

A Comparative Effect of Mouthwashes with Different Alcohol Concentrations on Surface Hardness, Sorption and Solubility of Composite Resins

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Abstract

The longevity and durability of composite resins are influenced by the actions of water, saliva, drinks, food and features of the oral environment.

Objective: The aim of this study was to evaluate the effect of mouthwashes containing alcohol on the surface hardness, sorption and solubility of composite resins.

Methods: Disc-shaped specimens were prepared with two composite resins Z250 (Z2) and Z350XT (Z3). Measurements of Vickers hardness were performed before and after immersion in Plax, PerioGard, Listerine, ethanol and distilled water for 12 h at 37°C, followed by a further 12 h at 37°C in artificial saliva. Sorption and solubility were performed according to ISO 4049. Data were analysed using one-way ANOVA and Tukey tests ($\alpha=0.05$).

Results: None of the mouthwashes significantly reduced the hardness of the resin Z2 ($p>0.05$). The greatest change in resin Z3 hardness was produced by PerioGard ($p<0.01$). Plax produced the lowest changes in the sorption and solubility of resins Z2 and Z3 ($p<0.01$), followed by Listerine and PerioGard.

Conclusions: The sorption and solubility properties of the composite resins were more altered by mouthwashes than the surface hardness.

Key words: Composite resins, Mouthwashes, Hardness tests, Absorption, Solubility, Introduction

Introduction

Health education is a priority for dental professionals and has an important role in prevention of oral health problems [1]. However, despite the emphasis on brushing and flossing, the prevalence of dental caries and periodontal disease remains high [2] and, for selected patients, chemotherapeutic agents in the form of mouthwashes may be indicated.

Mouthwashes are usually recommended for consumers to reduce halitosis, prevent, control dental caries and periodontal diseases [3,4]. Although mouthwashes are effective in reducing plaque induced gingivitis and providing fluoride to prevent dental caries, some studies have addressed the risks associated with the daily use of mouthwashes. These risks include dry mouth [5], an increase in the incidence of head and neck cancer [6], extrinsic pigmentation [5], and increase degradation of composite resin restorations [7].

The use of composite resin has increased in recent years and the development of new formulations has broadened the indications of adhesive restorative procedures [8]. Nevertheless, the replacement of unsatisfactory restoration remains the most frequently performed restorative work [9].

The longevity and durability of dental composite resins are substantially influenced by the characteristics of the oral environment [3,10] and the presence of degradation in composite restorations. As degradation also occurs in areas that are unexposed to abrasion and compression, chemical degradation must be present [11]. Water, saliva [12,13], drinks and food [14,15] are some of the factors that can lead to the degradation of composite resins, and these factors can cause changes in the mechanical properties of the resins [7,16,17].

The reduction in surface hardness can affect others

properties of the composites such as the wear resistance [18]. The decrease in wear resistance of any restorative material can result in increased roughness of these materials, which favour the accumulation of dental biofilm, pigments that lead to composite staining and premature failure of the restoration, requiring its replacement [17,19].

The alcohol concentration of mouthwashes influences the degradation of composite resins [3,7,17]. As a good dimethacrylate solvent, alcohol can dilate and soften the polymer matrix by increasing the amount of unreacted monomers and oligomers that diffuse out of the material [20]. Therefore, mouthwashes with alcohol may adversely affect the hardness, sorption and solubility properties of composite resins. However, studies on the relationship between alcohol and composite resin properties still remain contradictory [5,7,17,21]. The objective of this study was to evaluate the effect of mouthwashes with alcohol on the surface hardness, sorption and solubility of hybrid and nanoparticle resin composites.

Materials and Methods

The materials used in this study are described in *Table 1*. Two hundred twenty disc-shaped (5 mm diameter x 2 mm high) specimens of composite resin were prepared with the aid of a cylindrical matrix positioned between two strips of polyester matrix, and an axial load of 500 g was applied for 1 minute. Using the continuous conventional technique, the composites were irradiated for 20 seconds with a halogen light source (Optilux 400, Demetron Research Corporation, Danbury, CT, USA - 600 mW/cm²).

