FLUORIDE

Quarterly Journal of the International Society for Fluoride Research Inc.

In-vitro Evaluation of Topical Fluoride on the Optical Properties, Surface Texture, and Hardness of Restorative Materials

Unique digital address (Digital object identifier [DOI] equivalent):
https://www.fluorideresearch.online/epub/files/276.pdf

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Accepted: 2024 Jul 9
Published as e276: 2024 Jul 11

ABSTRACT

Purpose: This study aims to evaluate the interactions of fluoride-containing gels and varnishes on the optical properties and surface texture of composite resins, compomers, and bulk-fill composite resins. Thus, it seeks to address the current gap in the literature regarding the effect of fluoride agents on bulk-fill restorative materials.

Methods: Using Teflon moulds, 50 specimens of 6 mm diameter and 2 mm thickness were produced for each of the G-ænial Posterior, Filtek™ One Bulk-Fill and Dyract-XP materials. Baseline color and surface roughnesss measurements of the specimens were performed. The specimens were subjected to the following applications: Study groups (Topex NaF gel, Topex APF gel, ProShield varnish, and MI varnish with RECALDENT^{TM}) and Control group (distilled water). Color and surface roughness measurements were repeated after the application period. The surface microhardness of the specimens (study groups and control group) was measured using Vickers microhardness tester. Statistical analyses, including two-way analyses of variance (comparison of color, whiteness and microhardness parameters) and three-way analyses of variance (comparison of surface roughness parameter), and Pearson's correlation test (the relationship between surface roughness and color change and surface roughness), were applied.

Results: The restorative material significantly (p-value<0.001) influenced the color changes (ΔE_{00}), with the Filtek™ One Bulk-Fill/Topex APF gel interaction yielding the highest ΔE_{00} average value (2.1±1.15). Materials and fluoride agents exerted significant effects on the Whiteness Index (ΔWI_D) for Dentistry (p-value<0.001). Dyract-XP displayed lower initial surface roughness (1.05±1.38), with all materials exhibiting comparable values after fluoride treatment. Microhardness varied among the materials, with Filtek™ One Bulk-Fill having the highest total value (65.39±2.67) and G-ænial Posterior having the lowest total (42.97±3.32) value. When considering the interaction of restorative materials, fluoride treatment, Topex APF gel-treated G-ænial Posterior showed the lowest (40.56±1.78) microhardness, whereas Filtek™ One Bulk-Fill showed the highest (65.06±1.88) values.

Conclusions: Topical fluoride agents induce alterations in the color, roughness, and hardness of restorative materials, depending on the material type, fluoride agent, and duration of application.

Key-words: *Atomic force microscopy, color, composite resins, compomers, fluorides, hardness.*

INTRODUCTION

Despite being fundamentally preventable, dental caries remains one of the most prevalent chronic diseases affecting both children and adults.[1-3] In recent years, there has been a noteworthy emphasis on detecting early signs of caries and adopting minimally invasive approaches to preserve dental tissues.[4] The primary objective of protective applications is to proactively prevent demineralization or enhance the durability, esthetics, and functionality of dental hard tissues through remineralization in existing demineralized/hypomineralized areas.[5] Fluoride is a frequently used agent because it has a therapeutic effect by promoting the remineralization of early carious lesions, and a protective effect by preventing the demineralization of dental hard tissues.[2] Fluoride applications can be administered both systemically and topically; however, topical applications are recommended for caries prophylaxis due to their superior efficacy and safety.[6, 7] Topical fluorides are commonly divided into two categories: professionally-applied products, administered by healthcare providers (e.g., solutions, gels, foams, and varnish), and self-applied products, used by patients at home (e.g., toothpaste and mouth-rinse). The former typically contains higher concentrations of fluoride and requires less frequent application compared to the latter.[6, 8] These professionally-applied fluoride products transform hydroxyapatite crystals within tooth enamel into fluoroapatite crystals, thereby enhancing the enamel's resistance to acid demineralization.[2] Jackson et al.[9] emphasized in their study that patients who applied topical fluoride professionally required fewer restorative procedures and tooth extractions. These preventive agents are commercially available in acidulated and neutral forms[8, 10], fluoride formulations encompass sodium fluoride (NaF), acidulated phosphate fluoride (APF), stannous fluoride (SnF_2), and amine fluoride (AmF). Acidulated phosphate fluoride (APF) gels, containing 1.23% fluoride ions and hydrofluoric acid, demonstrate superior effectiveness in enhancing fluoride uptake by enamel and reducing demineralization.[11] For patients with esthetic restorations such as porcelain and composite restorations, NaF neutral gels are recommended over APF.[12] Additionally, various fluoride-containing products have been introduced to the market. Functionalized tricalcium phosphate combined with fluoride (f-TCP) has been found not only to promote remineralization but also to decrease the necessary fluoride dosage for achieving comparable remineralization efficacy.[13] Another promising product, MI Varnish with RECALDENT™ (CPP-ACP), biologically delivers calcium, phosphate, and fluoride to the tooth surface and synergistically interacts with sodium fluoride (NaF).[14]

When the initial carious lesion cannot be stopped and cavitation occurs, restorative treatments are used to treat carious lesions.[15] As esthetics have become extremely important to patients in recent years, tooth-colored resin-based restorative materials are often preferred by clinicians.[16] Nevertheless, the choice of resin-based restorative materials may fluctuate based on a variety of mechanical and clinical criteria, in addition to esthetic considerations.[15] For instance, nanohybrid composites with superior mechanical properties due to their high inorganic content in a cavity exposed to high occlusal stresses[17], bulk fill composite resins that facilitate the clinical process by allowing a placement depth of 4-6 mm in deep cavities[18], resin-modified glass ionomer cements (compomers) are preferred due to their ability to release fluoride (in high caries risk), in class 5 cavities in elderly individuals[19] or in deciduous tooth restorations in young children.[20] Depending on the risk of caries, topical fluoride agents applied to the teeth at different periods for prophylaxis also come into contact with the restorations in the mouth. However, these agents may have adverse effects on the surface roughness, hardness and color of resin-based composites.[11, 21-23]

Although these effects have been examined in the literature, new restorative materials and protective agents are constantly being developed. It is noteworthy that there is a lack of research specifically addressing the effect of fluoride agents on the surface properties of bulk-fill restorative materials. This study aims to contribute to the current knowledge by investigating the effects of fluoride agents on the surface roughness, hardness, and color stability of bulk-fill restorative materials and comparing them with other resin-based restorative materials. Furthermore, the interactions of NaF-, APF-, and CCP-ACFP-containing gels and varnishes on the optical properties and surface texture of composite resins, compomers, and bulk-fill composite resins are to be evaluated. The null hypotheses for this study were as follows: (a) topical fluoride agents do not affect the optical properties of various restorative materials; (b) topical fluoride agents do not exert an effect on the surface roughness of various restorative materials; and (c) topical fluoride agents do not affect the surface microhardness of different restorative materials.

