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ORIGINAL RESEARCH

In vitro Evaluation of Topical Fluoride pH and their Effect on Surface Hardness of Composite Resin-based Restorative Materials

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ABSTRACT

Aim: The aim of the study was to correlate the pH and fluoride ion uptake with surface hardness of composite resin based restorative materials after topical fluoride application.

Methodology: Forty disks of each of test materials Composite (Filtek Z350XT, 3M ESPE, St Paul, MN, USA), Resin modified glass ionomer (Vitremer) and Compomer (Dyract AP) were made and ten disks of each material were placed in different test solutions – 1.23% APF gel, Sodium fluoride mouth rinse, 0.9% neutral fluoride and distilled water (Control group). After 36 hours of immersion, specimens were subjected to microhardness testing machine for evaluation of surface hardness.

Results: The greater hardness deterioration for all materials resulted with 1.23% APF gel when compared to the control group. Composite (Filtek Z350XT, 3M ESPE, St Paul, MN, USA) showed 17.13 VHN (control group 59.11 VHN). Vitremer showed 9.71 VHN (control group 37.71 VHN). Compomer showed 19.22 VHN (control group 36.78 VHN).

Conclusion: 1.23% ApF gel significantly decreased hardness of composite, Vitremer and Compomer. Hardness deterioration associated with sodium fluoride mouth rinse and 0.9% neutral fluoride was less compared to 1.23% ApF gel.

Keywords: Composite, Vitremer, Compomer, Fluoride release, Microhardness.

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INTRODUCTION

'Fluorides— Prepared to be part of the cure or part of the problem'.

Dental caries is a major dental disease affecting a large proportion of the inhabitants of the world which impairs the quality of life for many people causing pain and discomfort.

The cariostatic efficacy of fluorides has been convincingly demonstrated and the recent decline in caries prevalence is primarily attributed to the increased use of various fluoride agents.[1]

Topical fluorides act in several different ways to prevent caries by inhibiting demineralization and promote remineralization, thus encouraging repair and arrest of carious lesions. Depending on its concentration and pH, fluoride can also exert a bactericidal and anti enzymatic effect.

Composite restoratives and adhesive technique have become the foundation of modern dentistry, which is an important advancement in the field of restorative dentistry[2] but, the composition and surface integrity of composites and other glass containing restoratives can be significantly changed when exposed to strong acids this is clinically significant as topical acidulated phosphate fluoride (APF) gels, recommended as a preventive strategy in dentistry, contain strong acids.[3] Also low pH increases sorption, solubility and surface degradation of resin composite.[4] The type of restorative material also influences the degradation of restorative materials.[5-10]

Therefore this study investigated the effect of professionally applied topical fluoride on the surface hardness of three different composite based restorations, i.e. Composite, Vitremer and Compomer.

METHODOLOGY

The study involved 2 parameters:

Part I: To evaluate pH of topical fluoride and fluoride ion concentration.

Part II: To evaluate surface hardness of composite resin based restorative materials after topical fluoride application.

To correlate, the effect of pH and fluoride ion concentration on surface hardness of composite resin based restorative materials.

Part I: To evaluate the pH of topical fluoride and fluoride ion concentration. The following topical fluorides were used in the study;

- 1.2 3% acidulated phosphate fluoride gel (60 seconds taste gel–Pascal)
- Sodium fluoride mouth rinse (S-Flo-Group Pharmaceuticals Ltd)
- 0.9% neutral fluoride
- Distilled water (Control group)
- pH of topical fluoride and fluoride ion concentration was evaluated by using Jenway 3330 research pH meter and ion analyzer with orion ion selective electrode prior to the placement of test material.

Part II: To evaluate surface hardness of composite resin based restoration materials after topical fluoride application.

The following test materials used in the study which were grouped as follows;

Group I: Composite (Filtek Z350XT, 3M ESPE, St Paul, MN, USA)

Group II: A resin modified glass ionomer (Vitremer – 3M)

Group III: A compomer (Dyract A/P dentsply).

Forty disks of each of the testing material were made having a diameter of 8 mm and thickness of 1.65 mm using Teflon mould.

