

## Comparing the Effect of Topical Acidulated Phosphate Fluoride on Micro-Hardness of Two Fissure Sealants and One Flowable Composite

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### Abstract

**Objective:** Probable effect of fluoride containing materials like APF on sealants is very important. The purpose of this study is to assess the effect of frequent application of APF (1.23%) on the surface microhardness of two fissure sealants and one flowable composite.

**Methods:** In this experimental study, 81 specimens of two fissure sealants and one flowable composite were prepared, using special polymer mold in three groups of 27 including unfilled resin sealant (Fissurite F, Voco), filled resin sealant (Fissurit FX, Voco) and one flowable composite (Arabesk Flow, Voco). Then, these three groups were divided into three sub groups of 9 as follows: Group 1, 4, 7 (control): No treatment- Group 2, 8, 5: Single application using APF- Group 3, 6, 9: Six times application using APF. The APF was applied on the experimental specimens every time for 4 minutes. Then, the samples were stored in the distilled water. Finally, the surface microhardness of the sealants was measured using Vickers test. The statistical analysis was performed by 2-way ANOVA & One-way ANOVA tests.

**Results:** Type of materials (without filler, containing filler and flowable composite) was effective on the surface microhardness and the difference between three types of materials was statically significant ( $p < 0.001$ ). Unfilled fissure sealants, showed minimum surface microhardness and flow able composite showed maximum surface microhardness. Also, the frequency of gel use (once or many times), had no effect on the average surface microhardness of the materials under review ( $p > 0.05$ ).

**Conclusion:** All three materials were resistant to the destructive effects of APF gel and it did not make a significant microhardness changes.

**Key words:** Acidulated phosphated fluoride, Dental caries, Fissure sealants, Flowable composite, Prevention, Surface microhardness.

**Please cite this article as follows:**

Mazaheri R, Pischevar L, Keyhanifard N, Ghasemi E. Comparing the Effect of Topical Acidulated Phosphate Fluoride on Micro-Hardness of Two Fissure Sealants and One Flowable Composite. *J Dent Sch* 2014; 32(2): 103-110.

Received: 12.03.2013

Final Revision: 11.08.2013

Accepted: 29.10.2013

### Introduction:

Caries prevention is considered as important particularly for children and adolescents. Various methods are available to prevent caries and stop initial caries including Fluoride and Fissure Sealant application.

Resin-based composites, Polyacid-modified resin composites (compomers) and glass ionomers are extensively used for restoration of

primary and permanent teeth. Fissure sealants are also used to protect teeth surface from caries and there are several studies indicating their great impact on preventing development and progression of dental caries. Now, various fissure sealants are available on the market, some of which lack filler and some others contain various amounts of filler particles (1).

Many patients, cured by above mentioned substances, receive other preventive treatments

such as local Fluoride in different forms. Laboratory studies have indicated that restorative materials, such as porcelain, resin composite, fissure sealant and glass ionomer, are susceptible to change in surface morphology while treating with local Fluoride compounds (1,2,3), so that their combination and surface characteristics may change when influenced by strong acids. One of these compounds which is considered as a preventive strategy for children and adolescents and can also be effective on surface morphology of restorative material is Fluoride Phosphoric Acid gel (1, 2). This type of gel etches enamel and increases Fluoride uptake (4).

Several investigations have indicated that Fluoride therapy leads in a reduction in microhardness of glass Ionomeric restoration (5, 6, 7). In addition, an increase in surface roughness and a decrease in hardness of resin composite after using Fluoride-containing compounds are reported in some investigations (8, 9, 10). Certainly, some research has indicated no impact of Fluoride therapy on surface characteristics of materials (11, 12).

Although the impact of APF gel on glass ionomer and composite material has been extensively reported, few articles are available on the impact of this gel on sealant material. Shafiei and Memarpour have argued that APF gel has no impact on surface hardness of resin sealant without filler (13). Moslemi, *et al.* (2009) also claimed that APF gel has no effect on surface microhardness reduction of fissure sealant containing filler (14).

As the impact of APF gel on flowable composites and their comparison with fissure sealant material has not been investigated, this study aims at investigating the impact of successive application of APF gel (1.23%) on surface microhardness of three materials including resin sealant without filler (Fissurit F, VOCO), resin sealant with filler (Fissurit FX, VOCO) and flowable composite (Arabesk Flow,

VOCO) and their comparison.

