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Original

Color stability, gloss, and surface roughness of indirect composite resins

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Abstract: This *in vitro* study evaluated the color stability, gloss, and surface roughness (Ra) of four indirect composites. Enamel and dentin shades of Radica, Sculpture Plus, Belleglass-NG, and Gradia Indirect were evaluated for color stability. Specimens were stored in a staining solution (coffee) and assessed every 3 days for a period of 21 days, after which color difference (ΔE*) was calculated. Only enamel shades were used for measuring gloss and surface roughness at 0, 5000, 10,000, and 20,000 cycles of simulated toothbrushing. The results were statistically analyzed using repeated measures analysis of variance (ANOVA) and Tukey's test. Statistically significant changes in ΔE* during the 21-day period were observed in all indirect composites for enamel and dentin shades, and ΔE* was greater than the clinically acceptable value of 3.3. Belleglass-NG showed the least change in ΔE*, while Sculpture Plus had large changes in ΔE*. In all groups, gloss was 75% lower than baseline values after 10,000 cycles of toothbrushing. After 20,000 cycles, surface roughness was highest in Radica and lowest in Belleglass-NG. Belleglass-NG had higher color stability and gloss retention, and lower surface roughness, as compared with the other composite systems. (J Oral Sci 55, 9-15, 2013)

Keywords: composite resin; gloss; roughness; staining.

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Introduction

New microhybrid composite resins contain high densities of filler and dramatically differ in form, size, and composition from the earlier generation of indirect composites (1). These indirect composites are used in several clinical applications, including inlays and onlays, laminated veneers and jacket crowns, implant-supported restorations, and prostheses (2,3). As compared with direct composite restorations, indirect composite technique has better potential to produce an appropriate anatomic form, as well as proximal contacts and contours, excellent occlusal morphology, and good marginal accuracy (3,4).

Color stability is vital for the longevity of the facing on removable or fixed partial dentures, crowns, or direct restorations in esthetically important areas (5). *In vitro* studies (6,7) showed that resin-based composites are susceptible to staining and discoloration, and clinical studies have confirmed these *in vitro* findings. In a double-blind pilot study (8), Setz et al. compared two composites used in veneering telescopic dentures and observed significant discoloration after 1 year. Rosentritt et al. (9) used a reflection spectrophotometer to measure the color stability of laboratory-made composite veneers and concluded that discoloration in the test material was clinically unacceptable.

Gloss originates from the geometrical distribution of the light reflected by the surface and is an attribute of visual appearance (10). Surface gloss affects the esthetic appearance of restorations (11). Differences in gloss between a restoration and the surrounding tooth structure can be detected even when colors are matched. Gloss is influenced by a variety of factors, such as the filler size distribution, the index of refraction of the fillers

Table 1 Polymerization methods for the four investigated commercial composites

Material (Shade: enamel)	Curing method
Radica (Dentsply) Lot #061101	Enterra Curing Light (heat [80ºC+ halogen light]): 5 min of initial cure plus 2 min of Pontic cure
Belleglass-NG (Kerr Corp) F-Lot #2714305	Belleglass Curing Unit (heat [140ºC and nitrogen pressure]): Initial light cure with LED visible light for 20 s followed by 20-min cure cycle under nitrogen pressure (413685 Pa).
Gradia Indirect (GC, Tokyo, Japan) Lot #0612251	Gradia Curing Unit and Step Curing Light (halogen light): 20 s under Step Curing Light followed by 5 min in Gradia Curing Unit
Sculpture Plus (Pentron Lab) Lot #156905	Sculpture Curing Unit (heat, pressure, light): build-up cycle and final cycle each 8 min (each cycle 5 min under nitrogen pressure [551581 Pa] and 3 min of halogen light)

present in the plastic, and the viscosity of the resin matrix components (12). Color stability and gloss may also be influenced by surface roughness (13).

It is essential to observe stain resistance and gloss retention in indirect composites. We evaluated color change in four commercial indirect composite resins after exposing the materials to concentrated coffee slurry and assessed the gloss retention and surface roughness of these materials after subjecting them to simulated toothbrushing. The null hypothesis was that there would be no color change, decrease in gloss, or increase in surface roughness over time.

Materials and Methods

The resin composites examined were obtained directly from the manufacturers. The polymerization parameters and lot number for each resin composite are shown in Table 1. Enamel and dentin shade A2 were used for evaluating color stability, while only the enamel shade was used for measuring gloss and surface roughness.

