Dental Research Journal

Original Article

Effect of bioactive glass-containing dentin adhesives on microshear bond strength of composite restorations

Sayed Mostafa Mousavinasab[1], Farzad Sarandi[1], Parsa Rezvanian[2], Mohammad Atai[3], Sepidehsadat Mousavinasab[4]

[1]Department of Operative Dentistry, Dental Materials Research Center, Dental Research Institute, Isfahan University of Medical Sciences, [2]Department of Animal Biotechnology, Cell Science Research Center, Royan Institute for Biotechnology, ACECR, Isfahan, [3]Department of Polymer Science, Iran Polymer and Petrochemical Institute, Tehran, Iran, [4]Department of Conservative Dentistry, Faculty of Dentistry, MAHSA University, Selangor, Malaysia

ABSTRACT

Background: In general, bioactive glasses (BAGs) can react with tissue minerals and promote remineralization. However, the application of BAG in bonding agents and its impact on bond strength remain uncertain due to insufficient information and limited research in this area.

Materials and Methods: This study employed a randomized controlled design to assess the effects of composite-bonding agents with varying BAG contents on shear bond strength and fracture pattern in sound and demineralized teeth, with and without thermocycling. Thus, 80 healthy third molars were randomly divided into two groups: sound teeth and demineralized teeth. Five bonding agents were applied to the prepared dentin surfaces, including four experimental composite-bonding agents with varying BAG content (0, 0.2, 0.5, and 2 wt%) and the Adper Single Bond commercial bonding as control. The shear bond strength of all samples was measured using a universal tester. The type of failure of each specimen was determined using a stereomicroscope. Kruskal–Wallis nonparametric test was performed on the obtained shear bond strength data followed by Mann–Whitney *post hoc* test with Bonferroni correction to determine statistical significance. The level of significance was considered $P \leq 0.05$ for all tests and was adjusted by Bonferroni correction.

Results: Demineralization significantly decreased shear bond strength in the teeth samples. Adper Single Bond exhibited the highest shear bond strength values. The addition of BAG did not have a significant influence on shear bond strength, regardless of demineralization or thermocycling condition. Adhesive failure was the predominant type of failure in all groups.

Conclusion: The incorporation of BAG filler up to 2 wt% did not result in significant changes in shear bond strength. Experimental adhesive bonding agents with 2 wt% BAG content demonstrated shear bond strengths comparable to the commercial bonding agent in sound nontreated, sound thermocycled, demineralized nontreated, and demineralized thermocycled groups.

Key Words: Dentin bonding agent, permanent dental restoration, shear strength, tooth demineralization

Received: 06-Dec-2022
Revised: 07-Jul-2023
Accepted: 17-Jul-2023
Published: 28-Aug-2023

Address for correspondence:
Dr. Parsa Rezvanian, Department of Animal Biotechnology, Cell Science Research Center, Royan Institute for Biotechnology, ACECR, Isfahan, Iran.
E-mail: parsa.rezvanian@gmail.com

Access this article online

Website: www.drj.ir
www.drjjournal.net
www.ncbi.nlm.nih.gov/pmc/journals/1480

How to cite this article: Mousavinasab SM, Sarandi F, Rezvanian P, Atai M, Mousavinasab S. Effect of bioactive glass-containing dentin adhesives on microshear bond strength of composite restorations. Dent Res J 2023;20:95.

INTRODUCTION

In recent years, there has been a soaring demand for esthetic dental restorations, which has led to the development of bonding restorations. Thus, systems such as self-etch (SE) and etch and rinse (E and R) have been introduced. In this regard, the use of SE systems has experienced a marked increase due to the elimination of the washing step and reduced clinical errors.[1] To achieve a durable bonding with high strength, different strategies are employed: (1) the use of several layers of adhesive for SE adhesive systems;[2] (2) the application of a hydrophobic layer after the SE adhesive in order to reduce water diffusion;[3] and (3) the employment of matrix metalloproteinase (MMP) enzyme inhibitors such as chlorhexidine, ethylenediaminetetraacetic acid, tetracycline, and galardin,[4-9] as well as collagen cross-linkers such as glutaraldehyde and proanthocyanidin, to improve the mechanical properties and reduce collagen degradation.[10]

One of the major factors affecting the durability of bonding agents is the remineralization of demineralized dentin. In this regard, intrafibrillar mineralization of fibrillar collagen by ion-releasing agents such as amorphous calcium phosphates and bioactive glasses (BAGs) could lead to the regeneration of apatite crystals and deactivate MMPs at the crown site.[11,12] BAG was first introduced by Professor L. Hench in the 1970s as the first bioactive material[13,14] and is composed of four main components: SiO_2, CaO, Na_2O, and P_2O_5.[15] This material was a type of glass that was able to bind to the hard tissue without any adverse effects. The discovery of the biocompatibility characteristics of BAG was a revolution in the health-care industry and soon found numerous clinical applications in the restoration of hard tissue in medical and dental fields.[13,16]

Recently, there has been an interest in the use of BAG materials.[17] This is due to the effectiveness of restorations and the remineralization of the hard tissue by BAG.

The antibacterial properties of BAG are considered a gold standard in clinical odontology applications. BAG was first used as bone replacement in dentoalveolar and maxillofacial restorations, as well as periodontal restorations and implants.[18,19] Afterward, the application of BAG was expanded to include remineralizing agent and filler in adhesives and dental composites.[20-25]

BAG has been used in the treatments of dentin hypersensitivity and also as a crown surface preparation agent in total etching systems. When BAG comes in contact with dental tissues, its particles diffuse into the tubules and interact with the adjacent tissue.[20-22] Recent studies have shown that the use of BAG as filler in dental composites and adhesives results in improvements in restoration durability through remineralization and antibacterial effects.[23] Moreover, BAG contributes to enamel hardness and inhibits white spots.[24] It has also been suggested that BAG is associated with the remineralization of demineralized tissue.[25] Khvostenko *et al*. suggested that dental composites containing BAG possessed superior mechanical properties such as bending strength and fracture toughness compared to those of Heliomolar composites. They also stated that these composites were comparable to Filtek Z250 and Filtek Supreme Plus.[26] Additionally, it has been suggested that adhesives containing BAG can prevent the occurrence of white lesions and secondary decays.[27] Moreover, decay prevention in orthodontic bonding agents containing BAG was superior to Transbond XT Plus, they could also prevent enamel softening adjacent to the brackets.[20] Furthermore, BAG-containing adhesives may exert beneficial effects on the durability of composite restoration bonds through the precipitation of minerals in the hybrid layer and inhibition of MMP enzyme.[28] The addition of bioactive calcium silicate fillers to H and R adhesives similarly increased bond durability in the resin–crown interface.[29] The presence of BAG in the adhesive structure reduces adhesive permeability and improves its mechanical properties.[30] Ultimately, it has been demonstrated that BAG-containing adhesives are capable of remineralization of dental pulp.[31]