## Methods:

In this experimental-interventional study, 81 disks were prepared in a special mold with 10 mm diameter and 2 mm height. Twenty-seven disks contained unfilled resin fissure sealant (Fissurit F, VOCO, Germany), 27 disks had filled resin fissure sealant (Fissurit FX, VOCO, Germany) and 27 disks had flowable composites (Arabesk Flow, A<sub>2</sub>, VOCO, Germany). The properties of the three materials can be found in Table 1.

**Table 1- Structural properties of the three studied materials**

	Fluoride content	4%
<b>Fissurit F (Voco)</b>	Resin matrix	Bis –GMA- di-urethane di- methacrylate BHI- Benzolderivate-
	Filler content	-
	Particle filler size	-
	Fluoride content	1%
<b>Fissurit FX (Voco)</b>	Resin matrix	Bis –GMA- di-urethane di- methacrylate BHI- Benzolderivate-
	Filler content	55%
	Particle filler size	microfiller
	Fluoride content	-
<b>Arabesk Flow (Voco)</b>	Resin matrix	Bis –GMA- urethane di- methacrylate Tri-ethylene glycol di-methacrylate
	Filler content	64%
	Particle filler size	0.7 Mm (Micro hybrid)

For samples preparation, first, cylinder molds made of a special polymer alloy with 10 mm diameter and 2 mm depth. The prepared mold was placed on a glass slab and was fixed with wax. Next, the study materials (Fissurit F, Fissurit FX, Arabesk Flow) were respectively injected into the mold until the mold was completely filled. Then they were covered with a

celluloid tape and a glass slide was placed on its surface to remove the extra material. The surface of Fissurit F and Fissurit FX samples was light cured (Fibop, FB-A3 LED, China) with 550 mW/cm<sup>2</sup> for 30 seconds (as per the manufacturer's instruction). The surface of Arabesk Flow samples was exposed for 40 seconds. After curing, the disks were removed from the mold. The surface of the disk which was close to celluloid tape (the surface used for fluoride therapy and measuring the superficial micro-hardness) was marked so that it became distinctive from other surfaces. At the beginning, the intensity of light curing units was measured, and recalibrated after curing 10 samples.

After all the samples (81 disks) were prepared, they were divided into 9 experimental groups (each substance was divided into 3 groups). Each group included 9 disks in coded plastic containers containing distilled water and kept at 37 °C (inside an incubator) for 48 hours for completion of the polymerization. Three of the nine groups (one group per substance) were used as control group (Groups 1, 4 and 7). In the control group, fluoride gel was not used on the surface of the specimens while in the other 3 groups (groups 2, 5, 8) APF fluoride gel (Sultan Topex, Sultan Dental Products, USA, 1/23%) was once placed on the surface of the samples with a cotton roll for 4 minutes in a way that the gel completely covered the surface of the disks. In the last three groups (groups 3, 6, 9), APF Fluoride Gel was applied to the sample surfaces 6 times for 4 minutes (every two weeks). After applying the gel, the sample surfaces were cleansed with a cotton roll and the samples were again placed in distilled water.

The surface of the samples was examined through the Vickers micro-hardness test and Micro met device (BUHLER Model). In order to measure the surface micro-hardness, 3 points were once more selected on each disk and each point was placed under a load of 10 gram for 10 seconds. The pyramid located at the tip of the

device was in the form of a diamond with equal sides which left a diamond-shaped impression on each of the three points. Eventually, calculating diameter mean of the resulting diamond and using the formula  $Hv = \frac{1/8544 \times F}{d^2}$ , hardness intensity was obtained for every point and hardness mean for every sample was resulted. The collected values were recorded in the checklists, and the data were entered in SPSS statistical software version 18. Analysis of each set of data was done through two-way analysis of variance test (2-way ANOVA) and Tukey test.  $p < 0.05$  was considered statistically significant.

## Results:

The mean surface micro-hardness in 9 test groups is shown in tables 2. Two-way ANOVA showed that the type of material used (unfilled fissure sealant, filled fissure sealant and flowable composite) was effective on the amount of surface micro-hardness and there is a statistically significant difference between the three materials. ( $p < 0.001$ ) According to table 2, the minimum surface micro-hardness belong to Fissurit F and the maximum surface micro-hardness belong to Arabesk Flow. ( $p < 0.001$ )

Also, two-way ANOVA showed that in all three studied materials, there was no significant relationship between the number of times of APF gel application and the mean surface micro-hardness. ( $p = 0.34$ )

In other words, the number of times gel is applied (one-time or several time application) does not affect the mean of surface micro-hardness of the studied materials (Table 2). Moreover, the experiment showed that there was no significant interaction between the type of material used and the number of times the gel is applied. ( $p = 0.142$ )

In addition, Tukey test showed that the mean surface micro-hardness in Arabesk Flow was

more than that of Fissurit FX ( $p<0.001$ ) and Fissurit F, and the mean surface micro-hardness

in Fissurit FX was more than that in Fissurit F ( $p<0.001$ ).

