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Micro-Hardness and Surface Roughness of Bulk-Fill Composite Resin: Effect of Surface Sealant Application and Two Bleaching Regimens

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Abstract

Objectives The aim of this study was to assess the impact of applying a surface sealant and two bleaching regimens, on the micro-hardness, and surface roughness of bulk-fill composite resin

Materials and Methods One-hundred twenty specimens were prepared with a diameter of 5 mm and thickness of 4 mm using bulk-fill composite. Specimens were subdivided into group 1: ($n = 60$), in which Fortify surface sealant was used, and group 2 (control): ($n = 60$), in which specimens were only finished and polished. After applying in-office and at-home bleaching, micro-hardness and surface roughness were measured before and after bleaching. Statistical analysis was performed using Student's *t*-test and paired test.

Results Hardness values of group 1 were lower than values of group 2 before and after bleaching, with different bleaching procedures. Applying surface sealant and different bleaching regimens led to a significant difference of surface roughness ($p < 0.001$).

Conclusion Applying surface sealant decreases both micro-hardness and surface roughness. The bleaching procedures significantly affect the surface roughness, but not the micro-hardness.

Clinical Significance Surface sealant can enhance the surface roughness of bulk-fill composite in nonstress bearing area as it reduces the surface micro-hardness.

Keywords
- bulk-fill
- composite
- at-home bleaching
- in-office bleaching
- micro-hardness
- surface roughness
- surface sealant

Introduction

Composite resins are extensively used in dental practice as a restorative material due to their optical and esthetic properties. Composites are available in different shades and opacities. They have appropriate handling, characteristics, and efficient working time. They also have lower cost compared to other indirect restorations.[1,2]

An incremental filling technique was effectively used for an extended period of time. This was in order to enhance the adaptation of margins and to decrease shrinkage stress.[3,4] Bulk-fill resin-based composites (RBCs) are restorative materials that are suitable for patients who suffer from restricted consistence.[5] It was proved that the bulk-filled composite resin has a minimal shrinkage stress, as well as a low shrinkage rate estimation, compared to nonflowable and flowable nano-hybrid composites.[6]

The bleaching procedure is an effective strategy that can be utilized to improve the shade and color of the teeth.[7]

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There are many dental bleaching regimens that have been identified in various studies. They include three major bleaching approaches which are at-home, in-office powered bleaching, and over the counter.[8]

Bleaching procedures are often requested by patients who have direct or indirect restorations.[9] Therefore, it is important to consider the mechanical, as well as the physical characteristics of the restorative material that affect the prognosis and survival of restorations.[10]

Bleaching can result in adverse effects on the existing restorations regarding hardness and surface roughness. Both features could essentially affect the clinical longevity of restorations, bacterial adhesion, biofilm formation, and staining. Previous studies reported contradictory outcomes in relation to the changes of composite micro-hardness after bleaching of composite resins.[11–13]

Th use of a surface sealant can fortify the organic matrix of composite resin and plug the microcracks in the surface. Additionally, it decreases surface porosity and creates a surface that is more thoroughly cured, improving the composite's stain resistance.[14] Although finishing and polishing procedures can provide a clinically acceptable surface, a surface sealant can fortify the organic matrix of composite resin and plugs the microcracks in the surface.[15]

The bulk-fill composite has become a successful restorative material. Due to continuous interest in aesthetic; and recorded adverse effects of the bleaching on the composite resin restoration, it was essential to investigate the ability of the surface sealant to protect the bulk-fill composite resin restoration after bleaching.

Consequently, the main goal of this study was to determine the impact of in-office as well as at-home bleaching on Vickers micro-hardness, and also the surface roughness of bulk-fill composite resins, either sealed or unsealed with a surface sealant. The null-hypotheses were that there's no difference in hardness or surface roughness of composite resin, whether sealed or unsealed, before or after applying the bleaching procedure. Also, the application of the bleaching regime has no significant effect on the changes in micro-hardness, neither the roughness of composite surface.

Materials and Methods

This in-vitro study was approved by the ethics committee of Faculty of dentistry Tanta University (#R-BIO-11-22-12).

The specimens were prepared and tested in the following procedure:

Specimens Preparation

Materials used in the current study are displayed in ►**Table 1**.

