



## The Effect of Acidulated Phosphate Fluoride (APF) on the Microleakage of Composite Flow and Fissure Sealant Restorations

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### ABSTRACT

Fluoride therapy and fissure sealant are the main methods in the prevention of caries in children. However, even though many studies have reported an increased use of these two treatment, there has been very little research reported on the effectiveness of such use. The aim of this study was to evaluate the topical effect of APF gel on the microleakage of composite resin that is used as fissure sealant. A total of 60 healthy premolar teeth extracted for orthodontic treatment were disinfected and brushed by pumice in accordance with APF composite (Sultan, USA) restorations and treatment instruction and were divided into 4 groups of 15 (4 x 15): Group1) nanohybrid composite (Grandio flow, Voco) + saline, Group2) nanohybrid composite (Grandio flow, Voco) + APF, Group3) microhybrid composite (Arabesk flow, voco) + saline, Group4) microhybrid composite (Arabesk flow, voco) + APF. All samples were subjected to a thermo-cycling process and then were immersed in a methylene blue solution with a 30 second dwell time. The samples were cut and the microleakage was analyzed and sectioned by stereomicroscope (Magnus) at 40x magnification. Data were analyzed by Mann-whitney test. Mann-whitney test indicated that no significant difference exists between the microleakage of groups 1 and 2 (P=0.775), 3 and 4 (P=0.436). Group 4 demonstrated higher microleakage scores than other groups whereas group1 showed the lowest microleakage value when compared with other groups tested. Although the results did not show statistically significant differences, it is suggested that low composite resin and smaller filler particles particularly nanohybrid composites were found to be the best products in this group. But, given the negligible effect of the composites, the non-acidic fluoride material is recommended for composite restorations.

**Key words:** Microleakage, APF Gel, Fissure Sealants, Microhybrid Composite, Nanohybrid Composite

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### INTRODUCTION

Oral health is essential to general health and well-being at every stage of life. Bacteria from the mouth can cause infection in other parts of the body. Many children have inadequate oral and general health because of active and uncontrolled

dental caries. Dental caries is the most common chronic infectious disease in the world and can lead to pain, periodontal disease, early tooth loss and other social and psychological problems [1]. Although only 12.5% of all the tooth surfaces are occlusal, the dental cavity and fissures in the biting surfaces of the teeth have the highest prevalence (60%) of all dental caries [2]. Cavities and fissures provide a unique colonization for microorganisms and also there is no adequate

mechanical access for cleaning and oral hygiene procedures in these two areas.

Another factor that is responsible for the high prevalence of caries in occlusal surfaces is inadequate access of saliva into the occlusal fissure surface which prevents remineralization and reduces the effectiveness of fluoride in these areas. *Streptococcus mutans* is a major pathogen of human dental caries and is followed by caries after 6-24 month intervals. So, fissure sealants are of value in the prevention of tooth decay and may enhance caries resistance. The placement of fissure sealants is a highly effective means of preventing pit and fissure caries [3, 4]. In order to improve fissure sealants' retention and effectiveness in caries several different materials and techniques were compared. Currently, restorative materials (flowable composites) due to high filler, less porosity, surface abrasion resistance, greater influence in the pit and fissure sealants, and less microleakage have also been used as fissure sealant [5-7].

Topical fluoride therapy is another standard prevention action in pediatric dentistry that can be professionally applied once every 6 months. The most widely used fluoride in dental practices today is stannous fluoride 8-10%, 2% sodium fluoride, and 1.23% of APF gel which has been proven to have a beneficial effect than other topical agents. The APF gel is a mixture of sodium fluoride, hydrofluoric acid and phosphoric acid, and pH range between 3.2-3.5 [2, 8]. Studies show that the mixture of APF due to hydrofluoric acid causes etching of the surface of the material, buildup of bacteria, discoloration, deterioration and loss of continuity of the edges wear, decomposition of materials, and ultimately reduce the longevity of restorative material [8, 9]. It also significantly increases the surface roughness of glass ionomer restorations, resin modified glass ionomer, and compomer and composites [10, 11]. The application of APF compound causes degradation of filler in microfilled fissure sealant and also the degradation of matrix and filler in the glass ionomer sealant; as well as it has a negative effect on the stability of the interface matrix-filler [12, 13]. The purpose of this study was to assess the effect of repeated application of APF (1.23%) gel on the surface roughness of two fissure sealants and one flowable composite..