There are little but promising data available on the microshear bond strength (μ-SBS) of BAG-containing adhesive bonding agents under demineralization and/or thermocycling conditions.[32,33] In a study conducted by Kim *et al*., the antibacterial and remineralizing effects of orthodontic bonding agents with BAG content were investigated. The results demonstrated superior antibacterial and remineralizing properties of orthodontic bonding agents with BAG content compared to conventional bonding agents.[23] Another study conducted by Skallevold *et al*. showed that

BAG-containing resin composites improved dentin remineralization and decreased enzymatic degradation in the crown interface.[34]

Compared to bonding systems without BAG, BAG-containing bonding systems decrease micropermeability at the resin–crown interface through remineralization of the demineralized region and increase elastic modulus and hardness.[34] Moreover, it has been demonstrated that BAG-containing orthodontic adhesives possess acid-neutralizing effects and improved SBS in enamel. It has been proposed that orthodontic adhesives containing 45S5F-B BAG considerably decreased white spot lesions.[35] Furthermore, the presence of BAG in the structure of composites acts as a deposit of calcium and fluoride ions and releases these ions over time.[36]

Based on the desirable properties of BAG mentioned above, the aim of this study was to determine the effect of adding BAG to a dentin-adhesive bonding agent in order to improve its μ-SBS and assess the impact of thermocycling and demineralization on μ-SBS of adhesive bonding agents. Thus, in this work, novel dentin bonding adhesive agents containing 0%–2% BAG were prepared, and their μ-SBS was compared against a commercially available bonding agent. Moreover, the effect of demineralization and thermocycling on the μ-SBS of the bonding agents was evaluated. Ultimately, an optimum concentration of BAG that produced the highest μ-SBS was determined.

MATERIALS AND METHODS

Study design

The study employed a randomized controlled design with multiple groups and levels of variables (explained below) to assess the effects of different composite-bonding agents with BAG content on shear bond strength and fracture pattern in sound and demineralized teeth, with and without thermocycling. This design allowed for the systematic investigation of the influence of these factors on the performance of the bonding agents.

Ethics

The study was reviewed by the Ethics Committee of Isfahan University of Medical Sciences and confirmed with the ethics code 399366. Two hundred third molar teeth were extracted from individuals aged between 18 and 25 years and used in this study. All participants were clearly notified about the study and provided informed consent by signing consent forms.

Tooth preparation

The teeth used in this study were obtained over the course of 3 months. After extraction, the teeth were cleaned in order to remove any remaining blood, saliva, or debris and preserved in 0.5% chloramine-T at 4°C for 1 week. Following preservation, the teeth were stored in distilled water at 4°C.

The surface of the teeth was cleaned using prophylaxis paste (ProphyCare, Directa, Upplands Väsby, Sweden), with a low-speed handpiece and rubber cap. Subsequently, the teeth were mounted in self-curing acrylic (Beta dent, Tehran, Iran) at the cementoenamel junction (CEJ) level and cut 3 mm above the CEJ using a water-cooled fine cutter (NSK, Kanuma Tochigi, Japan). The surface of the mounted and exposed samples was then polished for 30 s using 400, 600, and 800 grit silicon carbide sandpapers, respectively, in order to obtain a uniform and flat smear layer. After the preparation step, samples with apparent defects, fractures, or cavities were omitted from the study. Thus, out of 200 extracted third molars, only 80 were deemed suitable and included in the study (total number of samples $n = 80$). The prepared teeth were then randomly assigned to two groups: Sound teeth and teeth exposed to the demineralization process.

Four experimental composite-bonding agents

For the fabrication of the experimental dentin bonding agents, a combination of two resin monomers with 0.4%w camphorquinone and 0.8%w ethyl 4-dimethylaminobenzoate was used. The resin monomers included ethoxylated bisphenol A dimethacrylate (EOBPADMA) and BisGMA. The EOBPADMA-to-BisGMA ratio was 1:2.5 which demonstrated the highest strength and hardness compared to other ratios.[27] All the reagents were purchased from Sigma-Aldrich (St. Louis, MO, USA).

BAG 45S5 powder (Nikceram, Isfahan, Iran) was mixed with the bonding agents in concentrations of 0, 0.2, 0.5, and 2 wt% BAG, using SpeedMixer (FlackTek, Landrum, SC, USA) for 1 h. Subsequently, the samples were sonicated in an ultrasonic bath (Sonoplus UW 2200, Bandelin, Berlin, Germany) in order to achieve a uniform distribution of BAG particles. Mixing was performed until the workability of the mixture was similar to that of the Adper Single Bond commercial bonding agent. The concentrations

of 0, 0.2, 0.5, and 2 wt% were named A, B, C, and D, respectively. Each group "A to D" had 16 samples (n for groups A, B, C, and D = 16)

Control group

The fabricated bonding agents were compared with a commercially available bonding agent, Adper Single Bond (3M ESPE, Seefeld, Germany), which served as the control group. This group was labeled E and consisted of 16 specimens (n for group E = 16).

Grouping overview

Subsequently, the prepared 80 teeth were initially divided into five groups of different composite bonding (groups A, B, C, D, and E) named level 1 groups. Groups A, B, C, D, and E represent bonding agents with 0, 0.2, 0.5, and 2 wt% BAG and Adper Single Bond, respectively. Each level 1 group had 16 specimens and further divided into two level 2 groups, S (n = 8) and D (n = 8), which represent sound and demineralized teeth. Each of the level 2 groups was further divided into two level 3 groups, T and NT which indicate teeth treated with thermocycling and nontreated, respectively. Overall, there were 20 level 3 groups with n = 4 each. They vary in bonding composition (A-E), demineralization status (sound and demineralized), and thermocycling status (thermocycled and nontreated) [Figure 1]. The sample size determination was based on a previous study by Huang *et al*.[37]

Demineralization

For the demineralization process, the previously prepared teeth were exposed to 20 ml of demineralization solution at 23°C for 48 h. The demineralization solution contained 0.002 molar $CaCl_2$, 0.002 molar KH_3PO_4, and 0.002 molar glacial acetic acid (all from Sigma-Aldrich, St. Louis, MO, USA). The final pH was adjusted to 4.3 using a pH meter (Fisher Scientific, Hampton, NH, USA).[38] Overall, half of the prepared teeth (n = 40) underwent demineralization. In each group (A through E) half of the samples were demineralized.