Hundred and twenty composite resin discs were prepared with a diameter of 5 mm and thickness of 4 mm using a Filtek Bulk-Fill (3M ESPE, St. Paul, Minnesota, United States) with a Teflon mold. Every specimen was light cured based on the

Table 1 Materials used in study, type, manufacturer, and composition

Material	Type	Manufacturer	Composition
Filtek Bulk Fill	Resin-based composite	3M/ESPE, St. Paul, Minnesota, United States	The monomer, the resin matrix AUDMA, UDMA, and 1, 12-dodecane-DMA. The filler nonagglomerated/nonaggregated 20 nm silica filler, a nonagglomerated/nonaggregated 4 to 11 nm zirconia filler, nonaggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4–11 nm zirconia particles) and a ytterbium trifluoride filler consisting of agglomerate 100 nm particles filler load 76.5%wt(58.4%vol) Filler size 0.01–3.5 µm
Opalescence15%; take-home whitening gel	At-home bleaching material	Ultradent Products Inc., South Jordan, Utah, United States	A 15% carbamide peroxide gel, potassium nitrate, and fluoride
Philips Dash 30%	30% hydrogen peroxide concentration	Philips, Amsterdam, the Netherlands p.8–5.2 45 min	A 30% hydrogen peroxide, thickeners, glycol, inorganic load, and deionized water
Fortify composite surface sealant	Surface sealant	BISCO Schaumburg, Illinois, United States	Potassium hydroxide 0–5%, 1-hydroxyethylidene-1,1-diphosphonic acid 0–5%, polymaleic acid based polymeric 0–5%, hydroxy phosphonoacetic acid 0% to 5% Bis-EMA UDMA 0.4 µm amorphous silica

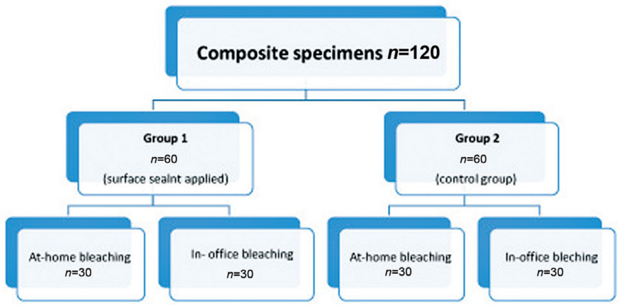

Fig. 1 Experimental study design.

manufacturer's instructions, via a Light C unit (Bluephase curing unit, Ivoclar Vivadent, Lichtenstein) at 650 mW/cm2 irradiance. Specimens were treated then stored in deionized water in an incubator, for 24 hours at 37°C.

Specimens were polished with a rubber disc (Kenda dental polisher Liechtenstein) followed by ultra polishing Super-Snap (Super-Snap, super buff set Shofu inc., Kyoto. Japan), then the polished discs were stored in deionized water for 24 hours before use.

Specimens were divided according to the application of the surface sealant into two main groups ($n=60$): 1. Fortify composite surface sealant (Bisco, Inc. United States) and 2. control (no sealant). Each group was further divided into two subgroups according to bleaching regime used into ($n=30$): In-office bleaching and at-home bleaching regimes.

The experimental study design is shown in ►**Fig. 1**.

Material Application

Surface sealing: Specimens were washed using 32%phosphoric acid (3M ESPE. St. Paul, Minnesota, United States) for 15 seconds, washed with distilled water, then air dried. Next, the sealant material was applied to the surface, and light-cured for 30 seconds based on the guidelines of the manufacturer.

Bleaching regimes: At-home bleaching gel (opalescence 15% take-home whitening gel) (Ultradent Products Inc., South Jordan, Utah, United States) was applied to the surface of the composite at 37°C for 6 hours a day, based on the directions of the manufacturer for 8 days. Dash In-Office Whitening System (Philips, Amsterdam, the Netherlands) was used with a concentration of 30% hydrogen peroxide (pH 4.8–5.2). The bleaching gel was applied according to the instructions of the manufacturer in three cycles, each cycle lasted for 15 minutes. After each cycle, the gel was carefully removed and the specimens were rinsed.

Vickers Micro-hardness Measurements

Vickers micro-hardness was measured before bleaching. It was performed to each specimen via a micro-hardness tester (ZwicRoell, West Midlands, England), with a load of 100 gm, and a steadiness time of 15 seconds per side, 3 points were taken on the surface. The average micro-hardness was computed based on the three-point values and documented for each specimen as an initial Vickers micro-hardness value. Initial micro-hardness measurements were calculated for the composite specimens before applying the bleaching procedure. After performing either at-home or in-office bleaching, the measurements of specimens were repeated.