MATERIAL AND METHODS

Restorative materials used in the study

Figure 1 presents a schematic diagram summarizing the study design. Three restorative materials (microfilled composite resin [G-ænial Posterior (GP)], compomer [Dyract-XP (DXP)], and bulk-fill resin composite [Filtek™ One Bulk-Fill (FBF)]) and four topical fluoride agents (Topex NaF gel, Topex APF gel, ProShield varnish, and MI varnish with RECALDENT™) were analyzed in this study (Figure 1- section 1). The manufacturers, shades, and compositions of restorative materials and fluoride agents are listed in Table 1.

Preparation of specimens

Fifty specimens, 6 mm in diameter and 2 mm thickness for each restorative material, were fabricated using Teflon molds (Figure 1- section 2). A Mylar strip (SS White Co.; Philadelphia, PA, USA) and a glass plate were lightly pressed onto the specimens to remove any superimposed resin composite and to obtain a smooth surface. Polymerization of all specimens was performed using a light-emitting diode (LED) curing light (D-Light Procuring light, GC, Japan) with an irradiance of 1200 mW/cm^2, 20 s duration, for each resin-based material. A radiometer (Bluephase Meter II, Ivoclar Vivadent, Schaan, Liechtenstein) was used to check the intensity of the curing light before polymerization of each group. A 1-mm transparent polyester tape was used to standardize the distance between the light unit and the specimen. All the specimens were polished using a multi-step finishing disc kit (Super-Snap Rainbow Technique, Shofu Inc., Kyoto, Japan) and a one-step polishing kit (One Gloss, Shofu Inc., Kyoto, Japan). A digital caliper gauge (N48AA, Maplin Electronics, UK) was used to maintain the final thickness of the specimens at 2±0.1 mm. A mark was made with a round diamond bur on the unpolished surface of the specimens to ensure that measurements could be consistently taken from the same surface at each stage of the process. For post-polymerization, the specimens were immersed in distilled water at 37 °C for 24 h.

To ensure calibration, color, surface roughness and hardness measurements of the specimens were performed by a single researcher.

Color measurement process

A digital spectrophotometer (Vita Easyshade V, Vita Zahnfabrik, Bad Säckingen, Germany) was set to "tooth" mode and used to perform the baseline color measurements (T_0) of the specimens (Figure 1- section 3). The probe was placed in the center of the specimens and the "L*, C*, H*, a*, and b*" values were measured separately on a white background under constant laboratory illumination. These values were measured thrice, and the average values were recorded.[24] After every nine measurements, the spectrophotometer was recalibrated according to the manufacturer's instructions.

Surface roughness measurement process

Baseline surface roughness measurements (T_0) were performed using a mechanical contact profilometer (Mitutoyo, Surftest SJ-410, Japan) with a cut-off length of 0.8 mm (Figure 1- section 3). “Ra” is the arithmetic average value of the absolute sum of all surface irregularities (height and depth) at a given measuring distance.[25] The measurement distance of the device was set to 4 mm and the cutting value to 0.8 mm. Before each measurement, the profilometer was calibrated using a reference block with a Ra value of 3.05 µm. The contact angle of the specimen with the tip of the reader profilometer was 90° and it was placed on the specimen plate. Three measurements were obtained at the center of each specimen surface, and the Ra values were recorded in micrometers. Three measured values were averaged for each specimen.

Topical fluoride agents applications

The specimens were randomly (https://www.random.org/) divided into five groups according to each tested fluoride agents (n=10): Distilled water (control), Topex NaF gel, Topex APF gel, ProShield varnish, and MI varnish (Figure 1- section 4).

In group 1, the specimens were exposed to 20 mL of distilled water for 14 days, which was used as a control to evaluate the intrinsic color changes of the restorative materials. The distilled water solution was renewed every day.

In the gel groups (group 2 and 3), 2 ml of fluoride gel was applied to the surfaces of the specimens for 4 min, and the gel was mixed with an applicator for 1 s at the beginning of each minute for 4 minutes. Subsequently, the specimens were left in contact with the fluoride gel for 26 min. The specimens were then cleaned using an electric toothbrush under running tap water and maintained in distilled water for 2 h. The cycle was repeated four times at two-hour intervals.[11]

In the varnish groups (group 4 and 5), 0.5-1 ml fluoride varnishes were applied to the specimens with a microbrush for 1 min and left to dry for 5 min. The specimens were maintained in distilled water at 37 °C for 24 h and cleaned using an electric toothbrush under running tap water. This cycle was repeated four times to simulate the recall periods of fluoride applications of individuals in high-risk caries risk group.[26]

Following the application (T_1) of topical fluoride agents, color, and surface roughness measurements were repeated (Figure 1- section 5). To evaluate the color differences after topical fluoride application, ΔE_{00} ($T_{1-}T_0$) values were calculated using equation (1).[27]

$$\Delta E_{00}=\left[\left(\frac{\Delta L}{k_L S_L}\right)^2+\left(\frac{\Delta C}{k_C S_C}\right)^2+\left(\frac{\Delta H}{k_H S_H}\right)^2 R_T\left(\frac{\Delta C}{k_C S_C}\right)\left(\frac{\Delta H}{k_H S_H}\right)\right]^{1/2} \quad (1)$$

where ΔL, ΔC, and ΔH denote the lightness, chroma, and hue differences, respectively, which are calculated based on the difference between the baseline and final color measurements. The weighting functions S_L, S_C, and S_H were included in the equation to overcome the inconsistencies of the CIE L*a*b system.[28] The rotation function was also included to compensate for chromatic differences in the blue region, thus improving the performance of the color-difference equation.[27] In addition, the parametric factors k_L, k_C and k_H were adjusted according to the different viewing parameters and experimental conditions. The CIEDE2000 (1:1:1) formula was used in this study. Finally, color changes were analyzed based on an acceptability threshold of 50:50% (AT:ΔE_{00}=1.8) and perceptibility threshold of 50:50% (PT:ΔE_{00}=0.8) for all resin-based materials.[29] A new CIELAB space-based Whiteness Index for Dentistry (WI_D) was calculated using equation (2) to assess the change in whiteness before and after application:[30]

$$(2) \quad WI_D = 0.511L^* - 2.324a^* - 1.100b^*$$

Changes (ΔWI_D) in tooth whiteness were calculated using Equation (3):

$$(3) \quad \Delta WI_D = WI_D\,(\text{application}) - WI_D\,(\text{baseline})$$

For whiteness change analysis, the 50:50% whiteness perceptibility threshold of 0.72 units (WPT=0.72) and the 50:50% whiteness acceptability threshold of 2.60 units (WAT = 2.60) were adopted.[31]

Topographic surface imaging with Atomic Force Microscope (AFM)

Atomic force microscope (AFM) images were acquired for two randomly selected specimens from each experimental group following the testing process. The specimens were viewed using an AFM device (Multimode AFM, Veeco Instruments Inc., California, USA) (Figure 1- section 5). Tapping mode was used, and a spring constant of 40 N/m was employed. The device was scanned in a 10 × 10 μm^2 area at a resonance frequency of 300 kHz, and 3D images were generated with a resolution of 512 × 512 pixels.[25]