Composite bonding

Initially, the surface of the teeth was etched using phosphoric acid 37% (Etch Royal, Watertown, NY, USA) for 15 s and the bonding agent was applied to the surface in two layers and cured for 20 s with light cure equipment Valo (Ultradent, South Jordan, UT, USA) placed over a distance of 1 mm from the surface of the tooth. Plastic tubes with an internal diameter of 1 mm and a height of 1 mm were placed over the prepared crown surface and restored using X-AP Clearfil (Kuraray CO, Okayama, Japan) composite resin with A2 color. Curing was performed for 40 s using light cure equipment placed over a distance of 1 mm from the surface with an intensity of 650 mW/cm^2. The Intensity of the used light was tested using an LED radiometer (Demetron 1, SDS/Kerr, Brea, CA, USA).

Thermocycling

After the bonding step, half of the samples in each group were subjected to thermocycling (THE-1100, SDM, Westerham, Germany). Thermocycling was performed after composite-bonding step, in deionized water and included 2000 cycles between 5 and 55°C with a submergence time of 30 s and transition time of 10 s.[39]

Bond strength measurement

All samples were placed in distilled water at 37°C and 100% relative humidity in an incubator for 24 h

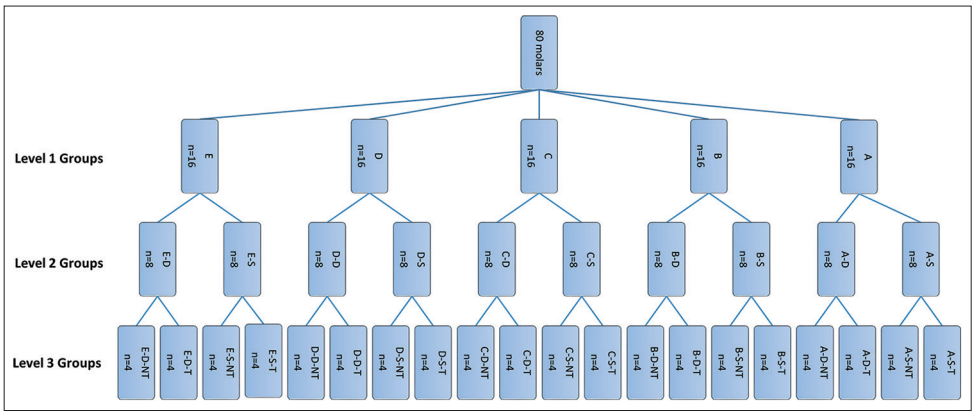

Figure 1: Different levels of sample groups used in this study. A, B, C, D, and E indicate composite-bonding agent with 0, 0.2, 0.5, and 2 wt% BAG content and commercial Adper Single Bond, respectively. S and D indicate sound and demineralized teeth. T and NT indicate thermocycled and nontreated.

after which the plastic tubes were cut and separated using no. 11 blade mounted on a scalpel. The shear bond strength of the samples was measured using a universal testing machine (Bisco Inc., Schaumburg, IL, USA) with a cross-head speed of 0.5 mm/min. Force was applied to the samples by a knife-like mandrel.

Determination of fracture pattern

Fracture type was analyzed using a stereomicroscope (Dino-Lite Pro, AnMo Electronics Corp., Taipei, Taiwan) under ×50 magnification. Different fracture types were defined as cohesive fracture (fracture in the crown or dentin), adhesive fracture (fracture in the bond between the crown and the composite), and a combination of adhesive and cohesive fractures.

Statistical analysis

Data were analyzed by SPSS 25 (IBM, Armonk, NY, USA) software. Since the obtained data did not follow a normal distribution, statistically significant differences between μ-SBS values for different composite-bonding agents with different BAG content (A through E) were determined using Kruskal–Wallis nonparametric test followed by Mann–Whitney *post hoc* test with Bonferroni correction. *t*-test was used to compare μ-SBS values for demineralized versus sound teeth and thermocycled versus nonthermocycled samples within level 2 groups. Chi-square and Fisher's exact tests were used to determine if there is a meaningful relationship (association) between fracture pattern and bonding type (A-E groups), fracture pattern and tooth condition (demineralized or sound), fracture pattern and thermocycle status (nontreated or thermocycled), and fracture pattern and subgroup type (20 subgroups). All data are presented as mean ± standard deviation for each of the 20 subgroups. The level of significance was considered $P \leq 0.05$ for all tests and was adjusted by Bonferroni correction.

RESULTS

μ-SBS was measured for all samples. Student's *t*-test was used in order to determine differences in μ-SBS between sound and demineralized (S and D conditions) within level 2 groups Comparing A-S with A-T, B-S with B-D, C-S with C-D, D-S with D-D and E-S with E-D). The results are presented in Table 1. It was revealed that μ-SBS for sound and demineralized groups without BAG content (A-S and A-D level 2 groups) were 13.53 ± 5.58 and 6.25 ± 4.98 MPa,

Table 1: Shear bond strength data in MPa for different bonding agent level 1 groups (A-E), level 2 groups, and level 3 groups

Level 1 Groups	μ-SBS (mean±SD)	Level 2 Groups	μ-SBS (mean±SD)	Level 3 Groups	μ-SBS (mean±SD)
A	9.90±6.34	A-S	13.53±5.58[a]	A-S-T	14.15±7.07
				A-S-NT	12.92±4.66
		A-D	6.25±4.98	A-D-T	4.90±3.88
				A-D-NT	7.60±6.15
B	11.11±7.22	B-S	12.17±8.27	B-S-T	7.94±1.70[c]
				B-S-NT	16.40±10.45
		B-D	10.04±6.37	B-D-T	8.20±1.67
				B-D-NT	11.88±9.10
C	6.65±4.39	C-S	7.07±5.83	C-S-T	6.90±3.27[d]
				C-S-NT	7.24±8.27
		C-D	6.06±2.61	C-D-T	5.08±1.53
				C-D-NT	7.04±3.31
D	7.13±4.57	D-S	9.60±5.24[b]	D-S-T	10.14±4.70
				D-S-NT	9.07±6.42
		D-D	4.66±1.80	D-D-T	4.09±2.45
				D-D-NT	5.22±0.81
E	18.09±9.58	E-S	21.83±6.36	E-S-T	18.65±2.24
				E-S-NT	25.00±7.91
		E-D	14.35±11.15	E-D-T	7.77±2.13
				E-D-NT	20.94±13.04