Surface Roughness Measurements

Initial surface roughness measurements of the specimens were recorded using a three-dimensional optical surface profilometer (ZYGO Maxim-GP 200 profilometer, United States).

In this technique, a pattern of dark lines and bright lines arises due to the difference in the optical pathways between a reference and a sample beam. Received light is split inside an interferometer with one beam going to an internal reference surface and the other to the sample. After reflection, the beams recombine inside the interferometer and undergo constructive and destructive interference, which produce the light and dark fringe patterns. An upright scanning transducer and camera produce a three-dimensional interferogram of the surface. This interferogram is managed by a computer and converted using frequency analysis that leads to a noncontact three-dimensional.

The surface roughness parameter (Ra: an arithmetic mean of the sum of roughness profile values) was selected in the current study.

The specimens were scanned via high-resolution optical beam. The optical profilometer shows the three-dimensional topography of the specimens. Surface roughness measurements of the specimens were repeated for each composite specimen after applying the bleaching materials either at-home or in-office bleaching agents.

Statistical Analysis

Statistical analysis of Vickers micro-hardness and surface roughness measurements was performed using *t*-test to compare group 1 with group 2, as well as the paired test, which was used to compare the measurements of each specimen done before and after bleaching. Analysis was performed via software version 25.0, SPSS (Statistical Package for Social Sciences). Level of significance was *p*-value less than or equal to 0.001.

Results

According to Vickers micro-hardness measurements, all hardness values of group 1 were lower than that of group 2, either before or after bleaching with different bleaching procedures. *t*-Test revealed that the application of surface sealant resulted in a significant difference between the two groups tested, either before or after bleaching, or whether in-office or at-home bleaching) ($p<0.001$) as shown in ►**Table 2**. In group 1 (where a surface sealant was applied) and group 2 (which was considered as a control group with no application of sealant), the paired t-test also showed that the different bleaching regimes had no noticeable effect on the micro-hardness. There was no significant difference between the initial measurements, after at-home bleaching and after in-office bleaching ($p>0.001$; (►**Tables 3** and **4**)

Table 2 Means, SD, and statistical differences of the Vickers microhardness of bulk-fill composite resin of group 1 and group 2 before, after at-home bleaching, and after in-office bleaching

Measurements of Vickers microhardness		Group						*t*-Test	
		Group 1			Group 2			*t*-Value	*p*-Value
Before bleaching	Range	50.45	–	53.66	66.15	–	66.65	–76.830	<0.001*
	Mean ± SD	53.177	±	0.934	66.433	±	0.146		
After at-home bleaching	Range	50.31	–	53.56	66.12	–	66.72	–49.658	<0.001*
	Mean ± SD	52.452	±	1.532	66.418	±	0.162		
After in-office bleaching	Range	50.31	–	53.62	66.13	–	66.93	–63.810	<0.001*
	Mean ± SD	52.841	±	1.151	66.420	±	0.181		

Abbreviation: SD, standard deviation.
*indicate statistically significant difference ($p < 0.001$).

Table 3 Comparison of Vickers microhardness in group 1 among different bleaching regimens (before, after at-home bleaching, and after in-office bleaching)

Group 1	Measurements of Vicker hardness						Comparison	Differences		Paired test	
	Range			Mean	±	SD		Mean	SD	*t*-Value	*p*-Value
Before bleaching	50.45	–	53.66	53.177	±	0.934	B-AH	0.725	2.010	1.975	0.058[a]
After at-home bleaching	50.31	–	53.56	52.452	±	1.532	B-AO	0.336	1.626	1.132	0.267[a]
After in-office bleaching	50.31	–	53.62	52.841	±	1.151	AH-AO	–0.389	1.207	–1.765	0.088[a]

Abbreviation: SD, standard deviation.
Statistically significant difference ($p < 0.001$).
Note: B indicates before bleaching, AH indicates after at-home bleaching, and AO indicates after in- office bleaching.

Table 4 Comparison of Vickers microhardness in group 2 among three different bleaching regimens (before, after at-home bleaching, and after in-office bleaching)

Group 2	Measurements of Vicker hardness						Comparison	Differences		Paired test	
	Range			Mean	±	SD		Mean	SD	*t*-Value	*p*-Value
Before bleaching	66.15	–	66.65	66.433	±	0.146	B-AH	0.015	0.230	0.357	0.724[a]
After at- home bleaching	66.12	–	66.72	66.418	±	0.162	B-AO	0.013	0.245	0.283	0.779[a]
After in- office bleaching	66.13	–	66.93	66.420	±	0.181	AH-AO	–0.002	0.256	–0.050	0.961[a]

Abbreviation: SD, standard deviation.
Statistically significant difference ($p < 0.001$).
Note: B indicates before bleaching, AH indicates after at-home bleaching, and AO indicates after in-office bleaching.

