

EFFECT OF APF APPLICATION ON THE MICROHARDNESS OF LIGHT-ACTIVATED RESTORATIVE MATERIALS

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ستعمل فلوريد الفوسفات الحمضي الهلامي القوام كوسيلة لتحسين مقاومة الأسنان للتسوس عن طريق زيادة نسبة الفلورين فيها. إلا أن هذه المادة الحمضية تسبب خدشا في أسطح بعض مواد الحشو التي قد تكون موجودة في تلك الأسنان مما يؤدي إلى التصاق اللويحات الجرثومية عليها. ولقد ظهرت حديثاً مجموعة من مواد الحشو ضوئية التفاعل والتي تتميز بسهولة استخدامها وجودة خصائصها مما حدا بأطباء الأسنان الى استعمالها بكثرة وبالذات لعلاج أسنان الأطفال. كان الهدف من هذا البحث هو دراسة تأثير استعمال فلوريد الفوسفات الحمضي على الصلابة السطحية الدقيقة لثلاث من مواد الحشو ضوئية التفاعل هي مادة الكومبوزايت (بريزما) والأسمنت الزجاجي المتأين (فوجي) والكومبومر (دايراكت).

تم تحضير قوالب بيلاستيكية لإجراء البحث تحتوي على فجوات أسطوانية (2×10م) ملئت كل أربع منها بوحدة من مواد الحشو المذكورة بعد 48 ساعة عولجت كل ثلاث منها بفلوريد الفوسفات بينما تركت الرابعة للمقارنة. وتم تكرار التجربة مرتين بفارق 48 ساعة بين كل مرة وفي نهاية كل استعمال تم تحديد رقم فيكر للصلابة لكل عينة ثم قورنت النتائج إحصائياً.

أظهرت نتائج البحث أنه بوجه عام لا تتأثر درجة الصلابة السطحية لأي من المواد الثلاث باستعمال فلوريد الفوسفات المحمض كما تأكد أن الصلابة السطحية للكومبوزايت (بريزما) أقوى منها في الأسمنت الزجاجي المتأين (فوجي) والكومبومر (دايراكت).

Acidulated phosphate fluoride (APF) gels are commonly used as caries preventive materials in Pediatric Dentistry. APF gels are acidulated with phosphoric acid for the intended purpose of etching the enamel to enhance the fluoride uptake of teeth. Existing dental restorations, particularly glass ionomers and composite resins undergo etching and increased surface roughness when exposed to APF gels. Little work has been reported on the effect of topical APF application on the mechanical properties of restorative materials. Whether the surface microhardness of composites and glass ionomers are affected by such application is not known for sure. The aim of this study was to compare the surface microhardness of a compomer material (Dyract, De Trey Denstply), a composite resin (Prisma AP.H, LD Caulk), and a resin-modified glass ionomer (Fuji II LC, GC) following the application of 1.23% acidulated phosphate fluoride (APF) gel. Experimental specimens were treated with APF gel three times starting at 48h after mixing and with an interval of 48h in between (T₁, T₂, T₃). The surface microhardness of each material was

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determined after each APF gel application and compared to that of the same material without APF gel application as control. The results of this study showed that APF gel application did not affect the surface microhardness of the tested materials. It was also observed that the surface microhardness of Dyract and Fuji II LC were not significantly different from each other, both being significantly lower than Prisma AP.H.

Introduction

The proper selection of restorative materials is an important factor for clinical success. Glass ionomer cements (GICs) have a wide range of uses in dentistry. They can serve as fillings, bases, liners, luting cements, core materials as well as fissure sealants.^{1,3} In addition, GICs have shown a promise as bone cements since they are bio-active and promote bone growth.⁴ GICs adhere to enamel and dentin,^{5,6} release fluoride^{7,8} and reduce the occurrence of recurrent caries^{9,10}. These advantages of GICs are beneficial in restorative dentistry, especially in children with primary teeth when simple-to-use, fast setting, strong and fluoride-releasing materials are most helpful. The major disadvantages of conventional glass ionomers are short working times, relatively long setting times, sensitivity to moisture contamination as well as desiccation during the reaction stages.^{11,12} Composite resins offer easier clinical manipulation and superior esthetics when compared to glass ionomers.¹ However, lack of adhesion of composite restorative materials to tooth structure as well as lack of fluoride release may result in the development of recurrent caries at the tooth/restoration interface and replacement of the restoration.¹⁴