Microhardness measurements process

The microhardness (polished top surface of each restorative material) of samples with topical fluoride application (study groups) and no application (control group) was measured using a Vickers microhardness (VHN) tester (Buehler MMT 3 Digital Microhardness Instrument; Lake Bluff, IL, USA) (Figure 1- section 5). Each specimen received three indentations: one at the center and two others around it, with a minimum distance of 0.5 mm between each indentation.[32] For microhardness testing, indentations were created under a force of 100 × g for 10 s. This specific load was selected to ensure that the diagonal indentations were as large as possible, thereby maximizing the measurement resolution. The two diagonal lines produced by each indentation were measured and the VHN values were calculated for the resulting indentations using the following equation (4):[11]

$$(4) \quad VHN = (1.8544\,x\,P)/d^2$$

In the equation, 'VHN' represents the Vickers hardness expressed in kg/mm^2, 'P' stands for the indenter load in kg, and 'd' denotes the diagonal length of the indentation in mm. To calculate the VHN value, we obtained the average of three Vickers hardness measurements for each specimen. Any indentations that resulted in asymmetric diagonal lines, a jagged or chipped edge, or a noticeable shift in the location of the indentation tip were excluded from the analysis.

Statistical analysis

The sample size was determined using G*Power software (version 3.1.9.4, Heinrich Heine University, Düsseldorf, Germany). A predefined significance level (α) of 0.05, a $1-\alpha/2$ value of 1.96, a z value of 1.28, and a targeted statistical power (p) of 90% were specified for subgroups. With a critical F value of 2.0213, the procedure yielded a minimum calculated sample size of 128 samples. To account for a prospective dropout rate of 20%, the sample size was increased to 10 per group, resulting in a total of 150 subjects.

Statistical analyses were performed using Statistical Package for the Social Sciences (SPSS Inc., Chicago, IL, USA). The skewness and kurtosis values were examined to analyze the normal distribution of the parameters. Two-way analysis of variance (ANOVA) with Bonferroni correction was used to determine the significant differences between ΔE_{00}, ΔWI_D, and VHN parameters. Three-way analysis of variance was employed to determine the surface roughness (Ra) according to the materials and application procedures. Pearson's correlation coefficient was used to examine the relationship between surface roughness and color change and surface roughness parameters. The results are presented as the average ± standard deviation, and the significance level was set of p-value<0.050.

RESULTS

Assessment of optical characteristics

The average ΔE_{00}, and ΔWI_D values and standard deviations of the restorative materials after fluoride exposure are shown in Table 2.

Statistical analysis revealed that the main effect of the restorative material was found to be statistically significant on ΔE_{00} values (p-value<0.001). Specifically, GP exhibited an average ΔE_{00} value of 0.71, in contrasting 1.18 to FBF and 1.09 DXP. Notably, the ΔE_{00} values for GP deviate significantly from the averages observed for the other materials. Furthermore, the main effect of fluoride agents on the ΔE_{00} values was statistically significant (p-value=0.014). Distinct ΔE_{00} values were observed: 0.77 in distilled water, 1.11 in Topex APF gel, 0.94 in Topex NaF gel, 0.93 in MI varnish, and 1.23 in ProShield varnish. Notably, the ΔE_{00} value in distilled water differed significantly from that in ProShield varnish.

The interaction between the restorative material and fluoride agent also exhibited statistical significance on the ΔE_{00} average values (p-value<0.001). The FBF/Topex APF gel interaction yielded the highest ΔE_{00} average value (2.10), whereas the DXP/ProShield varnish interaction was closest to this value at 1.92. In contrast, the GP/Topex APF gel interaction displayed the lowest ΔE_{00} average value of 0.61.

In terms of ΔWI_D values, a statistically significant main effect of restorative material was observed (p-value=0.012). The average ΔWI_D values were 0.10 in GP, -0.94 in FBF, and -0.31 in DXP. Notably, the ΔWI_D value in GP significantly differed from the average values observed in FBF.

Similarly, the main effect of fluoride agents on the ΔWI_D values was statistically significant (p-value<0.001). Varied ΔWI_D values were observed: 0.46 in distilled water, -2.66 in Topex APF gel, -0.85 in Topex NaF gel, 1.40 in MI varnish, and -0.26 in ProShield varnish. The highest value 1.40 was obtained in the MI varnish and was similar to that obtained in distilled water. The lowest value -2.66 in the Topex APF gel significantly deviated from that of the other agents.

The interaction between the restorative material and fluoride agent was found to have a statistically significant effect on ΔWI_D values (p-value<0.001). The GP/MI varnish interaction yielded the highest ΔWI_D value (1.75), whereas the FBF/Topex APF gel interaction had the lowest value of -5.45, differing significantly from all other interactions.

Assessment of surface roughness

Table 3 presents the average Ra values and standard deviations of the restorative materials after exposure to the fluoride agents. The main effect of the restorative material on the average Ra value was not statistically significant (p-value=0.300). Similarly, the main effect of fluoride agents on the average Ra values was not statistically significant (p-value=0.101).

In contrast, the main effect of application phase (AP_0: before application; AP_1: after application) on the average Ra values was statistically significant (p-value<0.001). The average Ra value at AP_0 was 0.48, whereas that at AP_1 increased to 1.47.

The interaction between the restorative material and fluoride agent yielded statistically significant effects on the Ra values (p-value= 0.030). The DXP/Topex APF gel interaction had the highest value of 1.59, whereas the GP/Topex APF gel interaction had the lowest value at 0.75.

Similarly, the interaction between restorative material and application demonstrated statistical significance for Ra values (p-value= 0.006). The highest average value of 1.73 was observed at AP_1 for the DXP material, and similarly for the FBF material (1.40). The lowest average value 0.36 was noted at AP_0 for the DXP material, which was similar to the values obtained at AP_0 for the other materials.

The interaction between the fluoride agent and application was not statistically significant for the Ra values (p-value=0.208). The interaction between the fluoride agent and application did not exhibit statistical significance with respect to Ra values (p-value=0.208). Conversely, the interaction between the restorative material, fluoride agent, and application phase was statistically significant for Ra values (p-value=0.038). The DXP / Topex APF gel / AP_1 interaction yielded the highest Ra value of 2.81, while the lowest value of 0.35 was identified in the DXP / NaF gel / AP0 interaction.

AFM examination

2D and 3D AFM images of a randomly selected specimen from distinct fluoride agent groups applied to the three resin-based restorative materials under investigation, namely GP, FBF, and DXP are presented in Figure 2-4. The outcomes of AFM analysis were found to be congruent with those obtained through contact profilometry, both indicating the presence of irregular surfaces. Notably, the subgroup featuring the combination of DXP/Topex APF gel manifested heightened porosity, as delineated in Figure 4. Conversely, the subgroup involving the GP/Topex APF gel combination exhibited comparatively fewer irregular surfaces than other combinations, as depicted in Figure 2. In a general sense, irrespective of the subgroup, all surfaces displayed a rough topography characterized by prominent protrusions and deep holes, as illustrated in Figure 2-4.

