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Effect of bioactive glass particles on mechanical and adhesion properties of resin cements

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Abstract

Purpose: The aim of this study is to evaluate the mechanical and adhesive properties of three different resin cements with bioactive glass (BAG) incorporated in two different ratios.
Methods: BAG was added to different resin cements (3M Rely-X Ultimate, GC Link Ace, and GC Link Force) in different ratios (5% and 10% by weight). The three-point flexural strength, microhardness, and bond strength properties were evaluated. The fracture types of the groups were then analyzed using a stereo microscope. The data were analyzed using a multifactorial analysis of variance and Tukey's post-hoc tests ($\alpha < 0.05$).
Results: The addition of BAG reduced the flexural strength of the resin cements ($P < 0.05$).The effect of BAG addition on the Vickers microhardness value was significantly different for each cement group ($P < 0.05$). In addition, with the exception of the GC link force group (10% BAG addition), the BAG addition decreased the bond strength of cements to dentin in all the groups ($P = 0.171$).
Conclusions: The results of this study confirmed that different resin cements comprising different ratios of BAG exhibited different flexural strength, hardness, and bond-strength properties. Since the bond strength values increased with the addition of 10% BAG in the GC Link Force cement group, the effects of different BAG compositions could be worth investigating in future studies.

Keywords: Bioactive glass, Resin cement

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1. Introduction

Conventional dental cements are based on mechanically locking the restoration to the tooth, while the main task is to fill the gap between the tooth and restoration[1,2]. One of the epochal events in dentistry in the last 30 years was the adhesive cementation of tooth-colored restorations prepared using minimally invasive techniques[3]. Adhesive resin cements are used for the cementation of many restorations such as inlays, onlays, fiber-supported bridges, crowns, and laminate veneers[4]. While the content of the majority of resin cements is similar to that of dental composites, commercially available products have various physical and mechanical differences owing to their type, size, and amount of diluent monomer or filler particles[4,5].

According to the matrix formation mechanism, adhesive cements are classified as follows: (1) light-activated curing, (2) self- or auto-curing, and (3) dual curing[1]. Compared with conventional cements, adhesive cements have improved adhesion and mechanical properties[6,7]. However, polymerization shrinkage of these cements causes marginal integrity deterioration and micro-leakage of the restorations, which could significantly shorten the lifetime of the restorations[6,8,9]. One method of ensuring the longevity of dental restorations is to provide a tight connection between the tooth and restoration and create an environment that prevents bacterial adhesion[10]. Few clinical studies have been conducted on resin cements to evaluate the survival and success rates of restorations. Moreover, studies have also mentioned that self-adhesive resin cements have similar survival rates to zinc phosphate cements, i.e., a survival rate of 97.6%, even in the cementation of metal–ceramic crowns. However, it has also been reported that the survival rate of partial all-ceramic crowns cemented with self-adhesive resin cements is lower than that of conventional resin cements[11–13].

For this aim, bioactive materials that can slowly release ions to form bonds between tissues and materials can be added to self-adhesive resin cements[10,14]. Among the diverse spectrum of materials that exhibit bioactive properties (bioactive glasses (BAGs), hydroxyapatites, calcium phosphates, and calcium aluminates), BAGs are biocompatible, osteoconductive, and osteoinductive calcium-silicate-based biomaterials[10,15,16]. The first BAG, developed in 1969 by Prof. Larry Hench, could form bonds with soft and hard tissues such as bone. BAG, known as 45S5 today, consists of 46.1 mol% SiO_2, 26.9 mol% CaO, 24.4 mol% Na_2O, and 2.6 mol% P_2O_5 and precipitates hydroxyapatite in aqueous solutions[16–18].

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Table 1. Materials used in the study (Manufacturer or published data)

