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Research Article

The Effects of pH Changes on the Microhardness of Three Fluoride Releasing Restorative Materials: An *In Vitro* Study

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Abstract

Objective: The aim of this study was to compare the microhardness of three different fluoride releasing materials after the pH changes of the oral environment in order to provide long term success.

Materials and Methods: Three restorative materials were used for the sample preparation: Chemfil Rock glass ionomer, Glasiosite compomer and Beautifil II giomer. Twelve sample were prepared for each of the three groups (n = 12), and the specimens standardized to 12 mm in dimeter and 2 mm in depth by using a sampler. A vickers microhardness test was used to determine the initial microhardness values of the samples. All the groups were submitted to pH cycling based on the Stephan Curve and the final microhardness tests were completed and recorded. Statistical analyses were made by SPSS 20.0 software.

Results: The initial and post application microhardness values of all the materials used showed a statistically significant decrease after the pH cycling procedure. Beautifil II had the highest decrease in the percentage among the groups.

Conclusion: The results showed that pH effects on the microhardness of restorative materials are restorative material dependent. İn addition, the pH cycling solutions changed the microhardness of all the materials tested.

Keywords: *Glass Ionomer; Compomer; Giomer; Microhardness; Stephan Curve*

Abbreviations

CGIS: Conventional Glass Ionomer Cement; HEMA: Hydroxyethylmethacrylate

Introduction

The oral cavity is a complex, dynamic environment that produces challenges for clinicians with respect to moisture, temperature, and acid levels. An ideal restorative material should be biocompatible, stable over long term, nonreactive to pH changes, compatible with other materials, esthetically pleasing [1-4]. The main reason for restorative replacement is inadequate tooth structure sealing, which ends in secondary caries [5]. Fluoride reservoir materials can prevent secondary caries by releasing fluoride into the saliva, plaque, and teeth [2].

Conventional glass ionomer cement (CGIS) is a biocompatible fluoride releasing material that can be placed in cavities without bonding; however, it exhibits low physical strength, water sensibility, and aesthetic limitations [6-8][GC]. To improve the physical and mechanical properties of CGIS, compomers have been developed that resemble composite resin, but the fluoride is released trough hybrid materials [2,3]. When compared to CGIS, compomers are less biocompatible because of their hydroxyethylmethacrylate (HEMA) content [8,9]. Giomers have recently been introduced as an alternative for direct restorative procedures. They are fluoride releasing reservoirs that are aesthetically pleasing, polishable and biocompatible [10-12]. Although there are many options for restorative procedures, the perfect material has yet to be found.

Because of the lack of literature on this subject, the objective of this research was to determine the effects of Stefan curve pH changes on the microhardness of these three fluoride releasing restorative materials. The aim of this study was to compare the microhardness of three different fluoride releasing materials to the pH changes of the oral environment in order to provide long term success.

Materials and Methods

Three restorative materials were used for the preparation of the samples: Chemfil Rock glass ionomer (Dentsply, Konstnz, Germany), Glasiosite compomer (VOCO, Cuxhaven, Germany), and Beautifil II giomer (Shofu, Kyoto, Japan). Twelve samples were prepared for each of the three groups (n = 12). The specimens were standardized to 12 mm in diameter and 2 mm in depth by using a sampler (Smile Line, Saint-Imier, Switzerland), which allows practitioners to determine the thickness of samples. Shade A2 was chosen for each group (Figure 1).

Figure 1: *Sample preparation.*

In group1, the Chemfil Rock was prepared by using an amalgamator (400/M; Linea Tac, Montegrosso d'asti AT, Italy) and the materials were inserted horizontally into the sampler. The samples were restrained using transparent polyethylene terephthalate (Mylar) strips with a glass slide cover. In group 1, the surface coating was not applied, but the samples were held for 20 minutes before removing the glass.

The polymerization in group 2 and 3 was performed with an Elipar FreeLight 2 curing unit (3M ESPE, St. Paul, MN, USA) for 20 seconds according to the manufacturer's instructions (Figure 2). After removing the samples from sampler, we waited 3 minutes for group 1, but group 2 and 3 were light cured again immediately.

Figure 2: *Elipar FreeLight II.*

The specimens were finished and polished with 12.7 mm aluminum oxide-coated discs (Sof-Lex, 3M ESPE) in decreasing grit size order by one single practitioner. Every sample was moistened with water prior to the use of each disc. Finally, the samples were numbered and stored in deionized water for 24 hours at 37°C in an incubator.

