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Effects of simulated gastric juice on color stability, surface roughness and microhardness of laboratory-processed composites

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Medical problems such as gastroesophageal reflux disease can cause considerable damage to restorations in the oral environment. This study evaluated the effects of gastric juice on the surface characteristics of different types of laboratory-processed indirect composites with different filler particles and polymerization modes. Specimens were prepared from Tescera (TES; Bisco), Sinfony (SIN; 3M ESPE), Solidex (SOL; Shofu), and Adoro (AD; Ivoclar Vivadent). Before exposing the specimens to simulated gastric juice for 24 h, color, surface roughness, and microhardness were measured on one half of the polished flat surface of each specimen. After exposure to the simulated gastric juice, the same tests were repeated on the other half of specimen surface. Results indicated that gastric juice had a significant impact on color change ($p<0.001$). AD showed the largest surface roughness change among the resins ($p<0.001$). Gastric juice also significantly affected the microhardness of the materials, and AD and TES showed statistically similar change in microhardness ($p>0.05$).

Keywords: Color, Dental resin, Gastric acid, Gastric juice, Surface properties

INTRODUCTION

Gastroesophageal reflux disease (GERD) is defined as involuntary muscle relaxing of the upper esophageal sphincter, which allows refluxed acid to move upward through the esophagus into the oral cavity[1]. The gastric contents affect the oral cavity and teeth as a result of gastroesophageal reflux, since pH of the gastric acid ranges between 1 and 1.5 —far below the critical pH of 5.5 at which tooth enamel will dissolve[2]. Gastric juice has been shown to demineralize enamel, dentin, and root cementum in *in vivo* and *in vitro* studies[3-6].

Laboratory-processed indirect resin composite systems may be the solution to some of the problems inherent to dental ceramics. These new-generation indirect resins contain a higher density of inorganic ceramic filler particles than the traditional direct and indirect resin composites[7]. Indirect resin composites are advocated for a wide range of fixed restorations, such as inlays, onlays, veneers, metal-free single unit crowns, and short-span anterior fixed dental prostheses (FDPs)[8]. For these materials, the post-polymerization process is pivotal to providing the superior flexural strength (when compared with feldspathic porcelain), minimal polymerization shrinkage, and wear rates comparable to enamel[9]. Indirect resin composites also boast of several other advantages: favorable aesthetics, reparability, and fast laboratory procedures[10]. These laboratory-processed resin composites are available in a wide variety with differences in terms of composition, polymerization modes, and polymerization conditions. Ultra-fine filler particles and polyfunctional methacrylate monomers are commonly used in these resin composites. They are processed using different laboratory procedures based on various combinations of light, heat, pressure, and vacuum to achieve a higher degree of polymerization[11].

The physical and mechanical properties of laboratory-processed resin composites[12-16] have been examined in numerous studies. However to date, no studies have investigated the effects of gastric juice on the optical properties and surface characteristics of these materials.

The objective of this study was to evaluate the effects of gastric juice on the color stability, surface roughness, and surface microhardness of four different types of laboratory-processed indirect composites which contained different filler particles and were processed using varied polymerization modes. The hypothesis to be tested in this study was that the resin composites would be affected in different degrees from the exposure to gastric juice.

MATERIALS AND METHODS

Specimen preparation

Resin composite materials used in this study are listed in Table 1, together with their chemical compositions. A polytetrafluoroethylene mold (diameter: 15 mm; thickness: 2 mm) was used to fabricate the specimens (Fig. 1a). For each resin composite, 10 cylindrical specimens of shade A3 (Vita Lumin shade guide, Vita Zahnfabrik, Bad Säckingen, Germany) were prepared according to manufacturer's recommendations (Fig. 1b). Material was packed into the mold using a plastic spatula. A glass slab was placed on the resin composite,

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Table 1 Brands, codes, chemical compositions, batch numbers, and polymerization protocols of laboratory-processed indirect resin composites used in this study (as provided by the manufacturers)

