

Original Article

Microshear Bond Strength of Nano-filled Resin Composite to Remineralized Tooth Tissues Using Nano-Bioactive Glass

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Abstract

Aim: This study aimed to assess micro shear bond strength of composite resin bonded using self-etch adhesive to dentin either before or after remineralization with Nano bioactive glass. **Materials and methods:** A total number of 25 extracted caries-free human permanent molars used. 5 specimens as a positive control (sound dentin), the remaining 20 specimens demineralized. The demineralized dentin divided into: 5 specimens' negative control (demineralized dentin) and the remaining 15 specimens divided into 3 groups (5 each) according to the period of application of Nano bioactive glass particles either one day, one week and one month. Composite resin was bonded to the dentin surface using universal self-etch adhesive and the teeth were tested for micro shear bond strength using a universal testing machine. **Results:** Nano bioactive glass showed statistically significant lower results than sound dentin and higher results than demineralized dentin. Different times of application showed more remineralization effect with the highest effect for the one-month application. **Conclusion:** Nano bioactive glass as remineralizing agent improved dentin bond strength after remineralization and the effect is greater with time.

Keywords: Nano bioactive glass, dentin, micro shear bond strength, remineralization, composite resin, self-etch adhesive.

I. INTRODUCTION

In the oral environment, the tooth substrates are usually exposed to episodes of demineralization and remineralization through different masticatory activities. In a neutral environment, the hydroxyapatite of the tooth is in balance with saliva, which is flooded with calcium and phosphate ions. Different minerals or ionic technology used for remineralization such as Bioactive glasses. Bioactive glasses are biocompatible materials, which can form hydroxyapatite layer if satu-

rated with the body fluids¹. Bioactive glasses can be used as porous or dense constructs in different purposes such as remineralization or hypersensitivity treatment by occluding the dentinal tubules. Adding different elements into the glass can stimulate other functions of bioactive glasses such as antibacterial properties, furthermore, can be used to adjust biocompatible materials turning them to be bioactive².

Bioactive glasses can be prepared with nanoparticles sizes which enhances its

properties more owing to the Nano size. Nanoparticles help in increasing the concentration of the material through more surface area available with smaller particle sizes³. Self-etch adhesives has been proposed to maintain mineral contents of remineralized as well as demineralized hard tooth tissues to fulfill the concept of minimal invasive dentistry. The simplicity of application of self-etch adhesive encourages most of dental practitioner to expand its use in many clinical applications⁴. By understanding the demineralization-remineralization process, together with making use of the Nanotechnology, and the benefits of the bioactive glass; it was beneficial to conduct this study to assess the bonding efficiency of resin composite bonded using self-etch adhesive to dentin either before or after remineralization with Nano bioactive glass.

II. MATERIALS AND METHODS

A total number of 25 extracted caries-free human permanent molars were used. 5 specimens as a positive control (sound dentin), the remaining 20 specimens demineralized. The demineralized dentin divided into 5 specimens' negative control (demineralized dentin) and the remaining 15 specimens divided into 3 groups (5 each) according to the period of application of Nano bioactive glass particles either one day, one week and one month.

Each tooth was marked on the lingual side with a pencil, so it could be easily distinguished after embedding it in rectangular transparent auto-polymerized acrylic resin block (Acrostone, Egypt). Using a grinding machine (EmmeviSpA- BadiaPolesine, Italy) with continuous water irrigation, flat dentin surface was obtained on the marked lingual side. Then, the exposed dentin surface was polished with 600-grit Silicon Carbide (SiC) paper to create a standard smear layer.

All dentin specimens- except the positive control specimens- were demineralized by suspending into glass tubes containing 20 ml of demineralizing solution for 72 hours, in an incubator at a temperature of 37 degrees Celsius. The demineralizing solution was prepared at the pharmacology department-faculty of pharmacy- Cairo University, Egypt. It is composed of Calcium Chloride = 2.2 mM, Monosodium Phosphate = 2.2 mM, Lactic acid

= 0.05 M Fluoride = 0. 2 ppm, adjusted with 50% Sodium Hydroxide to a pH (4.5)⁵.

Twenty milligrams (0.02 g) of Nano-bioactive glass (Nano Streams – 6th October city – Egypt), composed of 45 wt% Silica, 25 wt% Calcium Oxide, 25 wt% Sodium Oxide and 5 wt% Phosphorus Penta oxide, was mixed with one milliliter of the artificial saliva then the mixture applied on the demineralized surface of each specimen, rubbed with sterilized cotton bud for one minute. For the one-day group the procedure was done once, while for the one week and one-month groups the procedure was repeated for seven and thirty days respectively. The specimens were kept in artificial saliva (pH 7.2) between the applications.

The artificial Saliva was prepared at the pharmacology department- faculty of pharmacy- Cairo University, Egypt. It is composed of Trisodium Phosphate - 3.90 mM, Sodium Chloride - 4.29 mM, Pottassium Chloride- 17.98 mM, Calcium Chloride - 1.10 mM, Magnesium Chloride - 0.08 mM, Sulfuric acid - 0.50 mM, Sodium Bicarbonate - 3.27 mM, distilled water. The artificial saliva was changed every 24 hours⁶.