Microhardness measurements

A vickers diamond intender (HMG-G; Shimadzu, Kyoto, Japan) was used to determine the initial microhardness values of the samples (Figure 3). Five indentations were made in each sample, 2 mm apart and the average was calculated automatically by the device. Then, the samples were immersed in 10 different 5 ml experimental artificial saliva solutions were as follows: 0.7 mmol/l $CaCl_2.2H_2O$, 0.2 mmol/l $MgCl_2$, 4.0 mmol/l $KH_2PO_{4,}$ 30.0 mmol/l KCl and 20.0 mmol/l HEPES [4-(2-hydroxyetyhl))-1-piperazineethanesulfonic acid] buffer [12].

Figure 3: *Microhardness testing machine.*

The samples were placed in separate glasses with the direction of the indentations toward the bottom. Each time the solution was changed, all the samples were washed with deionized water. All the groups were submitted to pH cycling and then, the final microhardness tests were completed and recorded.

Solution	pH	Minutes
1	6.7	8
2	5.8	1
3	4.9	9
4	4.8	20
5	5	12
6	5.2	12
7	5.4	12
8	5.6	25
9	5.9	25
10	6.2	8

Table 1: *The pH cycling of the Stephan curve.*

Statistical analyses

Statistical analysis was performed using the SPSS 20.0 software. To analyse normality Kolmogrov-Smirnory test was used. One-way ANOVA and Post-Hoc Tukey tests were used to analyse the differences amoung groups and the differences in the groups ($p < 0.05$)

Results and Discussion

The results of the microhardness tests of materials are displayed in figure 4 and they showed statistically significant differences ($p < 0.05$). The initial and post application microhardness values of all the materials used showed statistically significant decreases after the pH cycling procedure. However, Beautifil II exhibited the highest decrease in the percentage among the groups ($p > 0.05$).

