Original Article

The Effect of Incorporation of 0.5 %wt. Silica Nanoparticles on the Micro Shear Bond Strength of a Resin Modified Glass Ionomer Cement

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ABSTRACT

Statement of the Problem: The clinical success of glass ionomer restorations depends on the strength of resin-modified glass ionomer (RMGI) cement bonding to dentin and there is limited information available regarding the bond strength of resin modified glass ionomers containing silica nanoparticles to dental structures.

Purpose: The aim of this study was to compare the microshear bond strength (μSBS) of RMGI with and without silica (SiO2) nanoparticles to dentin of permanent teeth.

Materials and Method: In this experimental study, the occlusal surfaces of 30 freshly extracted intact third molars were ground to expose the flat dentin and after conditioning with 20% poly acrylic acid, were randomly assigned to two main groups (n=15). The first group was filled with RMGI (Fuji II LC, GC) and the second group was filled with RMGI plus 0.5%wt. silica nanoparticles. Then, each main group was divided into three subgroups, and then stored in an incubator at 37 ^{o}C with 100% humidity for 1, 7, and 30 days. The μSBS test was performed using a universal testing machine (1 mm/min). The data were analyzed by t-test, repeated measures ANOVA and Tukey test ($p< 0.05$).

Results: There were no statistically significant differences between the mean μSBS of the groups with and without nanoparticles along the different storage periods ($p> 0.05$). There was significant difference in μSBS values among the three different storage periods in all the tested materials ($p< 0.05$).

Conclusion: Incorporation of 0.5 %wt. silica nanoparticles did not compromise the μSBS of Fuji II LC RMGI to dentin.

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Introduction

Glass ionomer cements have certain properties such as fluoride release and chemical bonding to the tooth substrate. However, glass ionomer cements have some limitations such as low wear resistance and poor physical and mechanical properties. Many attempts have been made to overcome drawbacks, which include the addition of different fillers [1-2]. To improve the mechanical properties of conventional glass ionomer, resin modified glass ionomer (RMGI) have been developed in 1980. Despite advantages of RMGI, its short comes have limited its usage only to non-stress bearing areas [3-5].

Many efforts have been made in order to improve the mechanical properties of RMGI, such as incorpora-

tion of silica as an inorganic fillers resulted in its physical and mechanical properties improvement including increasing the compressive strength, diametral tensile strength and flexural strength and decreasing marginal gaps, water uptake, water solubility and micro leakage [6-10]. However, inert particles of silica have no interfere in setting reaction of cement. Recent studies demonstrated its antibacterial effect and the ability to adhere to matrix by chemical bonding and reinforced the structural matrix of cement [11-12].

Some authors used different nanoparticles (nanoZnO, nanoHA, and nanoTiO_2) to improve the mechanical, physical, and biological properties of RMGI, and have obtained encouraging results [13-15]. It has also been suggested the incorporation of silica nanoparticles to resin matrix could improve mechanical and physical properties of composite resins [16-17].

The latest development in glass ionomer technology is nano ionomer that incorporated nanosilica / zirconia particles to RMGI [3]. Few studies demonstrate that nano ionomer have better properties, such as higher aesthetics, abrasion resistance, optical properties and adhesion [3, 16, 18]. Moreover, according to previous study, adding nanosilica can improve the mechanical properties of commerical GIC Fuji II by optimum concentration of 0.5 wt. percentage [19]. Apart from the obvious enhancements, until now, bonding properties of RMGI containing nanosilica particles has not been reported. On the other hand, the changes in material composition may affect its bonding characteristics, and makes it unsuitable as a restorative material. Thus, the research hypothesis that has been tested was that incorporating nanosilica to RMGI would adversely affect its μSBS to dentin.

Therefore, the objective of present study was to evaluate the effect of incorporation of 0.5%wt of nanosilica particles on μSBS of RMGI. Based on the null hypothesis of the study, nanosilica particles would have no effect on the bond strength of RMGIC to dentin even in long-term.

Material and Method

Preparation of experimental cement

A commercially available RMGI, Fuji II LC improved (GC, Tokyo, Japan), with a recommended powder to liquid ratio of 3.2/1 was used in this study as the control and base material. The specific amount of nanosilica particles Fumed SiO_2 (Aerosil 200, Evonik Germany) consists of spherical particles, with a specific surface area of 200 m^2 g^{-1} and mean diameter of 12 nm weighted by a digital scale (Mettler Toledo-AB 204) and added to glass powder in order to achieve the 0.5 weight percentages of nanosilica in glass powder. For the sake of homogenous distribution of particles, powders were hand mixed by mortar and pestle for 20 minutes [15]. Two experimental powders were prepared including RMGI with 0 wt. % of nanosilica (as control group), and RMGI plus 0.5 wt. % of nanosilica.

