

EFFECT OF TWO DIFFERENT BLEACHING TECHNIQUES ON THE MICROHARDNESS AND SURFACE ROUGHNESS OF TWO BULK-FILL COMPOSITE MATERIALS: AN *IN VITRO* STUDY

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Patients demand for frequent whitening of teeth, with different bleaching materials. The most common bleaching chemical used are carbamide and hydrogen peroxide. This study examines the effects of different bleaching regimens by the evaluation of surface roughness (Ra) and microhardness of two bulk-fill composite materials. Sixty disk-shaped (10 × 4 mm) specimens of either Filtek bulk-fill or Tetric N-Ceram bulk-fill were used. These specimens were casually assigned in five main classes in line with the restorative material present. All specimens were measured for their microhardness and Ra values (Baseline-T1). Each group was encountered with two dissimilar bleaching regimens, first refers to in-office bleaching using Opalescence Boost 40 %, and at-home bleaching using Opalescence 20 %. Statistical analysis was made via Statistical Package for Social Sciences version 23.0 at a significance level of $p < 0.05$. Findings of the study indicated significant alterations in the microhardness when exposed to at-home bleaching agent. While, in the Ra values no significant alterations were found between the groups except for versus in-office bleaching in Surface Arithmetical mean height (SA) for Tetric N-Ceram. The study concludes that surface roughness and microhardness of bulk fill composite resin were affected by bleaching agents.

INTRODUCTION

The concept of esthetic dentistry has evolved into different modalities, with less-invasive and cost-effective treatments becoming popular. Many patients request for tooth whitening, which is a simple and inexpensive procedure. Tooth whitening is beneficial for patients with intrinsic and extrinsic discoloration, as the etiology of tooth discoloration is multi factorial [1]. Each technique has its advantages, disadvantages, and degree of effectiveness, and the result varies depending on a range of features i.e., the kind of stain, the bleaching material employed, and the protocol of treatment [1].

Among various bleaching components available, hydrogen peroxide (HP) and carbamide peroxide (CP) are the most frequently used for removing stains and whitening tooth surfaces, although HP tends to be more effective than CP [2]. However, the chemical structures of composite filling materials may be altered upon bleaching due to modification of the organic resins of which they are composed [3]. Several studies documented the outcome of HP on the chemical composition, mechanical properties, and discoloration of restorative materials [4], including other undesirable effects, such as the unstiffening and deterioration of both restorative

compounds and teeth. As such, the researchers have evaluated the outcomes of bleaching components with regard to dental restorative materials to limit their adverse effects. For example, in a laboratory study, the application of 10 % CP over the period of 3 weeks was found to alter the surface roughness of a composite resin that can be packed, although the surface microhardness did not undergo any change [2]. According to a previous study, the process of bleaching teeth is safe in terms of managing the risks associated to the alteration of hard tissues. The process of bleaching involves the decomposition of peroxides into the free radicals, which includes break-down of large molecules through reduction reaction. These reactions contribute in altering the chemical structure of different organic substances of tooth which leads towards the change in tooth color [5]. In contrast to this, some authors reported that home bleaching agents may contribute in softening the resin composites, whereas other authors in their study found no definite changes in the hardness [6-7]. AlMaklafi et al. [8] mentioned that the use of bleaching tooth must be held in consideration with its effects on physical, corrosive and mechanical properties of tooth, since changes in the properties of materials used may have significant implications. Also, the prognosis and longevity are usually dependent upon these tissues.