Assessment of microhardness

The average VHN values and standard deviations of the restorative materials after exposure to the fluoride agents are shown in Table 4. The average VHN value was 42.97 in the GP, 65.39 in the FBF, and 54.99 in the DXP, and values indicated notable variations among the materials.

In contrast, the main effect of the fluoride agents was not statistically significant (p-value=0.194). However, the interaction between the resin material and fluoride agent caused a statistically significant difference in VHN values (p-value=0.001). Notably, the FBF/Topex NaF gel interaction yielded the highest VHN value at 66.38, while the GP/APF gel interaction was the lowest at 40.56.

Assessment of the correlation between roughness values (Ra) and color change ΔE_{00} and microhardness (VHN) values

Table 5 presents relationship between Ra and ΔE_{00} and VHN values. There was no statistically significant relationship between AP_0 values and color change values (p-value=0.951). A statistically weak positive relationship was found between AP_0 values and microhardness values (r=0.210; p-value=0.010). There was no statistically significant relationship between AP_1 values and color change values (p-value=0.686). There was no statistically significant relationship between AP_1 values and microhardness values (p-value=0.562).

Table 1. Chemical composition of restorative materials and fluoride agents used in the study

Restorative materials used in the study							
Product	**LOT No**	**Manufacturer**	**Shade**	**Classification**	**Composition**	**Particle size**	**Filler load (vol%)**
GP	1709223	Kuraray Noritake Dental Inc.; Okayama, Japan	A3	Micro-hybrid posterior restorative	Bis-GMA, TEGDMA, and hydrophobic aromatic dimethacrylate, Glass ceramics, surface-treated alumina micro filler, silica	16-17 µm >100nm <100nm	65%
DXP	17110000250	Dentsply, DeTrey, Konstanz, Germany	A3	Polyacid Modified Resin Composite (Compomer)	UDMA, carboxylic acid modified dimethacrylate, TEGDMA, trimethacrylate resin (TMPTMA), dimethacrylate resins, Camphorquinone, ethyl-4 (dimethylamino) benzoate, butylated hydroxy toluene (BHT), strontium-alumino-sodium-fluoro phosphor-silicate glass, highly dispersed silicon dioxide, strontium fluoride, iron oxide pigments and titanium oxide pigments	Average: 0.8 µm	47%
FBF	N878473	3M ESPE GmbH, Seefeld, Germany	A3	Bulk-Fill nanofilled composite resin	Aromatic dimethacrylate (AUDMA), Addition-fragmentation monomers (AFM), UDMA, 1,12-Dodecanediol dimethacrylate (DDDMA), Nonagglomerated/nonaggregated 20 nm silica filler, Nonagglomerated/nonaggregated 4 to 11 nm zirconia filler, Aggregated zirconia/silica cluster filler, Ytterbium trifluoride filler consisting of agglomerate 100 nm particles	0.01-4.5 µm	76.5%

Topical fluoride agents used in the study			
Material type	**Products**	**Manufacturer**	**Composition**
2% NaF gel	Topex	Sultan, NJ, USA	Sodium Fluoride, Ethanol
1,23% APF gel	Topex	Sultan, NJ, USA	Sodium Fluoride, Phosphoric Acid, Titanium Dioxide
5% NaF varnish	ProShield	President dental, Germany	Resin, Sodium Fluoride 5%, Ethanol, Xylitol, TCP (Tricalcium Phosphate)
1–8% NaF varnish	MI varnish with RECALDENT™	GC, Tokyo, Japan	30–50 % polyvinyl acetate, 10–30 % hydrogenated rosin, 20–30 % ethanol, 1–8 % sodium fluoride, 1–5 % CPP-ACP, 1–5 % silicon dioxide

Abbreviations: GP: G-eanial posterior composite, DXP: Dyract-XP compomer, and FBF: Filtek™ One Bulk-Fill composite. Bis-GMA = bisphenol-glycidyl methacrylate; TEGDMA = triethyleneglycol dimethacrylate; UDMA = urethane dimethacrylate; NaF: Sodium Fluoride, APF: Acidified Phosphate Fluoride, CPP-ACP: casein phosphopeptide- amorphous calcium phosphate.

Table 2. Descriptive statistics and multiple comparison results of optical parameters according to restorative materials and fluoride agents

Average (± SD)					Two-way Anova Results			
ΔE_{00}								
Fluoride agents	**Restorative material**							
	GP	**FBF**	**DXP**	**Total**	**Factor**	**F**	**p-value**	**Partial Eta Square**
Distilled water	0.62 ± 0.32^{C}	0.82 ± 0.39^{C}	0.88 ± 0.44^{C}	0.77 ± 0.39^{b}	**Restorative material**	10.99	**<0.001**	0.140
Topex APF gel	0.61 ± 0.4^{C}	2.1 ± 1.15^{A}	0.62 ± 0.29^{C}	1.11 ± 1^{ab}	**Fluoride agents**	3.26	**0.014**	0.088
Topex NaF gel	0.62 ± 0.31^{C}	1.22 ± 0.77^{BC}	0.98 ± 0.54^{C}	0.94 ± 0.61^{ab}	**Restorative material*fluoride agents**	8.66	**<0.001**	0.339
MI varnish with RECALDENT™	1.06 ± 0.51^{C}	0.67 ± 0.3^{C}	1.07 ± 0.66^{C}	0.93 ± 0.53^{ab}	*R Squared = % 43.59; Adjusted R Squared = %37.74*			
ProShield varnish	0.65 ± 0.5^{C}	1.12 ± 0.35B^{C}	1.92 ± 0.36^{AB}	1.23 ± 0.66^{a}				
Total	0.71 ± 0.44^{b}	1.18 ± 0.82^{a}	1.09 ± 0.64^{a}	1 ± 0.68				
ΔWI_D								
Fluoride agents	**Restorative Material**							
	GP	**FBF**	**DXP**	**Total**	**Factor**	**F**	**p-value**	**Partial Eta Square**
Distilled water	0.54 ± 0.86^{ABC}	1.08 ± 1.09^{ABC}	-0.24 ± 1.41^{ABC}	0.46 ± 1.23^{ab}	**Restorative material**	4.58	**0.012**	0.064
Topex APF gel	-1.27 ± 1.25 ^{BC}	-5.45 ± 3.89^{D}	-1.27 ± 0.8^{C}	-2.66 ± 3.06^{d}	**Fluoride agent**	23.69	**<0.001**	0.412
Topex NaF gel	-0.26 ± 0.93^{ABC}	-0.76 ± 3.31^{ABC}	-1.53 ± 1.15^{C}	-0.85 ± 2.09^{c}	**Restorative material*fluoride agent**	4.72	**<0.001**	0.219
MI varnish with RECALDENT™	1.75 ± 0.68^{A}	1.05 ± 1.53^{ABC}	1.38 ± 0.98^{AB}	1.4 ± 1.12^{a}	*R Squared = ; Adjusted R Squared = %*			
ProShield varnish	-0.28 ± 1.06^{ABC}	-0.61 ± 2.07A^{BC}	0.12 ± 0.76^{ABC}	-0.26 ± 1.4^{bc}				
Total	0.1 ± 1.38^{a}	-0.94 ± 3.47^{b}	-0.31 ± 1.46^{ab}	-0.38 ± 2.34				

