 7 (no erosion), immersion in orange juice (OJ) (Minute Maid, Cia de Bebidas Ipiranga, Ribeirão Preto, São Paulo, Brazil) at pH 3.77, Smirnoff Ice (SI) (Viti Vinícula Cereser Ltd., Jundiaí, São Paulo, Brazil) at pH 3.19, Gatorade (GA) (Ambev, Jaguariuna, Brazil) with pH 3.24. Then, all samples were brushed with Colgate Luminous White dentifrice (Colgate Palmolive Industrial Ltd., São Bernardo do Campo, SP, Brazil). The variable response was the quantitative analysis of the composite's surface microhardness using the Knoop hardness number (KHN) after the samples were submitted to the erosion and abrasion processes.

Eighty samples (40 MC and 40 SC) were made using a triangular sample silicon matrix with each side measuring 5 mm and 2 mm deep. The matrix was filled with one increment of either MC (Filtek Z250, 3 M ESPE, St. Paul, MN) or SC (Filtek P90, 3 M ESPE, St. Paul, MN), according to the experimental group. Photoactivation was carried out by a 600 mW/cm^2 light curing unit with a halogen lamp XL 2500 (3 M ESPE, St. Paul, MN) for 20 s for the MC groups and 40 s for the SC groups, according to the manufacturer's recommendations. The materials used in the study are shown in Table 1.

The samples were randomly divided into eight experimental groups ($n = 10$) according to the composite used. Groups 1–4 (MC) and groups 5–8 (SC) were treated as follows: G1 and G5 – control (C), without erosion but with abrasion; G2 and G6 – OJ and abrasion; G3 and G7 – SI and abrasion; G4 and G8 – GA and abrasion. Then, a double-face adhesive tape was placed on a glass slab, where 10 samples of the same group were inserted in a circular format. A PVC cylindrical tube of 3 cm in diameter was placed around the samples, such that all the samples were inside the cylindrical tube, which was then filled with polyester resin. Then, the samples were polished with 600, 1200 and 2500-grit silicon carbide paper (3 M ESPE, St. Paul, MN) using a water-cooled mechanical grinder (Aropol 2 V, Arotec, Cotia, SP, Brazil).

Each beverage used for the erosion process had its pH measured by a digital pH meter (Digimed DM 21, São Paulo, SP, Brazil), which had been previously calibrated with standard solutions of pH 7 and pH 4. The pH of the beverage was evaluated immediately after the recipient was opened.

For the erosion cycles, the samples of groups 2, 3, 4, 6, 7 and 8 were immersed in 120 ml of an acid beverage, for 2 minutes, statically. Then, the samples were washed with distilled water and immersed in 120 ml of artificial saliva for 60 min, then submitted to the abrasion process.[9] The abrasion process was performed by the same operator who simulated a manual brushing with 40 repetitions on each sample. Then, the sample was placed upon a plane surface, using a metal device, with a central space to hold the brush handle so that the bristles of the brush were overlying and perpendicular to the sample throughout the whole brushing process. The samples were brushed with 100 ml slurry prepared with a dentifrice (Colgate Luminous White, Colgate Palmolive Industrial Ltd., São Bernardo do Campo, São Paulo, Brazil) and artificial saliva in the proportion of 1:3.[9] The erosion/abrasion cycles were performed at room temperature three times a day for 5 days. During the intervals between cycles, all experimental groups were stored in artificial saliva.

After 24 h at the end of all cycles, the samples were measured using a microhardness tester (HMV-2, Shimadzu, Tokyo, Japan). Three indentations spaced 100 μm apart were made in the center of each sample, at a load of 200 g and an indentation time of 10 s, and the arithmetic mean of the measurements corresponded to the microhardness value of each sample. Data were statistically analyzed by Instat software, version 5.0.1(SAS Institute Inc., Cary, NC), and *two way* ANOVA, according to the two types of resin composite and the erosion/abrasion cycles of the different acid beverages and the abrasive dentifrice.