On the composite resin, substantial softening of surface was seen when undergone the treatment with 10 % CP for 14 days at normal body temperature (37 °C), whereas the surface microhardness was left with no alteration, following a similar treatment at the normal room temperature (25 °C). Nevertheless, upon subjecting the composite resin with extremely concentrated gel of CP gel, the surface microhardness increased [3], and a range of CP concentrations were found to have no harmful effects at the flexural strength and fracture toughness of the composite resin [4]. Additionally, in-office bleaching agents (i.e., ≥ 35 % CP and HP) had no effect on composite resin's tensile strength [9]. In contrast, Taher [10] reported that the hardness of surface values of bleached composite resins (i.e., Tetric Flow and Tetric EvoCeram, Ivoclar Vivadent, Schaan, Liechtenstein) decreased significantly both on the superficial layer and in the deeper layers of the composite materials. The findings were in relation with the corrosion and deterioration of the resinous matrices inside the composites.

Clearly, the standard composite material must not have any modification consequent to the whitening of tooth. In this context, bulk-fill composite materials have recently been reported as a direct resin-based restorative material that exhibits different optical and surface features compared to regular resin composites, including a superior translucency and favorable surface polish ability [11]. As such, bulk-fill composites were manufactured to streamline the composite resin placement technique, with manufacturers claiming that bulk-fill composites show developed light transmission features and produce shrinkage stress of a lower polymerization owing to lessening the degree of the scattering in light scattering at the interface of filler matrix via either a rise of the filler scope or a decrease in the filler quantity. Bulk-fill composites can therefore be employed to increase the thickness by up to 4-5 mm [7].

Dental bleaching is a controversial issue, and a degree of debate exists surrounding the consequence of bleaching agents at intraoral restorative components. Bleaching agents act by causing the corrosion to the organic materials inside the tooth structure and releasing free radicals, and to date, all such agents employed have been demonstrated to be safe and cause no harm to the dental filling materials. In addition, one such bleaching agent, HP, can create free radicals through both oxidation and reduction, in addition to permit diffusion.

Since their progression in the 20th century, composite resins have emerged as the potential synthetic resins for restoring decayed teeth. However, clinicians have experienced several challenges such as collapsing of the in bonding of layers, incorporating voids, enlarged time of treatment for placing the layers and their polymerization, incremental placement and contamination between increment layers. Bulk Fill composites have been manufactured to overcome these disadvantages as they can be cured in single increment that is

up to 4 mm. To date, several researches have examined the bleaching agent's impact on the composite resin. However, comparison of the results of these studies is difficult, owing to number of bleaching agents and restorative materials employed. Since a smooth surface helps in improving the appearance and longevity of resin materials, a rough surface might contribute in discoloration, gingival, irritation, plaque, and recurrent carries [12].

The present study investigates the effects of two different bleaching agents (i.e., in-office bleaching agent Opalescence Boost 40 %, applied twice over 20 min per application [24 samples], and the bleaching agent, used at home i.e., Opalescence 20 %, that was used for the period of 4 hours per day for 1 week, then for 4 hours every second day for a further week [24 samples]) for the values of two bulk-fill composite materials of their Ra and microhardness. The hypothesis stated for the study is as follows;

H: There is significant impact of bleaching agent on the surface roughness and microhardness of bulk-fill composite materials.

EXPERIMENTAL

Materials

Two dissimilar bleaching agents and two bulk-fill components were used in the present study. As outlined in Table 1, 60 disk-shaped specimens (10 × 4 mm) composed of the Filtek Bulk Fill Posterior composite (3M ESPE, St. Paul, MN, USA) and an additional set composed of Tetric N-Ceram Bulk Fill (Ivoclar Vivadent, Schaan, Liechtenstein) were used.

Study samples

A control group consisting of 12 samples of each material was used to measure the Ra and microhardness values. 48 samples of each material were exposed to two different bleaching protocols;

- In-office bleaching using Opalescence Boost 40 %, which was applied twice with 20 min per application (24 samples).
- At-home bleaching using Opalescence 20 %, which was applied for 4 h/d over 1 week then for 4 hours/day on alternate days for 1 week (24 samples).