Micro-shear bond strength (μSBS) and failure analysis

Thirty intact human thirds molar teeth extracted within past six months were selected, cleaned and stored in thymol solution for a week, then were stored in distilled water at 4^{o}C till the experiment. Each tooth was embedded in self-polymerizing acrylic resin in the manner that occlusal surface was accessible for testing. Occlusal surfaces of the teeth were transversally grounded by a diamond bur (Tizkavan, Iran) to 1 mm below the central groove, then polished with 600, 1000 and 1200 grit wet silicon carbide abrasive paper (3M, USA) to achieve a smooth dentin surface. Then occlusal surfaces were conditioned with cavity conditioner (GC, Tokyo, Japan) using a micro brush for 10 seconds based on manufacturer instructions, then washed for 20 seconds, and dried by cotton pellet. Lastly, teeth were divided in two group defined as RMGI without any additive (control), and RMGI with nanosilica. Then 0.5 %wt. nanosilica was added to RMGI powder and mixed with RMGI liquid according to manufacturer instruction. Silicon tubes with an internal diameter of 0.9mm and a height of 1mm were fixed on a thin glass slide by sticky wax, used as mold, and filled from the one side. Thereupon specimens were positioned on dentin and light cured for 40 seconds using light emitting diode (Demetron LC, SDS Kerr, USA). Each group subdivided into three subgroups (one day, one week, and one month) and stored in incubator (Automatic CO2 Incubator, NuAire, Inc.) at 37^{o}C with 100% humidity. Three tubes were placed per tooth and 15 specimens were prepared in each subgroup [20-21]. Just before testing, the mold was removed by a scalpel blade #11, and a thin steel wire with a diameter of 0.2mm was looped around each cylindrical sample in touch with the lower half-circle of

the cylinder [22]. The shear force was applied by pulling the wire loop up using a universal testing machine (Zwick Roell, Germany) at crosshead speed of 1 $mm.min^{-1}$ until failure occurred. The bond strength values were calculated as ratio of the maximum load required to the bonded surface area and reported in MPa [21]. Then the fractured specimens were observed under a stereomicroscope (40×) to find out the mode of failure, which were classified as adhesive, cohesive, and mixed failure.

Statistical analysis

Recorded values were analyzed with SPSS software (version 16) using the repeated measures ANOVA, t-test and Tukey post hoc HSD. The level of significance was set at 0.05.

Results

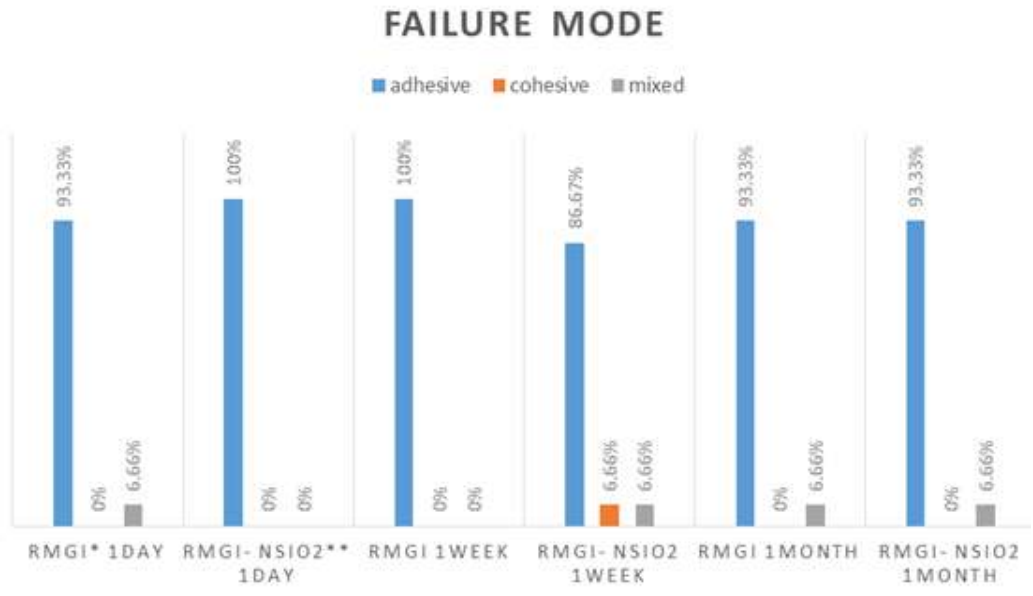
The means and standard deviations of µSBS of two groups (control and containing 0.5 wt. % $nSiO_2$) on different time intervals are shown in Table 1. The maximum and minimum µSBS mean values were recorded at third (one month) and first (24h) intervals for both groups, respectively. Concerning each tested group or material, as shown in Table 1, there were no significant differences between µSBS values of two groups along the three different storage periods (p= 0.563, p= 0.147 and p= 0.995, respectively). The statistical analysis of variance (using repeated measures ANOVA) considering the storage time as a factor revealed a significant difference in µSBS values among the three different storage periods in both tested groups as the amounts of bond strength increased by time pass. The µSBS of group1 at one month was significantly higher than 24h and one week (p< 0.001 and p< 0.006 respectively) which were significantly different compared to each other (p< 0.005). Also the µSBS of group 2 at one month was significantly higher than 24h and one week (p< 0.0001 and p< 0.002 respectively) which were significantly different compared to each other (p< 0.001).