Color stability

Six specimens were obtained for each group using a custom-made stainless steel mold with a diameter of 12.5 mm and a thickness of 2 mm. The material was placed in the mold in 1-mm increments. Each increment was polymerized according to the manufacturer's instructions, as described in Table 1. A polyester film (Mylar strip) was placed over the final increment and then pressed with a cover slide to ensure that the material was flush with the surface of the mold. Following polymerization, the specimens were carefully removed from the mold. After 24 h of dark storage in distilled water at room temperature, the baseline color of all the specimens was measured. CIE (Commision Internationale de l'Eclairage) L*a*b* values were obtained with a spectrophotometer (CM 2500d, Minolta, Osaka, Japan) and D65 light against a white background. All measurements were repeated three times, and the means for the L*a*b* values were calculated. After obtaining the baseline measurements, the specimens from each group were stored in a staining solution. The staining solution consisted of instant coffee and was prepared by adding 5 ounces (approximately 142 g) of coffee to 750 mL of boiling water.

After 72 h in the solution, color measurements were obtained again using the spectrophotometer, as described above. Before color measurement, the specimens stored in the staining solution were rinsed thoroughly under tap water and subjected to 10 strokes of brushing with a soft-grade toothbrush. The specimens were then gently dried with delicate paper wipes. The color measuring process described above was repeated every 72 h for 3 weeks. The change that occurred during each 72-h interval was calculated using the formula $\Delta E^* = [(\Delta L^*) 2 + (\Delta a^*) 2+ (\Delta b^*) 2]1/2$, where ΔE^* represents the color difference and ΔL^*, Δa^*, Δb^* represent changes in lightness, red-green coordinate, and yellow-blue coordinate, respectively. All measurements were obtained in the specular component-excluded (SCE) mode.

Gloss and surface roughness

Specimens (n = 10) of each composite material were fabricated using a custom-made stainless steel mold measuring 2 mm in thickness, 5 mm in width, and 20 mm in length. The material was placed in the mold in 1-mm increments. Each increment was then cured in the appropriate curing unit and polymerized as described in Table 1. A Mylar strip was placed over the final increment and pressed with a cover slide to ensure that the material was flush with the surface of the mold. After polymerization, the specimens were carefully removed from the mold and

Table 2 Mean (SD) ΔE* for enamel shade of composite resins at each time point

Measurement interval SCE (ΔE*)	Sculpture Plus	Radica	Gradia Indirect	Belleglass-NG
Baseline	0	0	0	0
Day 3	17.6 (3.5)[a]	5.2 (1.1)[b]	2.4 (0.9)[c]	2.5 (1.5)[c]
Day 6	23.0 (4.2)[a]	8.1 (2.2)[b]	4.4 (1.2)[c]	3.4 (2.5)[c]
Day 9	26.4 (4.8)[a]	7.6 (1.5)[b]	5.9 (1.4)[c]	2.9 (1.8)[d]
Day 12	28.7 (4.9)[a]	11.1 (1.7)[b]	7.3 (1.6)[c]	2.1 (1.0)[d]
Day 15	30.1 (4.8)[a]	10.7 (1.9)[b]	8.2 (0.2)[c]	3.2 (1.3)[d]
Day 18	31.9 (5.3)[a]	11.0 (1.7)[b]	8.0 (1.1)[c]	4.2 (1.8)[d]
Day 21	32.1 (5.0)[a]	11.4 (2.0)[b]	8.4 (1.0)[c]	4.6 (2.0)[d]

* In each row, values with the same superscript letter are not statistically different ($P > 0.05$, Tukey's test)

Table 3 Mean (SD) ΔE* for dentin shade of composite resins at each time point

Measurement interval SCE (ΔE*)	Sculpture Plus	Radica	Gradia Indirect	Belleglass-NG
Baseline	0	0	0	0
Day 3	14.5 (10.3)[a]	3.9 (1.9)[b]	3.1 (0.5)[b]	2.7 (0.9)[b]
Day 6	16.6 (10.5)[a]	6.6 (2.3)[b]	4.4 (0.8)[c]	3.1 (1.1)[c]
Day 9	16.9 (9.8)[a]	6.0 (2.6)[b]	5.4 (0.7)[b]	3.3 (1.5)[c]
Day 12	21.1 (11.3)[a]	6.9 (2.2)[b]	6.0 (0.8)[b]	3.8 (1.2)[c]
Day 15	22.4 (10.7)[a]	7.6 (2.4)[b]	6.6 (0.8)[b]	4.2 (1.1)[c]
Day 18	22.8 (11.3)[a]	8.0 (2.5)[b]	7.2 (0.7)[b]	4.6 (1.3)[c]
Day 21	22.9 (10.9)[a]	8.3 (2.6)[b]	7.1 (0.7)[b]	4.7 (1.1)[c]

