Shear Bond Strength of Resin Modified Glass Ionomer Cement to Dentine: Effect of Nano-Sized Hydroxyapatite Particles Incorporation and Storage

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Abstract: Abstract: Objective: The objective of this study was to determine the influence of incorporating of nano-sized hydroxyapatite particles in the level of 5% concentration to a resin modified glass ionomer cement (RMGI) material on the shear bond strength to dentin surface after twenty four hours and after six months. **Materials and methods**: sixty extracted molars teeth were utilized in the study. Teeth embedded in self cure acrylic resin mold after that the occlusal surface of each tooth was cut to expose a mid coronal dentine and form a flat surface, then the dentine surface was abraded with 600 grit silicon carbide paper. Hydroxyapatite particles weighted in exact amounts with digital balance to add it to the resin modified glass ionomer powder and in order to reach the mass percent of nanoparticles in glass powder to 5%. According to the incorporation of nano hydroxyapatite particles, the teeth were randomly divided into two groups. **Group A (control):** (30 specimens) resin modified glass ionomer (RMGI) without nano hydroxyappetite particles was used. **Group B:** (30 specimens) resin modified glass ionomer (RMGI) with nano hydroxyapatite particles was used. The powder and liquid were mixed according to the instructions of manufacturer. Then the mixed material was packed into a Teflon molds placed on the dentine surface of the tooth, and packed them until they were full to create resin modified glass ionomer sample measuring 4 mm diameter and 4 mm height. According to the time of storage each group subdivided into two subgroup .Subgroup1: (15 specimens) the specimens were stored in artificial saliva for a period of 24 hours. Subgroup 2: (15 specimens) the specimens were stored in artificial saliva for a period of six months. Shear bond strength was carried out using an Instron universal testing machine. **Results:** The highest mean shear bond strength (MPa) belonged to group B at 24 hours (11.75± 0.89) followed by group B at six month (11.32± 1.17) then RMGIC specimen of group A without additives (control group) at 24 hours (5.48± 1.25) then at six month of group A (4.81± 1.21). RMGIC specimen without any additives (control group) showed significantly lowest mean shear bond strength. The difference was significant between mean shear bond of group A and group B at 24 hours and at six month ($p<0.001$) .On the other hand the difference between mean shear bond strength (MPa) at 24 hours and at six month of group A and group B was insignificant $p>0.001$. **Conclusion:** Under the limitations of this study, it appears that we may add 5% nano-sized hydroxyapatite particles to the resin modified glass ionomer powder to improve its bond strength to dentine.
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Key words: Nano-sized hydroxyapatite, Resin modified glass ionomer, Shear bond strength.

1. Introduction

Glass Ionomer cement appeared in the field of dentistry in the 1970s by Wilson and Kent. Glass Ionomer Cements (GICs) are well thought-out one of the biomaterials which are used broadly in many and different situations in dental field; mainly in the minimally invasive dentistry for the managing of early carious lesions [1, 2].

The utilization of GIC is highly common in dentistry, because of its characteristic and unique properties of (GICs), where it is chemically bond to tooth structures, it has a coefficient of thermal expansion very close to that of the sound dentin, fluoride ion release which is considered one of the most important criteria, thus contributing to the remineralization process and it has a proper biocompatibility [3].

Preliminary conventional GICs had some disadvantages as its uses are limited to areas of low forces[4] because of their mechanical properties which can't withstand high greater masticatory force. An additional disadvantage is the time-consuming setting reaction which lead to delaying the finishing and polishing of restoration. Therefore, many improvements were developed [5].

Polymerizable functional groups were added to the structure of GICs in order to improve the clinical application and physical and chemical properties of conventional GICs, which yielded resin-modified glass-ionomer cements (RMGICs) [6]. Resin-modified glass-ionomers (RMGICs) are light cured material.

(RMGICs) have the advantage of a prolonged working time associated with a rapid setting reaction and high strength. It is also easily bonded to resinous materials. It has the strength properties nearly comparable to conventional glass-ionomer cements rather than composite resins, but it the disadvantage of containing free monomers like composite resin, so is not considered biocompatible material as conventional glass-ionomer cements. [7-9].

Hydroxyapatite (HAP) is a type of calcium-phosphate [Ca_{10} $(Po4)_6(OH)_2$] which is utilized due to its resemblance with the mineral content of enamel and dentin in addition to its properties as biocompatibility, bioactivity, low solubility in water and nontoxicity[10]. Also, it is especially important because of its potential for remineralization of primary carious lesions. Nano-Sized Hydroxyapatite Particles have a higher level of crystallinity and colloidal stability due to the smaller size and higher surface charge of particles that lead to their enhanced strength and ease of application[11].