## MATERIALS AND METHODS

A total of 60 freshly extracted human premolars teeth in the age range of 15-25 years were selected. All of the teeth were extracted due to orthodontic reasons and less than 3 months has passed since their extraction and were divided to four groups of 15 : Group1) nanohybrid composite (Grandio flow, Voco) + saline, Group2) nanohybrid composite (Grandio flow, Voco) + APF, Group3) microhybrid composite (Arabesk flow, voco) + saline, Group4) microhybrid composite (Arabesk flow, voco) + APF. The group 2 and 4 were assigned as study group and the group 1 and 3 were assigned as control group. The teeth were washed according to ISO / TS 11405 standard and any blood clots or excess tissue of the teeth were cleaned. The teeth were kept in distilled water (grade 3, ISO 3696) in refrigerator at 4 ° C and the liquid holder was replaced every week. The teeth were cleaned with pumice prophylaxis and fissures of the teeth were cleaned with a blunt probe.

The teeth in the first and second groups were etched for 20 seconds with 37% phosphoric acid gel (Vococid, Voco, Cuchaven, Germany) and were dried for 15 seconds to a certain extent that the dental gypsum model is formed. Then the total-etch adhesive bonding (solobond M, Voco, Germany) was used. The bonding agent was placed on the etched surface by a Microbrush. After about 30 seconds pause the bonding agent was air-dried thoroughly. Then, LED light cure unit (woodpecker, China) with intensity of 1400 mW/cm<sup>2</sup> was used for 20 seconds. The flowable nano composite (Grandio flow, Voco, Germany) was used in the grooves and then in order to achieve a maximum hardening was light-cured for 40 seconds. The teeth in the third and fourth groups like the two previous group had the etching and bonding work stages. The two groups and flowable microhybrid composite (Arabesk flow, voco, Germany) was used and light-cured for 40 seconds.

In order to complete the polymerization process the composite samples were stored in the physiological saline for 48 hours. In group 2 and 4 (the study group) the enamel and dentin surfaces were treated with 1.23% APF gel (Sultan, USA) for 4 minutes and at the same time group 1 and 3 (control group) were kept in the saline. After teeth removal of liquid gel and making additions to it, samples were washed with distilled water and were kept in water for half an hour and this

process was repeated for three times. This time is considered as the equivalent of two years of fluoride therapy (recall every 6 months interval recommended for fluoride therapy). Then the teeth were thermocycled for 1000 cycles according to ISO/TR 11405 criteria. So that each cycle includes 30 seconds exposure in the tank of hot water ( $2 \pm 55^\circ\text{C}$ ), 30 second exposure in cold water ( $5 \pm 2^\circ\text{C}$ ), and 10 seconds at the same time move from one tank to the other tank. After thermocycling the specimens were taken out and dab dried with a tissue paper and the root apex was sealed with sticky wax. The teeth were coated with a two layer of nail varnish, except for 1.0 mm of the restoration margins were kept free of any coating. The coated teeth were then immersed in 2% methylene blue solution dye for 24 hours. After removal from the dye, the coating was removed with acetone solution and the teeth were thoroughly washed under tap water for 10 minutes and dab dried with tissue paper. When the samples were mounted in self-cured acrylic resin and were bisected longitudinally in a buccolingual direction the extent of dye penetration was scored under a stereomicroscope (Labo Med CMZ4, India) at 40x magnification. The dye penetration was determined with the following code: Code0. No dye penetration, Code1. Dye penetration limited to 1/3 occlusal enamel interface and fissure sealant or restoration, Code2. Dye penetration limited to the middle third enamel interface and fissure sealant or restoration, Code3. Dye penetration limited to 1/3 apical enamel interface and fissure sealant or restoration. Data were analyzed by Mann-Whitney test.

