

ORIGINAL ARTICLE

## *In vitro* long-term degradation of aesthetic restorative materials in food-simulating media

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### Abstract

**Objective.** The aim of this study was to evaluate the effect of long-term food-simulating media storage on degradation of restorative materials through roughness measurements. **Materials and methods:** Sixty cylindrical specimens of each material (Filtek Z250, Esthet X, Filtek Flow, Dyract AP and Vitremer) were prepared, stored for 24 h, and polished. The surface roughness analysis was conducted using Surfcoorder (SE1700) roughness-measuring instrument. Three traces were recorded on each specimen at three different locations. The specimens were randomly distributed into five groups ( $n = 12$ ) according to the storage media: (water, ethanol, Coca-Cola<sup>®</sup>, citric acid and lactic acid). Roughness measurements were recorded after 1 week, 1, 3 and 6 months. The storage solutions were weekly changed. Data were submitted to ANOVA and Tukey test ( $p < 0.05$ ). **Results.** There was no significant increase on roughness means for Filtek Z250, Filtek Flow and Vitremer over time, regardless the storage media. Significant increase on surface roughness was observed for Esthet X after 1 month of storage in Coca-Cola<sup>®</sup> and after 3 months of storage in citric acid and for Dyract AP after 6 months in water. There were no significant differences on surface roughness between resin composites and compomer. Resin-modified glass ionomer showed a significantly higher surface roughness mean than the other materials. **Conclusions.** Food-simulating media affects the surface roughness of the materials. Biodegradation is material-, solution- and time-dependent.

**Key Words:** biodegradation, composite resin, diet, polycid-modified composite resins, scanning electron microscopy

### Introduction

Long-term clinical performance of restorative materials is related to a number of factors, including biodegradation in the oral environment [1]. The chemical degradation can be caused by acid challenges, including those produced by the cariogenic biofilm [2], acidic diet (soft drinks and acidic beverages) [3–6] and salivary enzymes [7,8].

Studies have shown the effect of the chemical environment of the oral cavity on the degradation of resin-based materials [2–8]. The effects are surface softening [2,3,9] and roughening [3,10,11] that can decrease the long-term durability of the restorations. As a consequence, degraded restorations encourage plaque accumulation, which may result in gingival inflammation, superficial staining and secondary caries [12,13].

The chemical degradation is material- and solution-dependent. The nature of resin matrix and size, type and distribution of filler particles and the resin-filler coupling agent regulated the degradation of the resin-based materials [3,10]. Oral fluids can promote disintegration of the silane-coupling agent at the resin-filler interface what leaches the filler particles to the oral environment, reducing the physical properties of the material [13,14]. In addition, organic acids and various food and liquid constituents can soften the resin matrix [2,3,9].

Each solution will mainly degrade one component of the resin-based material. Alcohol solutions act on the matrix. According to Yap et al. [3], the Bis-GMA matrix is susceptible to the softening effect of the food simulating liquids. Moreover, the inorganic filler can be damaged by the water and weak intra-oral acids, as

citric and lactic acids [15]. Thus, the media to which restorative materials are exposed can exert great influence in their chemical degradation.

Numerous *in vitro* [16–18] and *in situ* studies [19,20] have demonstrated the erosive potential of soft drinks and fruit juices on the tooth structure [17,21,22]. However, little is known about the influence of dietary habits on the degradation of the restorations in the long-term.

Considering that resin-based materials such as conventional resin composites, flowable resins, comonomers and resin-modified glass-ionomers are commonly used in aesthetic restorations in children and adolescents, who in turn are major consumers of acidic drinks such as fruit juice and soft drinks [23,24], it is important not only to compare the performance of different restorative materials but also to estimate their chemical durability. Therefore, this study aims to evaluate the long-term effect of food-simulating liquids on degradation of resin-based materials through roughness measurements and Scanning Electron Microscopy (SEM) observation. The hypothesis tested is that materials will have different behaviour in the long-term, depending on the acidic storage solutions.

## Materials and methods

### Specimen preparation

Three resin composites (Filtek Z250, Esthet-X and Filtek Flow), one polyacid-modified resin composite

(Dyract AP) and one resin-modified glass ionomer cement (Vitremer) were selected for this study. The technical profiles of the materials are described in Table I.

Materials were handled according to the manufacturer's instructions and inserted in a single increment into a stainless-steel mold (4 mm in diameter × 2 mm thick). A Centrix syringe (Centrix Inc., Shelton, CT) was used to insert Vitremer, whereas syringes supplied by the manufacturers were used for Dyract AP and Filtek Flow and a metal spatula was used for the other materials.

Sixty specimens of each material were made at 23°C at 50% relative humidity. First, the mold was filled with the material; then a piece of paraffin dental floss was incorporated into the materials during setting/curing to suspend the samples in the test medium; after, a polyester strip (Probem Ltda, Catanduva, SP, Brazil) was placed over the mold and the excess of the material was pressured with 1 kg load over a glass plate of 2 mm thickness to extrude it. The restorative materials were photoactivated for the recommended exposure times through the polyester strip using the photocuring unit Elipar Tri-light (ESPE–America Co., Seefeld, Germany) with a power density of 800 mW/cm<sup>2</sup>.