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Table 1. Composition and characteristics of the composite resins and solutions.

Material	Manufacturer	Composition	Alcohol Content	pH
Filtek Z250 [®] (Z2) hybrid	3M-ESPE, Dental Products, St. Paul, MN, US.	<i>Organic matrix:</i> bis-GMA, UDMA and bis-EMA. <i>Inorganic part:</i> zirconia/silica with 82% in weight (60% in volume).	-	-
Filtek Z350XT [®] (Z3) nanoparticle	3M-ESPE, Dental Products, St. Paul, MN, US.	<i>Organic matrix:</i> bis-GMA, UDMA, bis-EMA, TEGDMA and PEGDMA. <i>Inorganic part:</i> zirconia/silica with 78.5% in weight (63.3% in volume).	-	-
Plax [®]	Colgate-Palmolive Ind. Com. Ltd., São Bernardo do Campo, SP, Brazil.	Triclosan, sodium fluoride, PVM/MA copolymer (Gantex), alcohol, water, glycerine, sorbitol, sodium saccharine, sodium methyl taurate, sodium lauryl sulphate, sodium hydroxide, sodium phosphate, CI16035 and aroma.	8.7%	6.3
Listerine [®]	Johnson & Johnson Brazil Health Products Industry and Commerce Ltd., São José dos Campos, SP, Brazil.	Thymol, eucalyptol, methyl salicylate, menthol, water, alcohol, poloxamer 407, benzoic acid, sodium benzoate and caramel.	26.9%	3.9
PerioGard [®]	Colgate-Palmolive Ind. Com. Ltd., São Bernardo do Campo, SP, Brazil.	Chlorhexidine gluconate, water, glycerine, alcohol %, polysorbate 20, aromatic composition mint flavour, sodium saccharin, FD & C and Blue 1.	11.6%	5.2
Artificial saliva	-	KCl: 960 mg; NaCl: 674 mg; MgCl ₂ : 41 mg; K ₂ HPO ₄ : 274 mg; CaCl ₂ : 117 mg; Sorbitol: 24 g; Sodium carboxymethylcellulose: 8 g; Distilled water: q.s. 1000 mL.	-	6.9
Ethanol	Quimidrol Ind. Com. and Import Ltd., Joinville, SC, Brazil.	Ethanol, water.	92.8%	-

Bis-GMA: Bisphenol A Diglycidyl Methacrylate; Bis-EMA: Ethoxylated Bisphenol A Glycol Dimethacrylate; UDMA: Urethane Dimethacrylate; TEGDMA: Triethylene Glycol Dimethacrylate; PEGDMA: Polyethylene Glycol Dimethacrylate; KCl: Potassium Chloride; NaCl: Sodium Chloride; MgCl₂: Magnesium Chloride; K₂HPO₄: Dipotassium Phosphate; CaCl₂: Calcium chloride.

The specimens were stored in artificial saliva for 24 h at 37°C. The specimens were then ground on a water-cooled mechanical polisher (APL Arotec 4000, Arotec, Cotia, SP, Brazil) with 1200-grit silicon carbide (CSi) sandpaper for 30s. Finally, the specimens were polished with felt discs impregnated with 0.3 µm polishing diamond paste (Arotec, Cotia, SP, Brazil).

The specimens were immersed in Plax, Listerine and PerioGard mouthwashes and in ethanol (positive control) and distilled water (negative control). To simulate a year of mouthwash for 2 minutes per day, the specimens remained under constant stirring for 12 h at 37°C [21]. The specimens were thoroughly washed in water and stored in artificial saliva for 12 h at 37°C. The specimens were then washed in distilled water for 1 minute and dried with absorbent paper.

To measure pH, 20 mL of each mouthwash was placed in a beaker, and the pH was measured with a pHmeter (PROCYON AS720, Procyon Scientific Instrumentation Ltd., São Paulo, SP, Brazil). The pH value of each solution is shown in Table 1.

