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Effect of sports drinks on the surface properties of composite resins after prolonged exposure - *in vitro* study

Daniella Cristo Santin[1], Fabiana Scarparo Naufel[2,*], Rafael Francisco Lia Mondelli[1], Adriano Piccolotto[2], Vera Lúcia Schmitt[2]

[1] Department of Restorative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo (USP), Bauru, SP, Brazil.

[2] Department of Operative Dentistry, Western Paraná State University (UNIOESTE), Cascavel, PR, Brazil.

Aim: Using dietary supplements may affect the properties of composite resins due to their acidic pH. The present *in vitro* study aimed to assess the surface roughness and color stability of two composite resins - nanohybrid (Empress Direct) and nanoparticulate (Filtek Z350) - after prolonged exposure to dietary supplements. **Methods:** We produced 30 specimens from each composite (8x2-mm discs) and divided them into six groups (n=10). After the initial measurement of the surface properties (roughness and color), we exposed the specimens to a degradation process in Maltodextrin and Whey Protein for 22.5 and 7.5 days, respectively, using deionized water as the control solution. At the end of 22.5 days, we reassessed the specimens. After verifying data normality with the Kolmogorov-Smirnov test, we performed ANOVA followed by Tukey's test at 5%. **Results:** We found significant differences for materials immersed in the Whey Protein solution ($p<0.05$). The roughness of Empress Direct was higher (0.45+0.07) than Filtek Z350 (0.22+0.05). The composites tested also showed color change ($\Delta E>3.3$) after the immersion period ($p<0.001$). In Maltodextrin, the Empress Direct group presented (4.52+1.23) and Filtek Z350 (4.04+0.66), while after immersion in Whey Protein, they showed (5.34+1.68) and (4.26+1.02), respectively. **Conclusion:** Sports drinks changed the surface roughness and color stability of the composite resins studied. The Filtek Z350 group showed lower color variation than the Empress Direct composite in both solutions evaluated.

Keywords: Composite resins. Beverages. Dietary supplements. Surface properties.

Corresponding author:
Fabiana Scarparo Naufel
2069 Universitária Street, Cascavel, PR, 85814-110, Brazil
Phone +55 45 32203168,
e-mail: biberes@terra.com.br

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Introduction

Despite the improvements in the physical properties of composite resins, this restorative material is still subject to deterioration in the oral cavity. The unfavorable interaction between the oral environment and external factors such as dietary habits may compromise the longevity of restorations[1-3]. Some studies claim that ingesting acidic beverages may cause dental erosion and affect the properties of restorative materials[1,4,5]. In this context, the effect of regularly consuming dietary supplements requires further investigation. Consuming sports drinks serves for nourishing/hydration, aiming to improve athletic performance during physical exercise[4,5]. Nevertheless, they contain preservatives, acidulants, and large amounts of carbohydrates (glucose, fructose, sucrose, and maltodextrin), which create an acidic oral environment after consumption[1] and may change the structure of composite resins[6,7].

The chemical challenges provided by an acidic diet cause the degradation of the resin-based restorative materials, resulting in a softer organic matrix and increased surface roughness[6,8,9]. In this case, daily toothbrushing would mechanically remove superficial layers of the restoration and modify the surface roughness[7,10,11]. This would lead to dental biofilm deposition and reduce restoration brightness[7,10,12]. As a result, the restorations may suffer an extrinsic color change due to the penetration of pigments from sports drinks into the porosities, compromising the color stability and the longevity of restorations[6]. Thus, this *in vitro* study aimed to assess the effects of the daily exposure to dietary supplements on the surface properties (surface roughness and color stability) of composite resins. The null hypotheses tested in this study were: i) Dietary supplements do not affect the surface roughness and color stability of composite resins; ii) The composition of composite resin materials does not affect the surface roughness and color stability after the exposure to dietary supplements.

Materials and Methods

Experimental design

This study analyzed two factors: composite resin (in two levels) and solution of immersion (in three levels), whereas the surface roughness and color stability were the response variables.

Following the manufacturer's instructions for treatment, the consumption of Maltodextrin (Peter Food, São Paulo, Brazil) should be fractioned during training, while the consumption of Whey Protein (New Millen, São Paulo, Brazil) should occur immediately after training. Thus, for the daily use of the supplements tested in the present study, we established a daily 30-min exposure to Maltodextrin and 10-min exposure to Whey Protein. We calculated the exposure to sports drinks for three years of consumption, resulting in a total exposure time of 540 hours for Maltodextrin and 180 hours for Whey Protein.