* In each row, values with the same superscript letter are not statistically different ($P > 0.05$, Tukey's test)

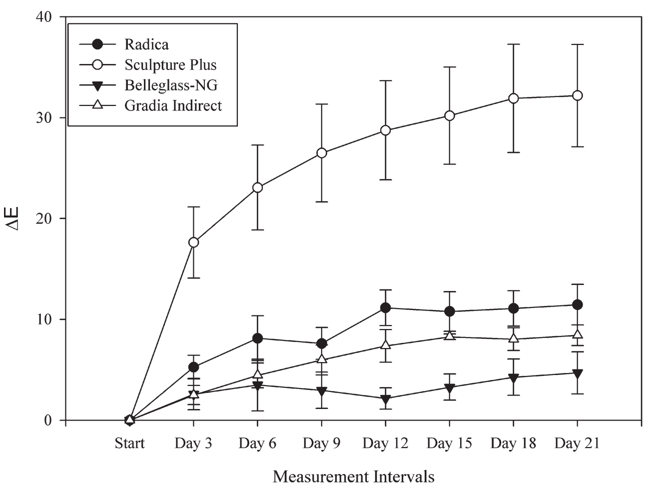

Fig. 1 Mean values for enamel shade of composite resins.

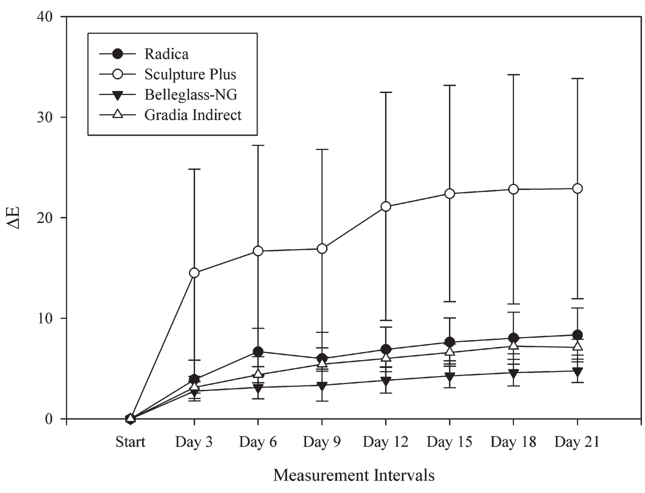

Fig. 2 Mean values for dentin shade of composite resins.

stored in distilled water at room temperature for 24 h. They were then finished with SiC discs (3M, St. Paul, MN, USA) on a polishing wheel using the sequence 400 grit, 600 grit, 800 grit, 1,200 grit, and further polishing with diamond polishing pastes (Buehler, Lake Bluff, Illinois, USA) with a particle size of 1 µm and 0.25 µm. Care was taken to observe the surface of the specimen using light microscopy (Nikon, Tokyo, Japan) at 5× magnification during every stage of finishing and polishing to ensure that the scratch lines were decreasing in size.

After the specimens were polished, baseline measurements were taken to record gloss and surface roughness. A glossmeter (Nikon) was used to measure gloss at a 60º angle from normal to the surface. Surface roughness (Ra) was measured along the length of the specimen, using a contact profilometer (Taylor Hobson, Leicester, UK).

Table 4 Mean (SD) values for gloss and surface roughness (Ra) of composite resins

Cycles	Gloss				Ra (µm)			
	Radica	Sculpture Plus	Belleglass-NG	Gradia Indirect	Radica	Sculpture Plus	Belleglass-NG	Gradia Indirect
0	87.3^{a} (1.5)	89.5^{a} (1.5)	81.5^{b} (1.9)	78.3^{b} (2.21)	0.023^{a} (0.008)	0.012^{b} (0.003)	0.011^{b} (0.007)	0.025^{a} (0.003)
5,000	35.8^{b} (9.9)	12.7^{a} (3.7)	45.3^{c} (9.11)	18.4^{a} (4.4)	0.226^{a} (0.06)	0.223^{a} (0.05)	0.134^{b} (0.03)	0.283^{c} (0.05)
10,000	15.5^{a} (6.0)	12.6^{a} (2.9)	26.2^{b} (10.2)	13.7^{a} (2.3)	0.3^{a} (0.06)	0.252^{b} (0.07)	0.204^{b} (0.04)	0.306^{a} (0.06)
20,000	9.5^{a} (2.4)	9^{a} (1.9)	19.8^{b} (7.9)	9.8^{a} (2.9)	0.447^{a} (0.07)	0.355^{b,c} (0.09)	0.3^{c} (0.07)	0.374^{b} (0.05)