After polymerization, specimens were individually stored for 24 h at 37°C at 100% relative humidity. Vitremer specimens were protected using Finishing Gloss, supplied by the manufacturer, to avoid water sorption during the first 24 h. Thereafter, specimens were submitted to standard finishing and polishing

Table I. Technical profiles and manufacturers of the materials evaluated.

Material	Category	Composition*	Mean filler size (µm)*	Manufacturer
Filtek Z250	Hybrid resin composite	Bis-GMA, Bis-EMA, UDMA, Inorganic filler – Zirconia/silica (60 vol. %); Photoinitiator	0.60	3M/ESPE Dental Products (St. Paul, MN)
Esthet-X	Hybrid resin composite	Bis-GMA, TEGDMA, barium-fluoro-alumino-silicate glass, silica, initiators/stabilizers	<1, glass 0.04, silica	Dentsply Indústria e Comércio Ltda (Petrópolis, RJ, Brazil)
Filtek Flow	Flowable composite	Bis-GMA, TEGDMA, dimetacrylate polymer, Inorganic filler – Zirconia/silica (47 vol%); Photoinitiator	0.60	3M/ESPE Dental Products
Dyract AP	Polyacid-modified resin composite	Cetylamine hydrofluoride acetone; UDM resin TCB resin; Polymerizable resins; Strontium fluoro-silicate glass (47 vol%); Strontium fluoride; Initiators/stabilizers	0.80	Dentsply Indústria e Comércio Ltda
Vitremer	Resin-modified glass-ionomer cement	Powder: fluoroaluminosilicate glass, redox catalyst system, pigments Liquid: aqueous solution of a polycarboxylic acid modified with pedant methacrylate groups, Vitrebond copolymer, water, HEMA, photoinitiators Primer: Vitrebond copolymer, HEMA, ethanol, photoinitiators	6.25**	3M/ESPE Dental Products

\*As disclosed by the manufacturers; \*\*According to Gladys et al. [28].

procedures, using medium, fine and superfine aluminum oxide abrasive disks, Sof-Lex (3M Dental Products, St Paul, MN) mounted in a low-speed hand piece (Kavo, Joinville, SC, Brazil). Each instrument was applied in a single direction for 15 s. After the finishing and the polishing steps, specimens were flushed with air-water spray and ultrasonically cleaned (Ultrasonic Cleaner, Model USC1400, UNIQUE Ind. e Com. Ltda., São Paulo, SP, Brazil) in distilled water for 10 min to remove polishing debris.

#### *Baseline surface roughness measurements*

Each specimen was gently dabbed dry with absorbent paper and the surface roughness analyses conducted using the SurfCorder SE1700 (Kosaka Corp., Tokyo, Japan) surface roughness-measuring instrument equipped with a diamond needle of 2- $\mu$ m radius. To record roughness measurements, the needle moved at a constant speed of 0.5 mm/s with a load of 0.7 mN. The cut-off value was set at 0.25 mm to maximize filtration of surface waviness [11]. The surface roughness was characterized by the average roughness (Ra) that is the arithmetical average value of all absolute distances of the roughness profile from the centerline within the measuring length. Ra values for each specimen were taken across the diameter over a standard length of 0.25 mm. Three traces were recorded on each specimen at three different locations—parallel, perpendicular and oblique to the finishing and the polishing scratch directions. The average of these three traces was used as the value for each specimen.

#### *Storage in acidic solutions*

Each group of 60 specimens was randomly subdivided into five groups ( $n = 12$ ) and stored at 37°C as follows: Group 1, distilled water; Group 2, 0.1 M lactic acid; Group 3, 0.1 M citric acid; Group 4, Coca-Cola®; Group 5, 50% ethanol-water solution [3]. The materials were individually immersed into 3 ml of these solutions immediately after the baseline surface roughness measurement and evaluated for surface roughness after 1-week, 1-, 2- and 6-months storage periods. During the immersion period, the specimens remained suspended by the dental floss in order to not touch the recipient walls, which guaranteed that all the surfaces were in contact with the solution. The specimens were lightly rinsed in water and gently dabbed dry with absorbent paper before each measurement, which was carried out as described before for the baseline surface roughness measurement. Solutions were changed weekly.

The pH values of each storage solution were determined using a pH meter (Orion Model 420A, Analyzer Com. e Ind. Ltda., São Paulo, SP, Brazil) immediately before the immersion of the specimens

and repeated after each roughness measurement. The pH values of the solution were 5.5 for distilled water, 5 for citric acid, 5 for lactic acid, 2.6 for Coca-Cola® and 7 for 50% ethanol-water solution.

#### *SEM evaluation*

Polished specimens and representative specimens of each material on each solution after 6 months storage were subjected to SEM (JEOL- JSM 5600LV, Tokyo, Japan) evaluation at  $\times 3000$  magnification to determine micro-structural surface alterations.

#### *Statistical analysis*

Analysis of variance with repeated measures (ANOVA) was carried out on roughness data, with restorative materials, storage solution and time as main effects and all possible combinations of these variables as interaction effects in the ANOVA model, with repeated measures, followed by Tukey-Kramer's test to carry out pairwise comparisons among these factors at 5% of significance. Statistical analysis was carried out by SAS/LAB (SAS Institute Inc. The SAS system release 8.2, software SAS/LAB. SAS Institute Inc, Cary, NC, 1999).