Specimen preparation

In the present study, we tested the following two composite resins of color A2 for enamel: nanoparticulate (Filtek Z350, 3M ESPE, Saint Paul, USA) and nanohybrid

(Empress Direct, Ivoclar Vivadent, Schaan, Liechtenstein). Table 1 presents details of the materials tested. We prepared 60 disc-shaped specimens (30 from each resin) using an addition silicone mold with 8 mm in diameter and 2 mm in height. We covered the set with a transparent polyester strip and pressed it with a glass plate. Then, we photoactivated the increment through the polyester strip with a LED device (Valo Ultradent, South Jordan, USA) for 20 seconds and measured the irradiance (1000 mW/cm^2) of the curing light with a power meter (Hilux Dental Curing Light Meter, Benlioglu Dental Inc., Ankara, Turkey). Then, we stored the specimens in deionized water for 24 hours at 37˚C.

We polished the surfaces of the specimens sequentially using #600, #1200, and #2000 grit silicon carbide abrasive papers under water cooling for 10, 20, and 30 seconds, respectively[4]. At each swap, we cleaned the specimens ultrasonically in deionized water for 5 minutes (U.S. Thornton Electronic Ltda., São Paulo, Brazil). After polishing, we identified the specimens and immersed them in deionized water for another 24 hours at 37˚C.

Surface roughness measurements (Ra)

We measured surface roughness (Ra, μm) with a Surfcorder SE1700 surface roughness measuring instrument (Kosaka Corp, Tokyo, Japan). We took the measurements before and after the immersion period. For standardizing the readings, we divided the disc-shaped specimens virtually into two parts, marking the side of the specimen at the 12-hour position. Then, on the left side of the disk, we took three readings from each specimen in three different directions (45°, 90°, and 135°) (Figure 1), always starting from the most central region of the disc towards the periphery, and traveling 1.25 mm with a cut of 0.25 mm; minimum T = 0.01 μm and maximum T = 8.00 μm. We determined the Ra value of each specimen by calculating the means of the three roughness readings for each time assessed (before and after immersion).

Assessment of color stability (ΔE)

We used a CM-700d spectrophotometer (Konica Minolta, Tokyo, Japan) to obtain the initial and final colors of the specimens according to the CIEL*a*b*. We transported the discs to a sample carrier and performed the readings in a light cabinet. We analyzed the color change by the difference between the initial and final ΔL, Δa, and Δb measurements[13], using the following formula: $\Delta E = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}]^{1/2}$.

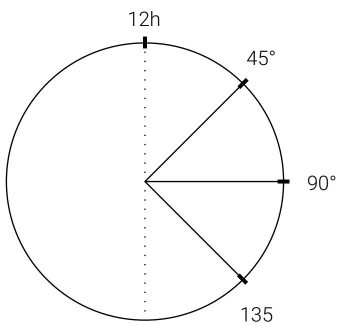

Figure 1. Description of the directions considered for measuring the surface roughness.

Immersion of specimens in the solutions

After measuring the initial roughness and color, we stored the specimens again in deionized water for 24 hours at 37˚C. Degradation started for the three solutions studied (n=10): deionized water (control), Maltodextrin, and Whey Protein (Table 1). Figure 2 shows the immersion protocol performed according to the groups evaluated.

Final roughness and color measurements

After 22.5 days of immersion, we performed the final measurements of surface roughness and color stability, as described previously (Figure 3).

Statistical analysis

For surface roughness, we tabulated the data considering the factors of resin, time, and treatment, while for color stability (ΔE), the factors were resin and treatment. Primarily, we analyzed the results using a Kolmogorov-Smirnov test to verify normal distribution. We continued with three-way ANOVA with repeated measures for roughness and two-way ANOVA for color stability (5%). Tukey's test (5%) determined statistically significant differences.

Table 1. Composition of composite resins and dietary supplements.

Material	Manufacturer and batch	Composition	Color/Taste	pH
Filtek Z350	3M ESPE (Saint Paul, MN, USA) - 1507000832HB004134407	Bis-GMA, UDMA, TEGDMA, PEGDMA, Bis-EMA. Non-aggregated silica and zirconia particles, silica/zirconia.	EA2	-
Empress Direct	Ivoclar Vivadent (Schaan, Liechtenstein) - U02602	Bis-GMA, UDMA, TEGMA, barium glass particles, ytterbium trifluoroethane, mixed oxides, silica dioxide, copolymers.	EA2	-
Maltodextrin	Peter food (São Paulo, Brazil) - 10686	Maltodextrin, citric acid, tangerine flavor, ascorbic acid, sodium saccharin, sodium hydrochloride, titanium dioxide, dye yellow dye.	Tangerine	2.67
Whey Protein	New Millen (São Paulo, Brazil) - 36807	Whey protein isolated and concentrated, hydrolyzed whey protein, modified waxy maize starch, cocoa powder, magnesium pyruvate, zinc L-aspartate, xanthan gum, flavoring and sucralose sweetener.	Black forest	5.99

*Abbreviations: Bis-GMA, Bisphenol glycidyl methacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; PEGMA, Polyethylene glycol dimetacrylate; Bis-EMA, ethoxylated bisphenol-A dimethacrylate.

