

## AN IN-VITRO ASSESSMENT OF MICROHARDNESS AND DENTINAL REMINERALIZATION OF ZIRCONOMER AND EQUIA FIL ON DENTAL SURFACE

Ibrahim Basha Eldossoky\*

### **ABSTRACT**

**Aim:** This research evaluates the effect of different types of glass ionomer restorations on surface microhardness and remineralization of dentin.

**Materials and methods:** Three of glass ionomer restorations (medi fil, equia fil and zirconomer) were used. A total number of 32 sound non-cariou human premolar teeth extracted for orthodontic reasons were used in this study. Standardized two class V cavities, one on the buccal and other on lingual surface of each tooth were prepared. The cavity preparation was 3, 2, and 1.5 mm for width, height and depth respectively. The teeth were sectioned mesio-distally to get (64) then divided into three equal main groups (21 teeth each) according to the restorative materials, group (M): medi fil group (E): equia fil group and (Z): zirconomer group. Each group was further subdivided into three equal divisions (7 teeth each) according to the storage times; one day (S1), one month (S2) and three months (S3). The specimens were sectioned longitudinally along the middle of the restorations to produce two blocks: One part was mounted in epoxy resin for microhardness testing, and the other for energy dispersive x -ray analysis (EDX). Specimens were stored in artificial saliva for one day, one month and three months before testing. A microhardness tester was used to measure the Vickers hardness number.

**Results:** It was found that the highest mean value of surface micro hardness and remineralization was recorded for medi fil group, while the lowest mean value was recorded for equia fil group. Pair-wise Tukey's post-hoc test showed no-significant between one month and three-month storage.

**Conclusion:** It can be concluded that all glass ionomer restorations show remineralization to tooth structures also zirconomer overpowered by conventional glass ionomer because its high fluoride release finally equia-fil show increase remineralization with time.

---

\* Lecturer, Department of Operative Dentistry Faculty of Dental Medicine, Boys, Cairo, Al-Azhar University

## INTRODUCTION

Glass ionomer cement (GIC) is acknowledged as a biomimetic material because it has mechanical properties similar to dentin<sup>(1)</sup>. Biocompatibility, fluoride release, and chemical bonding to hard tissues of the tooth render it an ideal material in many restorative situations<sup>(1,2)</sup>. Its ability to prevent and arrest carious lesions has been supported<sup>(3,4)</sup>.

High-viscosity glass ionomer cement (HVGIC) was used with restorative techniques. The powder-liquid ratios were higher than earlier conventional restoratives<sup>(5)</sup>. HVGICs are not so successful when it comes to the final polishing expectations, working time, and development of ultimate properties, as dehydration gives rise to a micro-cracks formation that reduces the cohesive strength compared with resin cements<sup>(6)</sup>.

Equia, produced by GC (Tokyo, Japan) is a newly introduced system consisting of a highly viscous conventional GIC (Equia Fil) combined with a novel nano-filled coating material (Equia Coat). This self-adhesive, nano-filled resin coating provides high hydrophilicity and minimal viscosity, which optimize the seal of a GIC to the dental surface. Compounded nanofillers protect the system against abrasive wear<sup>(7)</sup>.

With the ability of zirconia-reinforced glass ionomer cement ("zirconomer") to overcome these drawbacks, it is referred to as "white amalgam" as it provides amalgam-like strength and durability, in addition to the inherent protective properties of GIC, making it indicated in traumatic restorative techniques<sup>(8)</sup>.

Microhardness is one of the most important properties used to compare restorative materials subjected to high masticatory force. Microhardness testing is a simple and reliable in providing indirect information on mineral content changes in dental hard tissues<sup>(9)</sup>.

We used semi-quantitative energy dispersive X-ray (EDX) because it a reliable, precise, and retrievable method for quantifying the significant constituents present in a material or mixture. Analysis was performed by collecting line scans along the line: restorative material interface-enamel/dentin to determine the elemental distribution of minerals<sup>(10)</sup>.

## MATERIALS AND METHODS

- 1. Selection of teeth:** A total number of 32 sound non-carious human premolar teeth extracted for orthodontic reasons were collected from oral surgery clinic, free of cracks and any developmental defects, were used in this study.
- 2. Cavity preparation:** Standardized class V cavities, one on the buccal and one on the lingual surface of each tooth, were prepared at high speed using water as a coolant. The cavity preparation was 3, 2, 1.5 mm for width, height and depth, respectively. It was placed parallel to the cemento-enamel junction (CEJ), with the preparation extending 1 mm above the CEJ.
- 3. Grouping of teeth:** The teeth were sectioned mesiodistally to drill 64 cavitations. They were next divided into three equal main groups (21 teeth each) according to the restorative materials, group (M): medi-fil group (E): equia-fil group and (Z): zirconomer group.

