# The effect of acidulated phosphate fluoride application on dental enamel surfaces hardness

Edhie Arief P and Sri Kunarti Department of Conservative Dentistry Faculty of Dentistry Airlangga University Surabaya - Indonesia

#### ABSTRACT

Enamel demineralization by acid is the first step of caries process. It has recently been shown that acidulated phosphate fluoride (APF) can maintain the hardness of enamel surface. The aim of this study was examine the effect of APF application in the hardnest of enamel surface. Fifty extracted teeth were cut at their crown, 40 teeth were taken randomly then divided into 4 groups, group 1 as the control, group 2 was treated with APF for 1 minute, group 3 for 4 minutes and group 4 for 7 minutes, then all the samples were washed with demineralized water. To see the effect of APF, all of the samples were soaked in lactic acid demineralization solution with pH 4,5 for 72 hours., the hardness of the surfaces of those samples before and after the treatment was measured by Micro Vickers Hardness Tester. The data were analyzed using One-Way ANOVA and LSD tests. In conclusion, 1.23% APF gel can reduce higher enamel demineralization.

Key words: acidulated phosphate fluoride, surface hardness, enamel demineralization

*Correspondence*: Edhie Arief P, c/o: Bagian Ilmu Konservasi Gigi, Fakultas Kedokteran Gigi Universitas Airlangga. Jln. Mayjend. Prof. Dr. Moestopo no. 47 Surabaya 60132, Indonesia.

#### INTRODUCTION

Dental caries is a multifactorial disease which is caused by some factors such as microorganism substrate, dental surface and time.<sup>1</sup> Organic acid resulted from carbohydrate glycolisis by microorganism decreases pH plaque and pH liquid around the teeth. Along the next decades, many researches showing that there are acidogenic bacteria which can produce acid at pH lower than 12. Bacteria which mostly have the role in dental caries is *Streptococcus*, especially *Streptococcus mutans*.<sup>2,3</sup>

*Streptococcus mutans* metabolize carbohydrate to lactic acid. Extra cellular bacterial enzyme such as glucocyltransferase and fructocyltransferase break the carbohydrat. Than through glycolisis process, glucose is broke by bacteria and the results are two private molecules. This molecule is broke to lactit acid molecule. This lactit acid is strong acid which causes dental structure demineralization.<sup>4</sup> Many methods were done to prevent or to reduce caries risks, one of them is by making dental structure less dissolved towards acid through fluoride use.<sup>5</sup> Fluoride is effective anticaries agent when released into several forms or concentrations. Fluoride necessity can be well fulfill both by systemically through water, food, vegetables, or supplements and topically through toothpaste, mouth-wash and topical application.<sup>6</sup>

Topical fluoride is the prime protection agent after dental erosion. Topical fluoride application is recommended for the children after permanent dental eruption and the patients with high caries risk.<sup>7</sup> Topical fluoride application which is done to the children with dental permanent in non-fluoride area for six months reduce caries risks at permanent teeth for 26%.<sup>8</sup>

There are several agents used as topical application, one of them is acidulated phosphate fluoride (APF). APF has been used at the countries with high caries activities, low consciousness level in prevention, and the system of dental health treatment that is not well organized yet.<sup>9</sup> APF is sodium fluoride derivate in the form of solution, gel or powder which characterizes acid at pH 3 to 4 and characterizes buffer if it interacts with phosphate. APF has been tested in its use, both in the form of solution or gel, but gel is the form which is most frequently used. This agent has better ability in bonding to enamel calcium. It also non irritating and non staining that can be tolerated by adding the taste as well as easily accepted by patients. The affectivity of APF can be various, depends on the methods and frequency of its application.<sup>10</sup> This agent is a mineral which can strengthen the enamel surfaces and prevent root caries as well as inhibit caries risks as the effect of saliva product which is less because of radiation therapy or chemotherapy.

