



# Influence on the Hardness of Resin Modified Glass Ionomer Cements following Exposure to High and Low Fluoride Containing Toothpastes

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## ABSTRACT

**Objective:** Resin modified glass ionomer cements (RMGICs) are fluoride releasing restorative materials. Their recharge with sources of fluoride (like toothpastes) during their service in oral cavity is a common practice. The purpose of this study was to establish the influence of fluoride containing toothpastes on hardness of RMGICs after exposure to acidic and neutral fluoride containing toothpastes.

**Materials and methods:** Sixty specimens were made from two RMGICs (Vitremer and Fuji II LC). Initially hardness was measured without exposure to source of fluoride of 20 control specimens using Vickers microhardness tester. Rest of the 40 experimental specimens were exposed to fluoridated toothpastes for 60 days twice daily for 2 minutes, then hardness of specimens was measured again using Vickers microhardness tester. Data was statistically analyzed using one-way ANOVA on SPSS version 16.

**Results:** RMGICs which were exposed to low fluoride neutral source had lower values of hardness than RMGICs which were exposed to high fluoride acidic source.

**Conclusion:** High fluoride containing acidic toothpaste exhibited no detrimental effect on hardness of specimens over the period of study. Specimens exposed to high fluoride containing acidic source displayed higher values of hardness than the specimens at baseline without exposure to source of fluoride. RMGICs used in our study proved to be materials which exhibit increase in hardness after exposure to protocols practiced in our research for its recharge.

**Keywords:** Hardness, Resin modified glass ionomer cements, Fluoride containing toothpaste, Vickers microhardness.

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## INTRODUCTION

Glass ionomer cements introduced by Wilson and Kent had low tensile strength, flexural strength and fracture toughness.<sup>1</sup> Modifications including inclusion of resin resulted in the formation of a material with enhanced mechanical properties.<sup>2</sup> The importance of topically applied fluoride agents is verified in plummeting carious lesions.<sup>3</sup> Among these, the implication of the use of fluoride toothpastes is unequivocal.<sup>4-7</sup> The impact of relative regular exposure to provisions with low-dose of fluoride is proven through clinical trial.<sup>8</sup>

Resistance presented by a material to indentation or penetration is hardness of the material.<sup>9</sup> Indentation formed on a material's exterior is a consequence of interaction of properties like strength, proportional limit and capacity to abrade or being grinded by opposing dental structures and materials.<sup>10</sup> Restorative filling materials are expected to have enduring durability in the oral cavity.<sup>11</sup> Hence, prime most required physical property of restorative filling materials is surface hardness, that draws a parallel to compressive strength and abrasion resistance of material.<sup>11</sup> For this reason, enhancement or descent in hardness as a consequence of fluoride application through fluoride containing sources have clinical insinuations.<sup>12</sup> In a study, 'effect of topical fluoride gels on the microhardness of three glass ionomer cements' was explored.<sup>13</sup> One study reported that treatment with acidulated phosphate fluoride (APF) gel and contact with stannous fluoride (SnF<sub>2</sub>) gel does not reduce hardness.<sup>13</sup> In an another study, 1.23% APF gel and 2% sodium fluoride (NaF) gel were compared.<sup>14</sup> They also found that APF gel has detrimental effect on hardness of material while NaF caused no change in specimens' hardness.<sup>14</sup>

Use of fluoride containing toothpaste assures daily intake of fluoride. The purpose of our study was to explore change in hardness of RMGICs as a result of exposure to fluoride in the form of high and low fluoride containing, acidic and neutral toothpaste respectively. The hypothesis was that hardness will not change after exposure to these sources.