Self-etch adhesive system Single bond universal adhesive (3M ESPE, St Paul, MN, USA) was used according to manufacturer's instructions. Three micro-tubes (Tygon® tube) were mounted and held in place with a tweezer on the uncured adhesive to restrict the bonding area. The adhesive coat was then light cured for 10 seconds using a LED light-curing unit (Elipar S10, 3M ESPE, St Paul, MN, USA), After curing of the adhesive, the composite resin (Filtek™ Z350 XT) was packed in the micro-cylinders using endodontic plugger. After curing, the polyethylene micro-tubes were peeled away obtaining composite micro-cylinders of 0.8 mm diameter and 1 mm height bonded to the specimen surface. The specimens were stored in distilled water for 24 hours prior to micro shear bond strength (MSBS) testing. MSBS testing was done using a universal testing machine (Lloyd Instruments Ltd., Fareham UK).

For statistical Analysis, data presented as mean and standard deviation (SD) values. Data explored for normality using Kolmogorov-Smirnov and Shapiro-Wilk tests. μ -Shear Bond Strength showed normal

distribution, One Way-ANOVA test used to compare between different tested groups and follow-up periods on mean μ -Shear Bond Strength Followed by Tukey's' post hoc test for pairwise comparison. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM® SPSS® (SPSS Inc., IBM Corporation, NY, USA) Statistics Version 22 for Windows.

III. RESULTS

Mean values for MSBS in different groups and different application times are presented in Table 1. There was a statistically significant difference in MSBS values of sound dentin to demineralized dentin. Nano bioactive glass showed significantly different MSBS values compared to both sound and demineralized dentin in all periods of application except the one month, where it was only significantly different than demineralized dentin.

Table (1): Mean MSBS values for different groups and different application times

	Control samples		Nano bioactive glass			P value
	Sound dentin	Demineralized dentin	One day application	One-week application	One-month application	
Micro shear bond strength Mean (SD)	23.7 (1.54) ^a	11.63 (1.08) ^d	15.07 (1.72) ^c	17.4 (1) ^b	22.32 (2.08) ^a	$\leq 0.001^*$

IV. DISCUSSION

Bioactive glasses have unique remineralizing properties and are generally introduced into various dentifrices as very fine particles to provide calcium and phosphorus to the tooth surface. Bioactive glasses (BAGs) as opposed to most technical glasses are characterized by the reactivity in water and in aqueous liquids. The bioactivity of BAGs is derived from their reactions with tissue fluids resulting in the formation of hydroxy-carbonate apatite layer on the glass ².

Artificial saliva was used as a storage medium in this study to simulate the washing effect resembling the natural oral conditions. The storage medium was changed every 72 hours with fresh artificial saliva. It was also chosen, as the bioactive glasses need calcium and phosphorus rich medium to enhance the precipitation of hydroxy apatite crystals promoting the bioactive glass action ^{7,8}.

Regarding the adhesive system, Single Bond Universal (3M ESPE) was used in this study. It was developed by the manufacturer to be universally used in different approaches, either etch-and-rinse, self-etch or selective enamel-etch. This adhesive system offers a unique composition; it composed of 10-MDP, which is a phosphate monomer that gives the adhesive an acidic character enabling

simultaneous demineralization and monomer infiltration ⁹. In addition, 10-MDP is responsible for the chemical bonding that allows the superior bond strength over other adhesive systems as concluded by a recent systematic review and meta-analysis of in vitro studies of self-etch dental adhesives ¹⁰

Micro-shear bond strength test using specimens with reduced dimensions was obtained as a substitute for the conventional shear test. This test is better as it allows testing small areas thus permitting a regional mapping or depth profiling of different substrates and preparing multiple specimens on the same tooth surface. This testing method also provides better stress distribution that can be accomplished in smaller specimens, since the number of voids and stress- raising factors is lower than the ones that occur in larger areas used for shear or tensile bond strength tests ^{11,12}.

MSBS mean values showed the highest value in sound dentin and the lowest value in demineralized dentin. Sound dentin has normal mineral contents and calcification without any morphological changes that allowed the universal adhesive to bond to them excellently and the reduction of bond strength in demineralized samples might be due to mineral loss and changes in the calcium-phosphorus concentrations after demineralization¹³. Remineralization of the demineralized enamel

and dentin using Nano bioactive glass repaired the defective dentin surface both chemically and morphologically restoring some of the bond strength to the tooth substrate. These results agree with other previous studies¹⁴⁻¹⁶.

The one-month groups showed the highest MSBS values which was not statistically significant to the sound dentin values which could mean that after one month the dentin is probably back to normal mineral content. Second highest value was the one week followed by the one-day values which were significantly lower than sound dentin and higher than demineralized dentin. Which could mean that period of application is an important factor in remineralization and bond strength regaining over time of demineralized dentin. This could be explained by the increased amount, as well as the concentration of the remineralizing agent used over the application period. Additionally, the specimens were stored in artificial saliva between the applications of the remineralizing agents that allowed precipitation of the minerals present in the saliva on the tooth substrate improving the remineralization capability. These results are in agreement with previous studies^{6,17,18}.

The bond strength values were improved significantly with all application periods with maximum improvement after one month that nearly restored the values as with sound enamel (sound) and dentin (sound). This might be due to the efficient remineralization competence of the Nano bioactive glass that could be ascribed by its porous structure with the increased surface area improving the rate of HAP layer formation and more rapid and deep penetration into the demineralized enamel surface due to its small particle size. These results agree with previous studies^{19,20}.

V. CONCLUSION

Under the conditions of this study the following could be concluded:

1. Resin composite bond strength is significantly affected by the tooth substrate condition.
2. Using the nanotechnology in the form of Nano bioactive glass as remineralizing agent improved its remineralization capacity for dentin.
3. Application time of the remineralizing agent has a profound effect on its remineralization competence.

Conflict of Interest:

The authors declare no conflicts of interest.

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Ethics:

This study protocol was approved by the ethical committee of the faculty of dentistry-Cairo university on: 28 February 2023.

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