Each group was subdivided into three equal subgroups (7 teeth each) to be sorted according to the storage times; one day (S1), one month (S2) and three months (S3)

- 4. Statistical tests:** One-way ANOVA test and its corresponding p-value was measured. Post-hoc Tukey test and its p-values were calculated among the three groups.

**RESULTS**

We revealed that medi-fil group recorded the highest microhardness mean values, followed by zirconomer group, while equia-fil group recorded the lowest microhardness mean value (Table 1, Figure 1).

Other than the parameter of storage time, it was found that medi-fil group recorded the highest micro Ca, P and F remineralization mean values followed by the zirconomer group while the equia-fil group recorded the lowest microhardness mean value (Table 2, Figure 2).

TABLE (1) Hardness measures for the three groups

Hardness	M group	Z group	E group	P-value
D1 (Mean±SD)	85.00 ± 2.00	82.00 ± 1.00	53.33 ± 1.53	0.000
1M (Mean±SD)	74.00 ± 2.00	73.00 ± 1.00	56.78 ± 1.35	0.000
3M(Mean±SD)	66.00 ± 2.00	65.00 ± 1.00	59.33 ± 1.00	0.002

TABLE (2) Measures of Ca, P and F for the three groups

		M group (Mean±SD)	Z group (Mean±SD)	E group (Mean±SD)	P-value
<b>Ca</b>	1 Day	56.00 ± 2.00	40.00 ± 1.00	35.00 ± 1.00	0.000*
	1 Month	51.00 ± 1.00	34.33 ± 0.58	39.33 ± 2.52	0.000*
	3 Month	52.00 ± 2.00	27.67 ± 2.08	43.00 ± 2.00	0.000*
<b>P</b>	1 Day	33.00 ± 1.00	21.00 ± 1.00	17.00 ± 1.00	0.000*
	1 Month	25.33 ± 0.58	16.00 ± 1.00	21.67 ± 1.53	0.000*
	3 Month	25.00 ± 1.00	13.00 ± 2.00	25.67 ± 1.53	0.000*
<b>F</b>	1 Day	2.17 ± 0.15	1.00 ± 0.20	0.53 ± 0.31	0.000*
	1 Month	1.00 ± 0.20	0.53 ± 0.31	1.03 ± 0.21	0.079*
	3 Month	1.20 ± 0.20	1.00 ± 0.26	1.37 ± 0.21	0.219

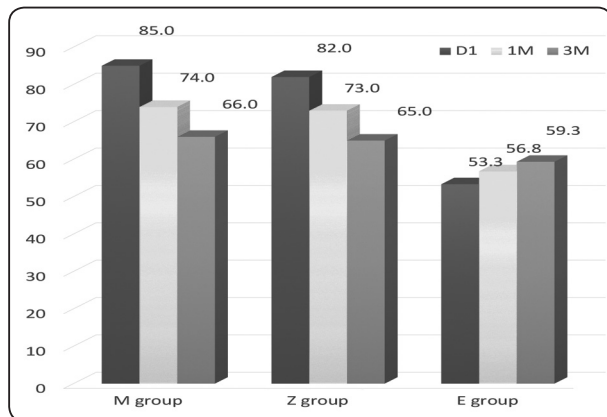


Fig. (1): Column chart of total micro hardness mean values of treatment groups.

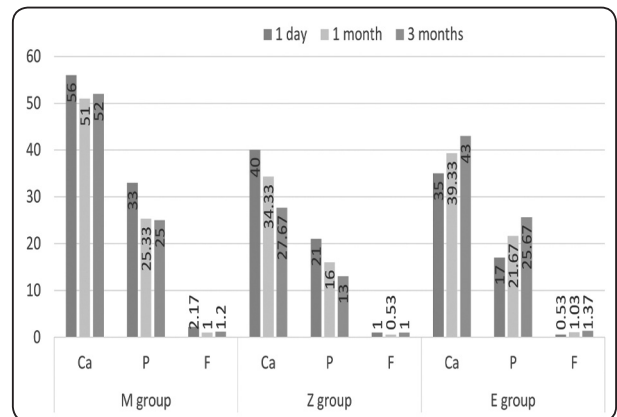


Fig. (2): Column chart of the mean values of Ca, P and F remineralization as function of treatment groups.