Resin Materials (Manufacturer)	Code	Batch number	Filler Type	Filler content (% weight)	Particle size	Monomers	Classification	Polymerization	Time
Tescera ATL (Bisco, Schaumburg, IL, USA)	TES	0700004328 0700004328	Glass, amorph silica	72	N/A	Ethoxylate bisphenol-A-dimethacrylate, bisphenol-A-diglycdyl methacrylate	Microfill	Heat-Light-Pressure (Tescera ATL)	2 min light/ pressure 13 min heat/ light/pressure
Sinfony (3M ESPE, Seefeld, Germany)	SIN	270630	Silicon dioxide strontium-aluminium borosilicate glass, quartz	50	50 nm-µm	Aliphatic, sikloaliphatic monomer	Ultra-fine hybrid	Light-Vacuum Visio Alfa (ESPE) Visio Beta Vario (ESPE)	5 sec light 14 min light/ vacuum
Solidex (Shofu, Kyoto, Japan)	SOL	020740	Silicon dioxide, aluminium oxide	53	1 µm	UDMA	Hybrid	Halogen Light Solidilite X	5 min light
SR Adoro (Ivoclar-Vivadent, Schaan, Liechtenstein)	AD	J23767 K05422	Silicon dioxide	63	1 µm	UDMA	Inhomogeneous microfill	Heat-Light (Targis Power Upgrade)	25 min heat/ light

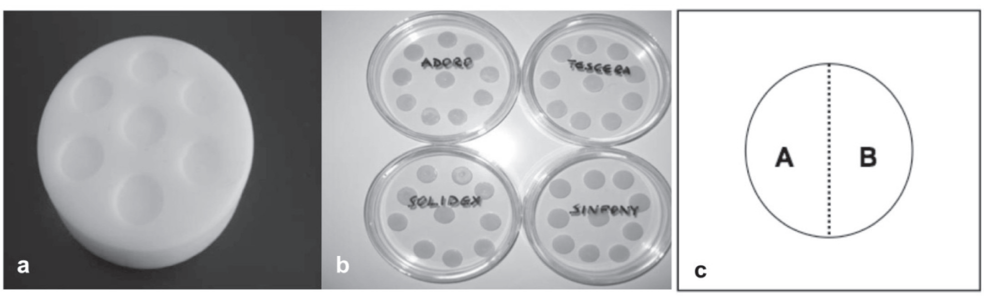

Fig. 1 Photos of: (a) poly(tetrafluoroethylene) mold (diameter: 15 mm; thickness: 2 mm) used to fabricate resin composite specimens; (b) specimens in glass petri dish prior to polishing; (c) schematic drawing of specimen surface used for color stability, surface roughness, and surface microhardness tests. Measurements were performed on Surface A prior to exposure to simulated gastric juice, and on Surface B after exposure.

and the latter was photo-polymerized from the upper and lower surfaces. After polymerization, the surfaces were ground-finished using 1,500-grit abrasive paper (English Abrasives, English Abrasives & Chemicals Ltd., London, UK) under water. One surface of each specimen was polished according to respective manufacturer's recommendations and stored in a dry dark environment until testing. Table 2 lists the polishing materials used.

Color stability, surface roughness, and surface microhardness tests were applied to one half of the polished flat surface of each specimen using a colorimeter, profilometer, and Vickers microhardness tester respectively, before exposing the specimen to simulated gastric juice for 24 h.

Simulated gastric juice was prepared by dissolving 2.0 g of sodium chloride and 3.2 g of pepsin (Pepsin, Darmstadt, Germany) in 7.0 mL of hydrochloric acid and

Table 2 Polishing materials of respective manufacturers were used correspondingly on the resin composites in this study

Polishing Material	Batch number	Manufacturer
Tescera Polishing Paste	REF T-1803	Bisco, Schaumburg, IL, USA
Sinfony Adjustment Kit	03128	3M ESPE, Seefeld, Germany
Sinfony Opal L Polishing Paste	520-0001	3M ESPE
Diamond Stick	004610	Shofu, Kyoto, Japan
CompoMaster Coarse	0503910	Shofu
Universal Polishing Paste	FL- 9494	Ivoclar Vivadent, Schaan, Liechtenstein

water to make up 1,000 mL[17]. The solution had a pH of 1.14 as measured by a pH meter. Each specimen was separately submerged in gastric juice in a glass petri dish and kept in an incubator (Dedeoğlu Ltd., Ankara, Turkey) at 37°C for 24 h in a dark environment. After 24 h, gastric juice was rinsed off from the specimens using air/water spray. The same tests were performed on the other half of the polished flat surfaces of the rinsed specimens (Fig. 1c).