## **Results**

Results of surface roughness are shown in Table II and Figures 1 and 2. ANOVA showed significant difference for materials, storage solutions and time ( $p < 0.000,01$ ) and for the interactions among these factors ( $p < 0.000,01$ ). As the interactions were significant, a comparison of the behavior under each storage condition and also the effect of different storage regimes on each material over time could be made. Within the material factor, significant difference was found between Vitremer and the other materials ( $p = 0.0024$ ). Vitremer showed the most significant rough surface on baseline, maintaining this behavior over time. No significant differences were observed for Ra values of Filtek Z250, Filtek Flow, Esthet X and Dyract AP on baseline ( $p = 0.087$ ) (Table II).

There was no significant increase on surface roughness over time for Filtek Z250, Filtek Flow and Vitremer, regardless the storage solution ( $p = 0.069$ ). There was a significant increase in surface roughness of Esthet X after 1 month of storage in Coca-Cola® ( $p = 0.034$ ) and the roughness values continue to significantly increase after 3 and 6 months (Figure 1). Esthet X after 3 months of storage in citric acid showed a significant increase on surface roughness, which was maintained after 6 months ( $p = 0.039$ ) (Figure 1). Dyract AP showed a significant increase on surface roughness after 6 months of storage in water ( $p = 0.041$ ) (Figure 2).

Table II. Means (SD) of roughness (Ra,  $\mu\text{m}$ ) for restorative materials stored in different solutions over time.

Time	Storage media	Material									
		Filtek Z250		Esthet-X		Filtek Flow		Dyract AP		Vitremer	
Baseline	Water	0.11 (0.02)	Aa	0.15 (0.04)	Aa	0.19 (0.02)	Aa	0.18 (0.05)	Aa	0.35 (0.13)	Ab
	Lactic acid	0.17 (0.05)	Aa	0.17 (0.03)	Aa	0.17 (0.02)	Aa	0.13 (0.03)	Aa	0.36 (0.09)	Ab
	Citric acid	0.17 (0.07)	Aa	0.18 (0.05)	Aa	0.16 (0.01)	Aa	0.12 (0.03)	Aa	0.34 (0.11)	Ab
	Coca-Cola®	0.11 (0.04)	Aa	0.14 (0.06)	Aa	0.20 (0.03)	Aa	0.24 (0.10)	Aa	0.39 (0.15)	Ab
	50% ethanol	0.17 (0.09)	Aa	0.13 (0.02)	Aa	0.17 (0.02)	Aa	0.22 (0.06)	Aa	0.35 (0.10)	Ab
1 week	Water	0.15 (0.09)	Aa	0.19 (0.18)	Aa	0.18 (0.02)	Aab	0.14 (0.02)	Aa	0.32 (0.13)	Ab
	Lactic acid	0.19 (0.14)	Aa	0.14 (0.02)	Aa	0.17 (0.07)	Aa	0.13 (0.04)	Aa	0.33 (0.11)	Ab
	Citric acid	0.17 (0.06)	Aa	0.19 (0.06)	Aa	0.16 (0.05)	Aa	0.16 (0.03)	Aa	0.47 (0.15)	Ab
	Coca-Cola®	0.13 (0.03)	Aa	0.23 (0.10)	Aa	0.20 (0.08)	Aa	0.22 (0.09)	Aa	0.40 (0.13)	Ab
	50% ethanol	0.17 (0.05)	Aa	0.22 (0.10)	Aa	0.19 (0.02)	Aa	0.19 (0.07)	Aa	0.41 (0.12)	Ab
1 month	Water	0.16 (0.06)	Aa	0.21 (0.09)	ABab	0.19 (0.02)	Aa	0.17 (0.07)	Aa	0.35 (0.10)	Ab
	Lactic acid	0.20 (0.11)	Aa	0.13 (0.01)	Aa	0.17 (0.03)	Aa	0.18 (0.06)	Aa	0.39 (0.12)	Ab
	Citric acid	0.17 (0.04)	Aa	0.16 (0.06)	Aba	0.17 (0.03)	Aa	0.17 (0.04)	Aa	0.46 (0.16)	Ab
	Coca-Cola®	0.14 (0.03)	Aa	0.26 (0.11)	Bb	0.21 (0.02)	Aab	0.20 (0.07)	Aab	0.36 (0.11)	Ab
	50% ethanol	0.18 (0.08)	Aa	0.22 (0.07)	ABab	0.20 (0.07)	Aa	0.20 (0.09)	Aa	0.38 (0.11)	Ab
3 months	Water	0.16 (0.07)	Aa	0.15 (0.07)	Aa	0.22 (0.05)	Aab	0.18 (0.07)	Aa	0.38 (0.16)	Ab
	Lactic acid	0.18 (0.03)	Aa	0.17 (0.04)	ABa	0.22 (0.05)	Aa	0.17 (0.05)	Aa	0.59 (0.27)	Ab
	Citric acid	0.18 (0.03)	Aa	0.31 (0.22)	CBa	0.20 (0.03)	Aa	0.17 (0.04)	Aa	0.50 (0.19)	Ab
	Coca-Cola®	0.15 (0.06)	Aa	0.34 (0.21)	Cb	0.23 (0.04)	Aab	0.18 (0.05)	Aab	0.56 (0.22)	Ac
	50% ethanol	0.19 (0.08)	Aa	0.19 (0.12)	ABCa	0.20 (0.02)	Aa	0.22 (0.07)	Aa	0.42 (0.14)	Ab
6 months	Water	0.15 (0.04)	Aa	0.15 (0.06)	Aa	0.17 (0.03)	Aab	0.30 (0.17)	Abc	0.35 (0.16)	Ac
	Lactic acid	0.16 (0.04)	Aa	0.23 (0.11)	ABa	0.18 (0.04)	Aa	0.19 (0.04)	Aa	0.57 (0.25)	Ab
	Citric acid	0.17 (0.05)	Aa	0.32 (0.19)	BCab	0.16 (0.01)	Aa	0.19 (0.07)	Aa	0.48 (0.16)	Ab
	Coca-Cola®	0.19 (0.10)	Aa	0.40 (0.13)	Cb	0.19 (0.02)	Aa	0.24 (0.10)	Aab	0.40 (0.15)	Ab
	50% ethanol	0.23 (0.04)	Aab	0.17 (0.07)	Aa	0.17 (0.01)	Aa	0.32 (0.09)	Abc	0.42 (0.12)	Ac