APF usually contains 2% sodium fluoride, 0.34% hydrogen fluoride and 0.98% phosphate acid. APF is in the form of solution, gel or foam.<sup>11</sup> APF can be used for children 6 years up and adults with high caries risks but has contraindication to the patients who have hypersensitive reaction, the patients who have dental implant, patients with composite restoration, porcelain, compomer, and ionomer glass.<sup>12</sup>

The objectives of this study is to know the ability of APF in maintaining dental enamel surfaces hardness and

give the information about the use APF in reducing enamel demineralization so that it can be used as the model for dentists in choosing topical fluoride agents.

#### MATERIALS AND METHODS

The materials that were used: 1.23% acidulated phosphate fluoride, 90% extra pure lactic acid (Merck), demineralization solution which concise of 0,1 M lactic acid and 0.1% thymol at pH 4.5<sup>13</sup> The instruments that were used: pH meter, micro Vickers hardness (Osaka, Japan).

All samples from extracted teeth, which were intact in labial side. Ten samples of group 1 ( $X_1$ ) were treated with APF agent by using cotton swab for 1 minute, 10 samples of group 2 ( $X_2$ ) for 4 minutes and group 3 for 7 minutes ( $X_3$ ). The teeth were cleaned using aquadest and dried, meanwhile 10 samples of controlling group ( $X_4$ ) are not put in to APF agent.

Forty samples were put in reaction tubes containing 5 ml of demineralization solution and closed tightly to avoid dehydration. Then the tube was put into incubator with temperature 37 °C for 72 hours. After 3 days, the teeth are taken from the solution, then washed with water and measured back for their enamel surfaces (last hardness). The measurement of enamel surfaces hardness uses Micro Surface Vickers Hardness Tester. Indentation result can be seen at Projector screen in the form of shadow shaping rhomb, the diagonal length is measured with micrometer. The measurement result, it could be known the hardness of enamel surfaces using the formula:

HVN = 
$$\frac{1.854 \times p}{d^2}$$

Description:

HVN = Sample hardness (kg/mm<sup>2</sup>) d = Measurement result (mm)

p = Weight is given (kg)

The measurement of enamel surfaces hardness was done for 3 times, at buccal side which was situated around the cut off vertical and horizontal line. Data collected was gotten from the measurement of surfaces hardness before and after the treatment so was the control sample.

The data were tested statically by using ANOVA test with significance standard/ level p = 0.05.

## RESULTS

The dental enamel surface hardness reductions after the application of APF were shown in table 1.

From table 1, it can be seen that there are the reduction of enamel hardness at the groups with APF application for 1 minute, 4 minute, and 7 minute, as well as group without APF application. By using Kolmogorov–Smirnov test, it shown that all of the research groups have bigger value than 0.05 or at 0.152 (p > 0.05) that means all of the research groups data are normal, and homogeneity test with Levene's test shows the value 0,081 (> 0.05), then continued with One-way ANOVA test to know the significance among the research groups.

The highest average of enamel hardness reduction is found at 7 minutes APF application, then using One-Way ANOVA test it can be seen that there are significance differences for comparison among the four groups (p < 0.05).

At table 2 there are significance differences for comparison among the groups, group 1, 4 and 7 minutes APF application as well as without APF application (p < 0.05). There are no significance differences between groups 4 and 7 minutes APF application.

#### DISCUSSION

The measurement of enamel surface hardness in this research is used as the evaluation towards chemical

Table 1.	Mean and standard deviation denta	l enamel surfaces	hardness reduction aff	ter APF application for 1	, 4, 7 minutes
	and without APF application in kg	g/mm <sup>2</sup>			

Groups	Ν	Mean	Standard Deviation
1 Minute APF	10	72.0875	8.31821
4 Minute APF	10	19.4375	3.66136
7 Minute APF	10	10.2461	7.48215
Without APF	10	202.4375	9.01775

Table 2. Test of the decreased differences at enamel surfaces hardness using One–Way ANOVA and LSD

	1 minutes APF	4 minutes APF	7 minutes APF	Without APF
1 minute APF	-	0.000*	0.000*	0.000*
4 minute APF		-		0.000*
7 minute APF				0.000*-

\* = significance difference (p < 0.05)

bondings in enamel treatment procedures. The surfaces hardness measurement is used to evaluate demineralization, because it is found the positive correlation between enamel surfaces hardness with the lost of minerals from the teeth.<sup>13,14</sup> APF gel has been used widely and its ability as anticariogenic agent has been evaluated through laboratory, clinically, and epidemiology studies.