## DISCUSSION

Measuring microhardness evaluates the mineral content of a tissue and assess the dissolution-base loss of mineral that is taking place secondary to losing the inorganic constituents, as is the case in initiating dental caries after demineralization<sup>(11, 12)</sup>. The present study revealed that the medi-fil treated group provided the highest microhardness mean value followed by zirconomer treated group. In comparison, the equia-fil group provided the lowest microhardness mean value. Ionic elements migrate to demineralized dentin. Perhaps a diffusion-driven activity is partly explained by the concentration gradient between the GIC and the dentin concerning these elements<sup>(13-15)</sup>. This finding goes hand in hand with the result obtained by Nurulnazra et al.<sup>(16)</sup>, who reported that conventional GIC showed high surface microhardness of dentin more than (encapsulated) Fuji IX GP Extra. This could be linked to the higher powder-to-liquid proportion in highly viscous preparation.

GICs provide more glass filler content with fewer ions cross-linking the polymer chains holding them close together, leading to less water transport and, consequently, releasing lesser fluoride. This finding was also consistent with what Dias et al.<sup>(17)</sup> reported as regards the increase in dentin hardness and changes in mineral content due to ion exchange from conventional GIC to dentin in the tooth-restoration interface. Ionic elements migrate to demineralized dentin. This mineral uptake promotes changes in the hardness of dentin<sup>(18, 19)</sup>. Also, this result was confirmed with the result obtained by Pereira et al.<sup>(20)</sup>. The resin-modified glass ionomer cements produced an inhibition zone with less hardness than conventional glass ionomer cement. We revealed that the equia fil treated group provided the higher microhardness mean value at three months than one month treated group while the one day group provided the lowest microhardness mean value. This may be due to a higher proportion of

powder liquid in high-viscosity GICs provides more glass filler content with fewer ions cross-linking the polymer chains holding them close together. Similar to Aykut-Yetkiner et al.<sup>(21)</sup>, who showed the microhardness of the dentin under GIC restored restorations using a traumatic restorative technique increased over time.

Although this increase was not sufficient for the microhardness to reach a value similar to that of healthy dentin, it may be attributed to the ionic exchange of fluorine and strontium process causing remineralization. Moreover, the result of this study was in agreement with Zoergiebel and Ilie<sup>(22)</sup>, who shows a gentle increase over the 1-year storage period with equia-fil restorations. Moreover, this result was in agreement with Santiago et al.<sup>(23)</sup>, who showed a considerable increase of microhardness of dentin according to the time with Fuji IX. This is attributed to the dynamic changes occurring after 180 days. The source of nutrition for the cariogenic bacteria is eliminated the microorganisms will not survive, putting an end to the carious process. GIC can establish a physical barrier against nutrients emanating from the communication with the buccal cavity.

The main ions involved in this process are calcium, phosphorous and fluoride. In particular, fluoride has always been a subject of research because it participated in remineralization. The medi-fil treated group, in our study, displayed the highest mean value of remineralization at one day, followed by zirconomer, while equia-fil provided the lowest mean value of remineralization. This result was explained by the greatest release of minerals from GIC that occurred on day one and diminished gradually. The Zirconia-reinforced samples took an exciting turn from a low fluoride release value after 24 hours to an increased value after seven days which again degraded after 28 days. After 28 days, there was no significant difference between the Fluoride release between the

conventional and zirconomer group<sup>(24)</sup>. This result was in agreement with Prabhakat et al.<sup>(25)</sup>, who established that conventional glass ionomer cement restorations showed an increased remineralization potential when compared to the experimental group containing Zirconia-reinforced GIC restoration. This may be due to maximum release of minerals, especially fluoride from conventional GIC, which led to increased remineralization. Also, this was in agreement with Kofman et al.<sup>(26)</sup>, who reported that minerals released from a GIC had the potential to enhance remineralization of the early carious lesion.

In the present study, the conventional GI (medi-fil) treated group provided a higher mean value of remineralization than equia-fil which provided the lowest mean value of remineralization. This result was explained by the most excellent release of minerals from conventional GIC, which act as nucleation sites for the calcium phosphate minerals to precipitate when dentin is stored in a solution rich with calcium and phosphate ions.