Note: ΔE_{00} and ΔWI_D values are based on the the Two-Way Analysis of Variance; a-d: No difference between main effects with the same letter; A-C: No difference between interactions with the same letter
p-values presented in bold font indicate statistical significance

Abbreviations: GP: G-ænial posterior composite, DXP: Dyract-XP compomer, and FBF: Filtek™ One Bulk-Fill composite, NaF: Sodium Fluoride, APF: Acidified Phosphate Fluoride

Table 3. Descriptive statistics and multiple comparison results of surface roughness values according to restorative materials and fluoride agents

Average (± SD) — Ra

Fluoride agents	Application phase	Restorative material: GP	FBF	DXP	Total
Distilled water	AP_0	0.52 ± 0.06^{CD}	0.6 ± 0.05^{CD}	0.38 ± 0.03^{D}	0.5 ± 0.1
	AP_1	1.32 ± 0.53^{BCD}	1.34 ± 0.41^{BCD}	2.16 ± 2.19^{AB}	1.61 ± 1.34
	Total	0.92 ± 0.55^{AB}	0.97 ± 0.47^{AB}	1.27 ± 1.76^{AB}	1.05 ± 1.09
Topex APF gel	AP_0	0.47 ± 0.08^{D}	0.61 ± 0.17^{CD}	0.36 ± 0.04^{D}	0.48 ± 0.15
	AP_1	1.04 ± 0.63^{BCD}	1.47 ± 0.68^{BCD}	2.81 ± 2.85^{A}	1.77 ± 1.84
	Total	0.75 ± 0.53^{B}	1.04 ± 0.66^{AB}	1.59 ± 2.33^{A}	1.13 ± 1.45
Topex NaF gel	AP_0	0.48 ± 0.05^{D}	0.51 ± 0.07^{CD}	0.35 ± 0.04^{D}	0.44 ± 0.09
	AP_1	1.09 ± 0.32^{BCD}	1.15 ± 0.38^{BCD}	1.18 ± 0.46^{BCD}	1.14 ± 0.38
	Total	0.79 ± 0.39^{B}	0.83 ± 0.42^{AB}	0.76 ± 0.53^{B}	0.79 ± 0.44
MI varnish with RECALDENT™	AP_0	0.53 ± 0.08^{CD}	0.59 ± 0.14^{CD}	0.35 ± 0.01^{D}	0.49 ± 0.14
	AP_1	1.72 ± 0.48^{ABC}	1.55 ± 0.53^{BCD}	1.24 ± 0.18^{BCD}	1.5 ± 0.46
	Total	1.12 ± 0.7^{AB}	1.07 ± 0.62^{AB}	0.8 ± 0.48^{B}	1 ± 0.61
ProShield Varnish	AP_0	0.49 ± 0.06^{D}	0.55 ± 0.06^{CD}	0.36 ± 0.02^{D}	0.47 ± 0.1
	AP_1	1.23 ± 0.38^{BCD}	1.5 ± 0.33^{BCD}	1.28 ± 0.4^{BCD}	1.34 ± 0.38
	Total	0.86 ± 0.46^{AB}	1.03 ± 0.54^{AB}	0.82 ± 0.55^{AB}	0.9 ± 0.52
Total	AP_0	0.5 ± 0.07^{C}	0.57 ± 0.11^{C}	0.36 ± 0.03^{C}	0.48 ± 0.12^{B}
	AP_1	1.28 ± 0.52^{B}	1.4 ± 0.49^{AB}	1.73 ± 1.7^{A}	1.47 ± 1.07^{Ag}
	Total	0.89 ± 0.54^{B}	0.99 ± 0.55^{AB}	1.05 ± 1.38^{A}	0.97 ± 0.91

Three-way Anova Results

Factor	F	p-value	Partial Eta Square
Restorative material	1.21	0.300	0.009
Fluoride agent	1.96	0.101	0.028
Application phase	142	**<0.001**	0.345
Restorative material*fluoride agent	2.16	**0.030**	0.060
Restorative material*application phase	5.16	**0.006**	0.037
Fluoride agent*application phase	1.48	0.208	0.021
Restorative material*Fluoride agent*application phase	2.08	**0.038**	0.058

R Squared =%42.85; Adjusted R Squared = %36.71

Note: *Ra values are based on the Three-way Analysis of Variance; A-D: No difference between interactions with the same letter*
p-values presented in bold font indicate statistical significance

Abbreviations: GP: G-ænial posterior composite, DXP: Dyract-XP compomer, and FBF: Filtek™ One Bulk-Fill composite, NaF: Sodium Fluoride, APF: Acidified Phosphate Fluoride, AP: Application phase

Table 4. Descriptive statistics and multiple comparison results of microhardness according to restorative materials and fluoride agents

Average (± SD) — VHN

Fluoride agents	Restorative material: GP	FBF	DXP	Total
Distilled water	41.24 ± 1.24^{D}	65.63 ± 2.43^{A}	55.77 ± 3.08^{B}	54.21 ± 10.44
Topex APF gel	40.56 ± 1.78^{D}	65.06 ± 1.88^{A}	54.32 ± 2.29^{B}	53.65 ± 10.77
Topex NaF gel	43.2 ± 1.75^{CD}	66.38 ± 1.52^{A}	54.61 ± 4.18^{B}	54.73 ± 9.98
MI varnish with RECALDENT^{TM}	43.35 ± 5.17^{CD}	64.67 ± 1.6^{A}	54.92 ± 1.79^{B}	54.31 ± 9.42
ProShield varnish	46.51 ± 1.24^{C}	64.22 ± 4.55^{A}	55.34 ± 3.46^{B}	55.36 ± 8.05
Total	42.97 ± 3.32^{c}	65.39 ± 2.67^{a}	54.99 ± 2.99^{b}	54.45 ± 9.66

Two-way Anova Results — VHN

Factor	F	p-value	Partial Eta Square
Restorative material	797.33	**<0.001**	0.922
Fluoride agent	1.54	0.194	0.044
Restorative material*fluoride agent	3.38	**0.001**	0.167

R Squared =%92.34; Adjusted R Squared = %91.55

Note: VHN values are based on the Two-Way Analysis of Variance; a-d: No difference between main effects with the same letter; A-C: No difference between interactions with the same letter
p-values presented in bold font indicate statistical significance