While taking into consideration the measurements of surface roughness (Ra), *t*-test indicated that both the application of a surface sealant and different bleaching regimes led to a significant difference. In addition, there was a noticeable difference between group 1 and group 2 in measurement of surface roughness, taking into consideration the initial measurements, as well as in-office and at-home bleaching ($p < 0.001$) as shown in ►**Table 5**. Furthermore, there was a significant difference when comparing the changes in surface roughness of group 1 and group 2 after bleaching application. There was a significant difference between the initial measurements of at-home and in-office, and also between at-home and in-office bleaching (►**Tables 6** and **7**). ►**Fig. 2A–C** shows interferometer image of group 1 and ►**Fig. 3A–C** shows interferometer image of group 2.

Discussion

The tested null hypothesis was partially rejected as the bleaching procedures had no significant changes in micro-hardness either in sealed or nonsealed group. On the other hand, application of surface sealant led to a significant lowering of micro-hardness, both before or after bleaching. The bleaching procedure led to a significant change in surface roughness measurements in both sealed and unsealed specimens. The second hypothesis was also partially rejected as the difference between in-office and at-home bleaching was observed in the measurements of surface roughness, but not detected in the analysis of micro-hardness measurements.

All composite resins are composed of organic matter (usually dimethacrylate). A silane coupling agent is used to

Table 5 Means and SD and statistical differences of the surface roughness measurements of bulk-fill composite resin of group 1and group 2 before, after at-home bleaching, and after in-office bleaching

Measurements of surface roughness		Group						*t*-Test	
		Group 1			Group 2			*t*-Value	*p*-Value
Before bleaching	Range	0.111	–	0.222	0.235	–	0.238	–32.404	<0.001*
	Mean ± SD	0.133	±	0.018	0.238	±	0.001		
After at-home bleaching	Range	0.322	–	0.326	0.603	–	0.633	–256.734	<0.001*
	Mean ± SD	0.323	±	0.001	0.629	±	0.006		
After in-office bleaching	Range	0.233	–	0.239	0.571	–	0.576	–935.299	<0.001*
	Mean ± SD	0.237	±	0.002	0.573	±	0.001		

Abbreviation: SD, standard deviation.
*indicate statistically significant difference ($p < 0.001$).

Table 6 Comparison of surface roughness measurements in group 1 among three different bleaching regimens (before, after at-home bleaching, and after in-office bleaching)

Group A	Measurements of surface roughness						Comparison	Differences		Paired test	
	Range			Mean	±	SD		Mean	SD	*t*-Value	*p*-Value
Before bleaching	0.111	–	0.222	0.133	±	0.018	B-AH	–0.190	0.018	–59.060	<0.001[a]
After at-home bleaching	0.322	–	0.326	0.323	±	0.001	B-AO	–0.104	0.017	–32.574	<0.001[a]
After in-office bleaching	0.233	–	0.239	0.237	±	0.002	AH-AO	0.086	0.002	235.086	<0.001[a]

Abbreviation: SD, standard deviation.
Statistically significant difference ($p < 0.001$).
Note: B indicates before bleaching, AH indicates after at-home bleaching, and AO indicates after in-office bleaching.

Table 7 Comparison of surface roughness measurements in group 2 among three different bleaching regimens (before, after at-home bleaching, and after in-office bleaching)

Group 2	Measurements of surface roughness						Comparison	Differences		Paired test	
	Range			Mean	±	SD		Mean	SD	*t*-Value	*p*-Value
Before bleaching	0.235	–	0.238	0.238	±	0.001	B-AH	–0.391	0.007	–322.589	<0.001[a]
After at-home bleaching	0.603	–	0.633	0.629	±	0.006	B-AO	–0.335	0.002	–1209.517	<0.001[a]
After in-office bleaching	0.571	–	0.576	0.573	±	0.001	AH-AO	0.056	0.007	46.526	<0.001[a]

Abbreviation: SD, standard deviation.
Statistically significant difference ($p < 0.001$).
Note: B indicates before bleaching, AH indicates after at-home bleaching, and AO indicates after in-office bleaching.

connect the filler to the organic matter. Finally, chemicals are used to encourage the polymerization reaction. Due to the increased interest in esthetic dentistry, the use of composite resins has increased, in addition to the use of bleaching procedures. In some cases, bleaching techniques are used in the presence of composite restoration in the teeth to be bleached.[16] Bulk-fill (RBC) has become one of the most widely used restorative materials in various situations in the oral cavity. In the current study, Filtek Bulk-Fill was the selected material.