**Table 2- The mean surface roughness and standard deviation of 9 studied groups**

	Without filler mean (SD)	Filled mean (SD)	Composite mean (SD)	Total mean (SD)
<b>Without gel</b>	15.96 (4.27)	20.70 (5.45)	35 (3.16)	24.01 (9.4)
<b>Gel applied once</b>	18.93 (3.77)	26.02 (7.91)	32.53 (3.45)	26.7 (7.6)
<b>Gel applied 6 timed</b>	15.97 (3.82)	23.63 (3.87)	34.27 (3.72)	24.6 (8.5)
<b>Total</b>	16.71 (4.03)	23.46 (6.06)	33.93 (3.48)	25.1 (8.5)
<b>Test results</b>	Gel effect: $F=1.1, p=0.34$ Group effect: $F=89.4, p<0.001$ Interactive effect: $F=1.79, p=0.14$			

**Discussion:**

Side effect potential of topical fluoride is expected for various aesthetic restorative materials, due to high reactivity of Fluoride containing materials particularly APF.(5,15,16) Through increasing evolutionary process of resin material, it appears that flowable composites also own characteristics that can be used as fissure sealant or applied for protective resin restoration (PRR). What is of major importance is their probable impacts on one another.

Some laboratory investigations indicated changes in some tooth-colored materials after applying fluoride containing compounds. Most of these researches have been done by changing the surface hardness of the materials, creating roughness on their surface or weight change or investigating with electron microscope. Reduction of material hardness leads to its higher degradation and finally there is the possibility material loss. Furthermore, increased surface roughness increased plaque adhesion, and surface discoloration and surface fatigue fracture occurs in the repair material. In fact there was a relationship between the increase in surface roughness and the decrease in material

hardness (7, 13, 17). So far many researchers have examined the effect of topical fluoride on surface roughness and the hardness of restorative materials such as resin composite, compomer and glass-ionomer restorations (6, 10, 18-23) but a few researchers have investigated the effect of topical fluoride therapy on resin fissure sealants and so far no research has been made on the effect of topical fluoride on flowable composites.

Thus, due to repeated application of topical fluoride in children, in this study the effect of topical application of Acidulated Phosphate Fluoride gel was studied on the mean surface micro-hardness of two fissure sealants both with and without filler and a type of flowable composite.

Several studies show various effects of APF on different types of composite resins, compomer and glass-ionomer (6, 10, 18-24). Fluoride compounds which possess acidic components have a higher reactivity compared to neutral fluoride compounds. The pH of APF gel was 3.5 and it contains 2% fluoride sodium, 0.34% acid hydrofluoric, 0.98% orthophosphoric acid (25). The acidic quality of APF gel increases the water binding into organic matrix and the

plasticizing property of water on resin reduces the roughness of resin matrix. In this way, the polymer base of materials is destroyed through the hydrolysis or oxidation, and the changes in pH can alter the organic composition of substance by hydrolysis of the ester groups of the matrix. Hydrolysis of these ester bonds forms free carboxylic acidic groups which reduce the pH of polymer matrix (13, 18).

The APF gel damages the resin/ filler interface as well as the organic resin matrix and separates the filler particles. This is due to the presence of acidic compounds in the APF that etch filler particles and cause surface and weight changes in the composite (9, 13, 26).

It seems that the effect of APF on the composite largely depends on the size and type of fillers and APF's exposure time. This effect is higher on composites containing barium aluminosilicate glass particles (that are sensitive to hydrofluoric acid) and lower in the microfilled composites in comparison with composites with larger macrofilled inorganic particles. (7, 8, 12, 13, 27) For the same reason, a number of researchers have suggested that neutral fluoride compounds be used in patients with composite fillings (8, 9).