Ten disks of each restorative material were placed in all four different test solutions. After 36 hours of immersion, the specimens were washed, dried and subjected to microhardness testing machine for surface hardness of composite based restorative materials using Vickers microhardness tester (Shimad Zu Japan). Again pH and fluoride ion concentration of topical fluoride was evaluated to quantify the fluoride ion uptake by the test specimens.

Statistical Analysis

Descriptive (Mean ± SD) and comparative statistics were used to illustrate and compare the results. One Way ANOVA was performed for multiple group comparisons followed by Newman-Keul's range test to determine whether significant differences were present between two materials or two applications. The difference was considered statistically significant when p-value was 0.05 or less.

Table 1: pH and fluoride ion concentration of topical fluoride before specimen immersion

Topical fluoride	*pH*	*Fluoride Ion concentration (PPM)*
1.23% APF gel	3.9	12,380.00
Sodium fluoride mouth rinse	6.5	907.00
0.9% neutral fluoride	7.01	910.00

RESULTS

The present in vitro study was aimed to evaluate pH and fluoride ion concentration before and after immersion of composite based restorative material specimens and to evaluate surface hardness of composite resin based restorative materials after topical fluoride application.

The study involved two parameters:

1. To evaluate pH of topical fluoride and fluoride ion concentration.
2. To evaluate surface hardness of composite resin based restorative materials after topical fluoride application.

Table 1 demonstrated the pH and fluoride ion concentration of APF gel, sodium fluoride mouth rinse and 0.9% neutral fluoride before immersion of composite based restorative material specimens.

The pH of all test solutions remained the same of pH 3.9 with APF gel, pH 6.5 and 7 with sodium fluoride mouth rinse and 0.9% neutral fluoride respectively.

The fluoride ion concentration in test solution treated with composite specimen remained the same as composite specimen neither released nor were recharged with fluoride ions. The fluoride ion concentration in test solutions treated with vitremer and compomer specimen showed increase in fluoride ion concentration indicating the increase fluoride release from vitremer and compomer.

Fluoride release in vitremer was more compared to compomer because glass ionomer content in vitremer is 80% compared to compomer 20%.[11] The greatest hardness deterioration for all materials resulted after treatment with 1.23% APF gel when compared to sodium fluoride mouth rinse and 0.9% neutral fluoride.

Table 2 showed mean surface hardness (VHN) of composite based restorative materials after various fluoride applications and Graphical representation is shown in Graph 1.

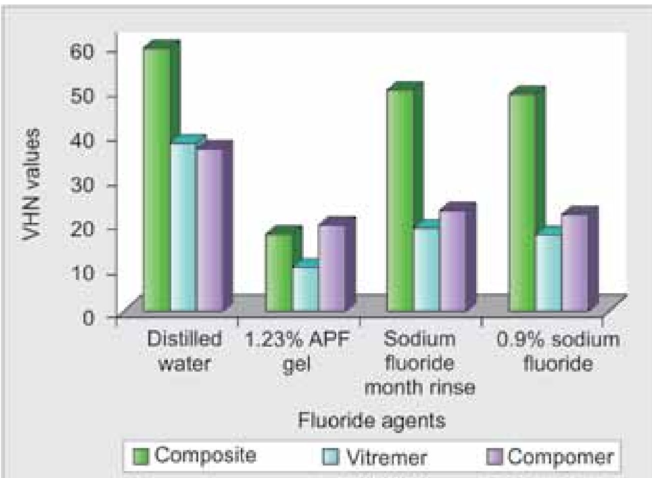
DISCUSSION

Scientific knowledge concerning caries has led to the establishment of preventive and therapeutic strategies in accordance with individual patient characteristics in regard to the risk and activity of caries. Measures to increase protective factors such as fluoride therapy may be adopted as part of these approaches, inhibiting tooth demineralization and enhancing remineralizing potential.[12]

In the 1960's, a new topical fluoride preparation APF was introduced by Brudevold and his co-workers at Forsyth Dental Centre. APF preparation contains sodium fluoride as the active ingredient. The general formula has 1.23% of

Table 2: Mean surface hardness (VHN) of the composite bases restorative materials after various fluoride applications