Current research suggests that the restorative versions of the light-cured GICs (hybrid glass ionomers) are promising.¹⁵ They possess the advantages of glass ionomers with the additional qualities of immediate resistance against water loss or uptake because of their light-activated resin component.¹⁶

A new system with properties that are claimed to be superior to other light-activated glass ionomers is now available and generically designated as a "compomer" and is marketed under the trade name Dyract.^{*17,18} Dyract is made up of strontium

fluoro-silicate glass that is contained in a newly formulated resin matrix of urethane dimethacrylate and another resin containing two methacrylate groups and two carboxyl groups (TCB). The material is directly extruded from compules into cavities previously treated with the Dyract primer/adhesive. Dyract is indicated for restoration of Class V cavities, cervical abrasion/erosion lesions, Class III cavities, as well as Class I and II cavities of primary molars.¹⁷ Pulp protection is required only in deep cavities.¹⁶ Dyract Restorative material exhibits an inherent ability to bond to enamel and dentin.¹⁶ Bond strength of 6.8 MPa to dentin, which is comparable to chemically setting glass ionomers has been reported.¹⁷ With the use of Dyract-PAS prime/adhesive, bond strength increases to 10.5 MPa.¹⁷

The surface of GIC can be significantly altered when etched with phosphoric acid^{18,19} or acidulated phosphate fluoride gel (APF).^{20,21} This is of clinical significance because APF gels are recommended as a preventive strategy in dentistry.²² The APF gels contain phosphoric acid which etches the enamel, enhancing fluoride uptake.²² Patients with glass ionomer restorations and receiving APF treatments, could be at risk of increasing the surface roughness of the materials due to their vulnerability to the acidic nature of the APF gel. This is a crucial feature regarding avoidance of plaque retention.²³ These changes in surface texture may affect the microhardness of the glass ionomer materials. It is also known that the prolonged setting reaction, dehydration or hydration of glass ionomers after the initial setting may influence their surface hardness and wear resistance.^{24,25} In addition, different protective coatings of GICS were unable to maintain the original surface hardness of the unprotected cement.²⁶

The purpose of this study was to compare the surface microhardness of three restorative materials (Dyract, Prisma APH and Fuji II LC) following application of 1.23% acidulated phosphate fluoride gel.

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Materials and Methods

Specimens were prepared by filling cylindrical molds (2 mm deep x 10 mm diameters) with each of the three tested materials. Six specimens were prepared of each material. Dyract and Prisma APH were placed directly without mixing. The hand-mixed material (Fuji II LC) was mixed and spatulated. The powder-liquid ratio was measured by use of the scoop and dropper bottle provided by the manufacturer. Immediately after insertion of the materials into the molds, the surface of the restoration was covered by a celluloid strip and pressed with a plastic slide and C-clamp to obtain a smooth and flat surface. After two minutes, the plastic slide was removed and the materials were cured in two different sites for 40 seconds exposure time each using a visible light curing unit.* The celluloid strip was removed and the specimens were placed in closed containers of deionized water for 48 hours at 37°C laboratory oven.+ Three specimens of each material were used for APF gel application and microhardness test (experimental group) and three specimens were used as control (no APF gel application). The APF gel,§ was applied to the test group of each material using cotton applicator for 4

minutes, rinsed with tap water for 20 seconds and dried with oil-free compressed air. The surface hardness for each specimen was determined at room temperature (24°C) after APF gel application [T,]. Three indentations were made on the top surface with a hardness tester^o equipped with a Vicker's indenter. All indentations were made at a load of 100 grams for 5 seconds and 10X objective. The length of the diagonals of each indentation was measured. The mean for each material was calculated and converted to Vicker Hardness Number (VHN) from the tables. The specimens were then stored in distilled water at 37°C. The application of APF gel and microhardness measurements were repeated two more times [T₂ & T₃] at 48 hours intervals. Statistical evaluation of the data was performed using Analysis of Variance and Duncan's Multiple range tests.