Statistical differences are expressed by upper case letters in columns (within each time interval) and by lower case letters in rows ( $p < 0.05$ ).

Within the storage solution factor, statistical differences were not observed over time in lactic acid and 50% ethanol-water solution storage, regardless of the

material ( $p = 0.756$ ). Coca-Cola® led to an increase on Ra values only for Esthet X after 1 month ( $p = 0.032$ ). Citric acid storage increased Ra values

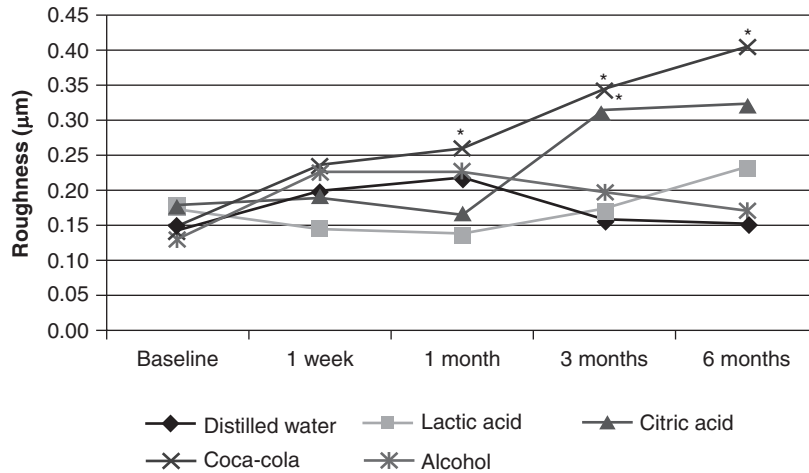


Figure 1. Means of roughness (Ra –  $\mu\text{m}$ ) for Esthet X stored in different solutions over time. Statistical differences are expressed by \* over the mean value in different periods.

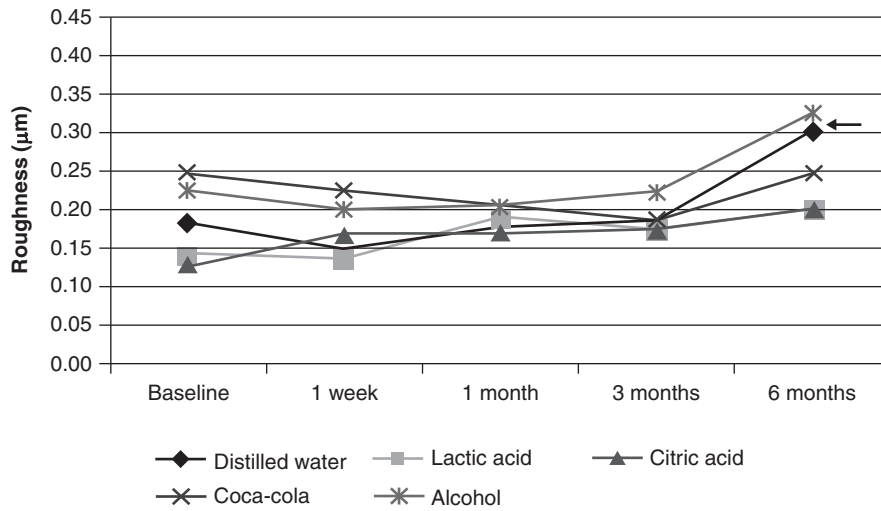


Figure 2. Means of roughness (Ra - μm) for Dyract AP stored in different solutions over time. Statistical differences are expressed by an arrow over the mean value in different periods.

only for Esthet X after 3 months ( $p = 0.040$ ) and water storage increased Ra values only for Dyract AP after 6 months ( $p = 0.041$ ).