* In each row, values with the same superscript letter are not statistically different ($P > 0.05$, Tukey's test)

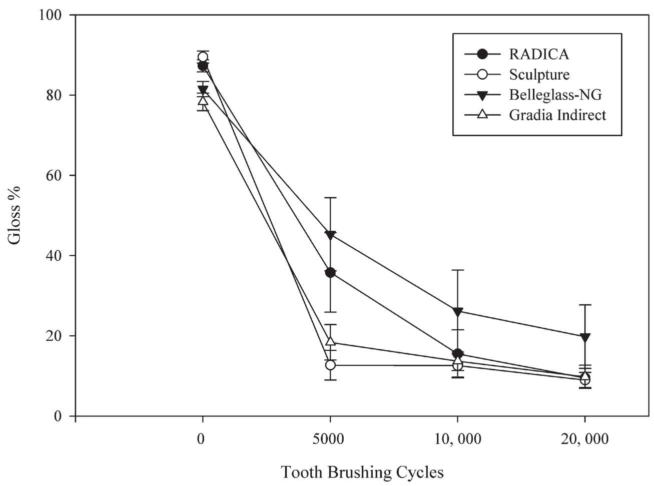

Fig. 3 Gloss of composite resins.

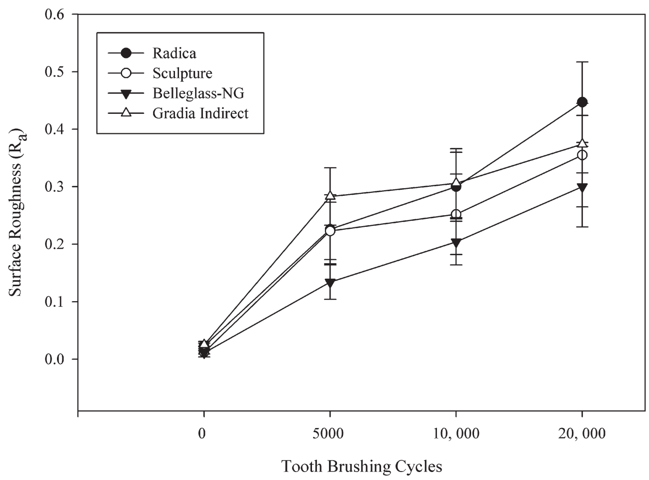

Fig. 4 Surface roughness of composite resins.

The length of the stylus scan was 4 mm. Three readings were taken in the central region of the specimen.

The specimens were then brushed with a Pepsodent toothbrush abrasion machine, using toothbrush heads with medium-grade bristles (Colgate-Palmolive Company, New York, NY, USA) and a slurry comprised of a 1:1 ratio of deionized water and dentifrice (Colgate Total Toothpaste, Colgate-Palmolive Company) with a relative dentin abrasivity (RDA) of 70. The specimens were brushed for 20,000 cycles at 170 cycles per minute and were removed after 5,000, 10,000, and 20,000 cycles. They were then cleaned with distilled water in an ultrasonic bath and dried with canned air and delicate-task wipes before measuring gloss and surface roughness. Images of the surface topography of the four indirect composites were obtained at 0, 5,000, 10,000, and 20,000 cycles using a light microscope.

Values for color change, gloss, and surface roughness were analyzed by repeated measures analysis of variance (ANOVA) and Tukey's test for pair-wise comparisons. The level of statistical significance was $\alpha = 0.05$.

Results

The mean (SD) values for the color difference (ΔE^*) in each group at each time point are shown in Tables 2 and 3 and Figs. 1 and 2. Discoloration worsened over time. A statistically significant difference was observed among composites when they were stored in the concentrated coffee slurry. Belleglass-NG had the lowest ΔE^*, while Sculpture Plus had the highest ΔE^*.