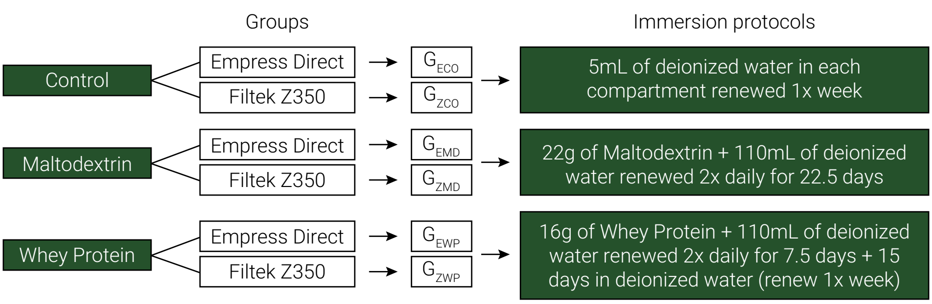

Figure 2. Description of the groups and immersion protocols evaluated.

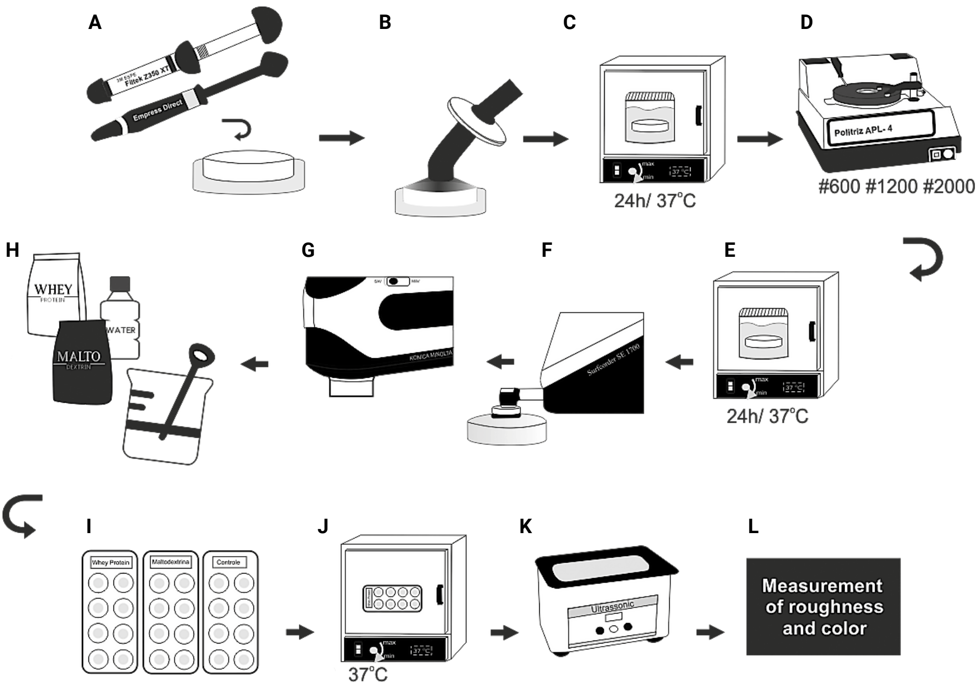

Figure 3. Flowchart representing the research steps: A - Insertion of composite resin in the matrix; B – Photoactivation; C – Storage of the specimens in deionized water for 24 hours in a 37˚C stove; D – Polishing with #600, #1200, and #2000 granulation files, followed by ultrasonic cleaning for 5 minutes; E – Storage of the specimens for additional 24 hours at 37˚C; F, G – Initial roughness and color measurements, respectively; H – Immersion protocol in solutions of deionized water, Maltodextrin, and Whey Protein; I – Specimens immersed in individual plastic compartments containing the solutions assessed; J – Storage in a 37˚C stove for 22.5 days for the control group and Maltodextrin, and 7.5 days for Whey Protein (to complete 22.5 days, the specimens were immersed in deionized water for 15 days); K – Cleaning of specimens in ultrasonic bath; L – Final roughness and color measurements.