Results

The means and standard deviations of the Vickers Hardness numbers (VHN) for the APF-treated (experimental) and untreated (control) specimens of the three tested materials at the three testing times T₁, T₂, and T₃ are presented in Tables 2-7.

For the hybrid glass ionomer (Fuji II LC), no significant differences in VHN values between the experimental and control specimens were observed at any of the three testing times.

For both, the compomer (Dyract) and the composite resin (Prisma APH), no significant differences in VHN values between the experimental and control specimens were observed except at the first testing time (T₁). AT₁, VHN values for the experimental Dyract specimens were

Table 1. Materials tested.

Product	Batch No.	Manufacturer
Dyract	940124	De Trey Dentsply, De-Trey-Str. I D-78467 Konstanz, Germany
Prisma AP.H	647202	The L.D. Caulk division - Dentsply International Inc., Milford DE 19963-0359, U.S.A.
Fuji II LC	251115	G.C. Corporation Tokyo, Japan

Table 2. Mean surface hardness (VHN) and standard deviation of all groups at the different testing times.

	DYRACT		PRISMA APH		FUJI II LC	
	Experimental	Control	Experimental	Control	Experimental	Control
T ₁	40.17 (4.97)	28.23 (2.25)	39.6 (3.67)	54.05 (5.96)	40.43 (9.7)	39.21 (5.3)
T ₂	32.38 (5.4)	33.59 (1.57)	41.0 (4.85)	39.91 (2.12)	30.78 (2.5)	33.98 (1.2)
T ₃	37.38 (3.21)	33.41 (1.93)	44.96 (4.03)	43.89 (2.31)	30.3 (5.8)	31.57 (1.92)

*Optilux 150, Demetron Research Corporation, USA.

^Laboratory oven, Imperial V. Lab-line Instruments Inc., IL, USA.

§Challenge Products Inc., Missouri, USA

nBuehler Micromet II, Lake Bluff, IL, USA.

significantly higher than those of the control specimens, whereas experimental Prisma APH specimens showed a significantly lower VHN values compared to the control specimens.

Comparing the VHN values of the tested materials to each other revealed that Prisma APH showed consistently higher surface hardness values than those of Fuji II LC and Dyract except for the experimental specimens at T_1 where all three materials were not significantly different.

Table 3. Dyract, Duncan's multiple range groupings.

Test Condition	Test Time	Mean VHN	Duncan Group
Experimental		40.17	A
Experimental	T_3	37.38	AB
Control	T_2	33.59	AB
Control	T_3	33.14	AB
Experimental	T_2	32.38	AB
Control	T_1	28.47	B

The means with the same letter are not significantly different from each other ($P < 0.05$).

Table 4. Prisma APH, Duncan's multiple range groupings.

Test Condition	Test Time	Mean VHN	Duncan Group
Control	T1	54.05	A
Experimental	T_3	44.96	AB
Control	T_3	43.89	AB
Experimental	T_2	41.00	AB
Control	T_2	54.05	AB
Experimental	T1	39.60	B

The means with the same letter are not significantly different from each other ($P < 0.05$).

Table 5. Fuji II LC, Duncan's multiple range groupings.

rest Condition	Test Time	Mean VHN	Duncan Group
		40.43	A
Experimental			
Control	T1	39.21	A
Control	T_2	33.98	AB
Control	T_3	33.57	B
Experimental	T_2	30.78	B
Experimental	T_3	30.30	B

The means with the same letter are not significantly different from each other ($P < 0.05$).

Both, the control and experimental groups of Fuji II LC and Dyract showed VHN values that were not significantly different from each other at all testing times.