Specimen preparation

60 disk-shaped specimens of Tetric N-Ceram Bulk Fill composite and a further set composed of Filtek Bulk Fill Posterior were arranged while using a custom Teflon mold (10 mm diameter, 4 mm depth). A bulk depth of 4 mm of each material was placed in the mold over a glass slab, and a glass plate (1 mm thick) was secured

Table 1. List of the materials employed and their compositions.

Material	Material type	Resin matrix	Filler
Tetric N-Ceram Bulk Fill (Ivoclar-Vivadent, Lichtenstein)	Packable hybrid bulk-fill composite	Bisphenol A glycidyl methacrylate (Bis GMA), bis[4-(2-ethoxy-3-methacryloyloxy propoxy)phenyl propane (Bis-EMA), and urethane-dimethacrylate (UDMA)	Filler content: barium glass, a prepolymer, ytterbium trifluoride, and a mixed oxide. Filler loading: 75–77 wt. %, 53–55 vol. %, inorganic filler particle size = 0.04–3 µm, mean particle size = 0.6 µm.
Filtek Bulk Fill Posterior restorative (3M ESPE, USA)	Packable nanofilled bulk-fill composite	ERGP-DMA, diurethane- DMA, and 1,12-dodecane- DMA	Non-agglomerated/non- aggregated 20 nm silica filler and 4–11 nm zirconia filler, aggregated zirconia/silica cluster filler, and ytterbium trifluoride filler agglomerate 100 nm particles Filler loading: 76.5 wt. %, 58.4 vol. %.

over the sample to flatten the surface. Polymerization was then performed by implementing the recommendation of manufacturer. In each case, the tip of the curing light was placed 1 mm away from the topmost exterior of the specimen, and the resulting specimen was deposited in water at 37 °C for 24 hours in a dark chamber. After this time, the composite disks were polished using polishing discs (Sof-Lex, 3M ESPE, USA) according to the manufacturer’s instructions.

Curing and bleaching process

The A1 or equivalent shade was selected for each composite resin and curing thickness was carried out using an Elipar DeepCure-S LED single wave instrument (3M ESPE, St. Paul, MN, USA). After each treatment, the specimens were cleaned through purified water for one-minute duration, aiming to eliminate the bleaching material. Further, it was placed in saliva substitute. Amid the test, the specimens were maintained at 37 °C. A generous amount of each bleaching material agent was used on the surface of disk directly while using the tip of the syringe and micro brush.

Microhardness measurements

The microhardness of each sample was measured using the Vickers hardness test (Buehler, Lake Bluff, Illinois, USA). A total of three random indentations (distance between indentations, 100 µm) were introduced at the top exterior of the specimen through a Vickers microhardness indenter under along with the pressure of

300 g, which was asserted for 15 seconds for each group. Average values were taken from the results of the three dimensions. The Vickers hardness (VH) was further computed through the equation below:

$$VH = 1.854P/D^2 \tag{1}$$

where *P* refers to the indentation load and *D* denotes the diagonal length impression.

The Ra tests

Following surface treatment, the arithmetic Ra values of the composite materials were measured using a Ra tester 3D Laser Profilometer designed for noncontact measurements. Average values obtained from measurements were carried out in triplicate.

Statistical analysis

All data were investigated using SPSS version 23.0 (SPSS Inc., Armonk, New York, USA). Continuous variables were reported as the mean and the SD. For comparing two means, a paired t-test was performed. P value of < 0.05 was determined as significant statistically.

RESULTS

Microhardness results

Two bulk fill constituents, namely Filtek Bulk Fill Posterior and Tetric N-Ceram Bulk Fill, were tested under two different bleaching regimens (i.e., in-office bleaching and at-home bleaching) (Figure 1). The third

Table 2. Microhardness result for the Tetric N-Ceram and 3M Filtek materials.

