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ORIGINAL RESEARCH

Effects of Bleaching Agents on the Microhardness and Surface Roughness of Bulk Fill Composites

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ABSTRACT

Aim: The aim was to compare the effect of 10 and 20% carbamide peroxide (CP) on microhardness (MHN) and surface roughness of two commercially available bulk fill composites (X-tra fill Bulk Fill and Tetric EvoCeram Bulk Fill).

Materials and methods: A total of 50 specimens with both type composites were prepared using brass molds of inner diameter 10 mm and a height of 4 mm. Each type of composite was further divided into three groups, control group stored in artificial saliva for 14 days, other groups bleached with 10 and 20% CP respectively, for 14 days. Microhardness of the composite resin was tested with a Vickers hardness tester, whereas surface roughness was tested with profilometer.

Results: All the analysis was performed using Statistical Package for the Social Sciences version 18. The value of $p<0.05$ was considered statistically significant. Both 10 and 20% CP significantly reduced MHN of experimental composite resins. However, the mean surface roughness was significantly higher in X-tra fill Bulk Fill than Tetric EvoCeram Bulk Fill at 20% CP concentration. The mean MHN of Tetric EvoCeram was reduced when compared with X-tra fill at 20% CP.

Conclusion: There was a significant reduction in the MHN of restorative materials observed after exposure to CP under a clinically simulated bleaching regimen. However, increase in surface roughness was seen only at 20% CP.

Clinical significance: The physical properties, such as MHN and surface roughness have a crucial effect on the longevity of restorations and, moreover, on the esthetic demands of patients, but they may be compromised by bleaching treatments. The aim of this research paper was to assess the reaction of home bleaching agents on the physical properties of two Bulk Fill composite resin restorative materials.

Keywords: Bleaching, Bulk fill composites, Carbamide peroxide, Microhardness, Surface roughness.

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INTRODUCTION

Over the past two decades, tooth whitening or bleaching has become one of the most popular esthetic dental treatments. Since the 1800s, the initial focus of dentists in this area was in office bleaching of teeth that were discolored as a result of trauma to the tooth or after endodontic treatment.[1] By the late 1980s, the field of tooth whitening dramatically changed with the development of dentist-prescribed, home-applied bleaching and other products and techniques for vital tooth bleaching that could be applied both in the dental office and at home.

Bleaching techniques can be classified by whether they involve vital or nonvital teeth or whether the procedure is performed in the office or has an at-home component, or both. In-office bleaching techniques include the use of a gel or paste composition that commonly contains 30 to 35% hydrogen peroxide, whereas 10 to 15% carbamide peroxide (CP) bleaching material in gel or paste form is generally used for at-home techniques.[2]

Low concentration of CP was proven to have minimal effects on the soft tissues of the mouth.[3] Data accumulated over the past 20 years, including some long-term clinical study follow-up, indicate no significant, long-term oral or systemic health risks associated with professional at-home tooth bleaching materials containing 10% CP (3.5% hydrogen peroxide).[4]

The introduction of CP at-home bleaching has created resurgence in the area of bleaching primarily because of its relative ease of application, the safety of the material, lower cost, easy availability, and the high percentage of successful treatment.[5]

The mechanism involved in bleaching is the decomposition of peroxides into unstable free radicals, which further break down into large pigmented molecules through oxidation or reduction reaction. This reaction changes the chemical structure of the interacting organic substances of the tooth, which results in color change.[6,7]

There are conflicting reports about consequences of bleaching material on hardness of restorative resins.[5] Some authors had reported that home bleaching agents may soften the resin composites,[8] while other authors found no significant changes in the hardness.[9-11]

The physical properties of restorative materials, such as microhardness (MHN), flexural strength, flexural modulus, and fracture toughness, influence the quality and durability of restorations.[12] A reduction in MHN due to organic matrix erosion may enhance the roughness of restorations and may decrease their wear resistance.[13]

There is a definite correlation found between surface roughness and vital bacterial adhesion.[14] It appears that bleaching agents may affect the adherence of certain cariogenic microorganisms to the outer surfaces of composite resin restorations by altering surface texture.[14] Surface roughness has a major impact on esthetic appearance, discoloration of restorations, plaque accumulation, secondary caries, and gingival irritation.[14]

Composite resins have taken the world of restorative dentistry by storm ever since their advent in the 20th century. However, ever since their introduction, clinicians have faced a variety of challenges, such as with incremental placement, incorporating voids, contamination between increment layers, failures in bonding between layers, and extended treatment time for placement of layers and their polymerization. To overcome these disadvantages, "Bulk Fill" composites have been introduced. According to manufacturer, they can be cured up to 4 mm in single increment. Thus, the aim of this study was to compare the effect of home bleaching agents on MHN and surface roughness of two Bulk Fill composites.