The most common bleaching materials used in dental practice are carbamide peroxide and hydrogen peroxide. The whitening agents that were used in this study were carbamide peroxide (15%) as at-home bleaching product (opalescence15% take-home whitening gel), and hydrogen peroxide (30%), as in-office bleaching (Philips Dash 30%).

It is obvious that the action of bleaching gels does not only affect the tooth structure, but also extend to the composite resin restoration. Several studies attempted to investigate those effects.[17]

The effect of bleaching agents on composite resin micro-hardness can be described as follows: carbamide peroxide degenerates into one-third hydrogen peroxide, and two-third urea when it interacts with water hydrogen peroxide, forming free radicals and resulting in the detachment of polymer chains, as well as breaking the double bonds in composite resin, which may be associated with the decreased surface micro-hardness.[18] In addition,

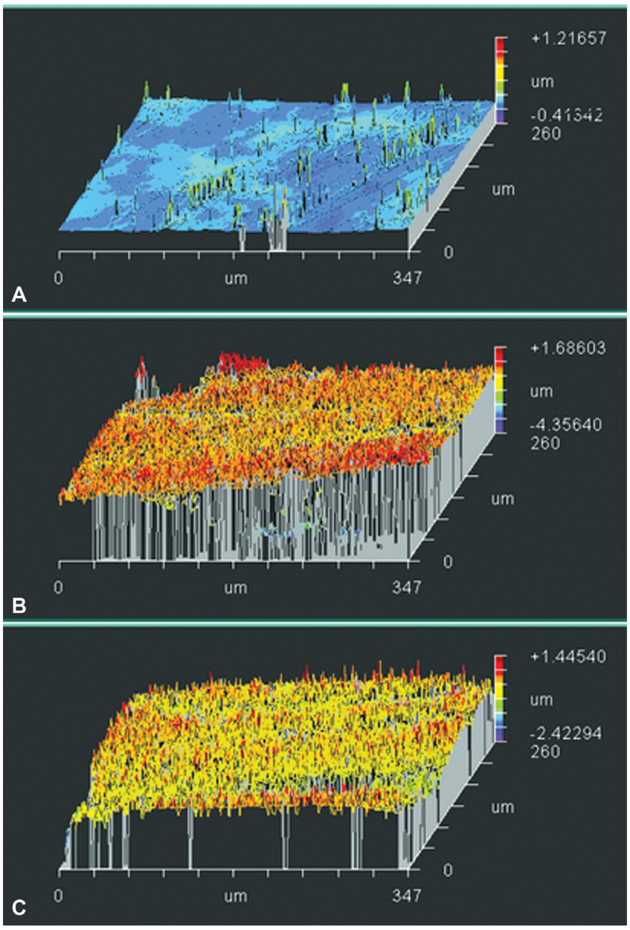

Fig. 2 (A before bleaching, B after at- home bleaching and C after in-office bleaching).