Figures 3–7 show the SEM micrographs of Esthet X and Dyract AP. Micrographs of polished Esthet X and Dyract AP surfaces are shown in Figures 3–6, respectively. The Esthet X specimen at 6 months stored in citric acid shows that a generalized roughening occurred with formation of pits on the material surface (Figure 4). Protrusion of filler particles can be observed in Figure 5, which shows a Esthet X surface after 6 months in Coca-Cola®. Figure 7 shows a Dyract AP surface after 6 months in water, a generalized roughening was observed.

**Discussion**

The roughness of intra-oral hard surfaces (teeth or restorative materials) had a major impact on the retention of oral micro-organisms, increasing the

risk for caries and periodontal inflammation [12]. In addition, a rough surface affects the teeth and restorative material wear [25] and increases the susceptibility to staining [13,26,27].

According to Bollen et al. [12], the roughness of all intra-oral hard surfaces should approximate a Ra value of 0.2 μm or lower to reduce bacterial retention. In this study, the resin-based materials Filtek Z250, Filtek Flow, Esthet X and Dyract AP showed initial Ra values lower than or very close to (Dyract) 0.2 μm (baseline, Table II). These materials showed no significant difference on mean surface roughness, corroborating with Gladys et al. [28] and Turssi et al. [10]. Vitremer was the roughest material, showing Ra values higher than 0.2 μm in baseline, maintaining a higher roughness over time, regardless of the storage solution, which concurs with el-Kalla and Garcia-Godoy [29]. The differences among materials regarding their means of

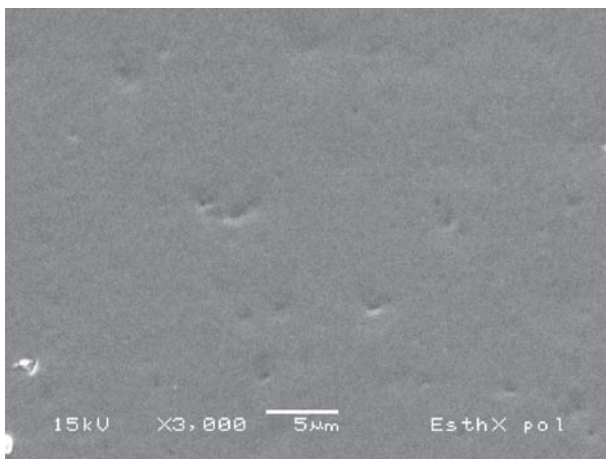


Figure 3. Scanning Electron Micrograph of the polished surface of Esthet X.

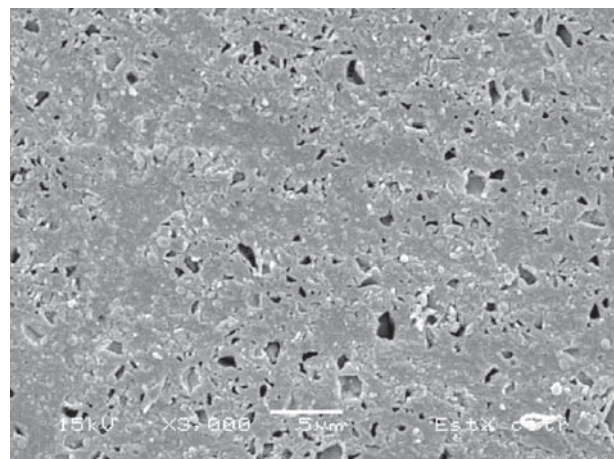


Figure 4. Scanning Electron Micrograph of Esthet X after 6 months of storage in citric acid.

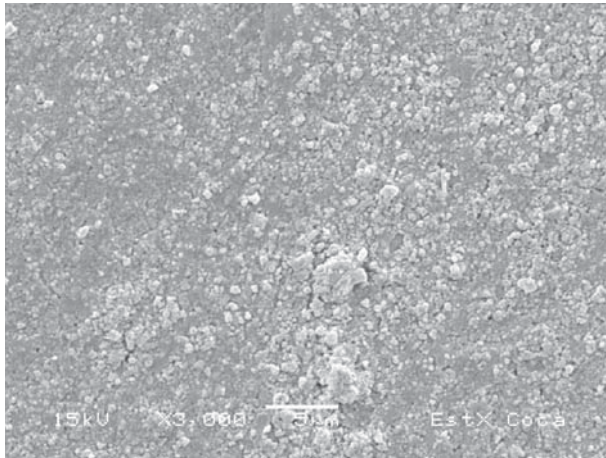


Figure 5. Scanning Electron Micrograph of Esthet X after 6 months of storage in Coca-Cola.

surface roughness on baseline are mainly related to differences in their filler particles size, shape, volume and distribution [28].

Roughening can be a consequence of the chemical dissolution of resin-based materials by the exposure of the surfaces to chemicals from drinks, food, microorganisms and saliva [2–8,25]. Some of the food-simulating liquids used to store the restorative materials in this investigation are among those recommended in FDA Guidelines to be used as food simulators. The ethanol-water solution and the citric acid simulate certain beverages, including alcohol, and vegetables, fruits, candy and syrup. Distilled water was included to simulate the wet intra-oral environment. Lactic acid is the main acid produced by the plaque microorganisms and is a product of the *Lactobacillus* fermentation process in fermented milk beverages. Coca-Cola® was selected because it is the soft drink frequently consumed by the youth population.