The results of this study agreed with Elizabeta<sup>(27)</sup>, who showed that the most remarkable effects were observed with the conventional glass ionomer cement, with the gap next to the materials being covered with crystalline deposits after 18 months of storage. In the present study, results revealed that the zirconomer-treated group provided the highest mean value of remineralization (fluoride release) in the first day then declined after one month. This agreed with Abdulsamee and Elkhadem<sup>(28)</sup>, in (2017), who reported that fluoride release by zirconomer was constant from 14 hours up to 10 days with a decline later.

We found that the zirconomer treated group provided the higher mean value of remineralization (fluoride release) than equia-fil. Similarly, Saxena and Tiwari<sup>(29)</sup>. Detected that fluoride release increased from day 1 to day 7 and then decreased on days 15 and 30. Vinasab and Meyers<sup>(30)</sup> reported similar results. Higher fluoride release in the first

few days is a typical feature of GICs<sup>(31)</sup>. This “Burst Effect” minimizes the number of residual bacteria in restored cavities and enhances the remineralization of enamel and dentin<sup>(30)</sup>. However, emphasis need to be placed on the various limitations of in-vivo and in-vitro methods of assessment frequently used in conservative operative dentistry. It should be also emphasized that in-vitro studies need to be supported by clinical trials, given that the oral cavity is a harsh environment that harbors a multitude of thermal, chemical and galvanic variables that cannot be provided experimentally in a perfect shape.<sup>(32)</sup>

## CONCLUSIONS

Under the circumstances and limitation of this study, all glass ionomer restorations show remineralization to tooth structures. However, zirconomer overpowered by a conventional glass ionomer because of its high fluoride release. Equia-fil show increase remineralization with time.

## RECOMMENDATIONS

- 1- Long-term (more than three months) and clinical studies are required to confirm these findings.
- 2- Further laboratory and in vivo investigations and re-evaluations to tooth structure are highly recommended in future.

## REFERENCES

1. Tyas M. Clinical evaluation of glass-ionomer cements restorations. *J Appl oral Sci.* (2006); 14:10–3.
2. Garcia-Contreras R, Scougall-Vilchis R, Contreras-Bulnes R, Sakagami H, Morales-Luckie R, Nakajima H. Mechanical, antibacterial and bond strength properties of nano-titanium-enriched glass ionomer cement. *J Appl oral Sci.* (2015); 23:321–8.
3. Raggio D, Tedesco T, Calvo A, Braga M. Do glass ionomer cements prevent caries lesions in margins of restorations in primary teeth? A systematic review and meta-analysis? *J Am Dent Assoc.* (2016); 147:177–85.