This in vitro study shows that APF is able to decrease the reduction of enamel surfaces hardness. The control group (without APF application) decrease enamel hardness higher than the other three groups. In control group, demineralization solution releases its hydrogen ion that reacts with apatite crystal teeth so that the apatite crystal becomes unstable. Furthermore, water and soluble phosphate are formed which finally destroy enamel membrane. The reaction can be written as follows:  $Ca_{10} + (PO_4)_6(OH)_2$ + 8 H <sup>+</sup>  $\rightarrow$  10  $Ca_+^2$  + 6 HPO<sub>4</sub><sup>2-</sup> + 2 H<sub>2</sub>O. Because of the destruction of enamel membrane, demineralization solution will penetrate deeper and dissolve apatite crystal. If acid atmosphere goes for long, demineralization will keep processing.

In clinical use, application fluoride gel for 1 minute is more general in use than 4 or 7 minutes application. The biggest absorbance happens in first minute of application but the maximum result is by treatment for 4 minutes then decrease at 7 minutes. This is probably because of dental satiation in absorbing APF.

APF is formulated in order the concentration of fluoride ion is sufficient to protect the teeth against the agents causing dental minerals lost. APF is used to increase fluoride absorbance in dental surfaces.<sup>15</sup> Many researches show that fluoride is more effective to protect enamel when the fluoride is in acid atmosphere than joining into enamel spaces.<sup>16</sup>

APF reacts with hydroxyapatite enamel forming fluoropatite which is more resistant towards acid if compared with the change of carbonate hydroxyapatite calcium. In short, APF helps to reduce the formation of cavity and increase of fluoride concentration in saliva quickly. Beside that, APF leaves calcium fluoride layer which characterizes temporary on enamel surface. The research shows that flour with high concentration that is found in APF can protect CaF2 on enamel surfaces so fluor reservoir along the remineralization process is formed when pH under goes the decrease. CaF2 is the main product reaction that is formed along the application of topical fluoride on dental hard tissues. CaF<sub>2</sub> formation is important because CaF2 roles in demineralization and remineralization phase on caries process. CaF2 acts as the control of pH from ions F which is released along against the caries on enamel or in dental plaque. Fluoride on CaF2 is produced when pH in oral cavity goes down as the consequence of acid production. Beside that the formation of  $CaF_2$  on enamel surfaces can be gone quickly but this causes the formation of fluor apatite by fluor ion in enamel minerals. The formation of product is when fluor reacts with enamel depends on fluor concentration duration, pH, frequency and

treatment methods. When fluoride concentration on enamel is above 100 ppm, CaF2 is formed with thaw mechanism below:  $Ca_{10}(PO_4)_6 (OH)_2 + 2 OF^- + 8H^+ \rightarrow 10 CaF_2 + 6 HPO_4^{2-} + 2 H_2O$ .

The above reaction is the reaction which is formed when the teeth is being applicated with topical fluoride. The higher fluoride concentration will produce bigger  $CaF_2$  formation. Furthermore, low pH in solution has strong influence in  $CaF_2$  formation. It is because of the low solution pH can increase enamel delve a little bit of enamel and settles  $CaF_2$  in big number on dental surfaces.<sup>17</sup>

Another research which is done using block enamel  $(5 \times 5 \times 2 \text{ mm})$  from incisive teeth which are soaked in 0.5 ml 1 M KOH in room temperature for 24 hours shows that is found significance differences at the number of CaF<sub>2</sub> which is formed on enamel among grasps of APF gel application and controlling group. At enamel groups with APF application, the number of the formed CaF<sub>2</sub> is  $31.72 \pm 22.18 \,\mu\text{g F/cm}^2$  meanwhile at controlling group is  $0.17 \pm 0.04 \,\mu\text{g F/cm}^2$ . These show that minerals in dental groups of APF application.