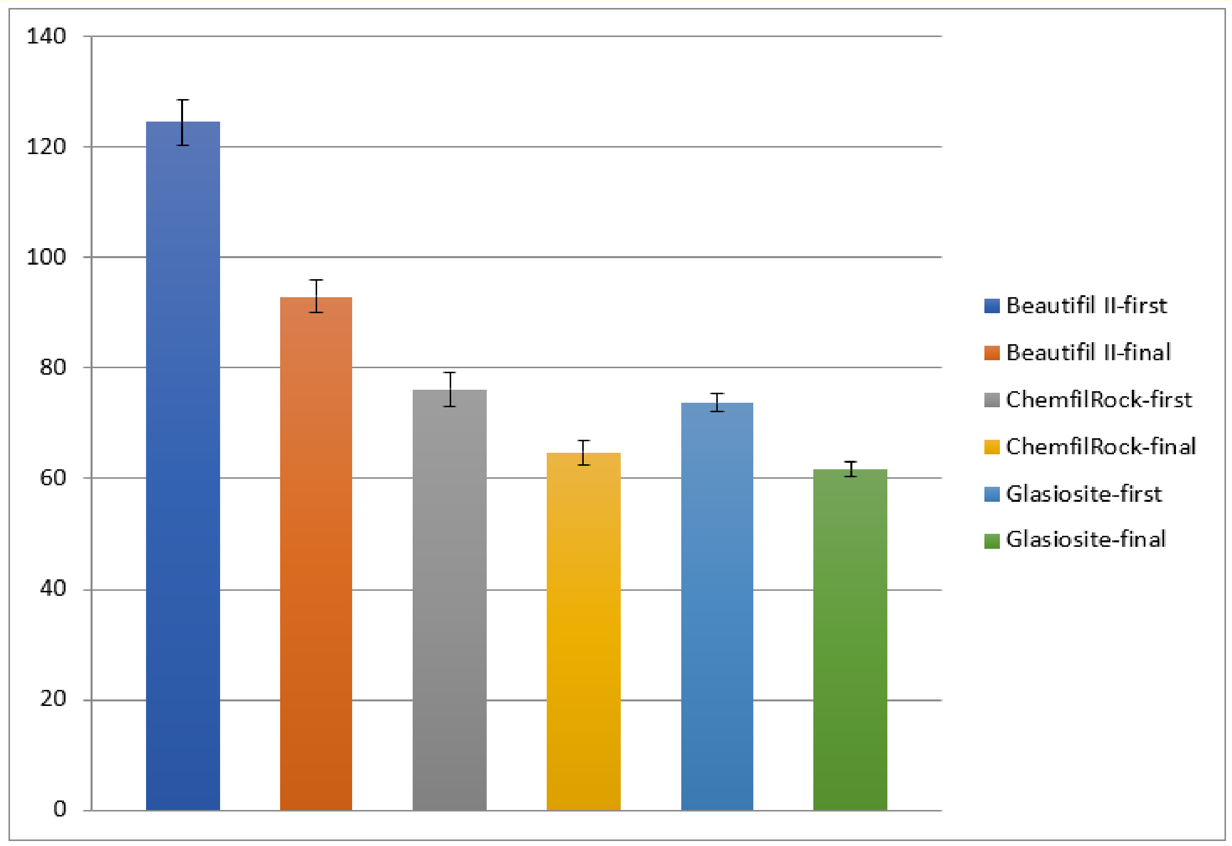

Figure 4: *The means and standard errors of the study results.*

For patients with a high caries risk level, the quality and the quantity of the ions released from restorative materials are important, as well as the physical and mechanical properties [13][F 2000, Dyract AP, experimental compomer]. When compared with the other dental materials, the fluoride releasing materials have demonstrated low plaque accumulation [14]. Even though the fluoride releasing dental materials are the proper choice when preventing caries [14], their low mechanical properties are still a big problem [15][Shofu, Kyoto, Japan]. Moreover, secondary caries under restorative materials have not yet been resolved.

Cariogenic microorganisms, acidic foods, dietary corbohydrates, salivary ionic strenght, bacteria, and enzymatic attacks are responsible for pH changes in the oral cavity [2,13,16,17][fluoride release and surface properties of restorative materials (Ketac-Fil Plus, Vitremer, Fuji II LC, Freedom and Fluorofil]. Thus, as long as sucrose is present in the plaque, acid forms, which results in tooth decay. In 1940, Stefan showed that the pH of the anterior theeth was reduced from 6.5 to 5 within 2 - 4 minutes after rinsing the mouth with a glucose solution. This way done by placing the electrodes in a plate on a flat surface and slowly reaching the initial pH value after 40 minutes. This showed that microorganismes can form acid from carbohydrates very quickly [17]. Because of this, we choose to use the Stefan curve to stimulate the pH changes.

It has been suggested that due to low pH levels or salivary enzyme actions in artifiacial saliva, the biodegradation of materials may increase [3,18] and the fluoride level may rise [18]. Therefore, the release of fluoride from dental materials is affected by the pH of medium [13][F 2000, Dyract AP, experimental compomer]. Due to the chemically active oral environment, the biodegradetion of materials is unavoidable. Thus, clinicians should be aware of the importance of selecting the appropriate restorative material for the patient based on their risk factors [2].

Chemical degradation can occur in many ways, through oxidative, thermolytic, photolytic, radiolytic, solvolytic, or hydrolytic deradation, the last of which iis most studied by clinicians. The biodegradation of materials results in decreased microhardness [4]. Similar to previous studies [4,19], the results of our study showed that the pH cycling procedure significantly reduced the microhardness of each material tested.

Even if the recharching mechanism has not been fully understood, it is believed that the hydrogel layer around the glass filler particles following the reactions between the glass and polyacid components is responsible for the fluoride release. The pH changes of the prereaction between the flora, aluminum silicate glass, and polyacid to form the CGIS matrix structure before blending with the resin [11,14]. Therefore, we suppose that the initial fluoride release of the giomer material played a major role in decreasing the microhardness value. In addition, the microhardness of the compomer material does not decrease as much as the giomer. One possible reason for this variation may be that the hydrogel layer of the glass filler in the giomer is more prevalent than in the compomer.

The microhardness of the CGIS material depends on the particle size, powder to liquid ratio, temperature, and humidity. To maintain standardization, one clinician prepared all of the samples on the same day by using an amalgamator based on the reaction time. Therefore the powder to liquid ratio changes, hand mixing differences, and environmental variations were avoided [7,19].

To overcome the early water exposure to the CGIS and increase the physical strenght, surface coating materials can be used. In addition, the loss of translucency and dimensional changes can be prevented. Varnishes, petroleum jelly, cocoa butter, and light cured resin have been recommended for coating; however, the application of resin takes time, increases costs, and reduces fluoride release [6][GC]. Shintome., *et al.* found that there was no significant difference between the material's microhardness values with or without surface coating [20]. However, one study reported that light cured self adhesive coating exhibited increased mechanical strenght when compared to the non-surface coated samples when the compressive tests were performed [21]. In our study, the surface coating was not applied, but he samples were held for 20 minutes with transparent polyethylene terephthalate strips and a glass slide cover as a coating effect.

Many investigators have found that the Sof-Lex disc performence is superior to that of other polishing materials [22]. Therfore, we choose Sof-Lex discsas the polishing material for our study.

This study points out importance of choosing the right restorative material based on the biodegradation of fluoride releasing restorative materials from the pH changes in an artificial oral cavity. Overall, it is clinically important to understand the correlation between the restorative materials and the pH variations in the oral cavity to maintain long term clinical success.