Vickers hardness

Sixty samples of each composite resin were prepared as described above and distributed in groups (n=10). Before (baseline) and after immersion in the solutions, Vickers hardness measurements were conducted with a Micro Hardness tester (HMV-2T, Shimadzu Corporation, Kyoto, Japan) by applying a load of 50 g for 10 s. The Vickers hardness number (VHN) for each depth was recorded as the average of three indentations.

Analysis of sorption and solubility

For the sorption and solubility tests, 50 samples of each composite resin were prepared as described above and randomly grouped (n=10). Each specimen was weighed on a precision analytical scale (AX200-Shimadzu Corporation, Kyoto, Japan) and transferred to a light-proof desiccator that contained dried silica gel (Fisher Scientific, Loughborough, Leicestershire, United Kingdom); each specimen remained on the desiccator for 22 h at 37 ± 1°C followed by 2 h at 23 ± 1°C. The specimens were re-weighed, and the conditioning cycle and drying were repeated until the weight loss was less than 1 × 10⁻³ g (ml). After the weight was stabilized, each specimen was measured with a digital micrometre model Digimes 110-250 (Digimes Precision Instruments Ltd., São Paulo, São Paulo, Brazil) to calculate the volume (V) in mm³. The specimens were then suspended individually by their diametric axis in 2 mL of distilled water, ethanol and mouthwashes under the same experimental conditions described above.

After each period of immersion in the solutions, the samples were removed from the flasks, dried with absorbent paper and kept at room temperature for 15 s to be re-weighed (m₂). The samples were then subjected to the conditioning cycle for drying until the weight loss was less than 1 × 10⁻³ g. The samples were weighed for a final time (m₃).

In µg/mm³, the sorption and solubility values of each specimen were calculated using equations 1 and 2:

$$(1) \text{ Sorption} = m_2 - m_3/V$$

$$(2) \text{ Solubility} = m1 - m3/V,$$

where

$m1$ = mass after initial specimen drying (μg),

$m2$ = mass after immersion in artificial saliva (μg),

$m3$ = final mass after drying (μg) and

V = volume in mm^3 .

Statistical analysis

The data were analysed with F-tests for one-way ANOVA and Tukey paired comparisons ($\alpha=0.05$). The software SPSS (Statistical Package for the Social Sciences) version 17 was used for all analyses.

Results

The average Vickers hardness values of the resins Z2 and Z3 are shown in *Table 2*. Comparing the results of hardness, sorption (*Table 3*) and solubility (*Table 4*), there were no statistically significant differences between the two composites tested ($p>0.05$). Overall, the resin Z2 showed lower hardness and higher sorption and solubility than the resin Z3 ($p>0.05$). Compared to distilled water, none of the mouthwashes significantly reduced the Vickers hardness of the resin Z2 ($p>0.05$).

PerioGard significantly reduced the surface hardness of Z3 ($p<0.01$) and increased the sorption ($p<0.01$) and solubility ($p<0.01$) of both resins. There were no statistically significant differences in the hardness and sorption of the resin Z3 between the PerioGard and ethanol groups ($p>0.05$).

In the Listerine group, there was a significant increase in solubility in the Z2 and Z3 resins ($p<0.01$). The values of sorption of Z2 showed no significant alterations for the Plax and Listerine groups ($p>0.05$). There were no significant differences in the solubility of the Z3 resin between the Plax and Listerine groups ($p>0.05$) or between the Listerine and PerioGard groups ($p>0.05$).

The resins immersed in Plax had the lowest recorded changes in the sorption and solubility properties. Resins Z2 and Z3 immersed in Plax showed significantly higher sorption and solubility compared to the resins immersed in distilled water ($p<0.01$). The specimens of the two composite resins immersed in ethanol showed the lowest hardness values and the highest values of sorption and solubility ($p<0.01$).