[a] and [b]Depict statistical significance versus A-D and D-D level 2 groups, respectively. [c] and [d]Depict statistical significance versus E-S-T level 3 group. A, B, C, D, and E indicate composite-bonding agent with 0, 0.2, 0.5, and 2 weight % BAG content and commercial Adper Single Bond, respectively. S and D indicate sound and demineralized teeth, and T and NT represent thermocycled and nontreated teeth, respectively. μ-SBS: Microshear bond strength, SD: Standard deviation, BAG: Bioactive glass

respectively ($P = 0.015$). Furthermore, the comparison of μ-SBS between sound and demineralized groups with 2 wt% BAG content (D-S and D-D level 2 groups) also revealed significant differences in μ-SBS. μ-SBS for D-D and D-S were 9.60 ± 5.24 and 4.66 ± 1.80 MPa, respectively ($P = 0.024$).

Moreover, the μ-SBS between thermocycled and nonthermocycled (T and NT conditions) samples were compared within level 2 groups. In this case, in the commercial adhesive bonding agent Adper Single bond (group E), there were statistically significant differences between the μ-SBS of thermocycled and nonthermocycled samples (13.21 ± 6.16 and 22.97 ± 10.22 MPa for thermocycled and nonthermocycled samples, respectively).

In order to determine the differences in μ-SBS in level 1 groups A-E with different bonding agents, nonparametric Kruskal–Wallis test was employed ($P < 0.001$). Mann–Whitney *post hoc* test after Bonferroni correction revealed that group E showed statistical differences with other groups (A-D). The values of μ-SBS for groups A-E are shown in Table 1.

With the aim of conducting a more detailed analysis of the results, each of the five level 1 groups of bonding agents (A-E) was divided into four level 3 groups (S-NT, S-T, D-NT and D-T, overall twenty level 3 groups) and analyzed by Kruskal–Wallis nonparametric test followed by Mann–Whitney *post hoc* test. In this case, the μ-SBS values for each level 1 group (A-E) were compared while the condition of tooth (sound or demineralized) and thermocycling condition was kept constant [comparison between A-S-NT, B-S-NT, C-S-NT, D-S-NT, and E-S-NT demonstrated in Figure 2a, then comparison between A-S-T, B-S-T, C-S-T, D-S-T, and E-S-T demonstrated in Figure 2b, then comparison between A-D-NT, B-D-NT, C-D-NT, D-D-NT, and E-D-NT demonstrated in Figure 2c and finally comparison between A-D-T, B-D-T, C-D-T, D-D-T, and E-D-T demonstrated in Figure 2d]. Figure 2 shows μ-SBS values of different dentin bonding agents for 4 testing condition as bar charts. As it can be seen in S-NT condition [Figure 2a], there were no statistically significant differences between the μ-SBS values of the bonding agents. In S-T condition [Figure 2b],

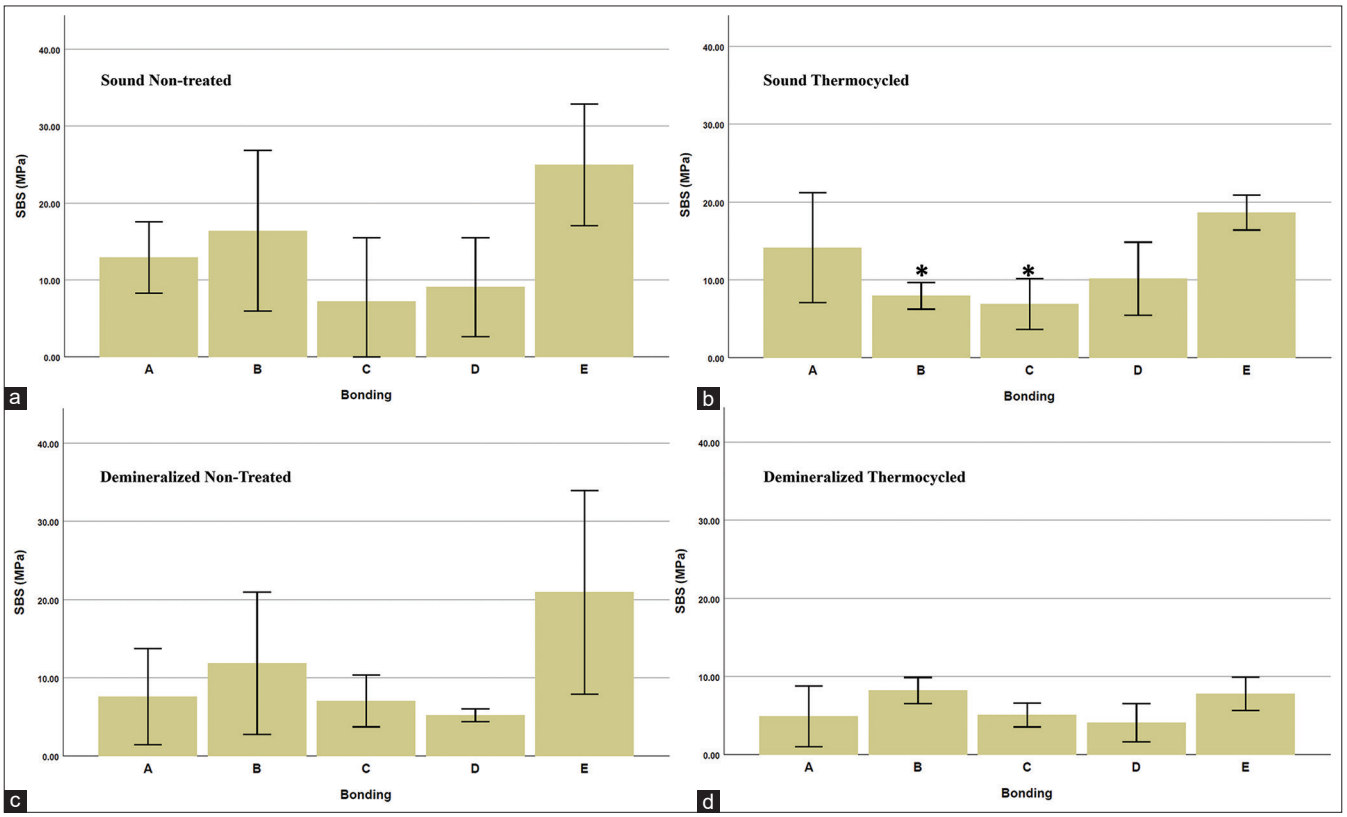

Figure 2: Microshear bond strength values for each subgroup presented as mean value ± standard deviation (a-d). Sound nontreated (a), sound thermocycled (b), demineralized nontreated (c), and demineralized thermocycled (d). A, B, C, D, and E indicate composite-bonding agent with 0, 0.2, 0.5, and 2 wt% BAG content and commercial Adper Single Bond, respectively. *Indicates statistical significance versus the corresponding E.

