Influence of Addition of Nano-sized Hydroxyapatite to a Type II Glass Ionomer Cement on the Microshear Bond Strength to Dentin

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Abstract: Nano-sized hydroxyapatite nano particles (nHA) have optimal biological properties and by incorporating them into the restorative materials, we can benefit from these important properties. The present study aimed to assess the effect of incorporation of nHA on the micro shear Bond Strength of type II GIC. The objective of this study was to determine the influence of incorporating (nHA) in the level of 5% concentration to type II glass ionomer cement material on the microshear bond strength to dentin surface after twenty four hours and seven days. Material and methods: This study was conducted on 60 samples using hydroxyapatite nanoparticles and resin modified glass ionomer cement. Flat dentin surfaces were prepared and cylindrical mold was used to be filled with type II GIC with nanoparticles of (nHA) and filled with type II GIC which considered as control. At 37 °C and 100% humidity the samples were kept for 24 hours and subjected to microshear bond testing universal testing machine at twenty four hours and seven days was used to determine microshear bond strength. Results: Microshear bond strength in conventional GIC increased from a mean of 2.25 Mpa at 24 hours to 3.29 Mpa on 7th day. Microshear bond strength of GIC with (nHA) increased significantly where it was 4.28 Mpa at 24 hours to 5.85 Mpa at 7th day. There was a significant difference in microshear bond strength at 24 hours between GIC and GIC-nHA. Conclusion: Within the limitations of this study, it appears that we may add 5% nano-sized HAP to the resin modified GI powder to improve its bond strength properties. Further studies are also required to evaluate other physical and mechanical properties of the obtained material.
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1. Introduction:

Recently, Glass Ionomer Cements (GICs) are considered one of the biomaterials which are used widely in many situations in dental field; particularly in the minimally invasive dentistry for the management of early carious lesions [1, 2].

The hermetic seal of the restorations is essential and the lack of it may lead to recurrence of caries especially when the therapeutic measures used in the management of carious lesions could not completely eradicate all the presence microorganisms in the remaining carious tissues. The recurrence of caries may occur due to the remaining cariogenic bacteria, which may cause failure and loss of the restoration[3]. In addition; altering concepts using atraumatic restorative techniques depend on removal of infected and disorganized dentin and conservation of the affected and less organized dentin which has the possibility to remineraliztion[1].

The utilization of GIC is highly common in dentistry, because of its characteristic and unique properties where (GICs) chemically bond to mineralized tissues of tooth structures, 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 [4, 5].

Glass ionomer cements presented and explored an added antibiofilm and antimicrobial effect; in addition to the efficacy. This would be of substantial clinical advantage and benefit. Glass ionomer cements could guard and protect the pulp tissue from bacterial invasion via its antibacterial seal under other restorative materials [6].

To date, several methods have been proposed to improve the strength characteristics of GI cements such as incorporation of various fillers like silver cermets and stainless steel powders without good outcome under in-vitro and in-vivo conditions. Therefore, at present, the main focus in research is on improving the biological properties of this cement to be helpful in patients at high risk of caries. A new method for improving the properties of GI cements is addition of hydroxyapatite to the glass powder.

Hydroxyapatite (HAP) is a type of calcium-phosphate [Ca_{10} $(Po4)_6(OH)_2$] which is used due to its similarity with the mineral content of enamel and dentin as well as its characteristics like biocompatibility, bioactivity, low solubility in water and nontoxicity[7-9]. Also, it is specifically important because of its potential for remineralization of primary carious lesions[10]. Nano-sized HAPs have a higher degree of crystallinity and colloidal stability due to the smaller size and higher surface charge of particles that result in their improved strength and hand ease of application.

Gu et al, in 2005 incorporated a combination of nano-sized HAP and zirconium dioxide (HA/ZrO_2) with specific percentages into the conventional GI powder and reported higher compressive and tensile strengths in 4% and 12% volume percentages[11].

Moshaverinia et al, in 2008 incorporated nanosized HAP into conventional GI 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[12]. 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 as filler 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 microshear bond strength of the new cement was evaluated under in-vitro conditions.