Conclusion

The effects of pH on the microhardness of restorative materials are material dependent. In this study, the pH cycling solutions changed the microhardness of all the materials tested. This change affected the beautifil II giomer material the most, but did not reduce the microhardness of the CGIS and compomer very much. Further clinical investigations are needed to clarify the biodegradation of CGIS in oral pH changes.

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Conflict of Interest

Nil.

Bibliography

1. Erdemir U., *et al*. "Surface hardness of different restorative materials after long-term immersion in sports and energy drinks". *Dental Materials Journal* 31.5 (2012): 729-736.

2. Silva KG., *et al*. "Effect of pH Variations in a Cycling Model on the Properties of Restorative Materials". *Operative Dentistry* 32.4 (2007): 328-335.

3. Gömeç Y., *et al*. "Effects of dietary acids on surface microhardness of various tooth-colored restoratives". *Dental Materials Journal* 23.3 (2004): 429-435.

4. El Gezawi M., *et al*. "Degradation Potential of Bulk Versus Incrementally Applied and Indirect Composites: Color, Microhardness, and Surface Deterioration". *Operative Dentistry* 41.6 (2016): 195-208.

5. Gordon W., *et al*. "The antimicrobial efficacy of the erbium, chromium: yttrium-scandium-gallium-garnet laser with radial emitting tips on root canal dentin walls infected with Enterococcus faecalis". *Journal of American Dental Association* 138.7 (2007): 992-1002.

6. Wang XY., *et al*. "Effect of early water exposure on the strength of glass ionomer restoratives". *Operative Dentistry* 31.5 (2006): 584-589.

7. De Moor RJ and Verbeeck RM. "Changes in surface hardness of conventional restorative glass ionomer cements". *Biomaterials* 19.24 (1998): 2269-2275.

8. Sidhu S and Nicholson J. "A Review of Glass-Ionomer Cements for Clinical Dentistry". *Journal of Functional Biomaterials* 7.3 (2016): E16.

9. Kanchanavasita W., *et al*. "Long-term surface micro-hardness of resin-modified glass ionomers". *Journal of Dentistry* 26.8 (1998): 707-712.

10. Chitnis D., *et al*. "Comparison of in-vitro bond strengths between resin-modified glass ionomer, polyacid-modified composite resin, and giomer adhesive systems". *American Journal of Orthodontics and Dentofacial Orthopedics* 129.3 (2006): e11-e16.

11. Harhash AY., *et al*. "A comparative in vitro study on fluoride release and water sorption of different flowable esthetic restorative materials". *European Journal of Dentistry* 11.2 (2017): 174-179.

12. Eisenburger M., *et al*. "The Use of Ultrasonication to Study Remineralisation of Eroded Enamel". *Caries Research* 35.1 (2001): 61-66.

13. Geurtsen W., *et al*. "Effect of storage media on the fluoride release and surface microhardness of four polyacid-modified composite resins ("compomers")". *Dental Materials* 15.3 (1999): 196-201.

14. Saku S., *et al*. "Antibacterial activity of composite resin with glass-ionomer filler particles". *Dental Materials Journal* 29.2 (2010): 193-198.

15. Gordan VV., *et al*. "A clinical evaluation of a self-etching primer and a giomer restorative material". *Journal of American Dental Association* 138.5 (2007): 621-627.

16. Bean TA., *et al*. "Effect of esterase on methacrylates and methacrylate polymers in an enzyme simulator for biodurability and biocompatibility testing". *Journal of Biomedical Materials Research* 28.1 (1994): 59-63.

17. Bowen WH. "The Stephan Curve revisited". *Odontology* 101.1 (2013): 2-8.

18. Munack J., *et al*. "Effects of various storage media on surface hardness and structure of four polyacid-modified composite resins ("compomers")". *Clinical Oral Investigations* 5.4 (2001): 254-259.

19. Nomoto R and McCabe JF. "Effect of mixing methods on the compressive strength of glass ionomer cements". *Journal of Dentistry* 29.3 (2001): 205-210.

20. Shintome LK., *et al*. "Microhardness of glass ionomer cements indicated for the ART technique according to surface protection treatment and storage time". *Brazilian Oral Research* 23.4 (2009): 439-345.

21. Sukumaran V., *et al*. "To Evaluate the Effect of Surface Coating on Three Different Types Glass Ionomer Restorations". *Biomedical and Pharmacology Journal* 8 (2015): 445-449.

22. Patil HA. "Effect of Various Finishing Procedures on the Reflectivity (Shine) of Tooth Enamel - An In-vitro Study". *Journal of Clinical and Diagnostic Research* 10.8 (2016): ZC22-ZC27.

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