the experimental bonding agents containing 0.2 and 0.5 wt% (B and C) showed significantly less μ-SBS compared to that of the commercial sample (E) while other experimental samples possessed μ-SBS values similar to that of the commercial control sample. In D-NT condition [Figure 2c], no statistically significant differences between the μ-SBS values of the bonding agents were observed which can be due to the high variation in the μ-SBS values measured for the control sample (E). Finally, in D-T condition [Figure 2d], there were also no statistical significance differences between the μ-SBS values for the experimental bonding agents (A-D) and the control bonding agent (E). In all 4 testing conditions (S-NT, S-T, D-NT, and D-T) there was no statistically significant differences between the μ-SBS values of experimental bonding agents (A, B, C, and D). The SBS data for the 20 level 3 groups are provided in Table 1.

Chi-square and Fisher's tests were used to determine if there is an association between fracture mode and bonding type (A-E groups). Chi-square method showed no significance (P = 0.072) while Fisher's exact test exhibited significance (P = 0.050). After performing pairwise comparisons, it was determined that there was an association between adhesive fracture mode and, A and E bonding types. The number of fracture types (adhesive, cohesive, or mixed) for each bonding group (A-E) can be seen in Table 2.

Chi-square and Fisher's exact tests were conducted to explore the potential meaningful dependency between fracture mode and tooth condition (sound or demineralized), as well as fracture mode and thermocycle condition (nontreated or thermocycled). The results indicated that there was no significant association between fracture type and tooth condition (P = 0.209 for both tests). Similarly, no significant association was found between fracture type and thermocycle condition (P = 0.787 for both tests).

The association between the twenty level 3 groups and fracture mode was also examined using Fisher's exact test and Chi-square test. In this case, the Chi-square test did not reveal any statistical significance (P = 0.088). However, in contrast, the fisher's exact test showed significance (P = 0.048). Pairwise comparisons were performed to determine which specific groups showed significance, but no significant associations were identified between any two groups. The number of each fracture pattern for the twenty studied level 3 groups is shown in Figure 3.

Table 2: Fracture type for different bonding agent groups (A-E) and subgroups

Level 1 Groups	Number of each fracture type			Level 3 Groups	Number of each fracture type		
	Adhesive	Cohesive	Mixed		Adhesive	Cohesive	Mixed
A	9	2	5	A-S-T	4	0	0
				A-S-NT	2	0	2
				A-D-T	1	1	2
				A-D-NT	2	1	1
B	13	0	3	B-S-T	2	0	2
				B-S-NT	3	0	1
				B-D-T	4	0	0
				B-D-NT	4	0	0
C	12	3	1	C-S-T	4	0	0
				C-S-NT	4	0	0
				C-D-T	3	1	0
				C-D-NT	1	2	1
D	12	1	3	D-S-T	4	0	0
				D-S-NT	3	1	0
				D-D-T	2	0	2
				D-D-NT	3	0	1
E	16	0	0	E-S-T	4	0	0
				E-S-NT	4	0	0
				E-D-T	4	0	0
				E-D-NT	4	0	0

A, B, C, D, and E indicate composite-bonding agent with 0, 0.2, 0.5, and 2 weight % BAG content and commercial Adper Single Bond, respectively. S and D indicate sound and demineralized teeth, and T and NT represent thermocycled and nontreated teeth, respectively. BAG: Bioactive glass

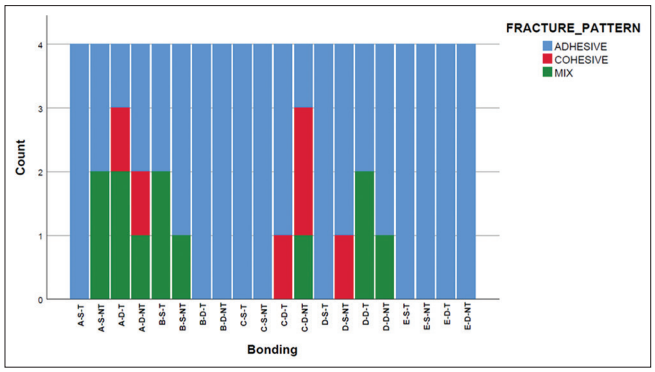

Figure 3: Number of each fracture type in subgroups. A, B, C, D, and E indicate composite-bonding agent with 0, 0.2, 0.5, and 2 wt% BAG content and commercial Adper Single Bond, respectively. S and D represent sound and demineralized teeth. T and NT represent thermocycled and nontreated.

DISCUSSION

In the current study, the effect of the addition of BAG to dentin bonding agents on μ-SBS of composite restorations was investigated. Primarily, in the general comparison between sound and demineralized teeth, it was demonstrated that the different dentin adhesives with 0 and 2 wt% BAG content (A and D) used in this study, possessed higher μ-SBS when applied on sound teeth. In a similar study, μ-SBS of short-term composite restorations with SE adhesive systems on sound and demineralized dentin was studied. The study revealed that μ-SBS values were highly dependent on demineralization status and were lower for sound dentin samples compared to demineralized samples.[40] The dissimilarity observed between the two studies could be related to the use of two different dentin bonding systems, as de-Melo *et al*. used SE system while in our study an E and R system was employed. In the mild SE system, increased demineralization, probably due to improved hybridization of the hybrid layer, leads to better bonding performance.