Gu et al, in 2005 incorporated a mix of nano-sized HAP and zirconium dioxide (HA/ZrO_2) with specific percentages into the conventional glass ionomer powder and reported superior compressive and tensile strengths in 4% and 12% volume percentages [12]. Moshaverinia et al, in 2008 incorporated nano-sized HAP into conventional GIC powder and showed that addition of 5 mass percent of this material caused a higher compressive, diametral tensile and biaxial flexural strengths compared to conventional cement [13, 14]. It seems that addition of nano-sized HAP to glass ionomer improves its biologic and mechanical properties. Thus, in this study we used nano-sized HAP with optimal biologic characteristics to improve the mechanical properties of resin modified glass ionomer cement. Nano-sized particles with specific mass percent in the form of needle shaped particles (which have not been studied so far) were added to the light cure glass powder and their effect on shear bond strength of the cement was evaluated under in-vitro conditions[11, 15].

In-vitro studies tend to simulate the clinical situation by different ways such as the use of thermocycling, load cycling and storage in different media and different condition during the experiment.

Durability of restorative materials can be measured by different methods. Shear bond strength considered on the most common method [16].

The aim of the current study was to determine the effect of incorporation of nano-sized hydroxyapatite particles on shear bond strength to dentin of a resin modified glass ionomer after 24 hours and after six months subsequent to storage in artificial saliva.

2. Materials and Methods

The materials used in the study were listed in table (1).

Table 1: materials used in the study

Material	Composition		Company
Resin modified glass- ionomer GC Fuji II LC® (Light cured glass ionomer restorative)	Powder: formulation –silicate glass Liquid: polyacrylic Acid: 2- hydroxyl ethylmethacrylate, urethanedimethacrylate, camphorquinone, distilled water.		GC Corporation, Tokyo, Japan
Nano –hydroxyapatite (Nano –HAP)	Calcium hydroxyphosphate		Nano Tech, Egypt
Artificial saliva	$CaCl_2$ $MgCl2.\ 6H_2O$ KH2PO4 KCl HEPES buffer pH 7.0	(0.7 mmoles/L) (0.2 mmoles/L) (4.0 mmoles/L) (30 mmoles/L) (20 mmoles/L)	Faculty of Pharmacy Zagazig University

Sixty extracted mandibular and maxillary molar teeth were utilized in the study. The teeth were selected free of any defects. All the debris were eliminated via a scaling instrument. The teeth then were kept in 0.2% thymol solution for 48 hours, after that the teeth were kept in distilled water until the study time.

Teeth then embedded in self cure acrylic resin (Vertex self-curing, Netherlands) till the cement-enamel junction level with help of custom made metallic mold. (figure1). The occlusal surface of each tooth was cut to expose a mid coronal dentine and form a flat surface, after that the surface of dentine was abraded with 600 grit silicon carbide paper (figure2). Nano-hydroxyapatite particles (table 1) weighted in exact amounts with digital balance (Mettler Toledo-AB 24) to add it to the resin modified glass ionomer powder (GC Fuji II LC®) (table 1) and in order to get to the mass percent of nanoparticles in glass powder to 5%. The obtained powder with a nanohydroxy appatite was mixed with a mortar and pestle for 20 minutes to attain homogenous of the particles in the powder [15]. Each tooth with flat dentine surface was placed in a custom made split Teflon mold supported by metallic mold to apply the resin modified glass ionomer cement (figure 3).

According to the incorporation of nano-sized hydroxyapatite particles, the teeth were randomly divided into two groups.

Group A (control): (30 specimens) resin modified glass ionomer (RMGI) (GC Fuji II LC®) without nano-sized hydroxyapatite particles was used.

Group B: (30 specimens) resin modified glass ionomer (RMGI) (GC Fuji II LC®) with nano-sized hydroxyapatite particles was used.

The powder and liquid were mixed according to the instructions of manufacturer. Then the mixed material was packed into a custom made Teflon mold placed on the dentine surface of the tooth after application of GC cavity conditioner to remove the smear according to the manufacturer's instructions. Material packed into the hole of the Teflon mold until they were full to create resin modified glass ionomer sample measuring 4 mm diameter and 4 mm height bonded to dentine surface. The specimens were immediately cured using a LED curing light (Bluephase N, Ivoclare Vivadent, India) for 10 seconds at a light intensity of 1000 mW/cm^2. The tip of the light-curing unit was placed 1 mm on top of the surface of the material [17]. Thermo-cycling was carried out for all specimens. Thermo-cycling used in the current study to simulat in-vivo thermal changes which occur intra-orally. Thermo-cycling was achieved by alternatively keeping the specimens in reservoirs of water at temperature of 5 °C and 55 °C, respectively, keeping in every reservoir for 30 seconds[18-20]. Artificial saliva was prepared for storage of specimens. The formulation for artificial saliva was depend on previous aging studies carried out by Dr. David Pashley research group (Pashley et al., 2004). The artificial saliva was prepared for the study and the pH was attuned to 7.0 with 1 N NaOH solution (table 1). It was preserved in the refrigerator until used in the study at temperature of 4C. The specimens were kept in an artificial saliva in an incubator at 37°C. The artificial saliva changed every 7 days to decrease the risk of bacterial growth [21].