Study Outline

Table 1 describes the materials used in this study. Twenty-five specimens each were prepared with two high-viscosity Bulk Fill resin composites (Tetric EvoCeram Bulk Fill – TECBF, IVA shade Ivoclar Vivadent, Liechtenstein, X-tra fill, VOCO GmbH, Cuxhaven, Germany Universal shade) which were again subdivided into three more subgroups representing the different bleaching protocol. Hence, there were a total of four groups with a sample size of 10 each and two groups with a sample size of five each. The groups were as follows:

- *Group I*: X-tra fill Bulk Fill composite resin treated with 10% CP for 8 hours/day for 14 days (n = 10)
- *Group II*: X-tra fill Bulk Fill composite resin treated with 20% CP for 2 hours/day for 14 days (n = 10)
- *Group III*: Tetric EvoCeram Bulk Fill composite resin treated with 10% CP for 8 hours/day for 14 days (n = 10)
- *Group IV*: Tetric EvoCeram Bulk Fill composite resin treated with 20% CP for 2 hours/day for 14 days (n = 10)
- *Groups V and VI*: Control group – the samples were not bleached and were stored in artificial saliva at 37°C for 14 days.

Specimen Preparation

Experimental composite resins were inserted into circular brass molds (10 mm inner diameter 4 mm thick) using a metal spatula. A mylar strip (3M ESPE, USA) was placed on top and pressed flat with a microscope slide to expel excess material and create a smooth surface.

Composites were then light-cured with a light-emitting diode curing unit (Bluephase Style, Ivoclar Vivadent) according to manufacturer's instructions. The output light intensity of the curing unit (1100 mW/cm^2) was measured using calibrated Bluephase Meter (Ivoclar Vivadent) and verified periodically during the experiments. The polishing of the samples was carried out with Sof-Lex polishing disks (3M ESPE) in the descending order of granulation. The disks were used on slow speed for 10 seconds each. Then, the samples were placed at 37°C in distilled water for 24 hours to assure complete polymerization.

The samples in groups 1, 2, 3, and 4 were taken out of the distilled water and air-dried with a jet spray for 60 seconds before bleaching. Thereafter, the bleaching agent was applied onto the surface of the specimen using micro-brush for the stipulated time (10% CP 8 hours a day, and 20% CP 2 hours a day) and was washed with a water jet spray for 60 seconds before placing it back into the artificial saliva for the bleaching cycle on the next day. The bleaching procedure continued for 14 days. The control group samples were immersed in artificial saliva. All samples are stored in plastic container at 37°C.

Table 1: Materials used in this study

Materials	*Manufacturer*	*LOT*	*Type*	*Composition*
Tetric EvoCeram Bulk Fill composite	Ivoclar Vivadent AG, Schaan, Liechtenstein	P84129	Bulk Fill composites	Bis-GMA, UDMA, bis-EMA; barium glass, YbF3, mixed oxide, prepolymerized fillers
X-tra fill Bulk Fill composite	VOCO GmbH, Cuxhaven, Germany	1245232	Bulk Fill composites	Bis-GMA, UDMA, TEGDMA
Opalescent PF 10%	Ultradent, product	B2HNF	Bleaching agent	Potassium nitrate, fluoride, water pH: 6.5
Opalescence PF20%	Ultradent, product	B25V3	Bleaching agent	Potassium nitrate, fluoride, water pH: 6.5

Bis-GMA: Bisphenol A-glycidyl methacrylate; UDMA: Urethane dimethacrylate; YbF3: Ytterbium (III) fluoride; TEGDMA: Triethylene glycol dimethacrylate; Bis-EMA: Ethoxylated bisphenol-A dimethacrylate

Table 2: Intragroup comparison of MHN between two Bulk Fill composite resins

	Group				
	Control	*10% CP*	*20% CP*		
Composite	*Mean±SD*	*Mean ± SD*	*Mean ± SD*	*p-value*	*Post hoc test*
X-tra fill					
MHN	86.46 ± 3.78	80.50 ± 8.94	63.29 ± 4.75	<0.001 S	10%, control >20%
Tetric EvoCeram Bulk Fill					
MHN	80.87 ± 2.13	75.01 ± 13.15	57.99 ± 4.61	<0.001 S	Control >20%

†ANOVA with *post hoc* Tukey's test; ‡ANOVA with *post hoc* Games–Howell test; SD: Standard deviation; S: Significant

Table 3: Intragroup comparison of surface roughness between two Bulk Fill composite resins

	Group				
	Control	*10% CP*	*20% CP*		
Composites	*Mean ± SD*	*Mean ± SD*	*Mean ± SD*	*p-value*	*Post hoc test*
X-tra fill					
Roughness	2.46 ± 0.81	2.66 ± 2.79	3.06 ± 1.59	0.784 NS	–
Tetric EvoCeram Bulk Fill					
Roughness	1.39 ± 1.81	2.58 ± 1.59	0.25 ± .05	0.064 NS	–