In another study, the effect of the addition of 0.2% sodium fluoride, MI Paste (CPP-ACP), and P11-4 peptide to Adper Single Bond and Clear Fill Bond adhesives in sound and demineralized teeth, on μ-SBS was addressed. The highest μ-SBS was observed in CPP-ACP adhesive and demineralized teeth. The μ-SBS values for Adper Single Bond and Clear Fill Bond adhesives in sound teeth were higher compared to those in demineralized teeth.[41] These results regarding the differences between sound and demineralized dentin may be comparable to the results obtained in our work. Likewise, in our study, the demineralization process led to a significant decrease in μ-SBS in the dentin adhesives with 0 and 2 wt% BAG. Moreover, increasing the concentration of BAG did not have any statistically significant effects on the μ-SBS of bonding agents in S-NT, D-NT, and D-T conditions. The low values of SBS for BAG-containing experimental samples could be related to the increase in adhesive viscosity in higher concentration of BAG and, in turn, its reduced penetration in the hybrid layer and tubule depth.

Another study conducted by De Morais *et al*. addressed the effect of the addition of Biosilicate BAG to several adhesive bonding systems on μ-SBS of composite restorations in sound and demineralized teeth. It was demonstrated that the addition of 10% Biosilicate as a suspension in pretreatment increased μ-SBS in both sound and demineralized teeth.[42] On the contrary, our findings showed higher μ-SBS values for sound teeth in the dentin adhesives with 0 and 2 wt% BAG. Moreover, instead of using a suspension, in our study BAG was mixed with the bonding agent.

It was also demonstrated that in the Adper Single Bond group, the thermocycled dentin adhesives possessed significantly lower μ-SBS compared to the nonthermocycled group. In a similar study, the effect of aging on μ-SBS of E and R dentin adhesives was investigated. The used adhesive was a two-step E and R system, Adper Single Bond identical to the present study. Their results demonstrated that thermocycling after 2000 and 10000 cycles significantly reduced μ-SBS when compared to the nonthermocycled group.[43]

When the μ-SBS values of different bonding agents were compared regardless of their demineralization or thermocycling status, it was observed that the commercial Adper Single Bond group (E) possessed significantly higher μ-SBS compared to the experimental groups (A-D).

Moreover, when the μ-SBS values were compared within each treatment condition S-NT (sound nontreated), S-T (sound thermocycled), D-NT (demineralized nontreated), and D-T (demineralized thermocycled), our results showed that in the S-NT, D-NT and D-T conditions, there were no significant differences in μ-SBS between the Adper Single Bond group (E) and the experimental bonding agent groups (A-D). However, in the S-T condition, the bonding agents having 0.2 and 0.5 wt% BAG (B and C) exhibited significantly lower μ-SBS compared to

the Adper Single Bond group (E). When comparing the different experimental bonding systems (A-D), under the mentioned conditions, no statistically significant differences were observed. It has been previously reported that the addition of BAG to dentin adhesives can increase the degree of conversion and Knoop hardness of dentin adhesives, but it may reduce microtensile bond strength as BAG acts as a polymer-strengthening mechanism.[44] However, this effect is typically observed when high amounts of BAG (approximately 60%) are added to the adhesive.[45] On the other hand, the addition of filler particles has not shown any significant effect on the mechanical properties of commercial adhesives.[46]

In our study, with increasing the BAG content in the adhesives, the same effect was observed independent of the condition of teeth (sound and demineralized) and aging (thermocycled and nontreated). Increasing the BAG content in the adhesives up to 2 wt% did not result in any significant effects on the μ-SBS value of the experimental bonding agents (A-D groups).

Specifically, when thermocycled sound teeth (S-T) were examined, the commercial adhesive exhibited higher SBS compared to the 0.2 and 0.5 wt% BAG groups (B and C). This suggests that the addition of 2 wt% BAG may increase the μ-SBS to a point where there are no statistically significant differences with the commercial adhesive (E).

In the case of nonthermocycled demineralized teeth (D-NT), the commercial adhesive showed roughly similar μ-SBS compared to all the other groups, and the addition of BAG at different concentrations did not have a significant effect on the μ-SBS values. Similarly, for thermocycled demineralized teeth (D-T), all the experimental bonding adhesives possessed approximately the same μ-SBS values as the commercial bonding.

In all of the studied groups, the increase in BAG content up to 2% did not result in any significant changes in the μ-SBS values of the experimental adhesive bonding agents. This observation is consistent with the findings of Panighi and G'Sell, who stated that there is a direct correlation between calcium content of the adhesive and its degree of conversion, hardness, and diffusion capacity, and it is related to the amount of wetting of the dentin surface by the adhesive.[47]

The examination of failure modes revealed that adhesive failures were predominant in all groups, which is consistent with the findings reported by Bauer *et al*.[48] The commercial bonding agent (E) exhibited the lowest number of cohesive and mixed failures, with all fractures being adhesive. In contrast, the experimental adhesive without BAG content (0%, A) showed the highest number of cohesive and mixed failures [Figure 3 and Table 2]. It can be deduced that increasing the BAG content in the fabricated adhesives, led to a decrease in the occurrence of cohesive and mixed failures, potentially due to the higher degree of conversion of these adhesives as previously stated.[48,49] Although some researchers have suggested that adhesive failures between dentin and bonding systems occur in lower SBS values,[50,51] others have found that there is no such correlation between failure types and SBS values.[52-54] Likewise, in our results no correlation was observed between mode of failure and SBS value. The discrepancy between the outcomes of the Chi-square and Fisher's exact tests could be attributed to the relatively low number of samples in each group.

CONCLUSION

In this study, the effect of incorporating BAG into dentin adhesive bonding agents was investigated. The experimental adhesives containing 0, 0.2, 0.5, and 2%wt BAG were bonded to prepared dentin surfaces, and μ-SBSs were measured under conditions of thermocycling and demineralization. It was determined that μ-SBSs of bonding agents in sound dentin groups were higher than that of the demineralized groups for bonding agents with 0 and 2 wt% BAG. However, there was no significant difference between the μ-SBSs of bonding agents with 0.2 and 0.5 wt% BAG and Adper Single Bond. μ-SBSs of the bonding agents in nonthermocycled samples were higher than those in thermocycled samples for the Adper Single Bond group. Additionally, the experimental bonding agent containing 2 wt% BAG showed comparable μ-SBS to the Adper Single Bond adhesive (commercial adhesive used as control) under all four conditions: S-NT, S-T, D-NT, and D-T. However, increasing the concentration of BAG filler in the adhesive from 0 to 2 wt% did not show any significant effects on the shear bond strength of the experimental samples. An analysis of failure modes of the samples revealed that adhesive failure was the predominant type of failure in all groups.