**RESULTS**

The extent of microleakage in Group1 (14.57) was lower than group3 (16.43) (Table1), but the difference was not significant according to the Mann - Whitney test ( $P=0.567$ ). The extent of microleakage in Group 2 (13.53) was lower than group4 (17.47), but the difference was not significant according to the Mann - Whitney test ( $P=0.233$ ) (Table2). The extent of microleakage in Group 1 (15) was lower than group2 (16), but the difference was not significant according to the Mann - Whitney test ( $P=0.775$ ) (Table3). The extent of microleakage in Group 3 (14.23) was lower than group4 (16.77), but the difference was not significant according to the Mann - Whitney test ( $P=0.436$ ) (Table4). The average microleakage

of composite in group one, two, three, four was 1.26, 1.4, 1.46, 1.86, respectively (Table5).

**Table 1: The average microleakage between Group 1 (nanohybrid composite + saline) and group 3 (microhybrid composite + saline)**

Group	N	Mean Rank	Sum of Ranks
1	15	14.57	218.50
3	15	16.43	246.50
Total	30		

Mann-Whitney= 98.5, Exact Sig= 0.233

**Table 2: The average microleakage between Group 2 (nanohybrid composite + APF) and group 4 (microhybrid composite + APF)**

Group	N	Mean Rank	Sum of Ranks
2	15	13.53	203
4	15	17.47	262
Total	30		

Mann-Whitney= 83, Exact Sig= 0.233

**Table 3: The average microleakage between Group 1 (nanohybrid composite + saline) and Group 2 (nanohybrid composite + APF)**

Group	N	Mean Rank	Sum of Ranks
1	15	15	225
2	15	16	240
Total	30		

Mann-Whitney= 105, Exact Sig= 0.775

**Table 4: The average microleakage between group3 (microhybrid composite + saline) and group 4 (microhybrid composite + APF)**

Group	N	Mean Rank	Sum of Ranks
3	15	14.23	213.5
4	15	16.77	251.5
Total	30		

Mann-Whitney= 93.5, Exact Sig= 0.436

**Table 5: The average microleakage, standard deviation, max/min value in each group**

Group	Value	
1	Mean	1.26
	SD	1.09
	Minimum rank	0
	Maximum rank	3
2	Mean	1.4
	SD	1.2
	Minimum rank	0
	Maximum rank	3
3	Mean	1.46
	SD	1.35
	Minimum rank	0
	Maximum rank	3
4	Mean	1.86
	SD	1.30
	Minimum rank	0
	Maximum rank	3

**Table 6: Frequency of dye penetration score**

Group	Sample size	Dye penetration code			
		code0	code1	code2	code3
1	15	6	0	8	1
2	15	5	1	7	2
3	15	6	1	3	5
4	15	4	1	3	7

## DISCUSSION

Despite recent advances in the field of public health and oral health, tooth decay remains the most common chronic disease in childhood and pit and fissure caries accounted for 80% of total caries in occlusal surfaces. Fortunately, dental disease and poor oral health can be easily prevented with regular access to fluoride therapy and fissure sealant treatment [1]. The aim of this study was to evaluate the topical effect of APF gel on the microleakage of microhybrid composite resin (Arabsk flow) and nanohybrid composite (Grandio flow) that is used as fissure sealant. The microleakage of microhybrid composite (Arabsk flow) in the present study before and after fluoride therapy was more than nanohybrid composite (Grandio flow); however the difference was not statistically significant (Tables 1 and 3). The most relevant factors related to micoleakage were polymerization shrinkage, thermal expansion coefficient, and modulus of elasticity, hydroscopic expansion, bond strength, bond conditions, factors associated with curing composite, flaw-related factors, and occlusal stress and thermocycling contribute to micrileakage [17].