This study sought to determine the effect of incorporation of nano-sized hydroxyapatite particles on microshear Bond Strength to Dentin of a resin modified glass ionomer.

2. Materials and Methods:

This study was conducted on 60 samples using hydroxyapatite nanoparticles (2090627, Nano Shell, India) and resin modified glass ionomer cement (Fuji II LC improved, GC Corporation, Tokyo, Japan).

Nano-sized HAPs was added to the glass powder after weight it in exact amounts with digital balance (Mettler Toledo-AB 24) and in order to reach the mass percent of nanoparticles in glass powder to 5% and the control group, nonanoparticles). The obtained powder was mixed with a mortar and pestle for 20 minutes to achieve homogenous distribution of particles.

The obtained powders were used to fabricate samples from the cements containing HAP. The powder according to the manufacturer's instructions (1 unit of liquid and 2 units of powder out of three mass percent) was mixed with liquid by a plastic spatula on a glass lab at room temperature and within a mixing time below 25 seconds. The prepared paste was used to fill up the related molds.

Human third molar teeth were collected for utilization in the current study, 60 caries-free freshly extracted teeth were used. The teeth were cleaned and any debris was removed using ultrasonic scalers and then at 37 °C the teeth were stored in distilled water until using in the study. 1.0 mm thick, dentin slice was prepared from each tooth. Using a low-speed diamond saw (Isomet®, Buehler, Lake Bluff, IL) the dentine slice was cut perpendicular to the long axis of the tooth from the upper-middle coronal portion section. During cutting water coolant was used. Silicon carbide paper up to #600 grit was used to ground the dentine slice to prepare a flat dentin surface [13-16].

The prepared dentin slices were then randomly divided into two main groups (containing 30 each) in relation to the incorporation of nanoparticle of hydroxyapatite in resin modified glass ionomer. Group A: resin modified glass ionomer without additives and Group B: resin modified glass ionomer with 5% hydroxyapatite nanoparticles [15].

The teeth were embedded in cold cure acrylic resin where the root of each tooth with prepared flat dentin surfaces was fixed in the acrylic resin till the height of CEJ. GIC was mixed according to the manufacturers' instructions, then The prepared GIC mixture was packed into a cylindrical plastic moulds placed on the dentine surface of the tooth, and packing them until they were full to create cylindrical GIC specimens measuring 5 mm diameter and 4 mm height [14].

Thermo-cycling was carried out after the specimens were kept in water at 37 °C for a period of 24 hours. Thermo-cycling which was used in the current study simulated in-vivo thermal changes which occur intra-orally. Testing was carried out, in accordance with the American National Standards Institute/American Dental Association (ANSI/ADA)[17] and the International Organization for Standardization (ISO) [18] for direct filling resins and dental adhesions. Thermo-cycling was achieved by alternatively keeping the specimens in water reservoirs at temperature of 5 °C and 55 °C, respectively, remaining in each reservoir for 30 seconds[19].

The microshear bond strength was calculated for each specimens whose diagram is shown in Fig 1. Each resin cylinder attached to dentin slice was located in the lower attachment part of the universal testing machine (Model LRX Plus II; Lloyd Instruments Ltd., Fareham, UK) for testing microshear bond strength. Around each resin cylinder a thin wire (diameter 0.20mm) was looped, creation contact throughout half of the cylinder base and was located as near as possible to the resin dentin interface [15]. A force of shear was subjected to each sample at a crosshead

speed of 0.5mm/min until failure takes place. The resin-dentin interface of the specimens and the wire loop were aligned as straight as possible to ensure that the same orientation in shear was maintained.

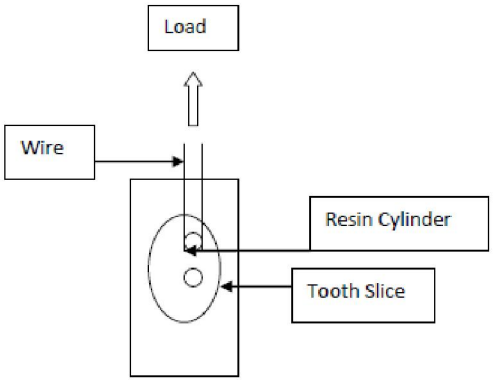

Fig 1: Diagram of the Microshear Bond Test

Half of each group, half of the samples (n=15) were tested and examined after 24 hours. The other half (n=15) were tested at the 7th day. (Area of the sample =19.64square mm).