In this study, most of the materials were not affected by the food-simulating media storage.

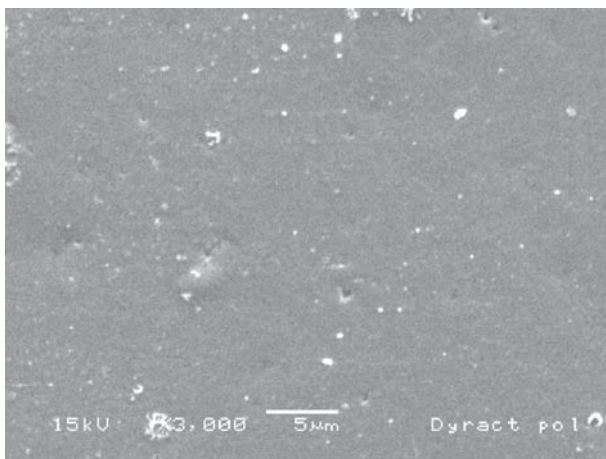


Figure 6. Scanning Electron Micrograph of polished surface of Dyract AP.

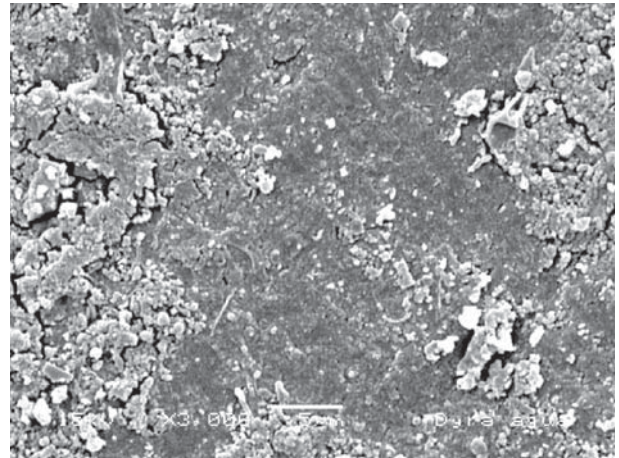


Figure 7. Scanning Electron Micrograph of Dyract AP after 6 months of storage in distilled water.

However, the long-term storage of resin-based restorative materials upon the acidic media trended toward an increase in surface roughness over time, especially for Esthet X, Dyract AP and Vitremer.

Composite resins Filtek Z250 and Filtek Flow had a similar behavior, not showing a significant increase on roughness over time, regardless of the storage solution. This is in agreement with Yap et al. [3,4]. Both materials have a similar composition, similar organic matrix and same filler size and type. The major difference between these materials is the filler content, lower in the flowable composite Filtek Flow. However, this difference seems to have no influence on the material roughness. The appearance of the specimens of Filtek Z250 and Filtek Flow did not change in any solution after 6 months storage, when viewed with the naked eye and by SEM observation. These specimens showed the same polished surface shine.

The analysis of the pH of the solutions that stored these materials (Filtek Z250 and Filtek Flow) did not show any marked change in the pH over time, compared with the pH of recently made solutions. This confirms that these materials were not affected by the different solutions, not leaching their components in the solutions. Karantakis et al. [30] demonstrated that composites are resistant to dissolution in acidic conditions.

The zirconia/silica filler particles from Filtek Z250 and Filtek Flow seem to be more inert in the different solutions compared to the barium-fluoroaluminosilicate glass filler particles from Esthet X, which could be the reason for the increase in surface roughness after storage in citric acid and in Coca-Cola® (Figures 4 and 5). Esthet X specimens showed loss of polished surface shine and the specimens stored in Coca-Cola® showed a slightly brown hue.

It has been established that the corrosive potential of an acidic solution is related to their pH, titratable ability, buffer capacity and the solution degree of saturation [31]. Coca-Cola® has low titratable ability

and low buffer capacity, reducing its erosive potential [31]. However, in this study the pH of this solution was very low (pH ~ 2.5), which is related with the erosive potential [31]. In addition, this soft drink has in its composition an inorganic and strong acid, phosphoric acid. Thus, the association of a low pH and the presence of a strong inorganic acid could have caused a sooner (1 month) and more aggressive attack of the Esthet X surface by this solution. Figure 5 shows some filler protrusions. This could be related to the resin matrix degradation. In addition, most specimens stored in Coca-Cola® showed a slightly brown hue, more evident in the case of the RMGI group.

Citric and lactic acids have high titratable ability and buffer capacity [31–34]. However, in this study their baseline pH was ~ 5.0, which is higher than that of Coca-Cola®. This could be related to the lack of significant effect on surface roughness for most materials tested in this study. The pH of these solutions (citric and lactic acids) was selected based on the pH to produce active caries [35].

The significant increase on Esthet X surface roughness after 3 months storage in citric acid could be related to dissolution of the inorganic fillers, as suggested by McKinney [15]. Figure 4 shows some dark pits, indicating filler debonding, on Esthet X surface after 6 months storage in citric acid solution, which is indicative of filler dissolution. In addition, the hydrolysis of the silane-coupling agent could have taken place, leading to the leaching of these fillers on the acidic solution, increasing the surface roughness of this material.