Gloss and Ra values are presented in Table 4 and Fig. 3 illustrate the decrease in gloss. Figure 4 shows Ra in relation to the number of cycles. Filler exposure and tracks produced by toothbrush bristles were noted after 10,000 cycles in all materials (Fig. 5). The results showed that all materials had high initial gloss. However, in all test materials gloss decreased and roughness increased as the number of toothbrushing cycles increased. As compared with the other materials, Belleglass-NG better retained gloss at 5,000 cycles. At 10,000 cycles, however, all groups showed a decrease of 75% from baseline gloss values. At 20,000 cycles, Belleglass-NG had the highest gloss of all tested materials, and surface roughness was

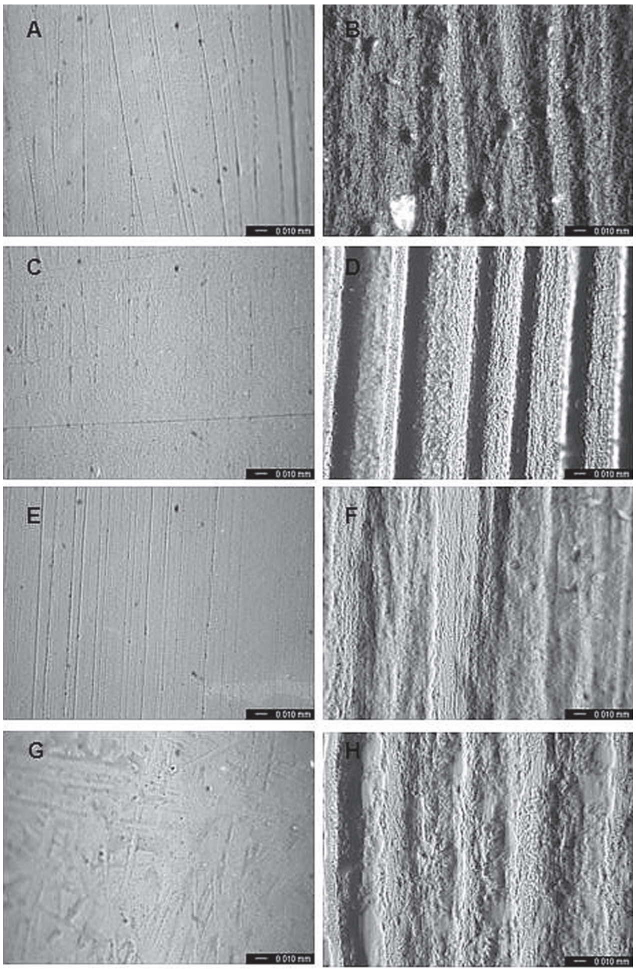

Fig. 5 Surface topography at baseline: A, Radica; C, Sculpture Plus; E, Belleglass-NG; G, Gradia Indirect. The polished surfaces can be seen. Surface topography at 10,000 cycles: B, Radica; D, Sculpture Plus; F, Belleglass-NG; H, Gradia Indirect. The tracks produced by toothbrush bristles and filler exposure are clearly visible.

highest in Radica and lowest in Belleglass-NG.

Discussion

Composite resins are restorative materials that are widely used in esthetic procedures due to their excellent properties and superior adhesion to enamel and dentin. However, one disadvantage of these materials is that their surface color changes over time, which is the main reason for replacing such restorations (14). There are many intrinsic and extrinsic factors that influence the color stability of composite resins. In regard to intrinsic factors, the resin matrix composition, filler loading, size and nature of the particles (13), quantity of photo-initiator or inhibitor (15), and the degree of polymerization (16) must be considered. However, exposure to food colorants, UV radiation (17), temperature changes, and water (18,19) are the main extrinsic factors in the hydrolysis and degradation of composites, which affect the appearance of these materials.

In the current study, the null hypothesis regarding color change was rejected: mean ΔE* increased in all four groups during the 21-day period. Perceptible color change (ΔE*) ranged from 4.69 to 32.18 for enamel and from 4.79 to 22.9 for dentin in SCE geometry. A spectrophotometer with an integrating sphere can operate in two different measuring geometries: specular component-included (SCI) and specular component-excluded (SCE). Lee et al. (20) showed that color changes measured with SCE geometry were greater than those measured with SCI geometry. For this reason, the measurements in this study were recorded using SCE geometry only.