Materials	Type	Manufacturer	Lot No.	Composition
Rely-X Ultimate	Adhesive resin cement	3M ESPE Dental Products, St. Paul, MN	6235394	10-Methacryloxydecyl dihydrogenphosphate (MDP) Dimethacrylate resins. HEMA. Vitrebond TM copolymer Filler. Ethanol. Water. Initiators. Silane Filler content: 43% Procedure: Place paste from automix syringe and then light cure from three directions (occlusal, mesial, and distal) for 20 s each.
	Scotchbond universal adhesive		90903A	MDP phosphate monomer. Dimethacrylate resins. HEMA. Vitrebond. Copolymer. Filler. Ethanol. Water. Initiators. Silane pH = 2.7 Procedure: Apply Scotchbond Universal Adhesive for 20 s, gently air dry for 5 s, and then light cure for 10 s.
G-Cem LinkAce	Self-adhesive resin cement	GC Corp., Tokyo, Japan	1904161	Urethane dimethacrylate surface-treated silica silane synergist Filler content: 52.5–62.5% Procedure: Place paste from automix syringe and then light cure from three directions (occlusal, mesial, and distal) for 20 s each.
	G-Multi Primer (Primer for glass ceramics, hybrid ceramics, zirconia, alumina, composites and metal bonding)		2012021	10-MDP, 10-MDTP, 3-methacryloxypropyltrimethoxy silane, ethanol. Procedure: G-Multi Primer for 20 s and then air dry strongly.
G-Cem Link Force	Adhesive resin cement	GC Corp., Tokyo, Japan	2101132	Bis-GMA, UDMA, dimethacrylate, barium glass filler, silica filler, pigment, initiator Catalyst: UDMA, Bis-MEPP, dimethacrylate, barium glass filler, silica filler, pigment, initiator. Filler content: 62% Procedure: Place paste from automix syringe and then light cure from three directions (occlusal, mesial, and distal) for 20 s each.
	G-Multi Primer (Primer for glass ceramics, hybrid ceramics, zirconia, alumina, composites and metal bonding)		2012021	10-MDP, 10-MDTP, 3-methacryloxypropyltrimethoxy silane, ethanol Procedure: G-Multi Primer for 20 s and then air dry strongly.
	G-Premio Bond (One component light-cured Adhesive)		2003121	10-MDP, 4-MET, methacrylate acid ester, distilled water, 10-MDTP, acetone, photo initiators, silica pH = 1.5 Procedure: Apply G-Premio BOND and wait for 10 s after drying for 5 s. Then light cure for 20 s.
Bioactive Glass 45S5	Bioactive glass	Schott, Mainz, Germany	G018-144	45.0% SiO_2 24.5% CaO 24.5% Na_2O 6.0% P_2O_5
Gradia Plus	Modular composite system for indirect restorations	GC Corp., Tokyo, Japan	210617C	UDMA, dimethacrylate, Inorganic fillers (71 wt%), Prepolymerized fillers (6 wt%), Photoinitiators, Stabilizers, Pigments

Previous studies have shown that BAG has a remineralization effect on artificial dentin caries, which is promising, and it may be a therapeutic option for caries treatment[19]. In another study conducted by Gillam *et al.*, it was concluded that the inclusion of BAG particles into an appropriately formulated agent may be effective for the treatment of dentin sensitivity by partially occluding the dentinal tubules[20]. However, there are concerns that the poor connection of the BAG particles added as a filler to the resin matrix and the released ions will negatively affect the stability of the resin material, which will adversely affect its mechanical properties[21,22]. Furthermore, calcium and phosphate released from bioactive materials in direct contact with dentin tissue can increase the life of restorations by clogging dentinal tubules or remineralizing secondary caries[23]. In addition, BAGs form an apatite-like phase in the marginal cavity and provide sealing, and by increasing the pH in the environment where they dissolve, they reduce the possibility of deterioration of the resin material caused by acid attacks[24].

The present study is thus aimed at investigating the bond strength and mechanical properties of BAG added to three different commercially available resin luting cements in two different amounts. The null hypotheses are as follows: (1) BAG addition would improve the mechanical properties of resin cements; (2) these mechanical properties are independent of the storage conditions; and (3) the bond strength of resin cements to dentin is positively affected by the incorporation of BAG into these cements.

2. Materials and Methods

2.1. *Preparation of the samples*

In this study, three commercially available resin cements, one self-adhesive (GC Link Ace) and two self-etch (3M Rely-X Ultimate, GC Link Force) were used. All the materials and resin cements used in this study are listed in **Table 1**. By measuring with a precision balance, BAG of 5% and 10% by weight was added to each resin cement and mixed manually according to the manufacturer's instructions. Samples were prepared for the three-point bending and Vickers hardness tests. The samples were sub-grouped according to their storage conditions. Half of the samples were stored in an incubator for 24 h, and the other half were stored in a simulated body fluid (SBF) solution for 2 weeks (n = 10/group). A single operator prepared all the samples.

2.2. Three-point flexural test

For the three-point flexural test (n = 10), samples of dimensions 25 × 2 × 2 mm were prepared according to ISO 4049:2019[25]. A 2 × 2 × 25 mm^3 steel mold was used to prepare the samples. The prepared experimental cements were placed in a mold, and finger pressure was applied using a transparent tape. The samples were then polymerized for 60 s from three different sides using an LED light-curing unit (Elipar S10, 3M Espe, St. Paul, USA) with an irradiance of 1200 mW/cm^2 in accordance with the manufacturer's instructions. The light device was calibrated after each polymerization process. The intensity of the light was checked regularly using a radiometer on the curing unit. The samples were sub-grouped into two groups after 24 h in an incubator at 37 °C. The first group was tested immediately, and the second group was kept in SBF for 2 weeks before testing.