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

3. Results:

The mean microshear bond strength values at each tested intervals both at 24 hours and 7 days for two groups (A and B) are presented in Table 1.

Table2. shows comparison of microshear 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 7 days.

The highest mean microshear bond strength (MPa) belonged to group B at 7 days (5.85+ 1.17) followed by group B at 24 hours (4.28+ 0.89) then RMGIC specimen Group A without additives (control group) at 7 days and at 24 hours (3.29+ 1.21), (2.25+ 1.25) respectively. This increase was statistically significant (P<0.05). RMGIC specimen without any additives (control group) showed significantly lowest mean microshear bond strength.

The difference was significant between mean microshear bond strength at 24 hours and 7 days (p<0.05)

Table 1. Mean values of microshear bond strength of samples of group A and B at 24 hours and 7 days.

Groups	Mean microshear bond strength value (Mpa)	
	At 24 hours	At 7 days
Group A	2.25	3.29
Group B	4.28	5.85

Table 2. Comparison and assessment of microshear bond strength between Group A (RMGIC) and Group B (RMGIC-nHA) from 24 hours to 7 days

Groups	Group A	Group B	Student's Unpaired 't' test value	P value
	Mean ± SD	Mean ± SD		
24 hours	2.25± 1.25	4.28±0.89	2.49	0.001*
7 days	3.29± 1.21	5.85±1.17	0.46	0.003*

**: Significant at P≤0.05*

4. Discussion:

A contemporary idea for increasing the strength and improving the mechanical properties of glass ionomer cements is addition of nanoparticles to the cement matrix. Glass ionomers containing hydroxyapatite were introduced to the field of dental materials awhile ago[12]. In the present study, we added hydroxyapatite nanoparticles to 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 [20].

Bonding can be explained as a criteria by which two surfaces are attached by chemical-physical or chemical means. One of the most important properties of GICs is its bonding ability to tooth structure with no surface pre-treatment of the tooth surface [21]. preceding studies have revealed that the shear bond strength values of the GICs to dentin are generally not high, ranged between 1 and 3 MPa and they seldom more than 5 MPa. The values be likely to be higher in the test of microshear as a result of the differences in reduction and stress distribution when compared with shear tests in bonding area [22,23]. Results of the current study demonstrated that addition of 5% nanoparticles to the resin modified GI cement powder significantly increased microshear bond strength of the cement compared to the control group as microshear bond strength increased from 2.25 to 4.28 MPa at 24 hours and increased from 3.29 to 5.85 MPa at 7 days [21].

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 [23]. This leads to ionic interaction among the nanoparticles and the positively charged Calcium ions of tooth structure leading to better adhesion [24].

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 higher occurrence of crystallization reactions 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 [26,27].

Barandehfard et al [28] found that the addition of nanosized hydroxyappetite 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. The results of this study came in agreement with many previous studies [29-31].

In our study, we used resin modified GI instead of conventional GI. Also, Moshaverninia et al, [12] used granular nanoparticles whereas in the present study, we used nanoparticles manufactured by Nano Shell Company that, according to the manufacturer, are rod-shaped. 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 aging on strength properties revealed that cement strength gradually increased from day one to day 7 and this trend of increase in strength occurred by addition of nano-sized HAP by up to 5 weight percent this findings came in agreement with the study conducted by Moheet et al., 2018 [32]. The study suggested that, the resin modified glass ionomer with nanosized hydroxyappetite can be considered as a prospective restorative material with improved properties.

Mixing the glass powder and acidic monomer is still relatively complete and it seems that progression of acid-base reactions eventually results in improved strength.

Conclusion

Within the limitations of this study, it appears that we may add 5% nano-sized HAP to the resin modified GI powder to improve its bond strength properties. Further studies are also required to evaluate other physical and mechanical properties of the obtained material.

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