	Tetric N-Ceram		3M Filtek	
	mean (SD)	p value	mean (SD)	p value
Control	54.17 (0.69)	*<0.001 (S)	73.45 (0.55)	*<0.001 (S)
At-Home bleached	52.22 (0.26)	**<0.001 (S)	65.83 (0.35)	**<0.001 (S)
In-Office bleached	47.78 (0.78)	***<0.001 (S)	56.57 (0.68)	***<0.001 (S)

Note: * *p* values between the control and the at-home bleached samples; ** *p* values between the control and the in-office bleached samples; *** *p* values between the at-home and the in-office bleached samples; S – significant; NS – not significant

group was the control group. Significant differences in the values of microhardness were marked in the control (no bleaching), at-home bleached, and in-office bleached. Following exposure to the at-home bleaching agent, significant alterations were detected in the microhardness values of the tested composite groups ($p = 0.05$).

Ra test results

Tables 3 displays the values of SDs and means and of the surface roughness values for the control group and for the in-office and at-home bleached specimens. After exposure to the at-home bleaching agent, substantial modifications were not noted in the Ra values of the tested composite groups ($p = 0.05$). However, no significant alterations were found in the Ra values between the groups, except for the control versus in-office bleaching in Surface Arithmetical mean height (SA) for Tetric N-Ceram (Figure 2).

DISCUSSION

This study has tested effects of different bleaching regimens in terms of the values of surface roughness (Ra) and microhardness of two bulk-fill composite materials. The findings revealed significant effect of bleaching agents on surface roughness and microhardness of bulk fill composite resin. Some of the previous studies reported that at-home bleaching agents can cause an increase [13] or a decrease [14] in surface microhardness; while, other

studies have revealed no substantial differences [15-17]. The material's microhardness hinges on its kind, structure, and tendency of degradation over time [18].

This study has compared the microhardness and Ra values of two recently reported bulk-fill composites after exposure to two different bleaching regimens, namely, in-office bleaching using Opalescence Boost 40 % and at-home bleaching using Opalescence 20 %. Similar to this, one of the previous studies has also narrated that the microhardness values of both Tetric N-Ceram and Filtek Bulk Fill Posterior composites were significantly altered due to a high degree of oxidation and subsequent degradation of the resin matrices of both materials. However, treatment with different concentrations of bleaching agents was found to have little influence on the microhardness of the surface. Polydorou et al. [13] also found that bleaching agent has no tendency to substantially reduce the composite materials' microhardness, and so it appears that the replacement of composite restorations is not required following bleaching treatment. Similar findings were proposed in the study of Ahmed et al. [19] who evaluated the effectiveness of bleaching with hydrogen peroxide on the microhardness and microleakage in the restoration of teeth. Cavities of class V were prepared focusing on the labial surfaces upon 60 extracted human upper central incisor teeth. The included teeth were divided under two groups. The restoration process in Group I were held through FiltekZ350XT, while in Group II, the restoration was held via Fuji II LC resin-modified glass ionomer. The teeth were divided into three different groups after

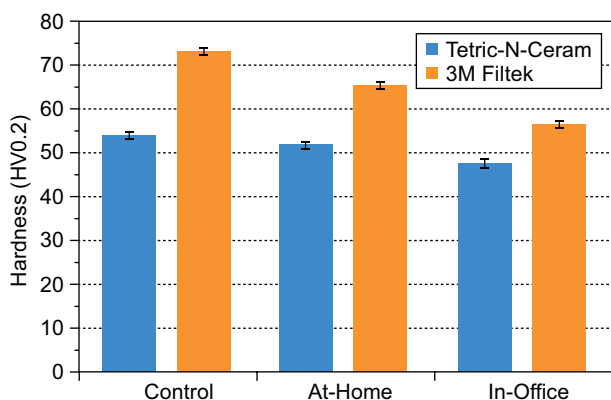


Figure 1. Microhardness results for Tetric N-Ceram and 3M Filtek.