Discussion

Topical fluoride application in the form of 1.23% APF gel has become an established preventive measure, not only for children, but also for the general population.²⁷ However, there is concern as to the effects of the acidic nature of APF on the surface properties of restorative materials. This has been shown to cause surface etching of conventional²⁸ and light-activated²⁰ glass ionomers as well as composite resins.²⁷ The effect of APF on

Table 6. Control groups, Duncan's multiple range groupings.

Material	Test Time	Mean VHN	Duncan Group
Prisma, APH	T1	54.05	A
Prisma, APH	T_3	43.89	A
Fuji II LC	T_2	39.91	AB
Prisma, APH	T1	39.21	AB
Dyract	T_2	33.59	B
Dyract	T_2	33.98	B
Fuji II LC	T_3	33.41	B
Fuji II LC	T_3	31.57	B
Dyract	T1	28.23	B

The means with the same letter are not significantly different from each other ($P < 0.05$).

Table 7. Experimental groups, Duncan's multiple range groupings.

Material	Test Time	Mean VHN	Duncan Group
Prisma, APH	T_3	44.96	A
Prisma, APH	T_2	41.00	A
Fuji II LC	T1	40.43	AB
Dyract	T_1	40.17	AB
Prisma, APH	T1	39.60	AB
Dyract	T_3	37.38	AB
Dyract	T_2	32.38	B
Fuji II LC	T_2	30.78	B
Fuji II LC	T_3	30.30	B

The means with the same letter are not significantly different from each other ($P < 0.05$).

Dyract has not yet been investigated but it is conceivable that some degree of etching could occur considering the similarity between its matrix and that of composites and glass ionomers.¹⁶ The resistance of Dyract to early acid attacks is also questionable considering that it finally sets by the typical acid-base reaction of conventional glass ionomers which occurs over many days.^{11,16,17}

Despite reports²⁷ that 1-minute APF gel application was as effective as 4 minutes, the latter was chosen in this study since it is more widely applied. All three applications performed were within the first week following mixing of the materials when they would be less resistant to acids^{11,16} and the effects would be more pronounced. The various degrees of surface etching that might have occurred in this study as a result of multiple APF gel application did not seem to affect the surface microhardness of the tested materials.

The VHN values for the experimental specimens of Fuji II LC were not significantly different from the controls at any of the testing times. In fact, they were found to be comparable to values reported in another study²⁶ for conventional glass ionomers. There was also a tendency for those values to decrease with time, regardless of whether they received APF gel or not. This might indicate deterioration of the surface properties of Fuji II LC on water immersion due to dissolution.

The superiority of Dyract over other light-activated glass ionomers as claimed by its manufacturers,¹⁶ was not reflected on its surface hardness values determined in this study. Generally, VHN values for Dyract and Fuji II LC were not significantly different. Experimental and control specimens of Dyract were also not significantly different from each other except at T_j where APF treated surfaces had significantly higher VHN values compared to un-treated surfaces. This might be beneficial but should be taken with reserve since those values dropped at T₁ and T₃ to become similar to control specimens. This observation cannot be explained within the scope of this work and will require further investigation.

VHN values for the composite resin Prisma APH were not affected by APF application except at T_j. This might call for delaying fluoride application to composites to at least four days following setting. Surface hardness of Prisma APH showed a general tendency to be higher than Fuji II LC and Dyract.

This confirms the need to further improve the strength properties of glass ionomer systems.

The surface hardness may be defined as the resistance of the materials to indentions and it reflects the abrasion resistance of the surfaces.^{22,29} It is therefore an important parameter in evaluating restorative materials, especially those intended to restore Class V cavities where they become subjected to abrasive forces. However, the effects of APF gel on other mechanical properties and over a longer period of time should be investigated prior to deciding on whether it is fully safe to apply APF gel on existing composite and glass ionomer restorations.

Conclusions

In general multiple APF gel application does not affect the surface hardness of light-activated glass ionomer, compomer and composite resin restorative materials. The surface hardness of Fuji II LC and Dyract are not significantly different from each other, both being significantly lower than Prisma APH.

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