According to the time of storage, each group subdivided into two subgroup:

Subgroup1: (15 specimens) the specimens were stored in artificial saliva for a period of 24 hours. Subgroup 2: (15 specimens) the specimens were stored in artificial saliva for a period of six month. The shear bond strength was carried out using an Instron universal testing machine (Conten Industries Inc, USA) the knife of the universal testing machine was directed perpendicular to the interface between the dentine and the resin modified glass ionomer [22]. The load was applied at a cross head speed of 0.5 mm/min until de-bonding occurred. The load of fracture was recorded and converted to the shear bond strength of each specimen in accordance with the subsequent equation:

Shear bond strength=Fractured load (kg) /surface area (cm^2)[19, 23]

The data were collected, tabulated and statistical analyzed by using Student's't' test for shear bond strength comparison (MPa).

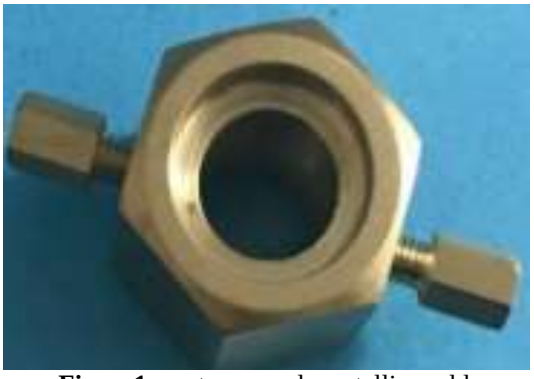

Figure1: custome made metallic mold

Figure 2: Tooth with flat dentine surface

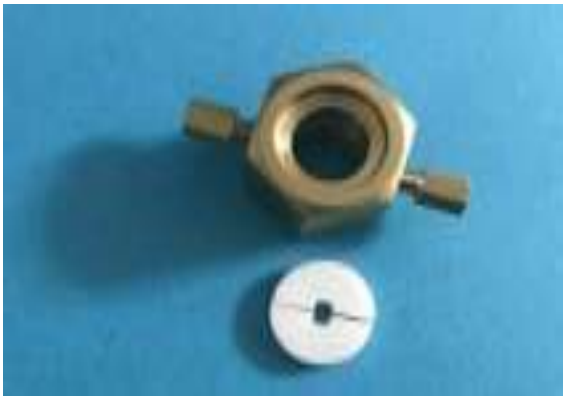

Figure 3: Teflon mold

3. Results

The mean shear bond strength values at each tested intervals both at 24 hours and at six month for two groups (A and B) and comparison of shear bond strength between; group A: Resin modified glass ionomer cement without additives and group B: Resin modified glass ionomer with 5% hydroxyapatite nanoparticles at 24 hours and at six month are shown in table 2 and figure 4.

Table 2: comparison of shear bond strength between; group A and group B at 24 hours and at six month

groups / storage time		Groups		t-Test	
		Group A (control)	Group B	t	P-value
After 24 Hours		5.48 ± 1.25	11.75 ± 0.89	-15.825	<0.001*
After 6 Months		4.81 ± 1.21	11.32 ± 1.17	-14.980	<0.001*
t-Test	t	1.492	1.133		
	P-value	0.147	0.267		

**: Significant at P≤0.001*

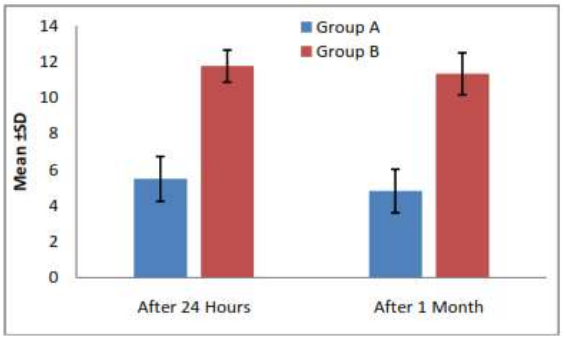

Figure 4: comparison of shear bond strength between; group A and group B at 24 hours and after six month.