In the present study most of the confounding factors had similar operation. For example, one type of bond and a curing device was used and the thermocycling range was similar. There was no difference between C. Factor and occlusal stress and among all the remaining factors, the polymerization shrinkage directly related to marginal leakage at the tooth-restoration interface [18]. Polymerization shrinkage leads to stress on adhesive layer leading to gap. The stress of 10 MPa leads to the marginal damage [19]. The main cause of polymerization shrinkage is a resin matrix that during the polymerization of monomer units returns to the original state. During pre-gel polymerization, the composite is able to flow, which relieves stresses within the structure. After gelation, flow ceases and cannot compensate for shrinkage stresses. Post-gel polymerization, therefore, results in clinically significant stresses in resin composite- tooth bond

and the surrounding tooth structure [20, 21]. Grandio Flow has more filler/weight content (80.2% WT) than Arabsk flow (64% WT), that is, the low resin matrix in the Grandio Flow composite can attributable to a significantly polymerization shrinkage and lower shrinkage stress [22].

The result of present study is similar to Hamouda *et al.*'s study that the microleakage of nano-composite was less than microhybrid composite, although the difference was not statistically significant [23].

Majeed, 2005 in a study compared the nanohybrid composite (Grandio flow) with ormocer-based composite and reported that microhybrid composite and nanofill composite had the lowest leakage. The result of study was in line with the present study [17].

Sharma, in a study in 2011 reported that the microleakage of nanofill composite was more than microhybrid composite which was incostinent with the result of the present study [24]. Sharma reasoned that the higher microleakage of nanofill composite associated with the higher filler in the composite and consequently higher stiffness and modulus of elasticity increase the polymerization shrinkage. The study also articulated that the cause of microleakage related to uniform size and small fillers in nanofill composite that make light distribution and reduce the light absorption during light curing and polymerization reduction and result to increased microleakage. In the present study the composite was nanohybrid composite composed of micrometer particles whose size range up to 3  $\mu\text{m}$  and affect light distribution and can be one of the reasons for the difference between the result of this study and Sharma's study [24]. Additionally, a number of studies cited that the combination of micron-sized fillers with the resin lead to thickness and viscosity. The nano-particles imbedded in a resin matrix do not behave like solid but it greatly resembles its liquids [2].

Benu *et al.*, 2012 in a study concluded that the Grandio flow composite have more filler percentage compared to other composites, but it has lower viscosity which in association with low modulus of elasticity represent lower solid- like property that lead to flow easily into the pits and fissures compared with other composites [26,27]. According to this fact adding

nano fillers does not increase stiffness and modulus of elasticity; moreover due to more flowable composite polymerization shrinkage will not increase.

The effect of fluoride on microleakage of two nanohybrid composites (Grandio flow and Arabesk flow) was not significant statistically (Tables 3 and 4). Tabari *et al.*'s study in 2012 also reported that APF has no effect on microhybrid composite [15]. Shabzendedar *et al.*'s study in 2011 concluded that the adhesive containing filler and microfiller were found to be wear resistant to the APF [28]. As well as, Seono *et al.*'s study in 2011 stated that APF had no effect on microhybrid composites which partially consistent with present study [13]. However, the result of study is inconsistent with the kula *et al.* and kargul *et al.*'s study that reported the adverse effect of APF on the surface roughness of all composites [29, 30].

The microleakage in nanohybrid composite before and after of fluoride therapy did not change, but a little increase in microhybrid composite microleakage was observed (Table 4) which is in line with Botta, ozdemir, and Dinonoscopy studies [31-33].

APF gel is the most effective agent in releasing fluoride compared to NaF 1% and SnF<sub>2</sub> 4% gel; However, APF gel contains hydrofluoric acid and phosphoric acid [30]. Phosphoric acid has the ability to etch glass particles. Hydrofluoric acid is more destructive than phosphoric acid because it can etch glass particles at lower temperatures [35]. APF has a pH of approximately 3.5 that causes damage and filler particles separation, destruction of resin matrix, etching surface area of the material and glass particles, allowed the ingress of bacteria leading to discoloration, and filler-matrix interface destruction and erosion. This results in a loss of marginal integrity destruction of materials and ultimately reduces the longevity of the restorative material [28, 33, 36].

