Effect of Fluoride Gel on Microhardness of Flowable Composites: An *In Vitro* Study

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Abstract

Objective: Adequate knowledge about restorative materials particularly flowable composite resins with favorable physical properties like optimal microhardness and resistance to environmental factors is a big concern for many dentists. Flowable composites have several applications for fissure sealant therapy and preventive resin restorations (PRR); which are common treatments in pediatric dentistry. Acidulated Phosphor Fluoride (APF) topical gels can cause surface degradation and weight loss in composite resins and decrease their wear resistance. This study aimed to assess the effect of APF gel on the microhardness of flowable composites.

Methods: This experimental *in vitro* study was conducted on 60 specimens measuring 2mm in thickness and 6mm in diameter fabricated of Tetric N-Flow (Ivoclar-Vivadent), PermaFlo (Ultradent) and Denfil (Vericom) flowable composites (n=20 for each group). Specimens were stored in artificial saliva at room temperature for one week. The 20 specimens in each group were randomly divided into 2 subgroups of test and control (n=10). Microhardness was measured using Vickers microhardness tester. In the control groups, 3 indentations were made on each disc on the periphery of a circle with at least 1mm distance from one another and from the specimen margin. The mean microhardness value was then calculated. The mean of all measurements was calculated as well. Test specimens were subjected to (Sultan) 1.23% APF gel for 4min and then rinsed and air-dried. Their microhardness was measured again as described above. Two-way ANOVA was used to compare the effect of APF gel and type of composite on the microhardness of different flowable composites.

Results: Based on the results, the microhardness of Denfil, PermaFlo and Tetric N-Flow was 16.5 (1.32), 37.36 (2.13) and 20.39 (0.52) before and 16.46 (2.20), 35.04 (2.43) and 19.13 (2.20) after the application of APF gel, respectively. The difference between the before and after values for each composite was significant (*p*=0.193) while the difference between different composites was statistically significant in this regard (*p*<0.001).

Conclusion: Exposure of Denfil, PermaFlo and Tetric N-Flow flowable composites to APF gel for 4min had no effect on their microhardness.

Key words: APF gel, Fissure sealant therapy, Flowable composite, Microhardness, PRR, Topical fluoride therapy.

Introduction:

Adequate knowledge about restorative materials particularly flowable composite resins with favorable physical properties like optimal microhardness and resistance to environmental factors is a big concern for many dentists. Use of flowable composites in dentistry was debuted in 1990 and gained increasing popularity for different clinical applications over time (1, 2).

Flowable composites have several applications for fissure sealant therapy and PRR; which are common treatments in pediatric dentistry. Periodic topical fluoride therapy was first introduced in 1940 for prevention of tooth caries (3). It is important to obtain a restorative material with optimal physical properties and adequate sealability of the pits and fissures. Any change in the structure, composition or physical properties of the restorative material will have consequences like microbial plaque accumulation on the restoration and its margins, color change, formation of porosities, gradual wear and eventual failure of the restoration and consequent recurrence of caries.

In vitro studies have indicated that restorative materials namely porcelain, resin-based composites, sealants and glass ionomer (GI) cements are susceptible to change in their surface morphology as the result of exposure to fluoride gels (4). APF topical gels can cause surface degradation and weight loss in composite resins and decrease their wear resistance. The amount of weight loss and surface degradation seem to be related to the type of filler particles and type of topical fluoride applied (5). Abate, *et al.* in 2001 reported that the microhardness of GI was significantly higher than that of resin-based composites following exposure to APF foam. The duration of foam exposure is the main factor in microhardness reduction (4). However, Mujdeci and Gokay in 2006 demonstrated that commercial bleaching agents had no adverse effects on the microhardness of tooth-colored restorative materials (6). Moslemi, *et al.* in 2009, showed that APF fluoride gel had no effect on the microhardness of filled composites used for sealant therapy. However, it decreased the microhardness of unfilled composites (7). Considering the controversial results and shortcomings of the previous studies, this study sought to assess the effect of topical fluoride therapy with APF gel (1.23%) on the microhardness of flowable composites.