The highest mean shear bond strength (MPa) belonged to group B at 24 hours (11.75±0.89) followed by group B at six month (11.32±1.17) then RMGIC specimen of group A without additives (control group) at 24 hours (5.48± 1.25) then at six month of group A (4.81± 1.21). RMGIC specimen without any additives (control group) showed significantly lowest mean shear bond strength.

The difference was significant between mean shear bond when comparison was carried out between group A and group B after 24 hours and after six months (p<0.001).

On the other hand the difference between mean shear bond strength (MPa) after 24 hours and after six months regarding group A and group B was not significant p>0.001, despite the values of shear bond strength (MPa) decreased in both groups as a result of storage.

4. Discussion

A current idea for enhancing the strength and improving the mechanical properties of glass ionomer cements is addition of nanoparticles to the material. Glass ionomer containing hydroxyapatite were introduced to the field of dental materials [24]. In the present study, we added hydroxyapatite nanoparticles to resin modified glass ionomer (RMGI) instead of the conventional glass powder. Nanoparticles due to their higher degree of crystallinity and colloidal stability have a greater reinforcing effect and easier application[25].

One of the most important properties of GICs is its bonding ability to tooth structure with no treatment of the surface before its application. Earlier studies have revealed that the shear bond strength values of the GICs to dentin surface are commonly not high, it ranged between 1 and 3 MPa and they not often more than 5 MPa.[3, 26, 27]. Results of the current study demonstrated that addition of 5% nanoparticles to the resin modified GI cement powder significantly increased shear bond strength of the resin modified glass ionomer to dentine compared to the control group [14]. These results indicate a strong reaction between cement matrix and nanoparticles. The reason for increased bond strength of GICs with nanohydroxyapatite particles may be due to different causes. One of these causes is the bonding mechanism of GICs that involves an ionic interaction among the negatively charged carboxylic groups of polyalkenoic acid and positively charged Calcium and/or Phosphate ions from the enamel surface or dentin [28]. This leads to ionic interaction among the nanoparticles and the positively charged Calcium ions of tooth structure leading to better adhesion [29].

There are other explanations which depend upon dissolution of nano-hydroxyappetite in the acidic monomer leading to the release of calcium ions from the surface of nanoparticles and increase of crystallization reactions take place that eventually results in higher strength of the cement [25].

The regularly shaped individual nanoparticles were indicated for atomic force. Nano particles would have made the mix denser which lead to enhanced and superior adhesion which considered an explanation of high bond strength [30].

Barandehfard et al., found that the addition of nano –size dhydroxyappetite to glass ionomer cement may improve the physical and biological properties of

the material and can be used for stress-bearing site cavities. The results came in coincide with the results of the current study [11].

The results of present study came in agreement with many previous studies [30-32]

Moshaverninia et al. [14] used granular nanoparticles whereas in the present study, we used nanoparticles it described according to the manufacturer as a rod-shaped paricles. In addition in our study, we used resin modified GICs instead of conventional GICs. It seems that both the above mentioned issues are responsible for the difference in values obtained in the two studies.

Evaluation of the effect of storage on the shear bond strength of resin modified glass ionomer to dentine revealed that the strength decreased with no significant difference in both tested groups after six months storage in artificial saliva and this tendency of decrease in strength was less by incorporation of 5% nano-sized hydroxyapatite particles up to 5 weight percent. The decrease in strength may be explained by the hydrolysis of the bond or dimensional shrinkage of the material due to storage and aging. The result of current study coincide with other studies [1, 11, 33, 34]. On the other hand other studies demonstrated that the bond increase by time, the dissimilarity of results may be attributed to the difference in the time of storage and storage media. It was reported that after twenty four hours of storage, maturation of the glass ionomer cement is not completely occurred which lead to the low values of bond strength. The enhancement of the bond strength after that may be due to aging which allow adequate time for complete maturation [35]. It was concluded that bond of glass ionomer to tooth structure is depend up on at first on hydrogen bonding and eventually matures and transformed into a stronger chemical bond [36]. Six months may be relatively long period of storage so, the increase in the shear bond strength was not detected.

Conclusion

Under the limitations of this study, it appears that we may add 5% nano-sized hydroxyapatite particles to the resin modified glass ionomer powder to improve its bond strength to dentine.

Recommendation

- Further studies are required to evaluate the effect of incorporation of nano-sized hydroxyapatite particles on other mechanical and physical properties of resin modified glass ionomer cement and conventional glass ionomer cement.
- Prolonged storage time is recommended in further studies.
- Different storage media is suggested to use in additional studies.

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