Abbreviations: VHN: Vickers Hardness Number, GP: G-ænial posterior composit, DXP: Dyract-XP compomer, and FBF: Filtek™ One Bulk-Fill composite, NaF: Sodium Fluoride, APF: Acidified Phosphate Fluoride

Table 5. Correlation between roughness values (Ra) and color change (ΔE_{00}) and microhardness (VHN) values

		Surface roughness (Ra0)	Surface roughness (Ra1)
Color change (ΔE_{00})	r	-0.005	-0.033
	p-value	0.951	0.686
Microhardness (VHN)	r	0.210	0.048
	p-value	**0.010**	0.562

r: Pearson Correlation Coefficient, p values presented in bold font indicate statistical significance

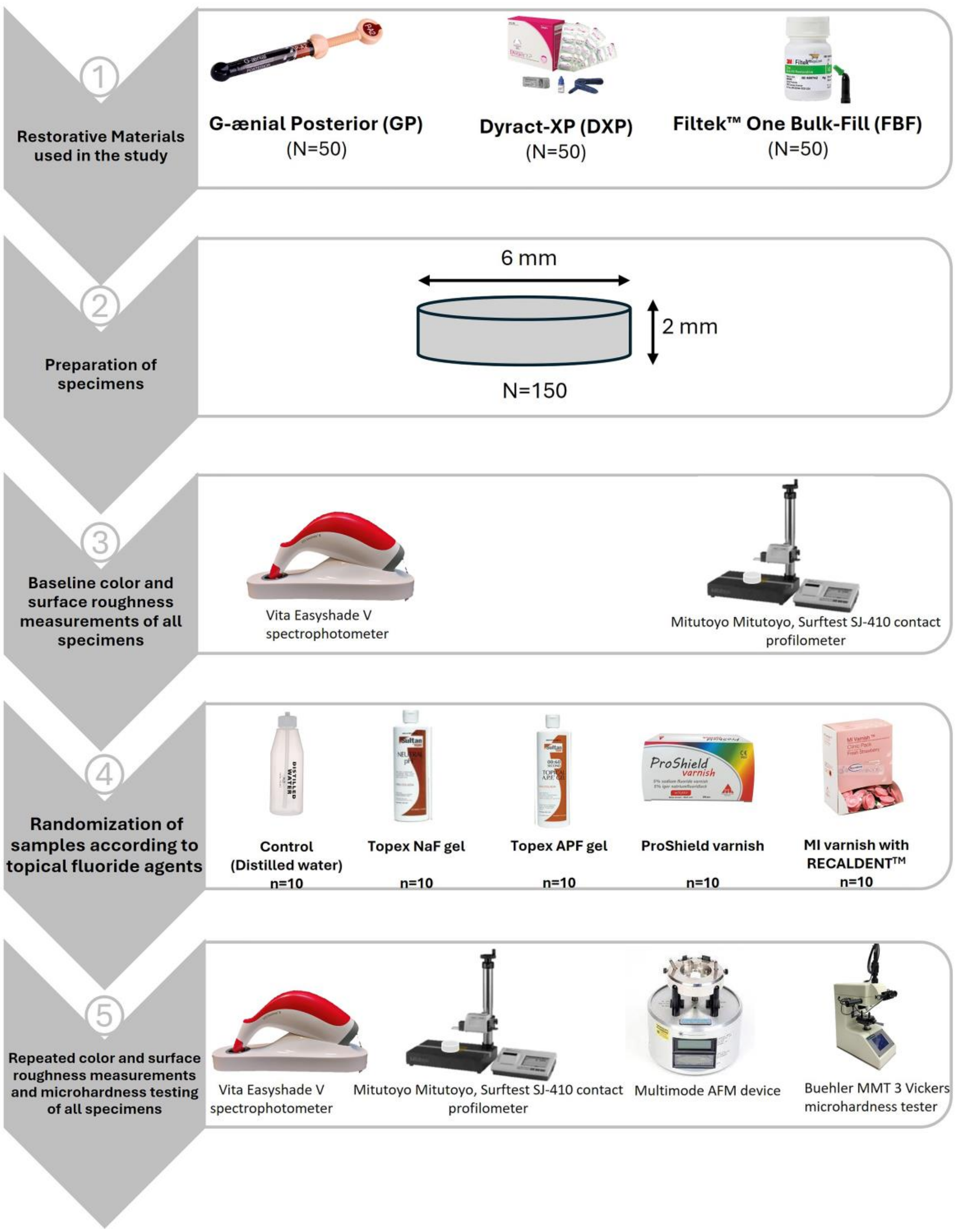

Figure 1. Schematic diagram of the study

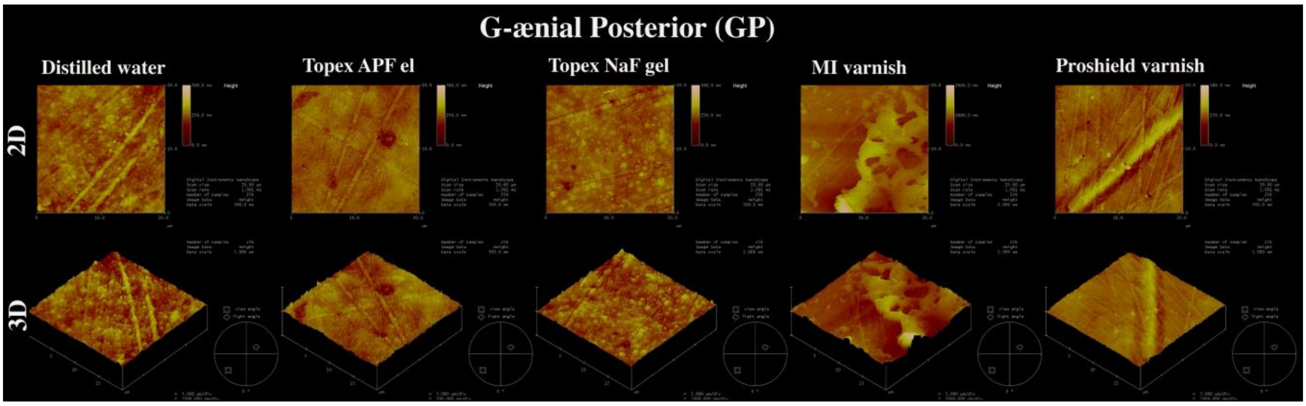

Figure 2. 2D and 3D atomic force microscopy images of G-ænial Posterior showing variations in surface topography.