SD: Standard deviation; NS: Not significant

Vickers Hardness Testing

After 24 hours, the composite specimens were placed under an MHN indentation device. Indentations were made at a fixed load of 1 kg for 15 seconds. This procedure was carried out thrice to produce three different indentations at three different points. Thereafter, the mean readings of the indentations were calculated accordingly. The indentations were observed under 100× magnification and the Vickers hardness number was digitally computed by measuring the dimensions of the indentation using the formula:

$$VHN = 1854.4 \times (F/d^2)$$

where, F = test load in Newton,

d = average of the two diagonals of an indentation in mm.

Surface Roughness Measurement

The surface roughness of the specimens was measured using a white-light three-dimensional (3D) noncontact optical profilometer (WYKO, Veeco, Plainview, NY, USA). On each specimen, scans were made at four randomly selected locations. The surfaces were studied at a magnification of 10.38× and an image size of 736 mm × 480 mm. The actual surface roughness was calculated from each measurement and data were expressed as Sa (arithmetic average of the 3D roughness surface profile).

RESULTS

All the analysis was performed using Statistical Package for the Social Sciences version 18. The value of $p < 0.05$ was considered statistically significant.

Intragroup Comparison

X-tra fill Bulk Fill composite resin showed a significant difference in the mean MHN among the control, 10 and 20% CP. *Post hoc* test showed that control and 10% CP had significantly higher mean MHN than 20% CP. No significant difference was seen between control and 10% CP (Table 2).

In Tetric EvoCeram Bulk Fill, there was overall significant difference in the mean MHN among the control, 10% CP, and 20% CP. *Post hoc* test showed that control had significantly higher mean MHN than 20% CP. No other significant differences were seen (Table 2).

There was no significant difference in the mean surface roughness among the control, 10% CP, and 20% CP in X-tra fill Bulk Fill and Tetric EvoCeram Bulk Fill respectively (Table 3).

Intergroup Comparison

X-tra fill Bulk Fill had significantly higher mean MHN than Tetric EvoCeram Bulk Fill in THE control group compared with 10 and 20% CP. However, there was no significant difference in the mean MHN between X-tra fill Bulk Fill and Tetric EvoCeram Bulk Fill at 10 and 20% CP concentrations (Table 4).

Table 4: Intergroup comparison of MHN between two Bulk Fill composite resins

	Composite		
	X-tra fill	*Tetric EvoCeram Bulk Fill*	
Group	*Mean ± SD*	*Mean ± SD*	*p-value*
Control			
MHN	86.46 ± 3.78	80.87 ± 2.13	0.02 S
10%			
MHN	80.50 ± 8.94	75.01 ± 13.15	0.462 NS
20%			
MHN	63.29 ± 4.75	57.99 ± 4.61	0.111 S

SD: Standard deviation; S: Significant; NS: Not significant

Table 5: Intergroup comparison of surface roughness between two Bulk Fill composite resins

	Composite		
	X-tra fill	*Tetric EvoCeram Bulk Fill*	
Group	*Mean ± SD*	*Mean ± SD*	*p-value*
Control Roughness	2.46 ± 0.81	1.39 ± 1.81	0.262 S
10% Roughness	2.66 ± 2.79	2.58 ± 1.59	0.954 NS
20% Roughness	3.06 ± 1.59	0.25 ± 0.05	0.017 S

Independent sample t-test, SD: Standard deviation; S: Significant; NS: Not significant

There was no significant difference in the mean surface roughness between X-tra fill Bulk Fill and Tetric EvoCeram Bulk Fill in control and 10% concentration. However, the mean surface roughness was significantly higher in X-tra fill Bulk Fill than Tetric EvoCeram Bulk Fill at 20% CP concentration (Table 5).

DISCUSSION

After the eruption of the tooth, aging, pulp necrosis, and iatrogenesis are the main causes of intrinsic discoloration. Coffee, tea, red wine, carrots, oranges, and tobacco give rise to extrinsic stain.[15] Exposure to high levels of fluoride, tetracycline administration, inherited developmental disorders, and trauma to the developing tooth may result in preeruptive discoloration.[15] Nowadays the demand for noninvasive treatment increasing from patients, bleaching is a conservative alternative treatment for discolored teeth.[16]

Most common bleaching products are hydrogen peroxide and CP-based products; 10% CP bleaching agent is the most commonly used at-home bleaching product. Carbamide peroxide decomposes to release hydrogen peroxide in an aqueous medium; 10% CP yields roughly 3.5% hydrogen peroxide. Range of concentrations of CP available includes 15, 20, 22, and 30% solution.[17]

Decrease in MHN leads to degradation of resin matrix which leads to discoloration of restorative resins.[6] Hence, the surface MHN of composite resins should not be changed after restoration in the mouth. Recently, due to increasing number of patients for bleaching treatment, the best restorative material should remain stable during chemical process of bleaching.