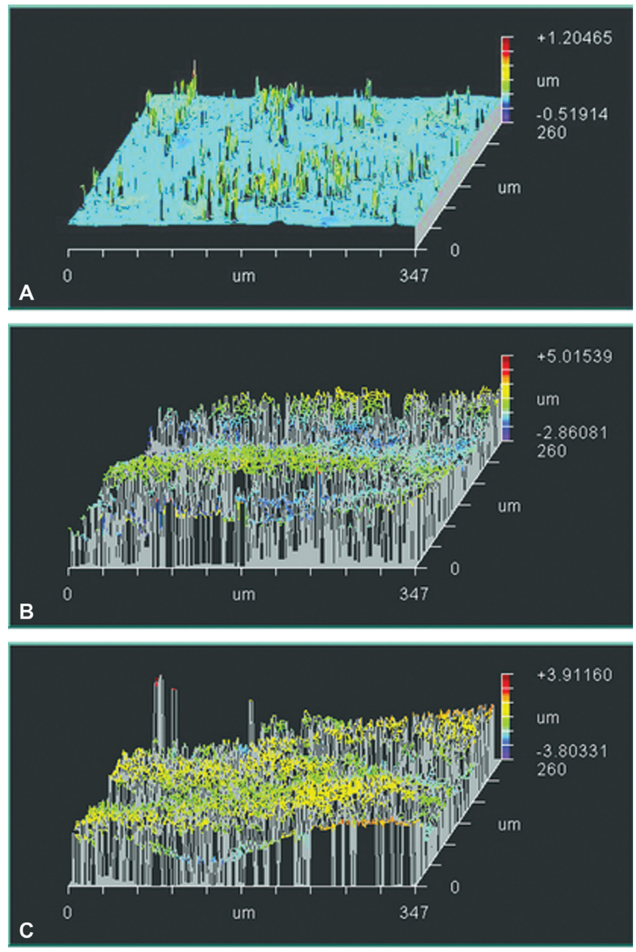

Fig. 3 (A before bleaching, B after at- home bleaching and C after in-office bleaching).

free radicals disturb the interface in the resin filler and cause microcracks.[19]

In the present study, regardless of the type of whitening regime, either hydrogen peroxide or carbamide peroxide, the Fortify sealed composite surface sealant showed a lower Vickers micro-hardness compared to the unsealed composite. This can be illustrated as resin monomers, which are the main component of surface sealant. Therefore, quantity of inorganic particles is relatively lower than the quantity of resin matrix. The fillers account for 17.3% of the Fortify sealant.[9]

Taking into consideration the impact of at-home whitening on micro-hardness, there had been no marked difference since the initial measurements. The results of the current study are consistent with the findings of a previous study which indicated that applying approximately 16% carbamide peroxide at room temperature had no effect on micro-hardness.[20]

Considering the impact of in-office whitening application on micro-hardness, the results of the current study demonstrated that there was no remarkable difference between the first measurements of the composite resin surface micro-hardness, and after the application of in-office bleaching. In addition, a comparison of 16% carbamide peroxide at-home bleaching with 30% hydrogen peroxide, as well as its effect on micro-hardness, showed no significant difference. This consequently means that the type of bleaching material used in the current study was not a factor of change in the micro-hardness values.

The current findings may be attributed to the increased breakdown of the composite resin material by in-office bleaching with higher hydrogen peroxide concentrations. The diversity between the current findings and those reported in previous studies is due the difference between the in-office whitening product applied in the present study and the type of bleaching in which the concentration of Philips Dash was 30% hydrogen peroxide concentration.[21]

The effect of the whitening materials depends on the properties of the compounds, the shape, the concentration, and the period of applying the bleaching agent/span. In addition, the substrate has a significant role as the bleaching agents have different effects on the resin matrix. Therefore, composite resins with a larger size than the resin matrix are more resistant to the adversarial actions of bleaching materials.[22]

The bleaching agent alters the surface roughness of the composite resin material mainly because of oxidation, which affects the organic matter function of the composite resin. The organic matter reduces the absorption of water and causes particle loss, so the surface roughness is increased.[23,24]

The organic content of composite resin was more resistant to the bleaching procedures. Also, the surface sealants often decrease the surface roughness.[24] Many studies[25–29] demonstrated that applying surface sealant to composite materials led to lower surface roughness values (Ra) when compared to a finished and polished surface without the use of a sealant. These results were consistent with the results of this study as group 1 (sealant was used) showed less surface roughness than group 2 (without sealant application), with a significant difference.

This study showed that at-home bleaching increased the surface roughness for both groups. This may be related to the repeated exposure and extended contact time, which would alter the resin matrix of the composite resin, while the inorganic filler is inert, even if in a highly acidic environment. Consequently, as presented, the matrix erosion occurs with the resulting dislocation of inorganic filler. This is supported by the findings that some types of composite resins are more affected by bleaching agents compared to other composites. These findings are partially consistent with the findings of Fernandes et al.[30] They concluded that bleaching with a bleaching agent with carbamide peroxide led to an increase in the surface roughness of the unsealed composite resin.

Conclusion

Under the limitations of this study including no thermocycling nor brushing simulation, the following findings can be concluded:

Applying surface sealant significantly decreased hardness and surface roughness of bulk-fill composite resin used in this study. The two different bleaching regimes used in the present study showed a significant difference in surface roughness. However, this did not significantly affect microhardness of the tested composite resin bulk-fill material.

Conflict of Interest
None declared.

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