## MATERIALS AND METHODS

Our study was an experimental, *in vitro* study with duration of 7 months. In which two RMGICs (Vitremer and Fuji II

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LC) were used (Table 1). Sixty specimens from these two materials were formed, i.e. (30) specimens from each type of RMGIC with dimension 10 mm diameter × 2 mm thick, amounting to a surface area of 1.42 cm<sup>2</sup>. Experimental group I is Vitremer, experimental II is Fuji II LC; each group had 30 samples: 10—control, 10—exposed to colgate, 10—exposed to close up toothpastes (Table 2). All specimens were prepared by a single operator. Before placing of material in the mold, the mold was lined using petroleum jelly, this facilitates easy removal of specimen following cure. The mold was lined on below with a polyethylene strip and a glass cover slip. After this arrangement was ready, mixing was commenced. The material was dispensed as powder and liquid on a clean mixing sheet following the ratios précised by the manufacturer. It was mixed with a cement spatula to accomplish a homogenous consistency, free of granules. The material was placed in plastic mold using cement spatula. It was ensured that material was a little brimful by putting more than required material every time in the mold. The microscopic slide was placed manually by the researcher. This led to the extrusion of excess material from the matrix and prevents the inclusion of air bubble. The glass cover slip was removed after 30 seconds. Specimens were cured from both the sides of the specimen following manufacturer's recommended time and light, at the recommended light intensity. Each specimen was stored in its mold for 1 hour after curing. After this finishing was executed with multifluted burs and polishing was achieved using Sof-Lex (Aluminums Oxide) disks and maintaining the material's surface moist. For every series, 10 strokes were made using a low-speed hand piece in one direction. Following this protocol 60 specimens were made, 20 (control) that is 10 from each RMGIC and 40 (experimental) that is 20 of each RMGIC. Out of these control 20 specimens, 10 were exposed to toothpaste I and remaining 10 were exposed to toothpaste II. After this each specimen was assigned to its plastic vial and was stored in 5 ml of distilled water for 24 hours in their individual plastic vials. Both sample and container were placed at

room temperature during the entire experiment. All control specimens after storage for 24 hours in distilled water were subjected to calibration of hardness using surface of specimen. Hardness was measured using Vickers microhardness tester (Vickers Series 402 MH Tester for HV 0.01-HV 2), by Wolpert W group micro Vickers hardness tester digital autoturret model number 402 MVD (at ambient room temperature (28 ± 2°C), with a diamond indenter having a 136° apex angle present at NED University's Department of Material Sciences. The samples before and after testing were inspected visually to substantiate the absence of any surface defects or pores. Three indentations were made on each specimen and were equally spaced over a circle and not closer than 1 mm to adjacent indentations or the margin of the specimen. A load of 300 gm was applied for a dwell time of 15 seconds. The two diagonals of the indentation left in the surface of the material after removal of the load are measured using a microscope and their average calculated. The area of the sloping surface of the indentation is calculated. After unloading, the diagonals of the indentation were measured and Vickers hardness number was evaluated using a microscope and computer analysis. Three indentations were measured for each specimen. Mean hardness value was determined. Hardness readings were directly obtained from dial. Objective lens used was of ×40 and the eye piece was of ×10 and the total magnification was ×400. The Vickers hardness is the quotient obtained by dividing the kgf load by the square mm area of indentation. Vickers hardness number can be calculated by using following formula:

$$HV = \frac{1.8544 F}{d^2}$$

F = applied load

d = arithmetic mean of two diagonals, d1 and d2 in mm

HV = Vickers hardness

The average hardness was calculated for each specimen.

The fluoride containing toothpastes employed in the study were colgate total advanced clean toothpaste and close up vitamin fluoride system milk calcium nutrient. Fluoride content and pH of both toothpastes was measured at PCSIR using fluoride ion selective electrode and pH meter respectively.