Color measurements

Color measurements were made using a colorimeter (Minolta Chroma Meter CR-321, Minolta, Osaka, Japan) according to CIE $L^*a^*b^*$ color system[18]. This instrument had a measuring head which provided 45-degree illumination and 0-degree viewing geometry (specular component included) for color measurements of glossy surfaces, with light provided by a pulsed xenon arc lamp over a 3-mm-diameter measuring area.

Three measurements were made on each specimen before and after exposure to simulated gastric juice. Before exposure to gastric juice, the mean value of each set of three measurements was designated as CIE $L_1^*a_1^*b_1^*$; after exposure, the mean value was designated as CIE $L_2^*a_2^*b_2^*$. Based on these data, ΔE value of each specimen was calculated. To position the probe tip of the colorimeter at the same location on each specimen, a polytetrafluoroethylene mold was prepared. Before each measurement session, the colorimeter was calibrated according to manufacturer's instructions using a white calibration cap (CR-A43, Minolta) provided by the manufacturer. Quantitative ΔE value of each specimen was calculated using the following formula[19]:

$\Delta L^*=L_2^*-L_1^*$

$\Delta a^*=a_2^*-a_1^*$

$\Delta b^*=b_2^*-b_1^*$

$\Delta E=[(\Delta L^*)^2+(\Delta a^*)^2+(\Delta b^*)^2]^{½}$

Surface roughness measurements

After color measurement, surface roughness of the same specimen was assessed using a profilometer (Perthometer M2, Mahr GmbH, Germany). To measure roughness profile in micrometers (µm), the diamond stylus (5-µm tip radius) was moved across the surface under a constant load of 3.9 mN at a speed of 0.12 mm/s over a range of 600 µm. This procedure was repeated three times at three different locations on each specimen to obtain the general surface characteristics of the specimen. The instrument was calibrated using a standard reference specimen. Before exposure to simulated gastric juice, the average value of each set of measurements was designated as Ra_1; after exposure, the average value was designated as Ra_2. Quantitative ΔRa value of each specimen was calculated using this formula: $Ra_2–Ra_1$.

Surface hardness measurements

Hardness was determined using the indentation technique with a microhardness tester (Wilson-Wolpert Tukon, Instron, Rozenburg, ZH, The Netherlands) under a load of 100 g for 10 s. Three indentations were made on each specimen using a Vickers diamond indenter to determine the mean microhardness value for each specimen. Indentation dimensions were measured using the eyepiece of a microscope, and hardness values were obtained from standard tables. Before exposure to gastric juice, the mean Vickers hardness value derived from each set of three indentation measurements was designated as VHN_1; after exposure, the mean value was designated as VHN_2. Quantitative ΔVHN value of each specimen was calculated using this formula: $VHN_2–VHN_1$.

Statistical analysis

Statistical analyses were performed using SPSS for Windows, Version 12.0 (SPSS Inc., Chicago, IL, USA). The Kolmogorov-Simirnov test showed that data were normally distributed (α=0.05). One-way analysis of variance (ANOVA) was used to determine the significant differences for color change, surface roughness and microhardness values between resin composite materials before and after exposure to gastric juice. The interactions and multiple comparisons were performed using a *post-hoc* Tukey's test. *P* values less than 0.05 were considered to be statistically significant in all tests.

RESULTS

Table 3 presents the mean ΔE values of the four resin composite groups and their standard deviations. One-way ANOVA revealed that gastric juice significantly affected color change (p<0.001). There were also significant differences in ΔE among the materials (p<0.001, Tukey's test). AD showed the lowest mean ΔE value (2.8±0.5) in comparison to the other resin composites (6.5±1.6–17.1±1.1).