Results

Table 2 presents the mean and standard deviation of roughness values before and after immersing the composite resins in the solutions tested. The ANOVA results showed statistically significant differences in material roughness for resin, immersion solution, and time ($p<0.001$). We observed a significant double interaction for the variables of immersion solution with time and resin and a triple interaction for all factors ($p<0.001$). Tukey's test (5%) revealed that, throughout the immersion protocol, the surface roughness of both resins decreased in all treatment solutions, except for the Empress Direct resin immersed in Whey Protein ($p<0.05$).

For color variation (ΔE), ANOVA indicated significant statistical differences for the factors of isolated resin and immersion solution and for the double interaction between the two variables ($p<0.001$). The data described in Table 3 (Tukey's test at 5%) show that both resins presented a visible color change in all solutions, considering they showed ΔE greater than or equal to 3.3. The Empress Direct resin showed a higher ΔE in the control solution than in both sports drinks, and the Z350 resin presented equivalent ΔE in all three solutions. For the variation of luminosity (ΔL) (Table 3), ANOVA showed statistically significant differences ($p<0.001$) for the isolated solution factor,

Table 2. Mean (µm) and standard deviation (SD) of surface roughness of the composite resins for the three factors studied.

	Deionized water		Maltodextrin		Whey Protein	
	Initial	Final	Initial	Final	Initial	Final
Empress Direct	0.66 (0.15) Aa	0.26 (0.03) Ba	0.36 (0.12) Aa	0.27 (0.04) Ba	0.32 (0.02) Bb	0.45 (0.07) Aa
Filtek Z350	0.37 (0.08) ABb	0.28 (0.04) Ba	0.39 (0.08) Aa	0.28 (0.08) Ba	0.44 (0.09) Aa	0.22 (0.05) Bb

*Different lower-case letters in the columns and upper-case letters in the rows indicate statistically significant differences (p<0.05).

Table 3. Mean and standard deviation (SD) of ΔE, ΔL, Δa* and Δb* of resins after immersion in solutions.

		Deionized water	Maltodextrin	Whey protein
ΔE	Empress Direct	7.54 (1.38) aA	4.52 (1.23) aB	5.34 (1.68) aB
	Filtek Z350	4.25 (2.14) bA	4.04 (0.66) aA	4.26 (1.02) aA
ΔL*	Empress Direct	-2.79 (0.65) bA	-1.57 (1.83) aA	-2.33 (1.17) bA
	Filtek Z350	-2.32 (2.62) bA	-0.71 (1.00) aA	-3.04 (0.71) bA
Δa*	Empress Direct	3.70 (1.08) aA	2.23 (0.89) bB	2.50 (2.00) aAB
	Filtek Z350	1.50 (1.01) bB	3.39 (0.61) aA	2.54 (0.72) aAB
Δb*	Empress Direct	-5.87 (1.12) bB	-2.88 (1.60) aA	-3.53 (1.54) bA
	Filtek Z350	-1.03 (2.66) aA	-1.50 (1.16) aA	-1.26 (1.02) aA

*For each variation, different lower-case letters in the columns and upper-case letters in the rows indicate statistically significant differences (p<0.05) for each factor.

and there was a lower luminosity reduction for both resins evaluated when immersed in Maltodextrin, compared to the control group and Whey Protein (p<0.05). For the red-green axis (Δa), ANOVA detected a significant difference for the interaction between the factors of resin and immersion solution (p<0.001). Positive values (Table 3) indicate a decrease in the green color and an increase in the red one. The interaction showed that for the a* ordinate, the Empress Direct composite resin showed greater variation in the control solution, while Filtek Z350 showed greater variation in the Maltodextrin solution (p<0.05). The Δa for Whey Protein was similar in both resins. For the Δb, ANOVA found a statistical significance for the variables of resin (p<0.001), immersion solution (p<0.05), and for the double interaction (p<0.001). The negative values of the b* ordinate (Table 3) indicate a decrease of blue coloration, which is mainly evident in the specimens of the Empress Direct resin immersed in the control solution. The Empress Direct resin showed higher Δb than Z350, with statistical significance in the control solution and Whey Protein (p<0.05).

Discussion

Aesthetic restorative materials are subject to the gradual degradation process in the oral environment due to changes in pH, temperature, chewing, brushing, and composition of the restorative material[2,3]. Because of the increased consumption of dietary supplements, this *in vitro* study aimed to assess the effects of sports drinks on the surface properties of composite resins.