The compomer Dyract AP had a similar behavior to composites, regarding the surface roughness. This is in agreement with other studies [10,11,28]. Dyract AP surface roughness was not affected by the storage on different acidic solutions over time, except for a significant increase on Ra values after 6 months of storage in water. Dyract AP specimens also showed loss of polished surface shine.

Dyract is an anhydride, which could react with water of the storage medium, showing the development of a carboxylate rich surface on the uppermost layer, turning this material less resistant to mechanical forces (wear) [36]. Conversely, in the presence of a low pH aqueous environment, water-soluble salts formed as a result of the increased solubility and selective dissolution of filler particles may retard or completely inhibit the carboxylate salt yields, increasing the abrasion resistance of the surface [36]. Thus, when Dyract AP specimens were subjected to an acidic condition the mean of surface roughness was lower than that observed for those immersed in distilled water after 6 months.

Also, the increased surface roughness after 6 months of Dyract AP stored in water could have been caused by a hydrolysis of the silane-coupling agent, or even the plasticizing process of the resin matrix. The SEM

photomicrograph of Dyract AP after 6 months storage in water shows a generalized rough surface (Figure 7). The poorer silanization of the filler particles could increase the potential for filler particle debonding.

The analysis of the pH of the solutions containing Dyract AP showed an increase on pH over time, especially in more acidic solutions, like Coca-Cola®, citric and lactic acids. This increase could be related to the release of the acid-base reaction products (fluoride and other ions) of this material that increases the degree of saturation of the solutions, increasing the pH, and decreasing the chemical degradation of this material. Nicholson et al. [37] demonstrated that Dyract AP is able to increase pH of lactic acid solution and this reduction on the pH was accompanied by a decrease on material mass. The more acidic the solution the higher is the fluoride release.

The resin-modified glass ionomer Vitremer had the roughest surface of all the materials. This is related to the highest filler particle size (Table I). However, the roughness of this material was not increased over time in any solution. The same process of increasing pH was observed for Vitremer. There was a marked change in the pH of the solutions, especially the more acidic ones. This increase was greater after 1 week, maintaining the pH over time. The fluoride and/or acid-base reaction products released in the acidic solution increased the saturation of the solution, decreasing the chemical attack of this material [38].

It should be considered that for both RMGI and compomer, the chemical degradation of material was accompanied by an increase in pH of the acidic solution [34,35]. Such a buffering effect is likely to be beneficial in protecting restored teeth from the development of secondary caries.

In this study, based on the results, the tested hypothesis must be accepted; most resin-based materials were resistant to chemical degradation over time, except for the composite Esthet X stored in citric acid and in Coca-Cola® and for the compomer Dyract AP stored in water.

Although chemical degradation is an important factor on resin-based material surface characteristics, other aspects such as the presence of mechanical forces (brushing and mastication), bacteria, saliva characteristics, etc., must be considered in order to determine the durability of the materials clinically. The association of chemical and mechanical challenges in the presence of saliva and microorganisms would determine the ultimate surface characteristics of resin-based materials in the oral environment.

## Conclusion

Based on the results of this study it can be concluded that different food-simulating solutions affect the long-term degradation of resin-based materials, depending on the material composition and type of

solution. A high consumption of beverages with low pH and high titratable ability or buffer capacity, as beverages containing citric acid in their composition and cola-based carbonated drinks should be avoided.