Additionally, it was concluded that the experimental adhesive bonding agents in the S-NT, D-NT, and

D-T conditions showed similar μ-SBS to that of the commercial Adper Single Bond adhesive bonding, regardless of their BAG content. However, in the S-NT groups, the experimental adhesives containing 0.2 and 0.5%wt BAG (B and C) possessed significantly lower μ-SBS compared to the Adper Single Bond. Ultimately, experimental bonding agent containing 2 wt% BAG performed similarly to the Adper Single Bond across all testes conditions.

Acknowledgments

The authors would like to thank the Isfahan University of Medical Sciences faculty of Dentistry for providing financial support.

Financial support and sponsorship

This study was partially supported by the research initiative of the Faculty of Dentistry, Isfahan University of Medical Sciences.

Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or nonfinancial in this article.

REFERENCES

1. Randolph LD, Palin WM, Leloup G, Leprince JG. Filler characteristics of modern dental resin composites and their influence on physico-mechanical properties. Dent Mater 2016;32:1586-99.
2. Ito S, Tay FR, Hashimoto M, Yoshiyama M, Saito T, Brackett WW, *et al.* Effects of multiple coatings of two all-in-one adhesives on dentin bonding. J Adhes Dent 2005;7:133-41.
3. Brackett WW, Ito S, Tay FR, Haisch LD, Pashley DH. Microtensile dentin bond strength of self-etching resins: Effect of a hydrophobic layer. Oper Dent 2005;30:733-8.
4. Pashley DH, Tay FR, Yiu C, Hashimoto M, Breschi L, Carvalho RM, *et al.* Collagen degradation by host-derived enzymes during aging. J Dent Res 2004;83:216-21.
5. Scaffa PM, Vidal CM, Barros N, Gesteira TF, Carmona AK, Breschi L, *et al.* Chlorhexidine inhibits the activity of dental cysteine cathepsins. J Dent Res 2012;91:420-5.
6. Thompson JM, Agee K, Sidow SJ, McNally K, Lindsey K, Borke J, *et al.* Inhibition of endogenous dentin matrix metalloproteinases by ethylenediaminetetraacetic acid. J Endod 2012;38:62-5.
7. Golub LM, Ramamurthy NS, McNamara TF, Greenwald RA, Rifkin BR. Tetracyclines inhibit connective tissue breakdown: New therapeutic implications for an old family of drugs. Crit Rev Oral Biol Med 1991;2:297-321.
8. Acharya MR, Venitz J, Figg WD, Sparreboom A. Chemically modified tetracyclines as inhibitors of matrix metalloproteinases. Drug Resist Updat 2004;7:195-208.
9. Hao JL, Nagano T, Nakamura M, Kumagai N, Mishima H, Nishida T. Galardin inhibits collagen degradation by rabbit keratocytes by inhibiting the activation of pro-matrix metalloproteinases. Exp Eye Res 1999;68:565-72.
10. Han B, Jaurequi J, Tang BW, Nimni ME. Proanthocyanidin: A natural crosslinking reagent for stabilizing collagen matrices. J Biomed Mater Res A 2003;65:118-24.
11. Niu LN, Zhang W, Pashley DH, Breschi L, Mao J, Chen JH, *et al.* Biomimetic remineralization of dentin. Dent Mater 2014;30:77-96.
12. Sauro S, Pashley DH. Strategies to stabilise dentine-bonded interfaces through remineralising operative approaches – State of the art. Int J Adhes Adhes 2016;69:39-57.
13. Baino F, Hamzehlou S, Kargozar S. Bioactive glasses: Where are we and where are we going? J Funct Biomater 2018;9:25.
14. Jones JR. Review of bioactive glass: From Hench to hybrids. Acta Biomater 2013;9:4457-86.
15. Farooq I, Imran Z, Farooq U, Leghari AA, Ali H, Patil S. Bioactive glass: A material for the future. World J Dent 2012;3:199-201.
16. Lu X, Kolzow J, Chen RR, Du J. Effect of solution condition on hydroxyapatite formation in evaluating bioactivity of B (2) O (3) containing 45S5 bioactive glasses. Bioact Mater 2019;4:207-14.
17. Simila HO, Boccaccini AR. Sol-gel bioactive glass containing biomaterials for restorative dentistry: A review. Dent Mater 2022;38:725-47.
18. Salonen JI, Arjasmaa M, Tuominen UA, Behbehani M, Zaatar EI. Bioactive glass in dentistry. J Minimum Interv Dent 2009;2:208-19.
19. Schepers E, de Clercq M, Ducheyne P, Kempeneers R. Bioactive glass particulate material as a filler for bone lesions. J Oral Rehabil 1991;18:439-52.
20. Tirapelli C, Panzeri H, Lara EH, Soares RG, Peitl O, Zanotto ED. The effect of a novel crystallised bioactive glass-ceramic powder on dentine hypersensitivity: A long-term clinical study. J Oral Rehabil 2011;38:253-62.
21. Tirapelli C, Panzeri H, Soares RG, Peitl O, Zanotto ED. A novel bioactive glass-ceramic for treating dentin hypersensitivity. Braz Oral Res 2010;24:381-7.
22. de Morais RC, Silveira RE, Chinelatti MA, Pires-de-Souza, Panzeri FC. Biosilicate as a dentin pretreatment for total-etch and self-etch adhesives: *In vitro* study. Int J Adhes Adhes 2016;70:271-6.
23. Kim YM, Kim DH, Song CW, Yoon SY, Kim SY, Na HS, *et al.* Antibacterial and remineralization effects of orthodontic bonding agents containing bioactive glass. Korean J Orthod 2018;48:163-71.
24. Manfred L, Covell DA, Crowe JJ, Tufekci E, Mitchell JC. A novel biomimetic orthodontic bonding agent helps prevent white spot lesions adjacent to brackets. Angle Orthod 2013;83:97-103.
25. De Caluwé T, Vercruysse CW, Ladik I, Convents R, Declercq H, Martens LC, *et al.* Addition of bioactive glass to glass ionomer cements: Effect on the physico-chemical properties and biocompatibility. Dent Mater 2017;33:e186-203.
26. Khvostenko D, Mitchell JC, Hilton TJ, Ferracane JL, Kruzic JJ. Mechanical performance of novel bioactive glass containing dental restorative composites. Dent Mater 2013;29:1139-48.
27. Brown ML, Davis HB, Tufekci E, Crowe JJ, Covell DA, Mitchell JC. Ion release from a novel orthodontic resin bonding