Although the test was conducted under stringent conditions, the mean ΔE* of all the indirect composites was higher than what is considered to be clinically acceptable (ie, ΔE* = 3.3) (18,21). It is believed that an ΔE* less than 3.0 indicates color stability of a composite material (22). Our findings showed that Belleglass-NG had higher color stability, possibly due to the curing mechanism of the material. Belleglass-NG specimens were cured under nitrogen pressure at 140°C for 20 min, which may have improved the degree of conversion (23). The color stability of composite materials depends on the conversion of matrix monomers (24,25), and insufficient monomer conversion induces absorption of staining substances (6). Samra et al. (26) also noted higher color stability in Belleglass specimens.

We expected that a secondary curing with heat and light would increase the degree of conversion (27) and thus possibly improve stain resistance. Although Sculpture Plus is cured under nitrogen pressure, heat, and light, it showed the highest discoloration. The composition and interaction of the resin and filler may have contributed to the stain resistance of this material. Overall, the present results suggest that post-curing at a higher temperature does not increase the stain resistance of composites to a clinically acceptable level. However, Stawarczyk et al. (28) showed that extrinsic discolorations caused by tea, coffee, and wine solutions were removed by polishing with a prophylaxis paste and that ΔE* decreased to less than 3.3.

The color changes of specimens were likely related to the staining solution used in the study. Coffee contains yellow colorants, which have different polarities. The sorption of colorants into the organic phase of the material was most likely due to the compatibility of the polymer phase with the yellow colorants of the coffee (21). As compared with other immersion media, coffee has the highest effect on the color stability of composite resins (29).

Restorations with high surface roughness are more susceptible to staining (13). To decrease the influence of roughness in the color stability test, the surfaces of specimens were polymerized under a polyester film (Mylar strip). However, according to Shintani et al. (30) and Patel et al. (31), the surface beneath the polyester film strip may not have the same degree of polymerization as the bulk of the resin material and, hence, may be vulnerable to staining.

Because daily toothbrushing with dentifrices changes the surface condition of composite materials (32-34), toothbrush/dentifrice abrasion testing was used to simulate clinical conditions in the oral environment in the present study. The specimens were brushed for 20,000 cycles, which simulates the use of a toothbrush for approximately 2 years (35). The composition of the dentifrice influences the loss of surface polish of the restorations (34). For this reason, only Colgate Total dentifrice was used, as it is a representative dentifrice for daily toothbrushing. According to the manufacturer's information, this dentifrice has medium abrasiveness (RDA, 70).

The null hypothesis for gloss and surface roughness was rejected because both had significantly changed after toothbrushing. The images in Fig. 5 illustrate the data shown in Table 4. Initially, all composite materials had high gloss. However, gloss had decreased by more than 75% in all materials at 10,000 cycles, perhaps due to surface disruption and filler exposure caused by toothbrushing. Toothbrushing produces microscopic and macroscopic roughness that causes incident light to be diffusely reflected, thus reducing gloss. Our results confirm those of Murakami (36), who found that the surface gloss of composite was significantly reduced by toothbrush/dentifrice abrasion.

Toothbrushing increases surface roughness of composite material and decreases the gloss of composite restorations (37). Belleglass-NG had the highest gloss retention and lowest R_a values at all time points, perhaps because the curing mechanism for Belleglass-NG improved surface properties, due to the higher degree of conversion (23). Another factor could be the composition of the composite resin, since both the resin matrix and filler particle type or content are thought to affect surface condition after toothbrushing, as long-term toothbrushing causes selective abrasion of the resin matrix and dislodgment of filler particles (32). Other studies that evaluated the effect of toothbrushing on deterioration of indirect composite resins showed a rapid increase in surface roughness as well as differences between materials (32,33).

Measurement of surface roughness was conducted using a contact profilometer with a stylus that moved along the length of the specimen, parallel to the direction of toothbrushing. Three measurements were taken, and the average was defined as mean Ra. Some authors have reported measurements taken perpendicular to the direction of toothbrushing (38), which may differ from those reported in the present study. Sophisticated instruments, such as non-contact optical interferometers and atomic force microscopes (AFM), are available to measure roughness at a much higher resolution and over a larger area. These devices might prove useful in future in-depth analyses.

In conclusion, the Belleglass-NG, Gradia Indirect, Radica, and Sculpture Plus indirect composite resin systems significantly differed in color stability, gloss, and surface roughness over time. Mean ΔE^* was greater than the clinically acceptable value of 3.3, and gloss had decreased by more than 75% in all investigated composites, at 10,000 cycles. However, as compared with the other composite systems, Belleglass-NG had greater color stability and gloss retention and less surface roughness. Within the limited scope of this study, our findings indicate that color stability and gloss remain a concern for these materials.

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