The bleaching protocol in the present study was designed to simulate treatment of teeth under cycling conditions of bleach and saliva exposure which is encountered under *in situ* conditions. It is observed that due to hydrolytic degradation by artificial saliva, the MHN of control group is less compared with baseline values.

The lower values of MHN postbleaching of restorative resins were mainly due to the oxidation and degradation of resinous matrix.[13] Furthermore, free radicals produced by peroxides may affect the resin-filler bond and result in filler-matrix debonding.[16] They result in a softening of composite resin and reduction in MHN. The decrease in MHN not only pertains to the surface of bleached composite but also to deeper layers up to 2 mm.[15] Tetric EvoCeram Bulk Fill composites have ivocerin incorporated in it which would result in softening after bleaching procedure and could be one of the reasons for reduced MHN when compared with X-tra fill Bulk Fill composites.[18] In X-tra fill Bulk Fill composite resin, the organic matrix is composed mainly of bisphenol A glycidyl methacrylate, urethane dimethacrylate, and triethylene glycol-dimethacrylate, while its inorganic particles comprise 70.1% of the volume.[19] However, according to Leprince et al[20] X-tra fill is similar to microhybrid restorative materials with high-filler loading possibly explaining high MHN, which is in accordance with the present study. The MHN of nanocomposites decreased with increase in concentration of CP.[21] This is in accordance with the present study.

Yap and Wattanapayungkul[14] showed that resin-based restoratives may be significantly roughened by the extended use of at-home bleaching agents. Bleaching may cause microscopic cracks, resulting in an increase in surface roughness, as shown in the scanning electron microscopic pictures.[2,14,22] Bailey and Swift[3] suggested that the surface changes mainly due to complex interactions inside the multicomponent bleaching materials and loss of matrix, rather than filler particles. Yap and Wattanapayungkul[14] suggested that differences between the materials could be a result of the difference in resin matrix components and filler size.

Lainovic et al[23] in a study on nanohybrid (FiltekZ550 and Tetric EvoCeram), nanofilled (FiltekZ250), and microhybrid (Gradia direct) materials concluded that all composites exhibited surface roughness. However, Tetric EvoCeram exhibited less surface roughness. This result supports the present study.

Tetric EvoCeram has glass fillers that have lesser wear and favorable polishing property, lower surface roughness, and high gloss compared with X-tra fill. The differences in the composite roughness and MHN values obtained after the same bleaching regime may be related to the different polymers in their organic matrix, and their filler content and particle size.[24,25]

There is limited scientific evidence regarding the effect of bleaching solutions on MHN of restorative resins.[3,26,27] Along with oxidizing agent used in the at-home bleaching agents, an additive called carbopol (carboxy polymethylene) may be added to increase the viscosity of gel that in turn improves bonding to the enamel surface and prolongs the liberation of oxygen. This additive makes the gel contained within the tray better and decreases the chemical interaction. The changes in the surface of restorative resins were mainly due to reactions inside the multicomponent bleaching materials rather than by one component.[3]

One factor which influences these interactions is pH value.[12] It will cause alterations in MHN of restorative resins.[11,16] Lower pH of bleaching agents influences the integrity of the dental hard tissue. It is mainly due to peroxide concentration in bleaching agents which increases the acidity. Hence, it is advised to maintain peroxide levels at optimal level so that pH will be maintained at 7.[24]

From this study, it can be inferred that MHN and surface roughness of Bulk Fill composite resin was affected by bleaching agents. Furthermore, since it is an *in vitro* study, the clinical scenario cannot be simulated completely. Besides MHN and surface roughness, degree of conversion, depth of cure, and surface roughness evaluation using atomic force microscopy can also provide substantial evidence with respect to the extent of polymerization and mechanical properties of dental composites.

It is always better to use bleaching agents under dentist supervision. However, we can replace fillings only where esthetics is priority.

CONCLUSION

Based on the results of this study, it can be concluded that:

- There was a significant reduction in the MHN of restorative materials observed after exposure to CP agents under a clinically simulated bleaching regimen.
- There was overall significant difference in the mean MHN among the control, 10% CP, and 20% CP in both groups. The mean MHN was significantly higher in X-tra fill than Tetric EvoCeram Bulk Fill at 20% CP concentration.
- The mean surface roughness was significantly higher in X-tra fill than Tetric EvoCeram Bulk Fill at 20% CP concentration.
- The clinicians make sure that their patients with composite restorations should be aware of the changes that may occur during whitening, as well as the possibility that their bleached restorations may need to be polished or replaced at the end of the treatment.

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