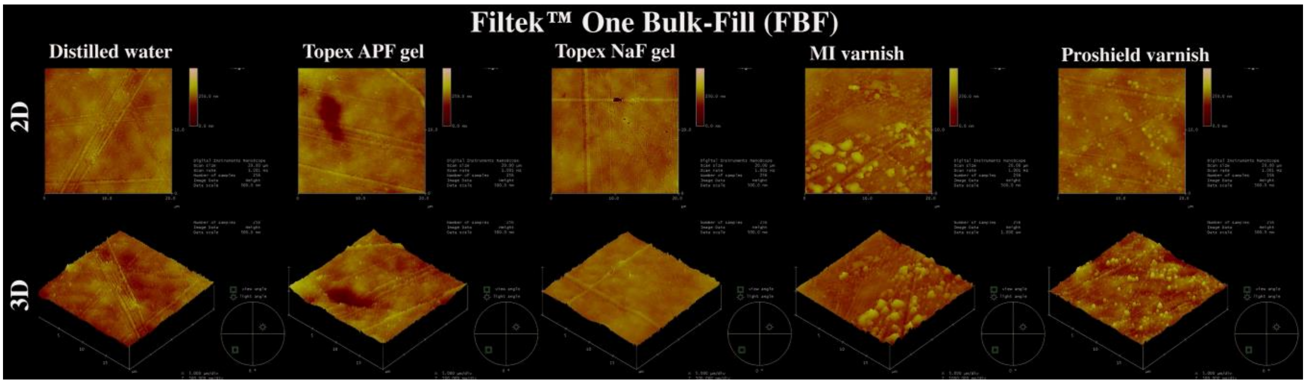

Figure 3. 2D and 3D atomic force microscopy images of Filtek™ One Bulk-Fill showing variations in surface topography.

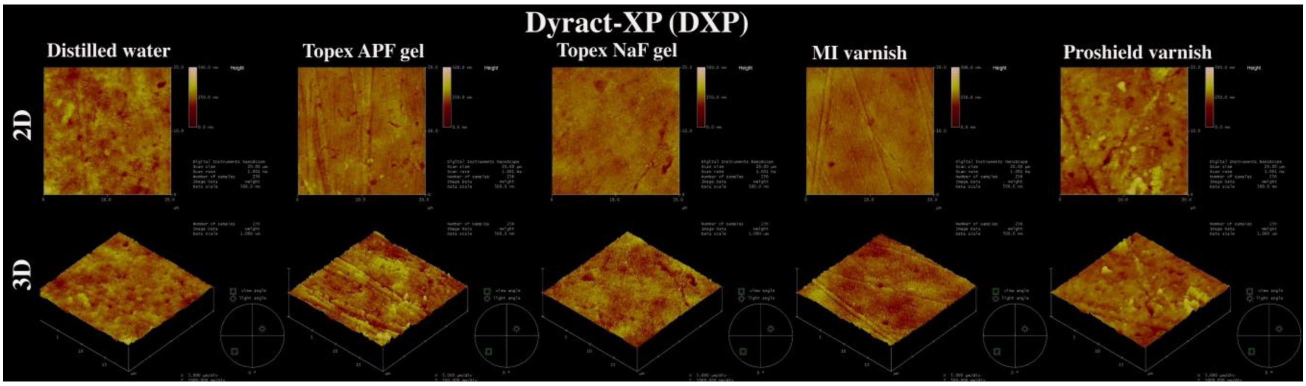

Figure 4. 2D and 3D atomic force microscopy images of Dyract-XP showing variations in surface topography.

DISCUSSION

The aim of this study was to investigate the effect of different topical fluoride agent applications on optical properties, surface roughness and microhardness parameters of resin-based restorative materials.

The findings of the current study revealed that the optical properties (color and whiteness) of restorative materials, were affected by the application of fluoride agents. Therefore, the first null hypothesis of our study stated that "topical fluoride agents have no discernible impact on the optical properties of diverse restorative materials", is rejected. The discoloration of the resin composites is directly related to the composition of the resin phase. Notably, urethane dimethacrylate (UDMA) exhibits superior stain resistance compared with bisphenol-glycidyl methacrylate (Bis-GMA) owing to its diminished water absorption and solubility properties.[33] The Bis-GMA monomer, characterized by heightened susceptibility to discoloration and possessing a "viscous and bulky bifunctional" matrix in GP, demonstrated minimal color alteration in the current investigation. This mitigation was achieved by dilution the Bis-GMA monomer with a more reactive monomer, namely triethylene glycol dimethacrylate (TEGDMA).[34] The dilution process facilitated the incorporation of a higher quantity of nanofiller into the resin matrix.[35] The heightened presence of fillers and monomers, particularly hydrophobic aromatic dimethacrylate, is posited as the rationale behind the observed diminished discoloration in the GP specimens, which is attributable to the resultant reduction in water absorption rates within the material.

Specifically, APF warrants careful attention because it possesses the potential to induce etching (owing to the hydrofluoric and phosphoric acid content) and staining of esthetic restorative materials.[36] In the present study, Topex APF gel caused more discoloration (not statistically significant) than Topex NaF gel. The foundational mechanism underlying the deterioration of resin-based composites by APF has been postulated to occur through three principal interaction pathways: the interaction of fluoride with reinforcing fillers, filler-matrix coupling agents, or the organic matrix.[37] The dissolution of composite filler particles is attributed to the presence of phosphoric acid and fluoride ions in the APF gel, which reduced the surface hardness.[23] The extent of damage to the resin surface is intricately linked to factors such as the type of fluoride gel used (acidulated or neutral), composition and size of filler particles within the resin composite, and the interplay between the resin matrix and inorganic fillers.[38] A previous scoping review study[39] reported that bulk-fill composite resins are characterized by matrix/filler ratio variability and filler heterogeneity in size and morphology, thus influencing their material properties. Numerous authors[40-42] have reported that the higher staining susceptibility in bulk-fill resin composites may be attributed to filler agglomerates that may not be perfectly silanized and integrated, resulting in higher staining susceptibility. Therefore, the reason why FBF/Topex APF had the highest discoloration in the current study can be attributed to the interaction between the zirconia agglomerates contained in the FBF and the phosphoric acid in the APF gel.

All combination of material/topical fluoride agents were below the clinically whiteness acceptability threshold (<2.60). No study has been found in the literature investigated the effect of topical fluoride application on the whiteness index of restorative materials. However, whitening effects have been reported to depend on the restorative material, shade, or type of chemical agents.[43] As mentioned earlier, the combination of a resin matrix, composition and size of filler particles within the resin composite, and the interplay between the resin matrix and inorganic fillers may have affected the ΔWI_D values.

The evaluation of the surface roughness involves both qualitative and quantitative methods. Qualitative assessments utilize optical or scanning electron microscopy (SEM), which provides visual observation of surface characteristics; however, these methods have limitations in direct three-dimensional (3D) reconstruction. In contrast, quantitative evaluations employ profilometry, a technique that yields numerical data on the surface roughness.[44] While profilometry offers valuable information, atomic force microscopy (AFM) has emerged as a superior option for investigations requiring a higher microscale resolution.[45] AFM facilitates precise topographical examination, enhancing the understanding of surface roughness and related interactions at a smaller scale.[44] In the present study, AFM imaging was conducted to complement and provide detailed insights into profilometry findings (Fig. 1-3).