Materials		*Composite I*	*Vitremer II*	*Compomer III*	*ANOVA 'F" p-value*	*Difference between materials*		
						I – II	*I – III*	*II – III*
1.	Distilled water	59.11 (1.02)	37.70 (1.89)	36.78 (1.06)	831.9 < 0.001	p < 0.01	p < 0.01 I > II, III	NS
2.	APF gel	17.13 (2.0)	9.71 (1.32)	19.22 (1.61)	89.6 < 0.001	p < 0.01	p < 0.05 III > I > II	p < 0.01
3.	NaF rinse	49.86 (1.39)	18.66 (1.67)	22.54 (1.95)	F = 1329.3 p < 0.001 Ve = 2.18	p < 0.01	p < 0.01 I > III > II	p < 0.01
4.	0.9% Neutral fluoride	48.86 (1.61)	17.14 (2.26)	21.86 (1.29)	F – 925.3 p < 0.001 Ve = 3.17	p < 0.01	p < 0.01 I > III > II	p < 0.01



Graph 1: Surface hardness (VHN) of the composite based restorative materials after treatment with different fluoride agents

fluoride as sodium fluoride buffered to a pH of 3 to 4 in a 0.1 M phosphoric acid, as greater fluoride uptake by enamel occurs under acidic conditions.[1]

Recent development and future trends focus mainly on composite based restorations. Hence, the aim of this study was to correlate the pH of topical fluoride and fluoride ion uptake with surface hardness of composite resin based restorative materials such as composite (Filtek Z350XT, 3M ESPE, St Paul, MN, USA), a resin modified glass ionomer (Vitremer) and a compomer (Dyract AP) after topical fluoride application.

Hardness is considered as the test parameter, as it is an important property for the restorative materials to have long-term durability in the oral cavity.[13] Hardness may be defined as the resistance of a material to indentation or penetration.[14] Strength, proportional limit and ductility are related to hardness. Hardness has also been used to predict the wear resistance of a material and its ability to abrade or be abraded by opposing dental structures and materials.[15] So a decrease in the hardness of a material may result in premature failure of a restoration requiring its replacement.

Group 1: Composite (Filtek Z350XT, 3M ESPE, St Paul, MN, USA).

Composite showed deterioration in microhardness in all test solution groups. The greater hardness deterioration resulted from treatment with 1.23% APF gel. The decrease in the hardness may result from a difference in the pH and fluoride ion concentration (Kula et al 1986,[21] Kula et al 1992). A 1.23% APF gel with greater fluoride and hydrogen ion concentration are expected to be more reactive than sodium fluoride mouth rinse and 0.9% neutral fluoride.

APF gel contains sodium fluoride and phosphoric acid. The typical dissociation reaction in the solution is:

$$NaF \rightleftharpoons Na^+ + F^-$$

$$H_3PO_4 \rightleftharpoons 2H^+ + HOP_4$$

$$H^+ + F^- \rightleftharpoons HF$$

H^+ and F ions are therefore present in the solution and forms hydrofluoric acid (HF) a known glass etchant which dissolves glass particles.[16]

Three major interaction pathways between composite and fluorides may be identified.

1. Interaction with organic matrix (Bis-GMA)
2. Interaction with filler matrix coupling agent
3. Interaction with reinforcing fillers.

The organic matrixes of the composite evaluated are all organic esters of methyl methacrylate derivatives. Organic esters undergo hydrolytic cleavage of the ester group in low pH.

$$R (CO) – OR + H_3O \longrightarrow R (CO) OH + R'OH$$

Papagiannoulis observed that as APF gel which is acidic (pH 3–4) so the amount of water bound to the organic matrix is increased resulting in the decreased hardness.[17] This reaction is acid—catalyzed and is pH dependent.

The decreased hardness after treatment with APF gel could also be attributed to the presence of HF (Cehrcli, Yazici and Garcia-Godoy 2000).[18] Hydrofluoric acid, a well known glass etchant, dissolves composite filler particles and fluorosilicate glass particles that contribute to decrease in surface hardness. Increased filler dissolution may result in increased exposure of the organic matrix and consequently an accelerated hydrolytic effect. Fluoride ion has been implicated in depolymerization reaction of the matrix filler interface.[3]

Three possible mechanisms may explain the interaction pathway of fluoride.

1. Rearrangement of the water monolayer adsorbed on filler where silanols form hydrogen bonds.
2. Hydrolysis of the organosilicon ester group.
3. Disorganization of the siloxane network formed from the condensation of intramolecular silanol group which stabilized the interface.