The chemical factor relevant for the erosion capacity not only depends on on the concentration of fluoride solution, but the time and frequency of fluoride immersion. A fast four minutes using of fluoride recommended for maximum use of the therapeutic properties of fluoride could have a devastating negative effect on on dental materials.<sup>29</sup> Filler components features such as composition, particle size, shape, type and method

of mixing the resin and filler composite resin behavior plays an important role in seeking to get topical fluoride treatment [12, 32, 37].

Different patterns have been seen on the loss of fillers. Kula *et al.*, 1996 in a study by using electron microscope observed the non-uniform areas of loss by APF which suggests that the components resin filler has a susceptible and non-susceptible heterogeneous phase [9]. According to studies, while composites include silica and zirconia still represent the lowest share of all APF priorities, composites containing barium aluminosilicate glass, silicon dioxide and barium glass fillers are the most prone to APF priorities. These composites also show more erosion. Reduced abrasion resistance causes hydrolytic degradation leading to leakage of fillers, silane debonded in the mid-level filler – matrix, and the formation of cracks which all are caused by increased osmotic pressure [9, 12, 15, 28, 33, 38-40].

It is also believed that the size of the filler particles affect APF impact on composite materials. Some studies confirmed that composites containing larger fillers are less affected than composites containing low size fillers.

According to different study conducted by Kargul *et al.* Seono *et al.*, and khalili *et al.*'s studies microfilled composite with smaller fillers has less affected [13, 30, 36]. Therefore, Seono recommended the application of microfilled composite in fluoride therapy [13]. Seono *et al.* and Botta *et al.*'s studies suggested that APF can attack the inorganic particles, and when these are very small, as in the nanofiller composite resins the size of the defects created by APF are practically imperceptible [13, 33].

Similarly, Ozdemir *et al.*, and Botta *et al.*'s studies confirmed that APF application did not cause surface alteration [32, 33]. Accordingly, in the present study the low and insignificant increase in the nano-hybrid composite leakage (Table 3) was due to the small size of particles which is consistent with other studies [13, 32, 33]. However, a slight increase in the nano-hybrid composite and increase in the microhybrid composite microleakage can be due to the effect of APF on the resin matrix [31, 36, 40, 41].

Mc cobe *et al.* study suggested that fluoride has no damaging effect on the matrix (42), but some other researchers have reported that the composites surface that have been immersed in

the APF has been clearly changed, and inflation in the matrix with a cracks and a view halo around the surrounding glass components glass has been observed. Resin shrinkage, air trapped (voids), and bubbles has been reported in the resin matrix due to high concentration of ions H and F in the APF. This view of swelling (inflammation) is the primary form of matrix degradation which caused through HEMA reaction [3]. On the other hand, Kula *et al.* and Yap *et al.*'s study confirmed the destructive effects of fluoride on the filler-matrix interface [29, 38]. Fluoride can influence the water layer contained in the fillers where hydrogen bonding silane should be established to connect to the matrix. It also hydrolyses the silicon ester groups and causes siloxane structure deformation which formed from density silanol groups that stabilize the interface between filler and matrix. All these mechanisms may weaken the particle-matrix interface that leads to reduction of filler size and decreasing surface roughness [15]. Prakki *et al.*'s study suggested that the low effect of pH on composites is due to hydrolysis of the ester groups in the resin matrix that makes up the free carboxylic acid groups and can decrease the pH in the polymerized matrix. Another reason for the higher microleakage of microhybrid than nanohybrid is due to the higher resin matrix composites in the microhybrid composite.

### CONCLUSIONS

1. Despite the insignificant difference observed between the nanohybrid and microhybrid composite microleakage, nanohybrid composite demonstrated significantly less microleakage that indicate the superior physical properties of the nanohybrid composite.
2. Given that the nanohybrid composites are affected by APF, it is recommended to use non-acidulated phosphate fluoride such as neutral sodium fluoride (NaF) or varnish fluoride when there is composite restoration on the teeth.
3. Composite that has smaller filler and resin particles like nanofill and nanohybrid are the best choice among acidulated phosphate fluoride agents.

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