After that for the next 60 days, everyday each specimen of the experimental group (total 40 specimens) was exposed to 4 ml of toothpaste slurry for 2 minutes twice a day. Slurry was made using the proportion 1 gm of toothpaste and 3 ml of deionized water per specimen. For a group of 10 specimens, 10 gm of toothpaste was mixed with distilled water in order to obtain 40 ml of slurry. Using a digital weight balance (Mettler Toldeo AL204) (0.01-210 gm) 10 gm of toothpaste was calculated, the quantity was then placed in a clean glass beaker and then distilled water was added up to

**Table 1:** Restorative materials used during the study

Product	Manufacturer	Presentation: Lot no.	Expiry
Vitremer	3M ESPE	20090114	Aug 2011
Fuji II LC	GC Corp	1011171	Nov 2011

**Table 2:** Toothpaste used during the study

Toothpastes	Manufacturer	Concentration of fluoride	pH
Colgate	Colgate Palmolive (Pakistan) Ltd.	197.91 mg/kg	7.06
Close up	Unilever Vietnam for unilever Pakistan	456.04 mg/kg	6.16

the mark 40 ml on the glass beaker. Afterwards the two were mixed to achieve a smooth paste. pH of toothpaste slurry was measured before it was exposed to specimens. Following exposure to toothpaste slurry, sample was grasped with clean metal forceps and rinsed with 1 ml deionized water. After rinsing, they were kept for the next 12 hours in 5 ml of fresh deionized water. Following storage specimens were again exposed to same protocol.

Results were collected and data was inserted in SPSS version 16. One-way ANOVA was applied on each material after the data was inserted to find out whether any group was statistically significant from others. Tukey honestly significant difference tests ( $\alpha = 0.05$ ) was applied to conclude which group/groups were significantly different. All analysis was carried out at significance level of 0.05.

## RESULTS

### RMGIC I (Vitremer)

After being stored for 1 day in 5 ml of distilled water, the mean value of hardness of the specimens was 31.798 (HV/300GF). After being exposed for 60 days to Colgate (Experimental I) and Close-up (Experimental II) toothpastes slurry and storage in 5 ml of distilled water, the mean value of hardness of the specimens was 49.56 (HV/300GF) and 47.9090 (HV/300GF) respectively. The hardness of control specimens was significantly lower than experimental groups ( $p$ -value 0) (Table 3). Both neutral and acidic mediums' exposure caused significant increase in hardness and these two groups were not significantly different from each other ( $p$ -value 0.928) (Graph 1).

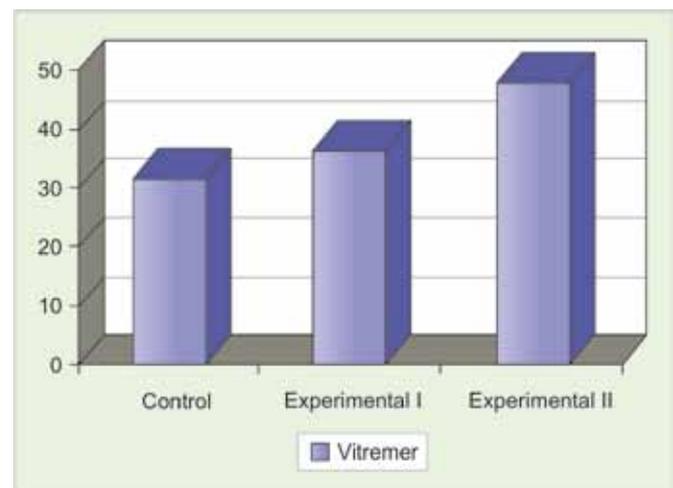
### RMGIC II (Fuji II LC)

After being stored for 1 day in 5 ml of distilled water, the mean value of hardness from the specimens was 36.36 (HV/300GF). After being exposed for 60 days to colgate (Experimental I) and close up (Experimental II) toothpastes slurry and storage in 5 ml of distilled water, the mean value of hardness from the specimens was 48.84 (HV/300GF) and 52.418 (HV/300GF) respectively. In control specimens, hardness was significantly lower than experimental groups ( $p$ -value 0). Both neutral and acidic mediums' exposure