Table 4 presents the mean values of Ra_1, Ra_2, and ΔRa of the four resin composite groups and their standard deviations. Although TES showed the highest Ra values (µm), the greatest surface roughness change in Ra occurred in AD (0.231±0.09), which was statistically significant (p<0.001) in comparison to the other resin composites (0.07±0.09–0.098±0.08 µm).

Table 5 presents the mean values of VHN_1, VHN_2, and ΔVHN of the four resin composite groups and their standard deviations. SOL showed the highest mean VHN_1 value (44.36±1.2) and the greatest change in VHN (14.05±1.28). ΔVHN was not significantly different between AD and TES (p>0.05).

DISCUSSION

Gastric juice has demineralization effect on enamel, dentin, and cementum[3–5]. Due to its low pH (less than pH 1), gastric juice may also dissolve glassy-matrix ceramics[20]. Results of this study showed that gastric juice had significant effects on laboratory-processed, methacrylate-based, indirect composites. Since significant differences were found in ΔE, ΔRa, and ΔVHN values among the resin composite materials

Table 3 Means and standard deviations (SD) of $L^*a^*b^*$ and ΔE values of resin composite groups as measured with a colorimeter. Different lowercase letters in the same column indicate significant differences (p<0.001)

Groups	Mean (±SD)						
	L_1^*	a_1^*	b_1^*	L_2^*	a_2^*	b_2^*	ΔE
TES	80.20±1.12	−3.82±0.69	10.60±2.27	92.14±1.82	−4.32±0.44	7.89±0.91	12.53±2.18[a]
SIN	93.89±2.54	−3.88±0.48	4.09±0.98	94.04±3.57	−4.62±0.74	8.39±1.12	6.51±1.57[b]
SOL	80.91±1.66	−3.54±0.17	18.64±0.76	93.00±0.96	−4.94±0.35	6.80±0.77	17.07±1.07[c]
AD	93.00±0.44	−4.49±0.55	6.90±0.52	90.79±0.49	−5.76±0.36	6.59±0.78	2.77±0.47[d]

Table 4 Means and standard deviations (SD) of Ra_1, Ra_2, and ΔRa values of resin composite groups as measured with a surface profilometer. Different lowercase letters in the same column indicate significant differences (p<0.05)

Groups	Mean (µm) (±SD)		
	Ra_1	Ra_2	ΔRa
TES	0.34±0.07[b]	0.41±0.09[b]	0.070±0.09[b]
SIN	0.18±0.03[a]	0.27±0.06[a]	0.094±0.06[b]
SOL	0.18±0.07[a]	0.27±0.09[a]	0.098±0.08[b]
AD	0.14±0.06[a]	0.37±0.08[ab]	0.231±0.09[a]

Table 5 Means and standard deviations (SD) of VHN_1, VHN_2, and ΔVHN values of resin composites groups as measured using Vickers hardness test. Different lowercase letters in the same column indicate significant differences (p<0.05)

Groups	Mean ± SD		
	VHN_1	VHN_2	ΔVHN
TES	42.18±0.34[d]	35.83±0.66[c]	6.15±1.02[a]
SIN	31.46±1.38[c]	26.78±0.60[a]	4.68±1.17[b]
SOL	44.36±1.20[b]	30.31±0.44[b]	14.05±1.28[c]
AD	33.09±0.87[a]	26.94±0.37[a]	6.15±1.02[a]

tested, the hypothesis that resin composites would be affected in different degrees from their exposure to gastric juice could be accepted.

In an *in vitro* study, extracted tooth crowns immersed in different acidic test solutions at 37°C for 3 to 24 h produced erosion visually similar to that clinically observed in all test solutions[21]. In another study, the surface roughness of filling materials was studied after 24-h exposure to simulated gastric juice[22]. In the present study, specimens were also immersed in simulated gastric juice for 24 h. This immersion duration without exposure to saliva at different intervals could be considered too long, but short enough to allow for water absorption. Taken together, these could be deemed as potential limitations of this study. Nonetheless, this experimental design represented a worse-case scenario to study the effect of gastric juice on the materials tested since reflux attacks may happen numerous times a day.