Studies have shown that the application of APF causes erosion, largely increases the surface roughness and reduces micro-hardness of glass-ionomers, This can be attributed to the sensitive structural nature of this substance (2, 6, 10, 21-23). However, erosion and decrease in micro-hardness of resin-modified glass-ionomers have been reported far less than conventional glass-ionomers which is due to the resin component which exists in this type of glass-ionomer (5). The studies conducted on the effect of APF gel on resin composites suggest changes in the morphological characterization of surface and increase in the surface roughness in various macrofilled or hybrid composites that contain macrofillers (19-21).

During their two studies using SEM and

evaluating surface roughness, Soeno, *et al.* (2000 and 2001) expressed that APF has less effect on microfilled composites compared to hybrid and macrofilled composites. Because the surface of hybrid and macrofilled composites contains inorganic macrofillers, their surface becomes rougher after applying APF. They suggested using microfilled composites or microhybrid composites (19, 20).

Penteado, *et al.* (2010) studies on surface roughness by AFM revealed that pH cycles did not affect microhybrid and nanofilled composites. The reason is the existence of small nano- and micro-fillers in the materials. These researchers recognized the short time of the sample's exposure to demineralized-remineralized solutions as another reason (18). Yeh, *et al.* (2011) also found the use of two different APF gels (Topex and Zap) ineffective on the surface micro-hardness and roughness of 3 types of nanocomposites and one type of microhybrid composite (11).

In this study, a significant difference was observed in the mean surface micro-hardness of the three studied materials (without application of APF gel) ( $p < 0.05$ ). The maximum surface micro-hardness belong to micro-hybrid flowable composite (Arabesk Flow, containing 64% Microfiller), followed by filled fissure sealant (Fissurit FX, containing 55% Microfiller) and the minimum surface micro-hardness belong to unfilled fissure sealant (Fissurit F), respectively. Since the monomer types that exist in the resin matrix of these three materials are exceptionally similar, the difference in surface micro-hardness of these materials can be attributed to the percentage of each monomer, the percentage of the existing filler (preferably) and the viscosity of each material. Boyer, *et al.* (1982) and Chung (1990) stated that the higher the filler content, the higher the hardness of the material (28, 29). Furthermore, in the present study, the application of APF gel (either once or repeatedly) did not affect the mean surface

micro-hardness of all three substances. Mazaheri, *et al.* also stated in their study that the application of this gel with this dosage administration has no impact on the mean of surface hardness of these three materials (30).

This can be attributed to the absence of filler in Fissurit F, the very small sizes of fillers (microfiller) and the proximity of filler particles to each other (14) to the two other materials. Another reason can be the short and insufficient duration of APF gel application for it to influence the polymer matrix even in frequent use.

Penteado, *et al.* (2010), besides the small size of filler particles, considers the samples' short exposure to acid solutions another reason that prevents changes in micro- and nano-filled composite surface roughness (18). Abate, *et al.* (2000) also reported the APF application time a major factor in reducing the material hardness (10). Furthermore, Kula, *et al.* (1992) and Shafiei and Memarpour (2010), similar to the current study, stated that APF did not affect the surface roughness of unfilled sealant resins (2, 13). Moslemi, *et al.* (2009) also found the application of APF gel ineffective on reducing micro-hardness of filled fissure sealant surfaces (microfilled) (14). de Alexander, *et al.* (2006) cited no impact of household bleaching material (Carbamide Peroxide 10%) on microhardness of sealants without filler. In this study, microhardness decrease of sealant with filler - Vitrosea Alfa- was also observed that can be attributed to long-term exposure to Carbamide Peroxide (4 hrs a day for 4 weeks) and

consequently dissolve of sensitive Silicon Dioxide glass particles present in this type of fissure sealant (12).

Kula, *et al.* (1992), in SEM studies, stated that losing fillers and the occurrence of surface changes in the filled sealants happen after APF is used. He considered the existence of silica glass macrofillers of sealants as its cause (2). It is necessary that further studies be conducted to clarify the complexity of this issue.

### **Conclusion:**

All the three materials of resin sealant without filler, with filler and flowable composite are resistant to the destructive effects of APF gel and no significant changes happen in their surface micro-hardness. This is important in their clinical application. Furthermore, considering that naturally the flowable composite surface has higher micro-hardness compared to sealant surfaces, the application of this material is preferred to sealant fissure.

### **Acknowledgements:**

Honorable professors and staff of Research Center of Isfahan University of Technology for measurement of material microhardness and also Mr. Hassanzadeh as statistical consultant of the research are sincerely appreciated and thanked.

### **Conflict of Interest: "None Declared"**

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