Table 1: Mean and standard deviation (SD) of micro shear bond strength (MPa) of two tested groups

Groups (n=15)	1 day	7 day	30 day
	Mean(SD)	Mean(SD)	Mean (SD)
RMGI*	10.6(2.66)^{aA}	12.66(2.31)^{aB}	16. 35(4.95)^{aC}
RMGI- 0.5 wt.% $nSiO_2$**	10.03(2.64)^{aA}	14.48(4.22)^{aB}	16.34(3.97)^{aC}

*RMGI: resin modified glass ionomer
**$nSiO_2$; Silica nanoparticles
Differences in capital letters indicate statistically significant differences within rows, and differences in lowercase letters indicate statistically significant differences within columns (p< 0.05).

The results of the microscopic investigation in the different subgroups are shown in Figure 1. Microscopic examination of interfacial debonding revealed that the majority of failure modes were adhesive failure followed by mixed failure, while cohesive failure was not found in any of the samples except for the group 2 at second interval that one cohesive failure was observed.



*RMGI: resin modified glass ionomer **nSiO2; Silica nanoparticles

Figure 1: Frequency bar chart of fracture modes of two sample groups at three different intervals

Discussion

In recent decade, the employment of nanotechnology in dentistry has vastly developed. Incorporation of different nanoparticles to the cement matrix is a novel idea for enhancing the strength and ameliorating the mechanical properties of glass ionomer cements [10, 14-15].

The composition of the restorative materials can interfere with their bond strength; some studies have demonstrated that incorporation of nanoparticles to glass ionomer would limit the acid base reaction of the RMGI and interfere with the chemical adhesion [21, 23-24]. There bond strength to dental structures decreases when a large amount of fillers is added. Since silica particles did not bond with the RMGI matrix, they may act as an impurity that prevents the reaction in cement [25].

This *in vitro* study was performed to evaluate the effect of incorporation of silica nanoparticles on μSBS of RMGI cement, since it was reported that bond strength testing could determine valuable clinical information, when accumulated in a well-controlled design [24]. *In vitro* studies, testing bond strength of glass ionomer cements, typically have large standard deviations that consequently challenge inter-study evaluation and appraisal [26].

Shear bond strength is a simple and widely used test to assess the bonding performance of restorative material, particularly regarding the glass ionomer cements, which present low bond strength [20-21, 24, 26]. Recently, the μSBS test has become popularized as an alternative to the conventional shear bond test. In the μSBS test, the stress distribution is more concentrated at the interface compared with the conventional shear bond test. This would decrease the chance of cohesive failure in the material or enamel/dentin that does not display the true interfacial bond strength [20-21, 27-28]. This method is an especially useful test for those substrates that are susceptible to the specimen preparation effects and micro tensile bond strength testing conditions, such as glass ionomer or enamel [21, 28-29]. However, there are some questions concerning the interdependence of multiple specimens from the same tooth in micro test, which may exaggerate the statistical significance levels for comparison between materials. It is highly possible that the measurements originating from one tooth would be biased by the individual features of the tooth, which the test was carried on [20].