Methods:

In this *in vitro* experimental study, three flowable composites namely Tetric N-Flow (Ivoclar, Vivadent), PermaFlo (Ultradent) and Denfil (Vericom) were used. Twenty disc-shaped specimens were fabricated of each composite resin using plastic molds measuring 2mm in thickness and 6mm in diameter. Composite resin was applied to the molds and covered with celluloid strips. Glass slabs were placed beneath and over the molds to pack the composite resin and prevent void formation. A 1.650g weight was placed over the specimens and removed after 30s (8). Specimens were light cured through the glass slab with one mm distance from the tip to the surface of specimens for 40s (6) using a light-curing unit (UDSM, Guilin Woodpecker) with an output power of 650 mV/cm^2 (6). Immediately after the polymerization, the celluloid strip was removed and disc-shaped composite specimens were stored in artificial saliva at room temperature for one week (5). The 20 specimens in each group were then randomly divided into 2subgroups of 10. The microhardness was measured using Vickers microhardness tester by making micro-indentations with 50g load for 30s (7). In the control group, each specimen was clamped in such a way that the tester tip was perpendicular to the specimen surface. Three indentations were made on each disc on the periphery of a circle with at least 1mm distance from one another and from the specimen margin. The mean microhardness value was then calculated. The mean of all measurements was calculated as well (8). The specimens were then rinsed with water and gently air-dried. (Sultan) 1.23% APF gel was applied to the surface of specimens for 4min using a microbrush, rinsed with water and gently air-dried. The microhardness of the test group specimens was measured as described above (8).

Two-way ANOVA was used to compare the effect of APF gel and type of composite on the microhardness of different flowable composites.

Results:

This study was done on 60 specimens fabricated of three different flowable composite resins namely Tetric N-Flow (Ivoclar, Vivadent), PermaFlo (Ultradent) and Denfil (Vericom). Two disc-shaped specimens were fabricated for

each test and control group.

Comparison of the microhardness value of test group specimens after exposure to APF gel with the control groups using two-way ANOVA revealed that the difference in microhardness for Tetric N-Flow, PermaFlo and Denfil was -0.04, -2.32 and -1.26, respectively ($p<0.001$). In both the test and control groups, the mean microhardness of PermaFlo and Denfil was higher and Tetric N-Flow had the lowest microhardness value (Table 1).

Table 1- Changes in microhardness of flowable composites after exposure to APF fluoride gel in comparison with the control group

Microhardness (VHN) Type of composite	Test mean (SD)	Control mean (SD)	p-value
Tetric N-Flow	16.46 (2.20)	16.50 (1.32)	
PermaFlo	35.04 (2.43)	37.36 (2.13)	
Denfil	19.13 (2.02)	20.39 (0.52)	0.193
Between group comparison	Permaflo> Denfil > Tetric N-Flow	Permaflo> Denfil > Tetric N-Flow	

The reduction in microhardness was 42.6% after the application of APF foam for 7 days. This rate was 16.36% after 7 days of storage in distilled water. The reduction in microhardness 24h after the application of APF gel was 23.71%. These values were not significantly different. The reduction in microhardness after 24h of storage in distilled water was 4.63%; which was significantly smaller than the values in other groups.

Discussion:

Hardness is defined as resistance to penetration or indentation. Indentation is made as the result of interaction of different characteristics of a material; thus, a widely accepted definition for it does not exist. Strength, proportional limit and the ability of the opposing teeth to cause abrasion in the material are among the hardness characteristics of a substance (7). Decreased microhardness of dental materials may enhance

their degradation, anatomical deformity and color change in the clinical setting (4, 8). Since this was an *in vitro* study, these factors could not be evaluated.

In general, hardness of a material depends on its multiple mechanical characteristics. Wear resistance is among these clinical characteristics. Hardness assessment relatively determines this behavior (8). Vickers microhardness test is among the most reliable microhardness measurement tests (7). Composite resins are made of silica compounds. Previous studies have suggested that presence of silica in ceramic dental materials makes them susceptible to hydrofluoric acid (HF); thus, type of filler can affect the surface microhardness reduction following the application of APF foam (4, 8).

Abate *et al*. in their study in 2001 evaluated the Barcol hardness of dental materials after APF foam application (4). They evaluated the hardness of Ariston PHC, Silux Plus and Filtek P60 resin-based composites as well as F2000

compomer and Vitremer and Lonofil Molar GIs after their exposure to APF foam. They concluded that the efficacy of APF foam depends on the restorative material, and the surface/treatment, surface/material and surface/treatment/material interactions were not significantly different. They found no significant difference in microhardness reduction between materials; which is in contrast to our results. This issue may be due to the difference in the type of storage media (distilled water, Oral-B Minute Foam) and duration of storage (1 minute, 24h and 7 days). In our study, the teeth were stored in artificial saliva for 7 days.