Discussion

Alcohol is a good polymer chain solvent, and solutions with high alcohol concentrations can degrade the mechanical properties and increase the wear of composite resins [22]. The findings of the present study are consistent with previous studies [17,18] that have tested mouthwashes with similar alcohol concentrations. Plax and Listerine mouthwashes caused no significant change in the hardness of micro-hybrid (Z2) and nanoparticle (Z3) composite resins. Although, the alcohol content of PerioGard (11.6%) is lower than that of Listerine (26.9%) but PerioGard was the only mouthwash that significantly affected the hardness of the resin Z3, which contradicts the results of a previous study [17]. These findings suggest that changes in the hardness of the composite resin are not strongly dependent on the alcohol concentration of the mouthwash.

The affinity of the composite resin matrix for dyes is modulated by properties such as the degree of conversion and sorption [23]. Previous studies [7,23,24] have reported that the colour of composite resin changes by the action of possible by-products of degradation of the polymer matrix, such as methacrylic acid, formaldehyde and specific molecules of methacrylates. These by-products result from physical-chemical reactions or the oxidation of residual monomers.

Both composite resins (Z2 and Z3) had visible color changes when immersed in PerioGard compared to the other

Table 2. Mean values (standard deviation) of the Vickers hardness of composite resins after immersion in the tested solutions.

	Baseline	Plax	Listerine	PerioGard	Ethanol	Distilled Water
Filtek Z250	82.98 (7.79) ^{Aa}	79.94 (8.48) ^{Aa}	83.37 (10.07) ^{Aa}	79.11 (12.59) ^{Aa}	60.82 (11.47) ^{Ba}	82.08 (4.37) ^{Aa}
Filtek Z350XT	83.97 (5.54) ^{Aa}	82.94 (11.17) ^{Aa}	83.87 (13.14) ^{Aa}	77.49 (7.81) ^{Ba}	66.13 (10.50) ^{Ba}	84.04 (8.68) ^{Aa}

Obs.: Different letters indicate statistically significant differences ($p<0.05$). Capital letters compare the solutions (horizontal), and lowercase letters compare the composite resins (vertical).

Table 3. Mean values (standard deviation) of sorption of composite resins after immersion in the tested solutions.

	Plax	Listerine	PerioGard	Ethanol	Distilled Water
Filtek Z250	15.76 (5.71) ^{Aa}	17.99 (4.47) ^{ABa}	21.78 (3.93) ^{BCa}	27.89 (5.69) ^{Ca}	5.18 (0.86) ^{Da}
Filtek Z350XT	11.97 (2.88) ^{Aa}	15.31 (2.19) ^{Ba}	20.08 (1.32) ^{Ca}	22.80 (2.86) ^{Ca}	4.77 (1.65) ^{Da}

Obs.: Different letters indicate statistically significant differences ($p<0.05$). Capital letters compare the solutions (horizontal), and lowercase letters compare the composite resins (vertical).

Table 4. Mean values (standard deviation) of solubility of composite resins after immersion in the tested solutions.

	Plax	Listerine	PerioGard	Ethanol	Distilled Water
Filtek Z250	6.43 (2.03) ^{Aa}	8.63 (0.71) ^{Ba}	9.10 (1.41) ^{Ba}	9.30 (1.25) ^{Ba}	4.19 (1.20) ^{Ca}
Filtek Z350XT	5.81 (1.01) ^{Aa}	7.02 (1.86) ^{ABa}	8.77 (1.39) ^{Ba}	8.93 (2.21) ^{Ba}	3.95 (0.76) ^{Ca}

Obs.: Different letters indicate statistically significant differences ($p<0.05$). Capital letters compare the solutions (horizontal), and lowercase letters compare the composite resins (vertical).

mouthwashes, like Festuccia [17], who found color change in the composites immersed in the same mouth rinse solution. PerioGard contains chlorhexidine, which is a substance that can act as a catalyst in non-enzymatic browning (Maillard reaction). The glycoproteins that is present in the acquired pellicle act as a substrate for a series of reactions of condensation and polymerisation, and results in the formation of dark substances known as melanoidins [25]. Chlorhexidine can promote protein denaturation and produce staining related to the formation of ferric and stannous sulphide, which suggests another theory of staining related to chlorhexidine [26]. However according the study methodology we cannot attribute the color change of composite resins exclusively to the presence of chlorhexidine in the PerioGrad solution, once those results were from an in vitro study, and natural factors, such as saliva and the acquired pellicle, were not reproduced. Because this group exhibited the highest values of sorption and solubility, this behaviour could be due to the most chemical degradation in the resin matrix.