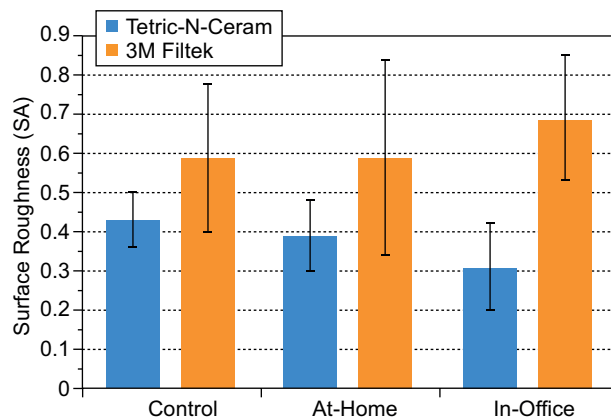


Figure 2. Surface roughness (SA) results for Tetric-N-Ceram and 3M Filtek.

Table 3. Surface roughness values for the bleached Tetric N-Ceram and 3M Filtek specimens.

	Tetric N-Ceram		3M Filtek	
	mean (SD)	p value	mean (SD)	p value
Control	0.43 (0.07)	*0.141 (NS)	0.59 (0.19)	*0.989 (NS)
At-Home bleached	0.39 (0.09)	**0.016 (S)	0.59 (0.25)	**0.152 (NS)
In-Office bleached	0.31 (0.11)	***0.155 (NS)	0.69 (0.16)	***0.367 (NS)

Note: * p values between the control and the at-home bleached samples; ** p values between the control and the in-office bleached samples; *** p values between the at-home and the in-office bleached samples; S – significant; NS – not significant

being thermocycled. The first group remained unbleached, and was regarded as control group. In the second group, the teeth were bleached with 14 % of hydrogen peroxide gel. While, the final group was bleached with 40 % of hydrogen peroxide gel. The teeth were immersed in die before analysis. Findings of the study indicated that bleaching had no effect on microleakage of Filtek Z350XT composite along with the compositions of Fuji II LC RMGI restorations while the microhardness of these restorations were also affected [19].

Özduman et al. [20] in their study evaluated the surface roughness and microhardness of two different bulk filled composites that were polymerized with light curing unit (LCU) at different time intervals after and before applying home bleaching agent. For surface and microhardness tests, six different groups were prepared with bulk fill materials Sonic Fill, Filtek Bulk Fill as per distinct polymerization times. Besides, 102 specimens were further prepared by the utilization of Teflon molds with the overall depth of (4 mm and 5 mm) and polymerized with LCU. Microhardness was evaluated in 30 specimens. The top/bottom microhardness ratio was also evaluated, prior to the application of home bleaching agent. Measurements of related to surface roughness were performed among 72 specimens with before and after application of bleaching. Findings indicated a significant decrease in FB30s, when microhardness values of the composites top surfaces were compared before and after bleaching. Findings of the study indicated no significant differences in the value of surface roughness, specifically when surface roughness were compared on the basis of bulk-fill materials. In addition, a significant influence of polymerization time was observed at the time when group were observed. Finally, the roughness values of surface were found as significantly increased in SF30s and SF20s [20], these findings are in line with those presented in the present study.

Magdy et al. [21] evaluated the surface roughness of different resin-based composites including one nanoceramic, one nanohybrid, one bulk fill resin. Disc specimens of cylindrical Teflon molds of 2 mm and 8 mm diameter were prepared. For different composite materials, fabrication of 15 discs were held, while a total of 60 discs were obtained. A glass slide of 1-2mm thickness was kept over the strip before curing with the light curing unit to make the surface flat. Mylar strip and the glass slide were then used to cure the specimens for 40 seconds. No finishing treatment was provided to five specimens per every material once cured with the Mylar strips. The specimens were further polished with Eve. These specimens were regarded as control. Findings of the study indicated that specimens of Tetric Evo Ceram and Tetro Evo Ceram Bulk Fill specimens revealed somewhat similar surface appearance as the Mylar strip. Following the findings, it was concluded that Bulk Fill and nanohybrid resin composites display smoothest surfaces in comparison with microhybrid resin composites and nanoceramics [21]