Moslemi *et al.*, in 2009 evaluated the effect of APF gel on the microhardness of unfilled (Clinpro) and filled (Helioseal) sealant composites using Vickers microhardness test. They found no significant difference in the microhardness of Helioseal composite before and after the application of APF gel. However, this difference for Clinpro composite was statistically significant. Thus, it seems that filled sealants are more resistant to APF gel than the unfilled sealants. They stated that HF acid in the APF gel affects the filler particles, and composite resins containing boro-aluminosilicate glass show the greatest surface changes after the application of APF gel. Insignificant effect of APF gel on Helioseal composite is due to the absence of gap between filler particles. It appears that if the distance between filler particles is less than 0.1mm, the protective effect of filler particles confers resistance against APF gel (7).

Yeh *et al.* in 2011 evaluated the microhardness of nanofilled composites after the application of topical fluoride gel. They showed that application of Topex, Zap and a pH7 gel did not cause morphologic changes in Premisa, Filtek Z350 and Grandio composite resins. However, Taste Gel with 60s application time decreased the microhardness of Premisa, Filtek Z350 and Grandio. The insignificant effect of fluoride solutions on microhybrid composites is due to the presence of Estelite Sigma fillers in their composition. Since the size of Estelite Sigma filler particles is 0.1 to 0.3 mm, it can have a protective effect and because the space between filler particles in nanofilled and nanohybrid composites is greater than in microhybrid, the effect of fluoride solutions on them will be greater (9).

Based on a study by Benderli *et al.* in 2005 application of fluoride gel alone on the test surfaces caused a moderate to severe reduction in microhardness except in Compoglass Flow. Also, the addition of pH circulation increased micromorphological changes on the surface of materials. The resistance of compomers to APF gel was greater than that of resin-reinforced glass ionomers (10); this finding is in accord with our obtained results because compomers have a resin base similar to the materials used in our study.

Wilde, *et al.* in 2006 evaluated the effect of two fluoride mouth rinses (Oral-B and Fluorgard) on the surface microhardness of two resin-reinforced GIs (Fuji II and Vitremer). The results showed that the microhardness of resin reinforced GI changed by both solutions ($p<0.01$). Fluorgard caused greater changes than Oral-B. Regardless of the type of solution and time of measurement, Fuji II had a higher mean microhardness than Vitremer ($p<0.05$). Also, microhardness increased during the first 7 days and maintained high for 30 days. Considering the interaction between the type of solution and time of assessment, specimens in artificial saliva (control) and Oral-B showed increased microhardness during the first 7 days and it remained high for 30 days. Specimens immersed in Fluorgard did not show a significant change in microhardness during the first 7 days. This rate decreased at 30 days. All solutions showed the same behavior for both restorative materials (8). This finding was in contrast to our results. This difference may be due to the different

methodology, assessment time points (24 and 48h, 7, 14, 21 and 30 days), type of storage media (Oral-B and Fluorgard) and the understudy materials (Vitremer and Fuji II).

Triana *et al*., in 1994 assessed the effect of Oral-B Minute APF gel on the surface of 2 light-cured GIs (Vivaglass, Vitrebond) with/without a protective glaze (Ketac Glaze). They indicated that APF gel significantly etches the surface of both GIs and the unfilled resin (Glaze) protects the GIs from the abrasive effects of APF gel (11). This difference may be attributed to the different understudy materials and different storage time in distilled water. They showed that application of glaze to the restoration surface prevents surface degradation as the result of exposure to low pH compounds and maintains their physical properties.

Yap *et al*. (2002) studied the effect of professional topical fluoride therapy (1.23% APF gel and foam, 0.9% neutral foam and 0.4% stannous fluoride)on the surface roughness of composite (Spectrum TPH), compomer (Dyract AP) and giomer (Reactmer) restorations (5). They showed that the effect of topical application of fluoride on surface microhardness depends on the type of material. Application of APF gel and foam compared to stannous fluoride gel and foam caused a significant reduction in surface microhardness (4.53 versus 15.97). No significant difference was seen between the 3 groups in microhardness after the application of stannous fluoride gel. However, the surface microhardness of compomer significantly decreased following the application of neutral foam; which is contrast to our findings probably due to the differences in the understudy materials, storage media and duration of fluoride application (36h). Long-term application of fluoride in the mentioned study compared to the application of APF gel for 4min in our study can explain the different results in terms of microhardness reduction.