All these mechanisms may weaken the particle matrix interface leading to the decrease in surface hardness of composites.[19]

Group II: A resin modified glass ionomer (Vitremer).

Vitremer showed deterioration in microhardness in all test solutions. The greatest hardness deterioration is also resulted with the treatment of 1.23% of APF gel.

The effect of 1.23% APF gel on resin modified glass ionomer cement (Vitremer) manifested itself in a new pattern leading to decreased in surface hardness.

Matrix dissolution: This was detected peripheral to the glass particles, leading to a 'halo' appearance surrounding the glass particles, the glass particles are sheathed by a silicons hydrogel layer (Smith, 1994; Mitra 1994). 'Halo' effect could be due to dissolution of the siliceous hydrogel layer as in the final set structure of conventional glass ionomer.

Since true resin-modified glass ionomers are reported to undergo an acid-base reaction as well as photopolymerization (Mika 1994), a siliceous hydrogel layer or reaction zone would be expected to a greater (or) a lesser extent sheathing the glass particles. Because of the improved resistance of the resin modified glass ionomer resinous matrix to dissolution and solubility, the siliceous hydrogel layer was the first to erode, leading to the 'halo' like appearance. It can be considered the weakest link in the system.

Generalized matrix changes that resulted in a swollen (or) expanded matrix appearance in resin modified glass ionomer with variable degree of severity.

Nickolson, Arstice and McLean (1992) reported that light cured glass ionomer prepared with HEMA copolymers behave like hydrogels and absorb water. Resin modified glass ionomer cements contained polyacrylic acid and HEMA (2-hdyroxyethyl methacrylate), a hydrophilic monomer, which acts as a solvent and a polymerizable monomer.

Nicholson and Arstice (1994) reported that because of the contrasting nature of these liquid components, there is a natural tendency for resin modified glass ionomers to undergo phase separation while still liquid. This tendency for phase separation increases as the setting reaction proceeds, i.e. as HEMA undergoes polymerization it ceases to be water soluble, which causes it to separate from the aqueous part of the formulation. In addition, as the acid-base reaction proceeds and the acid becomes more neutralized, more hydrophobic organic species become less soluble in the aqueous phase therefore the set product is expected to contain domains of different phases in its microstructure.

Nicholson and Arstice (1994)[17] also reported that almost certainly the sites of these hydrophilic domains are the centers to which water migrates as it is absorbed therefore resin modified glass ionomer showed a tendency to absorb water and swell which was particularly evident with APF gel but was observed lesser extent with neutral gel.

Group III: A Compomer (Dyract AP)

Compomer showed deterioration in microhardness in all test solutions. The greatest hardness deterioration is also resulted with the treatment of 1.23% of APF gel.

As APF gel contains hydrofluoric acid and phosphoric acid (El-Badrawy, McComb and Wood; (1993)[19] which has the ability to etch glass particle, (Council on Dental Materials, Instruments and Equipments, 1988) can lead to the chemical erosion by acid etching. Also surface degradation of resin component by APF gel is partly related to the filler particles present (Kula 1986).[21] Strontium fluorosilicate glasses present in compomer are more susceptible to acid erosion leading to filler dissolution and decrease in surface hardness of compomer.[20]

Dieb et al in 2007[6] who reported that mouth rinses with low pH are detrimental to the hardness of resin composites. Basically the low pH of mouth rinses may have acted in the polymeric matrix of the nanofilled resin composite used in the study, through catalysis of ester groups from dimethacrylate monomers present in the composition (Bis GMA, Bis EMA, UDMA and TEG DMA).[22] The hydrolysis of these ester groups may have formed alcohol and carboxylic acid molecules that may have accelerated the degradation of the resin composite.[23]

Hence, the type of restorative material also influences the degradation of restorative material.

CONCLUSION

The following conclusions were drawn from the study:

- All topical fluoride application showed significant deteriorative effect on resin based restorative material.
- The surface deterioration and decrease in microhardness was dependent on the pH of topical fluoride.
- 1.23% APF gel significantly decreased hardness of the Composite, Vitremer and Compomer.