Similarly, Yu et al. [15] reported no decrease in nanohybrid composite's microhardness subsequent to the apply 15 % CP, that may be related to the diluting impact of saliva to diminish the impact of the bleaching agent. In contrast, between in-office bleaching and at-home bleaching regimens, Leal et al. [22] found a significant discrepancy statistically in the case of SonicFill™ that was associated with Filtek™ Supreme XTE.15 Previous studies have showed significant effect on composite materials' microhardness by in-office bleaching [16] although, other studies reported a decrease in its microhardness.[23-25]

The characteristics of the composites also come under the effect of the features of filler, including the structure, bulk, and mass. For instance, the flexural strength, elasticity modulus, and hardness are improved, as the filler volume is increased [23-25]. In addition, the microhardness values of bulk fill composite constituents are positively correlated with the filler volume fraction. In this case, the microhardness values of the 3M Filtek bulk-fill materials were higher than those of the Tetric N-Ceram materials. Contrastive results for the effects of bleaching with regard to surface microhardness can be associated with various elements, i.e., the active agent's structure, concentration, and pH along with the application time. HP had tendency to put effect on the interface of resin–filler and brought about the debonding of the filler–matrix. The bleaching agent may therefore result in the development of minute fissures, thereby adding up the value of Ra, as confirmed by scanning electron microscopy. [26] From the results presented herein, the study has reported the reduction in microhardness for the two exposed tested groups in comparison with the control group, i.e., in the case of Tetric N-Ceram, a 3.6 % reduction for at-home bleaching, and a 11.78 % reduction for in-office bleaching, while for 3M Filtek, a 10.7 % reduction for at-home bleaching, and a 22.98 % reduction for in-office bleaching were observed.

The organic matrix composition of composite resins make them more inclined to chemical modification as related to inactive ceramic or metal restorations [25]. Various studies, regarding the Ra values of resin composites after bleaching, reported that in-office bleaching was found to either adversely affect the composites [20-22, 27], or have no effect [24]. Some studies reported that at-home bleaching increases Ra values; [28] while, other studies found that the various composites could be treated with the bleaching agent with no harm and damage to its roughness [29]. In addition, Mohammadi et al. [30] had found no remarkable changes in the Ra values after exposing the Sonic Fill bulk fill composite to a range of bleaching materials. The specific roughness parameters employed herein followed the ISO 4287-1997 standard [31]. Furthermore, the Ra values of resin-based restoratives increased significantly upon the extended use of at-home bleaching agents, likely due to the formation of microscopic cracks, as mentioned

previously. [29] Finally, Lainovic et al. [32] noted that upon examination of the values of Ra of nanohybrid (FiltekZ550 and Tetric EvoCeram Bulk Fill), nanofilled (FiltekZ250), and microhybrid (Gradia direct) materials, Tetric EvoCeram exhibited the lowest Ra values.

CONCLUSION

Following the findings of this study, it can be concluded that surface roughness and microhardness of bulk fill composite resin were affected by bleaching agents. In addition, the clinical scenario cannot be simulated comprehensively, since it is an in vitro study. The extent of alteration, surface roughness evaluation, and depth of cure using atomic force microscopy can further provide significant evidence in terms of mechanical properties and extent of polymerization of dental composites. The supervision of dentists is important when using bleaching agents. However, these agents can replace fillings only where esthetics is preferred.

The findings of the study reported the effects of different bleaching regimens on the surface roughness (Ra) and microhardness and values of two bulk-fill composite materials. Amid the settings, employed for this *in vitro* study, the use of bleaching agents has affected the microhardness values of both Tetric N-Ceram and Filtek bulk fill, while the Ra value of the Filtek bulk fill material remained unchanged. These results are of importance to ensure that the correct bleaching regimen is selected to prevent alteration of the composite material chemical structures.

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