Clinical use of composites and compomers has increased during the recent years due to improved formulation, simplifying the steps, higher esthetics and decreased popularity of amalgam due to fear of mercury toxicity and revisions in legal rules and regulations. APF topical gels can damage the surface, decrease weight and reduce the wear resistance of composites. The amount of weight reduction and surface damage depends on the filler particles of the composite resin and topical fluoride used. Barium boroaluminosilicate particles in composite resins are the most susceptible agents to surface changes due to the application of APF gel (5).

Oral-B Minute Foam is comprised of 2.14% sodium fluoride and 0.23% HF acid. Both these products are produced by the degradation of Oral-B Minute Foam after long exposure. However, in the oral environment, presence of salivary proteins may protect the restoration surface before and after the exposure to foam (8). On the other hand, it should be noted that in a clinical preventive program, a type of fluoride product may be used to increase or decrease the effect of APF foam and this issue should be further evaluated in future studies (4).

Hosoya *et al*. in 2011 evaluated the effect of APF gel on surface roughness, shine and color of composite resins and reported that EQ composite polished with 180 grit abrasive papers showed no significant difference while B2 and CM showed less hardness after the application of APF. Before the application of APF, EQ composite had significantly less hardness than CM and B2 and after the application of APF, the hardness of group polished with 3000 grit abrasive paper was less than that of the other groups. The difference between their results and ours may be due to the size of filler particles of the understudy composite resins (12).

In a comparative study of the effect of topical fluoride on the microhardness of different composites by Gill *et al*. in 2010, APF caused a significant reduction in the microhardness of all

restorative materials. This reduction was the highest in GI and the lowest in resin-modified GI. No difference was found in the microhardness of restorative materials after the application of NaF (13). Some studies have shown that acids produced by the biofilm bacteria affect the surface of composite resin restorations and cause surface changes. This effect is similar to the use of phosphate fluoride on esthetic restorations (8).

The effect of professional topical fluoride therapy on the surfaces depends on the type of material. Decreased hardness may be due to the different pH or fluoride concentration. Also, 1.23% APF and foam with higher fluoride concentration, have reactivity 0.9% more than the neutral foam and 0.4% more than stannous fluoride. Three main routes exist for the interaction of materials and fluoride. An interaction exists between the organic matrix, filler matrix coupling agents or reinforcing fillers. Organic matrix of some composites is organic esters derived from methyl methacrylate and organic esters due to hydrolytic differences are similar to low pH esters. This reaction is accelerated by acid and is pH dependent. Since APF gel and foam are both acids with a pH of 3 to 4, the water content of organic matrix increases leading to the observed reduction in microhardness. Decreased microhardness after treating with APF foam and gel may be due to the presence of HF acid. HF acid degrades the filler and fluorosilicate particles in composite resin that attribute to surface roughness. It also degrades the hydrogel cylsius layer and ionic matrix surrounding the fluorosilicate particles in the giomer. Degradation of filler can increase the contact area of organic matrix and enhance the hydrolytic effect. Fluoride ions are involved indepolymerizing the matrix-filler interface reactions. Fluoride can change the orientation of single layer water absorbed in filler where silicates form hydrogen compounds. This material can also hydrolyze the organosilicone ester and derange the siloxane network structure. This network is formed by the concentration of intramolecular silanol groups and stabilizes the interface. All these processes may weaken the filler-matrix interface and lead to filler loss and microhardness reduction. APF foam is less harmful than APF gel. Microhardness after treatment with neutral foam is much higher than microhardness after treatment with APF foam and gel. Thus, for intraoral composite restorations, 0.9% fluoride neutral foam is topically used. The pH of stannous fluoride is 4.5; which is lower than that of the neutral fluoride foam. Thus, this finding may be due to the higher concentration of fluoride in the neutral foam. Also, it can be sufficient for polymerizing filler-matrix interface reactions. However, the uncreative fluoroaluminosilicate glasses in the compomer may be damaged (5).

Conclusion:

Based on the results, we may conclude that exposure of Denfil, PermaFlo and Tetric N-Flow flowable composites to APF gel for 4min had no effect on their microhardness.

Conflict of Interest: "None Declared"

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