REFERENCES

1. Sober Peter. Fluorides in preventive dentistry. Ist ed. Mr Sudhir Kumar Arya. For Arya (Medi) Publishing House, 1999 April.
2. Harry F, Albers. Tooth colored restoratives principles and techniques. 9th ed. Bc Decker Inc 2002.
3. Yap AUJ, Mok BYY. Effects of professionally applied topical fluorides on surface hardness of composite-based restoratives. Operative Dentistry 2002;2(6):576-581.
4. Almeida GS, Poskus LT, Guimaraes JG, Da silva EM. Effect of mouth rinses on salivary sorption, solubility and surface degradation of a nanofilled and a hybrid resin composite. Oper Dent 2010;35(1):105-111.
5. Indrani DJ, Triaminingsih S, Nurvanita LN, Yulanti AN. Effect of ethanol in mouth rinses on the surface hardness of a dental resin composite material. Padjadjaran J Dent 2009;21(3):8-13.
6. Diab M, Zaazou MH, Mubarak EH, Olaa MI Fahmy. Effect of five commercial mouth rinses on the microhardness and color stability of two resin composite restorative materials. Aust J Basic Appl Sci 2007;1(4):667-674.
7. Miranda DA, Bertoldo CE, Baggio Aguiar FH, Leite Lima DA, Lovandino JR. Effects of mouth washes on knoop hardness and surface roughness of dental composites after different immersion times. Braz Oral Res 2011;25(2):168-173.
8. Ateyah NZ. The effect of different mouth rinses on micro hardness of tooth colored restorative materials. J Pak Dent Assoc 2005;14(3):150-153.
9. Gurgan S, Onern A, Köprülü H. In vitro effects of alcohol. Containing and alcohol free mouthrinses on microhardness of some restorative materials. J Oral Rehab 1997;24(3):244-226.
10. Yap AU, Tan BW, Tay LC, Chang KM, Loy TK, Mok BY. Effect of mouth rinses on microhardness and wear of composite and compomer restoratives. Oper Dent 2003;28(6):740-746.
11. Soeno K, Matsumura H, Atsuta M, Kawasaki K. Influence of acidulated phosphate fluoride agent and effectiveness of subsequent polishing on composite material surfaces. Operative Dent 2002;27(3):305-310.
12. Burgess J, Norling B, Summitt J. Resin ionomer restorative materials. The new generation. J Esthet Dent 1994;6(5):207-215.
13. Okada K, Tosaki S, Hirota K, Hume WR. Surface hardness change of restorative filling materials in saliva. Dent Mater 2001;17(1):34-39.
14. O'Brien WJ. Physical properties of dental materials and their selection. Illinois: Quintessence Publishing Company 1977.p.18.
15. Anusavice KL. Mechanical properties of dental materials in phillip's science of dental materials. 10th ed. Philadelphia: WB Saunder's Co. 1996.p.69.
16. Turssi CP, de Magalhaes CS, Serra MC. Effect of fluoride gels on micromorphology of resin-modified glass-ionomer cements and polyacid-modified resin composites. Quintessence Int 2001; 32(7):571-577.
17. Papagiannoulis L, Tzoutzas J, Eliades G. Effect of topical fluoride agents on the morphologic characteristics and composition of resin composite restorative materials. J Prosthet Dent 1997;77(4):405-413.
18. Cehreli ZC, Yazici R, Garcia-Godoy F. Effect of 1.23% APF gel on fluoride releasing restorative materials. J Dent Child 2000 Sep-Oct;67(5):330-337.
19. El-Badrawy WA, McComb D. Effect of home use fluoride gels on resin modified glass ionomer cements. Oper Dent 1998;23(3):2-9.
20. Yip KHK, Peng D, Smales RJ. Effects of APF gel on the physical structure of compomers and glass ionomer cements. Operative Dent 2001;26(3):231-238.
21. Kula K, Nelson S, Kula T, Thompson V. In vitro effect of acidulated phosphate fluoride gel on the surface of composites with different filler particles. J Prosthet Dent 1986;56(2): 161-169.
22. Dental manufacturers/products/dental restorative materials/ dental composites/dental-nanocomposites/#tab2.Available from: htpp//solutions.3mae.ae/wps/portal/3M/en-AE/3M-ESPE [Last accessed on 2011 Sep 20].
23. Gopferich A. Mechanism of polymer degradation and erosion. Biomaterials 1996;17(2):103-114.