In the current study, the mean μSBS of Fuji II LC RMGIC to dentin in control group at first interval (24h) was 10.6±2.66 MPa that was similar to previously reported values for RMGIC [5, 30]. On the other hand, the mean μSBS of RMGIC plus nanosilica at first interval was 10.03±2.64 MPa that was nearly similar to control group and higher than previously reported values for nano ionomer [21, 30]. It indicates that incorporation of nanosilica had no influence on the short-term μSBS of RMGIC. Furthermore, μSBS was recorded after three different storage periods (24 h, 1 week, and 1 month) in order to evaluate the effect of time on bond strength of glass ionomer materials. Both groups showed a significant increase in the μSBS over time. This result can be explained by incomplete maturation of the glass ionomer cement after 24 h of storage that reasons for the lowest values of bond strength. The improvement of the bond strength after one week and one-month storage was due to aging that allows sufficient time for complete cement maturation. This was supported by a previous study [24], which reported that adhesion between glass ionomer cement, and tooth structure is based initially on hydrogen bonding, matures over time, and develops into a stronger chemical bond [23-24, 31].

There was no significant difference between two groups at each test time. Therefore, the null hypothesis regarding the long-term μSBS was accepted. This finding may be explained by the incorporation of inert particle of nanosilica, which not silanized, could not interfere with bonding reaction of cement. On the other hand, the amount of nanoparticles added was very small and might not have detrimental effect on bond strength of cement. Similar finding obtained with nanoTiO_2 addition in Garcia-Contreras *et al.* [14] and Elsaka *et al.* [32] studies, which reported incorporation of nanoTiO_2 to powder of glass ionomer does not interfere with the shear bond strength to dentin. Dissimilar results were obtained in previous studies that evaluated bonding effectiveness of a commerical nano-filled RMGI (Ketac N100) which is claimed contains nanoclusters of silica fillers and stated that the non-primed nano-filled RMGI bonded less effectively than conventional RMGI. This incongruity might be due to different utilized test methodology and materials. In spite of conventional RMGI cement, nano ionomer is not able to bond to dental

structure simultaneously and need to Ketac primer (Ketac primer; 3M ESPE, USA) to improve the wettability of dentin and monomer penetration into dentin substrate. It could be speculated that nano ionomer perhaps behaves more like a resin composite rather than a true glass ionomer [3, 33-34]. Additionally, Ketac N100 (Ketac N100/Ketac Nano; 3M ESPE, USA) is based on a prior RMGI from the same manufacturer (Vitremer), which was lower than Fuji II LC in terms of shear bond strength [3, 5, 34]. In other ways, lower bond strength of non-primed nano ionomer to dentin, may be related to the very superficially interaction of nano ionomer with dentin without evidence of demineralization and/or hybridization [3, 33]. In our study, we utilized poly acrylic acid as cavity conditioner that led to partially demineralization of smear layer and enhanced HEMA penetration to dentin [3, 35]. Generally, the various bond strength values found in different studies can be possibly attributed to several factors such as the type of material, application method, tooth preparation methods, storage conditions, and aging protocols [21, 24, 26].

In this study, all specimens were assessed under a stereomicroscope following μSBS testing to assess the mode of fracture and it was revealed that most fractures were of adhesive type, which indicates that the values obtained were clearly the strength of the bonded interface. This finding is similar to previous studies [6, 21-22, 27]. It may be related to the method of testing, which produced fewer cohesive failures. In addition, the results of stereomicroscopic assessment confirmed the results of μSBS tests. It was also suggested that under higher magnifications, the incidence of cohesive and mixed failure modes might have been increased. Scanning electron microscope examination is surely advisable but costly and time consuming. However, it remains contemplative how failure site descriptions should be figured out [24, 26].

At last, the obvious effect of incorporation of 0.5 %wt. nanosilica on flexural and compressive strength of RMGI [19] cannot be neglected. Along with the conclusions drawn from the present *in vitro* study, it can be fairly said that nanosilica added RMGI holds a promise to be employed as a restorative material particularly on high stress bearing areas. It is noteworthy that the silanized spherical silica fillers strongly interact with RMGI matrix and decrease the diffusion rate of fluoride [25]. Although in present study non-silanized nanosilica particles have been used, it may have influenced the fluoride release properties of RMGI. This effect should be taken into consideration and could be assessed in future studies.

Although we tried to simulate clinical conditions, there are always limitations associated with any *in vitro* studies such as the inability to simulate the biologic changes such as masticatory forces and chemical attacks by acids and enzymes that challenge the durability of restoration in the oral cavity. In order to assess the validity of novel cement in clinical application, further *in vitro* and *in vivo* investigations should be carried out to test the effect of the complex oral environmental conditions on the mechanical properties and chemical adhesion to the different tooth substrates.

Conclusion

On the basis of this study, it was determined that incorporation of certain weight percentage of silica nanoparticles to Fuji II LC RMGI cement had no significant change in its μSBS.

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Conflict of Interest

There is no conflict of interests in this research.The research was funded by Dental Research Center of Shahed Dental School (Tehran, Iran).

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