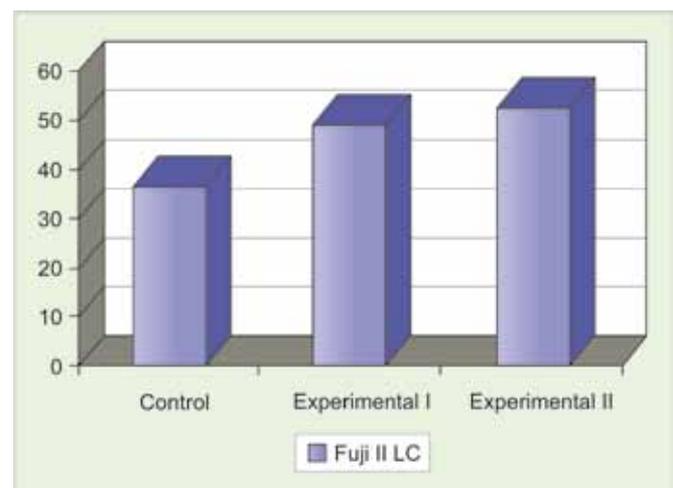
caused significant increase in hardness and these two groups were not significantly different from each other ( $p$ -value 0.512) (Graph 2).

## DISCUSSION

Hardness is an important property as in researches it is used to determine the setting/curing behavior and depth of cure of RMGIC.<sup>15-17</sup> With regard to polymerizable dental materials hardness is the characteristic which is an indicator about degree of conversion that is extent of polymerization of monomer to polymer.<sup>18</sup> Microhardness test is used for the



**Graph 1:** Comparing Vickers microhardness (HV/300GF) of RMGIC (Vitremer) of control and experimental groups



**Graph 2:** Comparing Vickers microhardness (HV/300GF) of RMGIC (Fuji II LC) of control and experimental groups

**Table 3:** Mean, highest and lowest value along with standard deviation and  $p$ -value for Vitremer and Fuji II LC

RMGIC	Experimental conditions	Mean HV/300GF	Highest value HV/300GF	Lowest value HV/300GF	Standard deviation	$p$ -value
Vitremer	Control	31.798	35.23	24.93	$\pm 2.81573$	0
	Experimental I	49.56	62.6	36.43	$\pm 8.68777$	0.769
	Experimental II	47.9	58.96	38.63	$\pm 6.31272$	0.928
Fuji II LC	Control	36.36	37.8	35	$\pm 0.97622$	0
	Experimental I	48.8411	57.06	29.6	$\pm 8.48997$	0.001
	Experimental II	52.4189	58.83	47.33	$\pm 4.55097$	0.512

indirect study of polymerization of resin based materials and for the assessment of curing unit efficiency.<sup>15</sup> In our study, Vicker's microhardness is used to determine the change in surface integrity/characteristics of RMGIC before and after exposure to fluoride rich toothpaste.

It is a capability of GIC based materials that they can release fluoride, in addition to that they can acquire fluoride ions following exposure to topical fluorides and can act as rechargeable fluoride release systems.<sup>19,20</sup> Nevertheless, the elevated reactivity of fluoride sources used in topical fluorides was found to deteriorate surface properties of esthetic restorative materials.<sup>8,13</sup> This can compromise the clinical longevity of the restoration. In our study, aim was to explore sources for recharge of RMGIC without causing deterioration in their mechanical properties. The sources used in our study did not cause deterioration in material's integrity. Both the RMGICs' hardness increased over the period of time with exposure to fluoridated toothpastes. In case of Vitremer, acidic high fluoride containing toothpaste caused more increase in hardness. While in Fuji II LC, both toothpastes were able to cause significant increase in hardness and the values for hardness of specimens after exposure to the two toothpastes were not different significantly. For the material Fuji II LC the recommended powder: liquid mass ratio is 3.2:1, and the composition has 100% Fluoroaluminosilicate glass in powder and liquid is consisted of 20 to 22% polyacrylic acid, 35 to 40% 2-hydroxyethyl methacrylate, 5 to 15% 'proprietary ingredient', 5 to 7% 2, 2,4, trimethyl hexamethylene dicarbonate, and 4 to 6% triethylene glycole dimethacrylate. This yields a filler level mass fraction of 76.2% for Fuji II LC while in case of Vitremer filler level is approximately 71% of mass fraction.<sup>21</sup> The high strength exhibited by Fuji II LC in our study as compared to Vitremer can be attributed to this relatively high filler level. The matrix of Fuji II LC has 2-hydroxyethyl methacrylate this also adds to the strength of the material.<sup>21</sup> Therefore, it is speculated that the composition of Fuji II LC is responsible for the better mechanical properties of Fuji II LC after day I during our study.