Results

The KHN results (Table 2) showed the highest means ($p < 0.001$) for the Z250 (MC) groups compared to the P90 (SC) groups. For the Z250 groups, G3 (SI) showed the lowest mean compared with those of G1 (C) ($p = 0.003$) and G2 (OJ) ($p < 0.001$), which presented the highest means. The G4 group

Table 1. Materials used in the study.

Material	Composition	pH	Manufacturer
Filtek Z250	Bis-GMA*, Bis-EMA (6)*, UDMA* and TEGDMA*. Zirconia and silica – 78.5%	–	3 M – ESPE, St. Paul, MN, USA
Filtek P90	Silorane (3,4-epoxy cyclohexylethyl siloxane,bis-3,4-epoxy cyclohexylethyl phenylmethyl silane). Quartz and yttrium – 76% – 0.47 μm	–	3 M – ESPE, St. Paul, MN, USA
Orange Juice (OJ)	Contains pure filtered water, premium concentrated orange juice, sugar, natural flavor, citric acid, ascorbic acid.	3.77	Minute Maid, Cia de Bebidas Ipiranga, Ribeirão Preto, SP, Brazil
Smirnoff Ice (SI)	4.5% alc/vol, water, sugar, citric acid, tartaric acid, sodium citrate, malic acid, sodium benzoate.	3.19	Viti Vinícula Cereser Ltd., Jundiaí, SP, Brazil
Gatorade (GA)	Water, saccharose, glucose, sodium chloride, sodium citrate, monobasic potassium phosphate, citric acid, flavorant.	3.24	Ambev, Jaguariuna, SP, Brazil
Colgate Luminous White	Aqua, hydrated silica, sorbitol, glycerin, pentasodium triphosphate, PEG-12, tetrapotassium pyrophosphate, sodium lauryl sulfate, aroma, cellulose gum, cocamidopryl betane, xanthan gum, sodium saccharin, sodium hydroxide, sodium fluoride (1100 ppm), RDA = 175.	–	Colgate Palmolive Industrial Ltd., São Bernardo do Campo, SP, Brazil

*Bis-GMA: bisphenol glycidyl methacrylate, TEGDMA: triethyleneglycol dimethacrylate, UDMA: urethane dimethacrylate, Bis-EMA: bisphenol ethoxylate dimethacrylate. Bis-EMA (6) is analogous to Bis-GMA; however, the former has six ethylene oxide units substituting the two hydroxyl groups between the aromatic backbone and the insaturates.

Table 2. Knoop hardness (KHN) means (standard deviations) in function of material and treatment.

Material	Control (G1–C)	Orange Juice (G2–OJ)	Smirnoff Ice (G3–SI)	Gatorade (G4–GA)
Filtek Z250	88.6 (9.6) Aa	89.7 (5.0) Aa	78.4 (9.4) Ba	83.6 (8.4) ABa
Filtek P90	55.6 (2.1) Ab	54.4 (3.7) Ab	54.9 (2.5) Ab	59.3 (2.3) Ab

Within the same material, means marked with the same capital letter were not significantly different. Within the same treatment, means marked with the same case letter were not significantly different.

(GA) presented an intermediary microhardness mean similar to the others. On the other hand, the means of all P90 groups showed similar KHN means with no significant difference among them ($p > 0.05$).

Discussion

In the present study, G3 (SI) was the only treatment that yielded a KHN value that decreased after the erosion/abrasion cycles. The alcoholic drinks (SI) contain 4.5% vol of alcohol in its composition, which may contribute to a reduction in microhardness. The ethanol penetrates into the MC resin matrix leading to an expansion of the polymeric chains, and the unreacted monomer may diffuses into the medium,[10,11] followed by a decrease of the resin mechanical properties. Furthermore, SI beverages contain acidulants, such as tartaric acid and citric acid, which lead to a low pH, and stimulates the matrix softening by the erosion process.