About their relationship in clinical practice, demineralization of enamel as the result of acid which contacts with the teeth does not happen continuously and undoes for along time, it usually happens in 1 to 2 minutes. Bitter taste usually stimulates the flow of saliva which is very effective as buffer system, but in permanent hyposalivation condition, neutralization from acid reflux will require longer time. Effectiveness of APF application in  $CaF_2$  formation which is useful in reducing enamel demineralization depends on the pH and duration of agent application so that can maintain the hardness of enamel surfaces. It concluded that APF gel can reduce high enamel demineralization as the consequence of acid, so it can maintain the hardness of enamel surface.

### REFERENCES

- Kidd EAM, Bechal SJ. Dasar-dasar karies penyakit dan penanggulangannya. Jakarta: EGC; 1992. p. 58–93.
- Roth GI, Calmes R. Oral biology. St Louis, Baltimore, Boston: The Mosby Company; 1981. p. 354–7.
- Bisla S. Dental caries–a student project. 2000. Available at: http: //www.sciweb.hfcc.com. Accessed January, 2007.
- Burt BA, Eklund SA. Dentistry dental practice and the community. 5<sup>th</sup> ed. Philadelphia: WB Saunders Company; 1999. p. 319–20.
- Samanarayake LP. Essential microbiology for dentistry. 2<sup>nd</sup> ed. London: Churchill Livingstone; 2002. p. 217–23.
- Samuel SMW, Rubinstein C. Microhardness of enamel restored with floride and non–flouride raleasing dental materials. Braz Dent J 2001; 12(1):35 8.
- Hayacibara MF, Paes Leme AF, Lima YBO, Goncalves NCLAV, Queiroz CS, Gomes MJ, Kozlowski FC. Alkali-soluble flouride deposition on enamel after professional application of topical fluoride in vitro. J Appl Oral Sci 2004; 12(1):18–21.
- Robinson DS, Bird DL. Essential of dental assisting. 3<sup>rd</sup> ed. WB: Saunders; 2001. p. 274–8.
- Hiratsuka K. Regulation of sucrose–6–phosphate hydrolase activity in streptococcus mutans: characterization of the scr-Rgene. 1998. Available at: http://rudyct.tripot.co.id. Accessed January, 2007.

- Delbem ACB, Brighenti FL, Mello Vieira AE, Cury JA. In vitro comparison of the cariostatic effect between topical application of fluoride gels and fluoride toothpaste. J Appl Oral Sci 2004; 12(2):54–58.
- Craig RG. Dental Materials Properties and Manipulation. 7<sup>th</sup> ed. St. Louis: Mosby, Inc; 2000. p. 34.
- 12. Barnes CM, Hlava Gl. Review of CDC recommendations for fluoride use. The Preventive Angle 2001; 2(2):22–4.
- Groenveld A, Arends J. Influence of pH and demineralization time on mineral content, thickness of surface layer and depth of artificial caries lesions. Caries Res. 9 St Louis: Mosby Inc; 1975. p. 36–44.
- Featherstone JD. The caries balance: contributing factors and early detection. J California Dent Assoc 2003 February; 31(2):17–20.
- Delbem ACB, Brighenti FL, Mello Vieira AE, Cury JA. In vitro comparison of the cariostatic effect between topical application of fluoride gels and fluoride toothpaste. J Appl Oral Sci 2004; 12:20.
- Jones L, Lekkas D, Hunt D, Mcintyre J, Rafir W. Studies on dental erosion: an in vivo-in vitro model of endogenous dental erosion–its application to testing protection by fluoride gel application. Australian Den J 2002; 47(4):304–8.
- Fejerskov, Kidd E. Dentistry dental practice and the community. 5<sup>th</sup> ed. Philadelphia: WB Saunders Company; 2003. p. 252–60.