In this study, we selected Maltodextrin and Whey Protein because they are often consumed for increasing the physical performance and for muscle gain, respectively[4]. The results showed that the composites resins exposed to Whey Protein were mostly affected, potentially for being protein-based. The literature reports that the composite resin exposed to the oral environment allows adsorbing proteins on its surface[14], which may have contributed to the increase in surface roughness and consequently greater retention of pigments.

The tendency to staining is one of the disadvantages of composite resins[1,8,9], therefore this study assessed whether resins immersed in dietary supplements would present color changes. Hence, the study results rejected the first null hypothesis, considering that the analysis of ΔE values showed that both Maltodextrin and Whey Protein solutions changed the color of the resins assessed. This resulted in ΔE values equal to or greater than 3.3, which is clinically perceptible to the human eye[13]. We may justify increased ΔE values after immersion in all solutions, including deionized water, because of the resin matrix potential of absorbing liquids[8,15]. The water absorbed carries the pigments of sports drinks and may stain the composites[8].

The results of the present study agree with the studies by Erdemir et al.[6] (2016), which state that the acidity of beverages may dissolute the organic matrix and increase the absorption of dyes that stain composite restorations, impairing their longevity. Low-pH beverages (Maltodextrin: pH=2.67; Whey Protein: pH=5.99) can solubilize restorative materials[1,16]. Increased surface wear may occur due to organic matrix softening, resulting in the loss of structural ions and decreased resistance[1,17,18]. However, we did not assess the pH effect of the experimental solutions on wear resistance, which requires further investigation to evaluate such an effect.

When comparing the two composite resins, the Empress Direct showed higher ΔE values than Filtek Z350 in all treatment solutions, rejecting the second null hypothesis, which we may attribute to the chemical composition[19]. Potentially, against chemical and/or frictional wear, the inorganic particles of larger size (0.04–3 μm) in the Empress Direct release from the organic matrix, leaving craters on the surface and increasing roughness. This may have contributed to the increase of the extrinsic pigmentation and loss of brightness of the specimens[6,8,19]. In addition, its organic matrix based on Bis-GMA and UDMA appears to be more susceptible to dissolution[1,17,18]. It is worth noting that, in the oral cavity, the salivary cleaning action, toothbrushing, and polishing of restorations may reduce the staining susceptibility of composite restorations, which would contribute to greater clinical longevity.

At the end of 22.5 days, the immersion in the solutions assessed reduced the surface roughness in all groups, except for the nanohybrid resin immersed in Whey Protein, which presented increased surface roughness values. According to previous studies[1,2,19], the effect of treatment solutions varied depending on the type of material, rejecting again the second null hypothesis. For the Empress Direct composite resin, we verified the highest roughness value in Whey Protein, whereas for Filtek Z350, it occurred in deionized water and Maltodextrin. This may occur because roughness is a property affected by the water sorption capacity of composite resins. Composites with increased loads tend to absorb less liquid[20].

The simulation of the frequent consumption of sugary and/or acidic solutions (Whey Protein and Maltodextrin) did not affect surface roughness. The organic phase of the composite resins, which is subject to water sorption, potentially determined the hygroscopic expansion, which relieves the stresses generated during polymerization shrinkage and may consequently reduce cracks and irregularities, as well as the roughness of composites[1,21,22].

The wear resistance of the Filtek Z350 nanoparticulate composite may relate to its chemical composition. The formulation based on nanoparticles and nanoclusters provides less interstitial space between the inorganic particles, making it more difficult to displace them against wear[7,11]. The smaller particles released leave minor surface defects, affecting the surface roughness and color stability of the restorations to a lesser extent[11,19].

The literature states that resins formulated with Bis-EMA tend to be less susceptible to dissolution when compared with those formulated with Bis-GMA and UDMA[1,17,18]. The resin-based composites made of inorganic particles of silica, zirconia, or barium glasses tend to suffer greater degradation than quartz particles[1,16]. Analyzing the composition of the Empress Direct resin allows inferring that its exclusive composition of Bis-GMA and UDMA, in addition to the larger and soft spherical particles (silica and barium glass), determined an increased softening and leaching of inorganic fillers[1,17].

In situ and *in vivo* studies are required to complement the findings of the present study regarding the degradation of composite resins exposed to dietary supplements.

Under the experimental conditions described, sports drinks changed the surface properties of the resins assessed, considering there were color changes after the immersion in the beverages tested and increased surface roughness for the Empress Direct resin immersed in Whey Protein. The nanoparticulate composite resin presented increased resistance to surface degradation, showing the best results for both properties evaluated.

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