On color difference after exposure to gastric juice, all laboratory-processed indirect composites, with the exception of AD, demonstrated color differences significantly above the value of 3.3, a threshold value considered as clinically noticeable to the human eye and thus unacceptable under clinical conditions[23]. ΔE values ranging between 1 and 3.3 represent a perceptible and clinically acceptable color difference. Different monomers might cause resin composite materials to vary in color. Although AD and SOL consisted mainly of UDMA as a matrix monomer, SOL showed the highest ΔE value whereas AD showed the lowest ΔE value. Hence, the matrix alone could not be held responsible for the color change of resin composites. In the case of AD, its color change might be associated with the extended polymerization time (25 min of heat/light) performed using the Targis Power Upgrade polymerization unit.

It has been reported that materials with high filler content had less color change[24]. Although TES had higher filler content than the other materials tested, its color change was greater than those of SIN and AD. Thus, inorganic filler amount alone could not be held responsible for color change. The color of esthetic restorative materials is determined not only by more macroscopic phenomena such as matrix, filler composition and filler content, but also by minor aspects such as pigment additions and potentially by all other chemical components of these materials, including the initiation monomers and silane coupling agents[25]. Nevertheless, future studies should focus on the polymerization mode which might have more impact on the degree of conversion, and hence color change[25-27].

Among the tested resin composites, TES showed the highest Ra_1 value (p<0.001). This might be associated with its higher inorganic filler content compared to the other materials. One possible reason for the differences in surface roughness among the resin composites could be attributed to the polishing systems as recommended by the respective manufacturers. Each polishing system consisted of its own polishing paste and polishing stick. The use of adjustment kit alone or preceding polishing paste or polishing stick application might have a different effect on the surface roughness of specimens. Although TES showed the highest Ra_1 value, it yielded the lowest mean ΔRa value. This result indicated that gastric juice had a greater impact on the surface roughness of TES than the other resin composites. It was reported that the surfaces of resin-based restorative materials became significantly rougher after they were subjected to the pH-cycling regime. This result could be ascribed to the capability of acid media to soften resin-based restorative materials[28]. Topographical analysis may clarify the depth of erosion in each material.

The hardness of resin composites depends on the degree of conversion of the polymer matrix and the volume of reinforcing filler particles[26]. Additional polymerization using special devices could increase cross-linking, which then enhances the hardness of the polymer matrix[29]. While some studies reported a positive correlation between increased microhardness and inorganic filler content of resin composites[9,29], others could not find any correlation between filler content and mechanical properties of composites[26,30-32]. Similarly, no correlation could be established between surface microhardness and inorganic filler content or size of the resin composites tested in the present study. Significant differences in microhardness values might be the result of their individual matrix polymer components[32]. Despite the high microhardness value of SOL prior to its exposure to gastric juice, it was the most affected material with the largest ΔVHN value. Its chemical composition influenced the degree of conversion during polymerization, resulting in lower resistance to indentation[33]. The material least affected by gastric juice was SIN, whereby its polymerizable monomer had no hydrophilic group. The absence of hydrophilic character in SIN might be the reason for its higher microhardness values among the resin composites.

In the present study, specimens were polished using the polishing method and instruments recommended for the respective resin composites. It was probable that a single polishing method for all resin composites could yield more uniform surface roughness. This is an aspect which needs to be investigated in future studies, although this approach may not comply with manufacturers' instructions. Further clinical investigations are also needed to examine if the tested laboratory-processed indirect composites fulfil mechanical and optical expectations in patients suffering from GERD.

CONCLUSIONS

Based on the results of this study, the following conclusions were drawn:

1. Exposure to simulated gastric juice for 24 h affected the color stability, surface roughness and microhardness of four different, laboratory-

processed, indirect resin composites.
2. Except for AD, the color change of all other resin composites was perceivable to the human eye and was clinically unacceptable.

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