In a research entitled as 'Fluoride releasing restorative materials: effects of pH on mechanical properties and ion release', when flexural strength of Fuji II LC and Vitremer was contrasted prior and subsequent to 84 days of immersion in solution at pH 4, 5.5 and 7.<sup>21</sup> It was found that solution's pH has little effect on strength of material.<sup>21</sup> In our study, acidic and neutral pH of source of fluoride had no deteriorating effect on specimen.

With regard to diametral tensile strength of three different RMGICs, it was found that all three displayed augment in strength when comparison was made after storage for an hour to storage following one week.<sup>22</sup> After 1 hour of

storage, Fuji II LC showed better strength than Vitremer.<sup>22</sup> Following storage for 24 hours and 1 week both materials exhibited similar values of strength.<sup>22</sup> This reflects that mechanical properties of RMGICs improve after storage in water. Similar findings were observed during our study. In our study increase in hardness was observed over the period of study for both the RMGICs.

Wang et al<sup>23</sup> measured top surface hardness of several dental materials including three RMGICs.<sup>23</sup> There results following six months period established that hardness is material dependent.<sup>23</sup> It was instituted that Fuji II LC's and Photac Fil's top surface hardness decreased but Vitremer's increased.<sup>23</sup> In the case of our study, differing results to this were accomplished. As in our study, Vitremer exhibited low hardness values than Fuji II LC.

Kanchanasita et al<sup>24</sup> during a 12 months study testimonies that hardness for both Fuji II LC and Vitremer increased during the study. They investigated the consequence of long-term storage in aqueous solutions on hardness.<sup>24</sup> The specimens were either kept in distilled water or artificial saliva and hardness was computed using the Wallace microindentation tester at regular time intervals up to 360 days.<sup>24</sup> In our study also hardness was found to increase for both the materials over the period of study. They elucidated that the post-hardening reaction taking place in material overcomes the plasticising effect of water on the specimens.<sup>24</sup> It was put forward by us that the post hardening reaction led to increase in hardness witnessed in our study. When Ellakuria and coresearchers<sup>15</sup> in a 12 months study compared Fuji II LC, Photac Fil Quick and Vitremer.<sup>15</sup> They described that top surface's hardness is dependent on the material.<sup>15</sup> This was supported by the results of our study. During our study also specimens were subjected to analogous storage conditions. The disparity in values of hardness observed consequently could be characterized to the composition, filler content and quality of RMGIC.

Increase in hardness in GIC based material can be explained by exploring setting reaction which occurs in material.<sup>25</sup> After mixing, in 5 to 7 minutes, calcium polycarboxylate was structured.<sup>25</sup> In the next 24 hours aluminum polycarboxylate's was created.<sup>25</sup> It is complementary, unwavering and perks up the mechanical properties.<sup>25</sup> After photo initiation, polymerization of HEMA continued for 24 hours.<sup>25</sup> In RMGICs there was a lack of water as a result of replacement of water with water/HEMA system.<sup>25</sup> The initial set is via polymerization of HEMA and acid base reaction proceeded gradually.<sup>25</sup> Therefore, a long working time, rapid setting and resistance to water contamination was achieved.<sup>25</sup> This leads to enhancement in mechanical properties over the period of time.

## CONCLUSION

The RMGICs used in this study exhibited increase in hardness following exposure to fluoride containing toothpastes. An increase in hardness was observed in the Fuji II LC specimens exposed to toothpaste that had higher fluoride content and acidic in nature, while the toothpaste that was neutral with lesser amount of fluoride was able to strengthen Vitremer significantly. Hence, the use of fluoride-containing toothpaste can be recommended as it may recharge fluoride containing restorative materials thereby inhibit caries and enhance the mechanical properties.

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