Water sorption in the composite resin is a controlled diffusion process that occurs primarily in the resin matrix [27]. When the composite resin is immersed in water, two different mechanisms occur. First, water sorption produces a mass increase via the accumulation of water molecules in micro-spaces at the interface between the filler and resin and in small morphological defects. This accumulation of water molecules can cause hygroscopic expansion, reduction in the mechanical properties such as colour changes, degradation of the filler/matrix combination, reduction of hardness and wear resistance [24,28-30]. Second, the leaching of components, such as particles or residual monomers, small polymer chains and particle ions, results in a loss of mass and characterises the phenomenon of solubility [24].

In the present study, the increasing values of sorption and solubility of composite resins did not directly correspond to the alcohol content of the mouthwashes. However, alcohol content is not the only factor that can lead to the modification of polymers [7,31]. Even mouthwashes without alcohol have shown to affect the hardness of restorative materials [23]. Alcohol is known to cause softening of the composite resin surface by removing monomers from the polymer structure. It also opens up the polymer structure that facilitates the diffusion of water and saliva that can lead to decrease in hardness, increase in material wear and change in other physical properties [20]. Therefore, alcohol has a clear influence on the hardness properties, sorption and solubility of composite resins, but the effect of alcohol does not happen by its own alone, there must be a simultaneous interaction of other factors that affect the physical properties of composite resins. Although Listerine has in its composition more than twice alcohol content of PerioGard, Listerine showed better

performance in the studied properties. One explanation may be that some ethanol was used for the emulsification of essential oils present in its composition, which would reduce the total amount of ethanol available.

The low pH of mouthwashes can also change the composite resin matrix by acting as a catalysis for the ester groups that are present in dimethacrylate monomers [7,24]. This process may cause degradation of the polymer network and lead to a phenomenon known as plasticization, which reduces the micro hardness of the composite resin [24]. However, the pH parameter only provides the initial concentration of H⁺ ions and does not represent the presence of undissociated acid in the medium [32].

The composition of the composite resins can interfere with the resistance to the action of chemicals, which may make the materials more or less susceptible to softening and degradation. In addition to the chemical composition, the chain type and crosslink density that are formed during the polymerization process [10] and the type and size of the filler particles [24] are also responsible for the resistance of the dental composite resin.

The uniform distribution of filler particles in the resin matrix is important for the performance of this material in humid environments as bubbles and voids at the filler/matrix interface can increase the amount of water absorption by the composite resin [33]. In aqueous and hydro-alcoholic solutions, differences in the composition of resins, such as the filler/matrix ratio, size and distribution of inorganic particles may affect hardness of the composite resin.

The salinization process and the homogeneous filling of the resin matrix by filler particles make the composite resin more resistant to hydrolysis [34]. The combination of nanoparticles for the formation of nanoclusters reduces the interstitial space of the filler particles, which increases the amount of composite loading and may improve the physical properties such as surface roughness, micro hardness, sorption and solubility compared to micro-particle or micro-hybrid composites [24].

Clinically, the effects of mouthwashes on the restorative material may be different. Clinical studies provide more accurate results as in vitro studies do not consider certain variables, such as natural saliva, food, drinks and the pH of the oral environment [3]; however, laboratory studies may suggest questions and direct future clinical studies.

Conclusion

The sorption and solubility properties of the composite resins were more altered by mouthwashes than the surface hardness. However, the alcohol content of the mouthwashes did not determine the recorded alterations. Nanoparticle and micro-hybrid resins exhibited similar performances.

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