agent for the reduction and/or prevention of white spot lesions. An *in vitro* study. Angle Orthod 2011;81:1014-20.
28. Profeta AC, Mannocci F, Foxton RM, Thompson I, Watson TF, Sauro S. Bioactive effects of a calcium/sodium phosphosilicate on the resin-dentine interface: A microtensile bond strength, scanning electron microscopy, and confocal microscopy study. Eur J Oral Sci 2012;120:353-62.
29. Profeta AC, Mannocci F, Foxton R, Watson TF, Feitosa VP, De Carlo B, *et al.* Experimental etch-and-rinse adhesives doped with bioactive calcium silicate-based micro-fillers to generate therapeutic resin-dentin interfaces. Dent Mater 2013;29:729-41.
30. Sauro S, Osorio R, Watson TF, Toledano M. Therapeutic effects of novel resin bonding systems containing bioactive glasses on mineral-depleted areas within the bonded-dentine interface. J Mater Sci Mater Med 2012;23:1521-32.
31. Wang Z, Shen Y, Haapasalo M, Wang J, Jiang T, Wang Y, *et al.* Polycarboxylated microfillers incorporated into light-curable resin-based dental adhesives evoke remineralization at the mineral-depleted dentin. J Biomater Sci Polym Ed 2014;25:679-97.
32. Yun J, Tsui KH, Fan Z, Burrow M, Matinlinna JP, Wang Y, *et al*. A biomimetic approach to evaluate mineralization of bioactive glass-loaded resin composites. J Prosthodont Res 2022;66:572-81.
33. Pires-de-Souza FC, Tonani-Torrieri R, Geng Vivanco R, Arruda CN, Geraldeli S, Sinhoreti MA, *et al.* Effect of incorporation of bioactive glass-ceramic into self-etch adhesives. J Adhes Dent 2022;24:195-202.
34. Skallevold HE, Rokaya D, Khurshid Z, Zafar MS. Bioactive glass applications in dentistry. Int J Mol Sci 2019;20:5960.
35. Yang SY, Kim SH, Choi SY, Kim KM. Acid neutralizing ability and shear bond strength using orthodontic adhesives containing three different types of bioactive glass. Materials (Basel) 2016;9:30125.
36. Davis HB, Gwinner F, Mitchell JC, Ferracane JL. Ion release from, and fluoride recharge of a composite with a fluoride-containing bioactive glass. Dent Mater 2014;30:1187-94.
37. Huang X, Xie J, Zhang M, Sun Z, Xu Y, Yang W, *et al.* Effect of 45S5 bioactive glass on the microshear bond strength of dental fluorosis. Microsc Res Tech 2022;85:1663-70.
38. Yang B, Flaim G, Dickens SH. Remineralization of human natural caries and artificial caries-like lesions with an experimental whisker-reinforced ART composite. Acta Biomater 2011;7:2303-9.
39. Ahmed T, Fareen N, Alam MK. The effect of surface treatment and thermocycling on the shear bond strength of orthodontic brackets to the Y-TZP zirconia ceramics: A systematic review. Dental Press J Orthod 2021;26:e212118.
40. de-Melo MA, Goes Dda C, de-Moraes MD, Santiago SL, Rodrigues LK. Effect of chlorhexidine on the bond strength of a self-etch adhesive system to sound and demineralized dentin. Braz Oral Res 2013;27:218-24.
41. Barbosa-Martins LF, de Sousa JP, de Castilho AR, Puppin-Rontani J, Davies RP, Puppin-Rontani RM. Enhancing bond strength on demineralized dentin by pre-treatment with selective remineralising agents. J Mech Behav Biomed Mater 2018;81:214-21.
42. de Morais RC, Silveira RE, Chinelatti M, Geraldeli S, de Carvalho Panzeri Pires-de-Souza F. Bond strength of adhesive systems to sound and demineralized dentin treated with bioactive glass ceramic suspension. Clin Oral Investig 2018;22:1923-31.
43. Hariri I, Shimada Y, Sadr A, Ichinose S, Tagami J. The effects of aging on shear bond strength and nanoleakage expression of an etch-and-rinse adhesive on human enamel and dentin. J Adhes Dent 2012;14:235-43.
44. Kim JS, Cho BH, Lee IB, Um CM, Lim BS, Oh MH, *et al.* Effect of the hydrophilic nanofiller loading on the mechanical properties and the microtensile bond strength of an ethanol-based one-bottle dentin adhesive. J Biomed Mater Res B Appl Biomater 2005;72:284-91.
45. Martins GC, Meier MM, Loguercio AD, Reis A, Gomes JC, Gomes OM. Effects of adding barium-borosilicate glass to a simplified etch-and-rinse adhesive on radiopacity and selected properties. J Adhes Dent 2014;16:107-14.
46. Giannini M, Liberti MS, Arrais CA, Reis AF, Mettenburg D, Rueggeberg FA. Influence of filler addition, storage medium and evaluation time on biaxial flexure strength and modulus of adhesive systems. Acta Odontol Scand 2012;70:478-84.
47. Panighi M, G'Sell C. Influence of calcium concentration on the dentin wettability by an adhesive. J Biomed Mater Res 1992;26:1081-9.
48. Bauer J, Carvalho EM, Carvalho CN, Meier MM, Souza JP, Carvalho RM, *et al.* Development of a simplified etch-and-rinse adhesive containing niobiophosphate bioactive glass. Int J Adhes Adhes 2016;69:110-4.
49. Tauböck TT, Zehnder M, Schweizer T, Stark WJ, Attin T, Mohn D. Functionalizing a dentin bonding resin to become bioactive. Dent Mater 2014;30:868-75.
50. Courson F, Bouter D, Ruse ND, Degrange M. Bond strengths of nine current dentine adhesive systems to primary and permanent teeth. J Oral Rehabil 2005;32:296-303.
51. Price RB, Dérand T, Andreou P, Murphy D. The effect of two configuration factors, time, and thermal cycling on resin to dentin bond strengths. Biomaterials 2003;24:1013-21.
52. de Araujo FB, García-Godoy F, Issáo M. A comparison of three resin bonding agents to primary tooth dentin. Pediatr Dent 1997;19:253-7.
53. Pashley DH, Sano H, Ciucchi B, Yoshiyama M, Carvalho RM. Adhesion testing of dentin bonding agents: A review. Dent Mater 1995;11:117-25.
54. Watanabe LG, Marshall GW Jr., Marshall SJ. Dentin shear strength: Effects of tubule orientation and intratooth location. Dent Mater 1996;12:109-15.