4. Tedesco T, Bonifacio C, Calvo A, Gimenez T, Braga M, Raggio D. Caries lesion prevention and arrestment in approximal surfaces in contact with glass ionomer cement restorations-A systematic review and meta-analysis. *Int J Paediatr Dent.* (2016); 26:161-72.
5. Yilmaz Y, Eyuboglu O, Kocogullari M, Belduz N. A one-Year Clinical Evaluation of a High-Viscosity Glass Ionomer Cement in Primary Molars. *J Contemp Dent Pract.* (2006); 7:71-8.
6. Anusavice KJ. 11th ed. USA: Phillip's Science of Dental Materials; 471-86.
7. Bonifacio C, Werner A, & Kleverlaan C Coating glass-ionomer cements with a nanofilled resin *Acta Odontologica Scandinavica* (2012) 70 (6) 471-477.
8. Zirconomer Zirconia Reinforced Restorative [Last cited 2016 Mar 11].
9. Imfeld T. Dental erosion, definition, classification and links. *Eur J Oral Sci.* (1996); 104: 151-155.
10. Giovanna A, Rodrigo N, Rached, Rui F, Mazur, Sergio Vieira, Evelise M, Souza. Effect of open-sandwich vs. adhesive restorative techniques on enamel and dentine demineralization: An in situ study(2013)872-880.
11. Kodaka T, Debari K, Yamada M, Kuroiwa M. Correlation between microhardness and mineral content in sound human enamel. *Caries Res* (1992); 26: 139-141.
12. Samuel S, Rubinstein C. Microhardness of enamel restored with fluoride and non-fluoride releasing dental materials. *Braz Dent J* (2001); 12: 35-38.
13. Nicholson J. Glass ionomer dental cements: update. *Materials Technology.* (2010)Mar; 25(1):813. doi:10.1179/175355509.
14. Sidhu S. Glass-ionomer cement restorative materials: a sticky subject? *Aust Dent J.* Jun; 56(Suppl 1):23-30. doi:10.1111/j.1834-7819.2010.01293.x. PMID: (2011) 21564113.
15. Baliga M, Bhat S. Effect of fluorides from various restorative materials on remineralization of adjacent tooth: an in vitro study. *J Indian Soc Pedod Prev Dent.* 2010 Apr-Jun; 28(2):84-90. doi:10.4103/0970-4388.66742. PMID: (2066)0973.
16. Nurulnazra M, Mahyuddin A and Sockalingam SNMP. In-vitro Comparative Evaluation of Cariostatic Potential and Marginal Microleakage of Commonly-used Glass-Ionomer Restorative Materials as Interim Therapeutic Restorations. *SM J Dent.* (2017); 3(1): 1010.
17. Gisele D, Ana Cláudia C, Fábio S, Viviane H, Fabiana A, Denise W The hardness and chemical changes in demineralized primary dentin treated by fluoride and glass ionomer cement *Rev Odontol UNESP.* - ISSN (2015)1807-2577.
18. Ten Cate J, Duijsters P. Alternating demineralization and remineralization of artificial enamel lesions. *Caries Res.* 1982; 16(3):201-10. doi:10.1159/000260599. PMID: 6953998.
19. Marquezan M, Corrêa F, Sanabe M, Rodrigues Filho L, Hebling J, Guedes-Pinto A, et al. Artificial methods of dentine caries induction: a hardness and morphological comparative study. *Arch Oral Biol.* 2009 Dec; 54(12):1111-7. doi:10.1016/j.archoralbio.2009.09.007. PMID: 19878926.
20. Pereira P, Inokoshi S., Yamada T, Tagami J. Micro hardness of in vitro caries inhibition zone adjacent to conventional and resin-modified glass ionomer cements (1998) *Dent Mater* 14:179-185 .
21. Aykut-Yetkiner A, Sim ek D, Eronat C, Çiftçio lu M. Comparison of the remineralization effect of glass ionomer cements versus a resin composite on dentin of primary teeth *European Journal of Paediatric Dentistry* (2014) vol. 15/2-.
22. Julius Z, Nicoleta I. An in vitro study on the maturation of conventional glass ionomer cements and their interface to dentin *Acta Materialia Inc*(2013) 1742-7061/\$.
23. Santiago B, Ventin D, Primo L. And Barcelos R. Microhardness of dentine underlying ART restorations in primary molars: an in vivo pilot study *British Dental Journal* (2005); 199: 103-106.
24. Bertolini M, Zaghete M, Gimenes R, Padovani G & Cruz C Preparation and evaluation of an experimental luting glass ionomer cement to be used in dentistry *Journal of Material Science: Materials in Medicine*(2009) 20(9)1781-5.
25. Prabhakar A, Kalimireddy L, Sugandhan. S, Saraswathi V. Evaluation of Fluoride Related Traits of Zirconia Infused GIC. *Int J Oral Health Med Res* (2016); 2(6): 17-20.
26. Hatibovic-Kofman S, Suljak JP & Koch G Remineralization of natural carious lesions with a glass ionomer cement *Swedish Dental Journal* (1997) 21(1-2) 11-7.

27. Elizabeta G, John W, Aleksandar T. Ion migration from fluoride-releasing dental restorative materials into dental hard tissues. *J Mater Sci: Mater Med* (2012)23:1811–1821.
28. Nagy Abdulsamee and Ahmed Hosny Elkhadem. “Zirconomer and Zirconomer Improved (White Amalgams): Restorative Materials for the Future. Review” *EC Dental Science* 15.4 (2017): 134-150.
29. Sudhanshu S, Sonia T. Energy dispersive X-ray microanalysis, fluoride release, and antimicrobial properties of glass ionomer cements indicated for atraumatic restorative treatment *J Int Soc Prevent Communit Dent*: (2016) 6; 366-72.
30. Mousa vinasab S, Meyers I. Fluoride release by glass ionomer cements compomer and giomer. *Dent Res J* (2009); 6:75-81.
31. Rao B, Moosani G, Shanmugaraj M, Kannapan B, Shankar B, Ismail P. Fluoride release and uptake of five dental restoratives from mouthwashes and dentifrices. *J Int Oral Health* (2015); 7:1-5.
32. Basha E. Assessment of composite leakage using optical coherence tomography: A systematic review. *Adv Clin Exp Dent*. 1(1):19-32. doi: 10.21608/aced.2020.150766