In the present investigation, it was noted that the restorative material exhibited no discernible impact on surface roughness. In this context, the second hypothesis of the present study is accepted. Despite the variations in filler size, loading, and shape among the three materials, they all share a commonality in being resin matrix-containing materials. Although no substantial difference in the initial surface roughness was identified between the three materials, the DXP material exhibited the lowest surface roughness. It has been reported that urethane dimethacrylate (UDMA)-based resin composites exhibit lower roughness than bisphenol A-glycidyl methacrylate (Bis-GMA)-based composites.[46] Moreover, a lower proportion of filler (47 wt. %) and soluble strontium fluoride salts, which are not homogeneously incorporated in the polymer matrix, may be the reason for the lower surface roughness in DXP.

The four fluoride agents used in the current study, Topex APF gel, Topex NaF gel, MI varnish, and Proshield varnish, did not yield significant variations in surface roughness. Nevertheless, upon examination of the disparities in partial eta squared values, it can be asserted that fluoride agents exhibit greater efficacy in influencing surface roughness than restorative materials. Yeh *et al.*[11] conducted a comparative analysis of the impact of three distinct topical fluoride gels on the surface roughness of composites. They observed that two fluoride gels containing magnesium aluminum silicate (MAS) clay did not induce surface changes, whereas the MAS clay-free fluoride gel resulted in an increase in surface roughness. Through ICP-mass elemental composition analysis, the researchers identified the presence of MAS clay elements (Mg, Al, and Si) in the two gels (which did not affect the roughness). MAS clay, characterized by a structure with positive and negative layers, neutralizes by binding H ions in hydrofluoric and orthophosphoric acids through its positive layer. Consequently, APF gels with MAS clay content have been proposed to contribute to surface alterations. The absence of alterations in surface roughness following the application of fluoride agents in the present study is attributed to the fact that the agents in the study consisted of APF and neutral NaF gels containing MAS clay.

Unlike the restorative material and fluoride agent, the application phase (AP_0 and AP_1) variables had a significant effect on roughness. Regardless of the fluoride agent used, the roughness values of the materials before and after application were between 0.36-1.73µm. DXP showed the highest change in surface roughness after application. The increase in surface roughness of DXP can be attributed to the lower proportion of fillers (47 wt.%) and soluble strontium fluoride salts (not homogeneously incorporated in the polymer matrix).[47] Similar to the results of the current study, Avşar and Tuloglu[48] stated that fluoride varnish applications on different compomers caused a general tendency to increase in Ra values in all samples compared to those before application. Similarly, Dionysopoulos *et al.*[47] stated that the application of APF gel to the specimen surfaces affected the surface roughness of the enamel and the tested dental composite restoratives, depending on their composition. It was emphasized that significant changes in roughness depended on the fluoride agents.[11] However, in this study, the roughness was evaluated by comparing the experimental groups with the control group (not by measuring the Ra values of the samples before and after application). The discrepancies in the results of these studies may be due to the use of different composite materials and APF gels as well as different methods and study designs.

Considering the interactions of all three main variables (material-fluoride agent-application phase), it was observed that the roughness increased significantly after the application of Topex APF gel to the DXP material. This increase in the surface roughness of the compomer may be attributed to its inferior mechanical properties and higher solubility compared with other restorative materials.[49] Although it has been reported that 1.23% APF agents should be used with caution in patients with composite resin restorations, it has also been reported that weight or surface loss may vary in different resin-based materials.[36] Although there was an increase in the roughness values before and after the application of the other fluoride agents to the restorative materials, these increases were not significant. In light of this knowledge, this study's results showed that using agents with neutral pH in preventive fluoride treatments may be recommended, especially in patients with compomer restorations, since APF gel applications cause an increase in surface roughness.

There is no consensus threshold value for VHN hardness value. However, according to some authors, resin composites with a hardness value above 50 VHN are considered ideal.[50] In the current study, the restorative material variable had a significant effect on the VHN value, and while FBF showed the highest microhardness value, DXP was the closest (both were above 50 VHN). GP's VHN value was 42.97. Fillers such as zirconia, ytterbium, and strontium in FBF and DXP may contribute to their high microhardness values. Consistent with the findings of our study, Rizzante *et al.*[51] reported that bulk-fill nanohybrid resins exhibited superior surface microhardness values compared with conventional resins characterized by a lower filler content. The reduction in the microhardness observed for the GP material can be attributed to the dilution of the Bis-GMA monomer with the TEGDMA monomer. This phenomenon is substantiated by the findings of a prior investigation that demonstrated a negative correlation between microhardness and the release of TEGDMA monomers in resin-based materials.[46]

In a study conducted in Peru evaluating the changes caused by fluoride varnishes on composites, it was reported that MI varnish containing tri-calcium phosphate increased the microhardness of the composite, while NaF varnishes with NaF and CPP-ACP caused a decrease in microhardness.[52] When the significant effect of fluoride agents on microhardness was evaluated in the current study, ProShield varnish significantly increased microhardness in the GP material compared to the control and Topex APF gel. Therefore, the third hypotesis of the current study is rejected. The differences in the results of these studies may be due to the use of different resin composite materials as well as different methods.

In numerous studies[17,18,22,25,26,28,44,45] have investigated the effects of topical fluoride agents on surface alterations of restorative materials, and diverse fluoride agents with neutral and acidic pH values have been employed to facilitate comparisons. In our study, we selected the most frequently used clinical fluoride gels and varnishes. The rationale behind the selection of different application phases in this study lies in the reported variation in the surface loss of restorative materials depending on the application phase, coupled with discrepancies in the application phases recommended by different manufacturers. Although the long-term clinical effects of these applications remain unclear, they provide insight into the average potential effects of topical fluoride agents. Since the oral environment cannot be fully replicated in in-vitro studies, the findings need to be confirmed through clinical studies and long-term clinical follow-up. These limitations highlight the necessity for further research in this area.

CONCLUSIONS

In conclusion, topical fluoride agents may significantly affect the optical properties, surface roughness, and microhardness of different restorative materials, depending on their type, content, and chemical structure. The bulk-fill composite resin /APF gel interaction exhibited a notable color change (2.1) and whiteness change (-5.45), which surpasses the acceptability threshold. The compomer/APF gel interaction exhibited the highest increase in surface roughness (1.59), whereas the micro-hybrid resin composite/APF gel interaction showed the lowest roughness (0.75). The interaction between the resin material and the fluoride agent significantly demonstrated higher microhardness in the Bulk-Fill nanofilled composite/APF gel combination (66.38).

This investigation provides valuable insights into the nuanced influence of fluoride agents on diverse restorative materials, underscoring the significance of meticulous consideration of material-specific factors in the context of dental applications. Future studies should take into account the presence of salivary proteins in the oral environment, as they may exert a protective effect on the material surface. Additionally, environmental pH fluctuations could potentially influence surface properties, and these factors should be considered in subsequent research.

ACKNOWLEDGEMENTS

The authors thank Ihsan Akşit, Erciyes University Technology Research and Application Centre lecturer, for AFM images and Assoc. Prof. Dr. Naci Murat for his kind support during the statistical analysis.

FUNDING

“Not applicable”.

CONFLICT OF INTERESTS

“None”.

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