In addition, toothbrushing has an influence on enamel and composite resin wear, mainly by the dentifrice's abrasiveness.[12] In the present study, a high abrasive dentifrice, containing tetrapotassium pyrophosphate, hydrated silica and pentasodium triphosphate (Colgate Luminous White, Colgate Palmolive Industrial LTDA, São Bernardo do Campo, Brazil), was used in the manual brushing process. The bleaching dentifrice have a high abrasiveness, and their association with the acidic beverages can lead to dental wear.[9] However, G4 (GA) presented an intermediate KHN mean and G2 (OJ) did not differ from the control group. In the present study, the samples were immersed into the acidic beverages for a short period of time, which may have contributed to the methacrylate composite not showing a significant decrease in microhardness, when ethanol was not present in the beverage.

Hamouda [7] noted that KHN decreased after the methacrylate composite was exposed to six different beverages containing citric acid with pH between 2.85 and 3.49. These findings suggest that the ingestion of acidic beverages can promote composite softening, leading to the materials' surface degradation, which may directly interfere with restoration longevity. In addition, Gomec et al. [8] also reported a KHN decrease in the methacrylate composite that was exposed to erosion cycles. In contrast, Yu et al. [13] related that there was no significant difference between the KHN values of silorane and methacrylate composite after erosion/abrasion cycles. In another study, Wongkhantee et al. [14] found no KHN differences in the composite Filtek Z250 after erosion cycles with various acidic beverages, and the methodological differences in this study also may explain these findings. This is because the author performed 10 erosion cycles of 5 s each, which is less exposure time compared to that of the other studies.

In the present study, the KHN means of SC were lower than those of MC. These findings corroborates those from previous studies [15,16] and this result could be correlated to various aspects, such as resin matrix differences between MC and SC, their photopolymerization process, or even their polymeric resilience.[16] However, the SC groups submitted to erosion/abrasion cycles maintained their KHN means, regardless of the beverage used. It is like that the silorane composites were more resistant to the erosion process promoted by the acidic beverages, as well as the elution actions of alcohol, when compared to the methacrylate composites.

According to Tistrou et al. [17] SC did not present eluted monomers in alcoholic medium when compared with a MC, but the authors suggested that this could be possible. Nevertheless, Kopperud et al. [18] evaluated the substance elution from a SC immersed in water or ethanol solution and detected that silorane monomers eluted only in the ethanol medium. The authors also reported that the SC has a lower polarity than that of MC, which may influence its water sorption and solubility. Clinically, it is relevant to state that composites, mainly the silorane composite, presented the highest durability under the erosion and/or abrasion processes, with lower structure loss when compared to the enamel surface and the polyacid-modified composite or the conventional ionomer cement restorations.[13]

The diversity of results from the studies previously described may be due to differences in each methodology such as samples immersion in acidic solutions for a longer period of time [7,8] or even submission to only a slight erosion cycle.[14] The lack of standard processes inspired an article that evaluated many exposure times, between 3 and 30 min of immersion in acidic beverages, with significant differences in the results.[19] The present study was performed with the erosion/abrasion cycles suggested by Engle et al. [9], due the similarity of the erosion/abrasion challenges that would be found in a clinical behavior. In order to simulate the patients' regular ingestion of beverages, the erosion cycle was performed with the samples immersed in an acid solution three times/day for 2 min each. After that, the samples were immersed in artificial saliva for 60 min before brushing with a high abrasive dentifrice.

Considering the limitations of this *in vitro* study, the authors concluded that methacrylate composite had higher KHN than silorane composite, regardless of the abrasion/erosion process. However, methacrylate composite when submitted to the acidic beverages' erosion challenge combined with the abrasion process, showed an alteration in its surface microhardness. Additionally, the beverages used in the present study were not able to interfere in silorane composite surface microhardness.

Acknowledgements

Declaration of interest

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