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## References

- [1] Oilo G. Biodegradation of dental composites/glass-ionomer cements. *Adv Dent Res* 1992;6:50–4.
- [2] Asmussen E. Softening of BISGMA-based polymers by ethanol and by organic acids of plaque. *Scand J Dent Res* 1984;92:257–61.
- [3] Yap AU, Low JS, Ong LF. Effect of food-simulating liquids on surface characteristics of composite and polyacid-modified composite restoratives. *Oper Dent* 2000;25:170–6.
- [4] Yap AU, Tan DTT, Goh BKC, Kuah HG, Goh M. Effect of food-simulating liquids on the flexural strength of composite and polyacid-modified composite restoratives. *Oper Dent* 2000;25:202–8.
- [5] Yap AU, Tan SHL, Wee SSC, Lee CW, Lim ELC, Zeng KY. Chemical degradation of composite restoratives. *J Oral Rehabil* 2001;28:1015–21.
- [6] Yap AU, Chew CL, Ong LFKL, Teoh SH. Environmental damage and occlusal contact area wear of composite restoratives. *J Oral Rehabil* 2002;29:87–97.
- [7] Larsen IB, Munksgaard EC. Effect of human saliva on surface degradation of composite resins. *Scand J Dent Res* 1991;99:254–61.
- [8] de Gee AJ, Wendt SL, Werner A, Davidson CL. Influence of enzymes and plaque acids on in vitro wear of dental composites. *Biomaterials* 1996;17:1327–32.
- [9] Wu W, McKinney JE. Influence of chemicals on wear of dental composites. *J Dent Res* 1982;61:1180–3.
- [10] Turssi CP, Hara AT, Serra MC, Rodrigues AL, Jr. Effect of storage media upon the surface micromorphology of resin-based restorative materials. *J Oral Rehabil* 2002;29:864–71.
- [11] Turssi CP, Magalhães CS, Serra MC, Rodrigues AL, Jr. Surface roughness assessment of resin-based materials during brushing preceded by pH-cycling simulations. *Oper Dent* 2001;26:576–84.
- [12] Bollen CM, Lambrechts P, Quirynen M. Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: a review of the literature. *Dent Mater* 1997;13:258–69.
- [13] Bagheri R, Burrow MF, Tyas M. Influence of food-simulating solutions and surface finish on susceptibility to staining of aesthetic restorative materials. *J Dent* 2005;33:389–98.
- [14] Roulet J, Wälti C. Influence of oral fluid on composite resin and glass ionomer cement. *J Prosthet Dent* 1984;52:182–9.
- [15] McKinney JE. Environmental damage and wear of dental composite restoratives. Posterior composite resin dental restorative materials. Netherlands: Peter Schulz Publishing Co; 1985.
- [16] Devlin H, Bassiouny MA, Boston D. Hardness of enamel exposed to Coca Cola and artificial saliva. *J Oral Rehabil* 2006;33:26–30.
- [17] Seow WK, Tong KM. Erosive effect of common beverages on extracted premolar teeth. *Aust Dent J* 2005;50:173–8.
- [18] Parry J, Shaw L, Arnaud MJ, Smith AJ. Investigation of mineral waters and soft drinks in relation to dental erosion. *J Oral Rehabil* 2001;28:766–72.
- [19] Johansson AK, Lingstrom P, Imfeld T, Birkhed D. Influence of drinking method on tooth-surface pH in relation to dental erosion. *Eur J Oral Sci* 2004;112:484–9.
- [20] Gedalia I, Dakuar A, Shapira L, Lewinstein I, Goultschin J, Rahamim E. Enamel softening with coca-cola and rehardening with milk or saliva. *Am J Dent* 1991;4:120–2.
- [21] Amaechi BT, Higham SM. Dental erosion: possible approaches to prevention and control. *J Dent* 2005;33:243–52.
- [22] Rees J, Loyn T, Gilmour A. Does low acid orange juice equal low erosion? *Dent Update* 2006;33:242–4.
- [23] Shaw L, Smith A. Erosion in children: an increasing clinical problem? *Dent Update* 1994;21:103–6.
- [24] Yip KH, Smales RJ, Kaidonis JA. The diagnosis and control of extrinsic acid erosion of tooth substance. *Gen Dent* 2003;51:350–3.
- [25] Turssi CP, Hara AT, Magalhães CS, Serra MC, Rodrigues AL, Jr. Influence of storage regime prior to abrasion on surface topography of restorative materials. *J Biomed Mater Res B Appl Biomater* 2003;65:227–32.
- [26] Dietschi D, Campanille G, Holz J, Meyer JM. Comparison of the color stability of ten new-generation composites: an in vitro study. *Dent Mater* 1994;10:353–62.
- [27] Hachiya Y, Iwaku M, Hosoda H, Fusayama T. Relation of finish to discoloration of composite resins. *J Prosthet Dent* 1984;52:811–14.
- [28] Gladys S, Meerbeek BV, Braem M, Lambrechts P, Vanherle G. Comparative physico-mechanical characterization of new hybrid restorative materials with conventional glass-ionomer and resin composite restorative materials. *J Dent Res* 1997;76:883–94.
- [29] el Kalla IH, Garcia-Godoy F. Mechanical properties of compomer restorative materials. *Oper Dent* 1999;24:2–8.
- [30] Karantakis P, Helvatjoglou-Antoniades M, Theodoridou-Pahini S, Papadogiannis Y. Fluoride release from three glass ionomers, a compomer, and a composite resin in water, artificial saliva, and lactic acid. *Oper Dent* 2000;25:20–5.
- [31] Jensdottir T, Bardow A, Holbrook P. Properties and modification of soft drinks in relation to their erosive potential in vitro. *J Dent* 2005;33:569–75.
- [32] Lussi A, Jaeggi T. Chemical factors. *Monogr Oral Sci* 2006;20:77–87.
- [33] Hannig C, Hamkens A, Becker K, Attin R, Attin T. Erosive effects of different acids on bovine enamel: release of calcium and phosphate in vitro. *Arch Oral Biol* 2005;50:541–52.
- [34] Edwards M, Creanor SL, Foye RH, Gilmour WH. Buffering capacities of soft drinks: the potential influence on dental erosion. *J Oral Rehabil* 1999;26:923–7.
- [35] Hojo S, Takahashi N, Yamada T. Acid profile in carious dentin. *J Dent Res* 1991;70:182–6.
- [36] Eliades G, Kakaboura A, Palaghias G. Acid-base reaction and fluoride release profiles in visible light-cured polyacid-modified composite restoratives (compomers). *Dent Mater* 1998;14:57–63.
- [37] Nicholson JW, Millar BJ, Czarnecka B, Limanowska-Shaw H. Storage of polyacid-modified resin composites (“compomers”) in lactic acid solution. *Dent Mater* 1999;15:413–6.
- [38] Nicholson JW, Czarnecka B, Limanowska-Shaw H. A preliminary study of the effect of glass-ionomer and related dental cements on the pH of lactic acid storage solutions. *Biomaterials* 1999;20:155–8.