Three-point flexural tests were performed using a universal testing machine (Lloyd Instruments, Fareham Hants, UK). The samples were placed on two support points at a distance of 20 mm, and the test was performed using a 2-mm indentation with a cross-head speed of 1 mm/min. The software Nexygen 4.0 was used to record the load–deflection curves.

2.3. Vickers hardness test

Three samples (n = 3) were selected from each cement group, and the surface hardness of the cements was measured using a Vickers microhardness device (Duramin-10, Struers, Copenhagen, Denmark). A diamond pyramid indentation was applied to 10 different points in each sample for 10 s under a load of 1.96 N. The Vickers hardness numbers (VHNs) were calculated from the average of 30 different measurements (Duramin Video Measurement System, version 3.0, Struers).

2.4. Shear bond strength test

The ethical permission required for our study were obtained from the relevant committee (GÜDHKAEK. 2020.20/5). Ninety intact, extracted, without-carious human third molars were disinfected with a 0.5% chloramine-T solution. For the bond strength testing (n = 10), the molar teeth were ground using a grinding machine (Struers RotoPol 11, StruersA/S, Rodovre, Denmark) under water cooling just below the enamel-dentin junction. Subsequently, silicon carbide abrasive paper no. 600 (FEPA) was used to obtain a standard smear layer. Composite resin tubes of 3.6-mm diameter and 4-mm height were fabricated using Gradia Plus (GC, Tokyo, Japan) to act as an indirect restoration. These tubes were placed in the center of the dentin while leaving 2 mm of enamel on each side, and the adhesive was then applied to the determined region. Resin composite materials that were prepared before cementation were sandblasted using a Cojet system (3M Espe, St Paul, MN, USA).

During the cementation procedure of Rely-X Ultimate, Scotchbond Universal Adhesive was applied for 20 s after gentle air drying' for 5s, which was then followed by light-curing for 10 s. In the cementation procedure for G-Cem Link Ace, G-Multi Primer was applied for 20 s, and the composite tube surface was then strongly air-dried. In addition, during the cementation procedure of the G-Cem Link Force, the G-Premio Bond was applied to the dentin surface after waiting for 10 s. The dentin surface was then dried for 5 s and light cured for 20 s. G-Multi Primer was used for 20 s and then air-dried. Following this stage, the resin cement was placed on the surface of the composite tubes and bonded to the dentin surface. The samples were then light-cured from three directions (mesial, distal, and occlusal) for 20 s each. The prepared samples were placed in SBF. The SBF used in our study was prepared as described by Kokubo *et al.*[26]. The SBF was changed once a week and placed in a 37 °C shaker incubator for 2 weeks.

To maintain their stability during testing, the samples were placed in an apparatus (Bencor Multi-T Shear Assembly, Danville Engineering Inc., San Ramon, CA, USA). The samples were placed in the apparatus on a circular cutting blade in contact with a dentin surface of diameter 3.6 mm. They were loaded with a universal testing machine (Lloyd, Fareham, Hants, UK) at a crosshead speed of 1.0 mm/min with a circular cutting tip in the shear mode. The shear bond strength was calculated using Nexygen 4.0 by dividing the maximum load at failure (N) by the bonding area (mm^2) and was recorded in megapascals.

2.5. SEM and EDS analysis

Scanning electron microscopy (SEM; JEOL-6060LV, JEOL Ltd., Japan) and energy-dispersive X-ray spectroscopy (EDS) (IXRF Instruments, IXRF, Inc., USA) were performed. After the shear bond strength test, two randomly selected samples from each group were dried in an incubator and coated with Au using a spray coater (Polaron Range CA7625 Carbon Accessory/Sputter Coate, Kolzer, Italy). SEM images were obtained at an acceleration voltage of 20 kV and a working distance of 12 mm in vacuum.

2.6. Statistical analysis

The homogeneity of the data was evaluated using Levene's test. The flexural and hardness properties were analyzed using a three-way analysis of variance (ANOVA) to evaluate the effects of the storage conditions, material, and BAG addition. The bonding properties were analyzed using a two-way ANOVA to evaluate the effect of the material and BAG addition. Statistically significant differences were determined using Tukey's post-hoc test. A *P*-value of less than 0.05 was considered to be statistically significant (SPSS version 23, IBM Corp., Chicago, IL).

3. Results

3.1. Three-point flexural test results

The flexural strength test results for the tested materials are presented in **Figure 1**. Accordingly, the storage conditions, BAG addition, and resin cement type had an effect on the flexural strength ($P < 0.05$). A significant two-factor interaction was observed between the material and the addition of BAG ($P < 0.05$). However, the relationship between the storage conditions and material was not significant ($P = 0.142$). No statistically significant differences were observed between the storage conditions and BAG amounts ($P = 0.057$).

No significant differences were found between the resin cements in the control groups under the different storage conditions. In addition, resin cements without BAG were not affected by the storage conditions ($P > 0.05$).

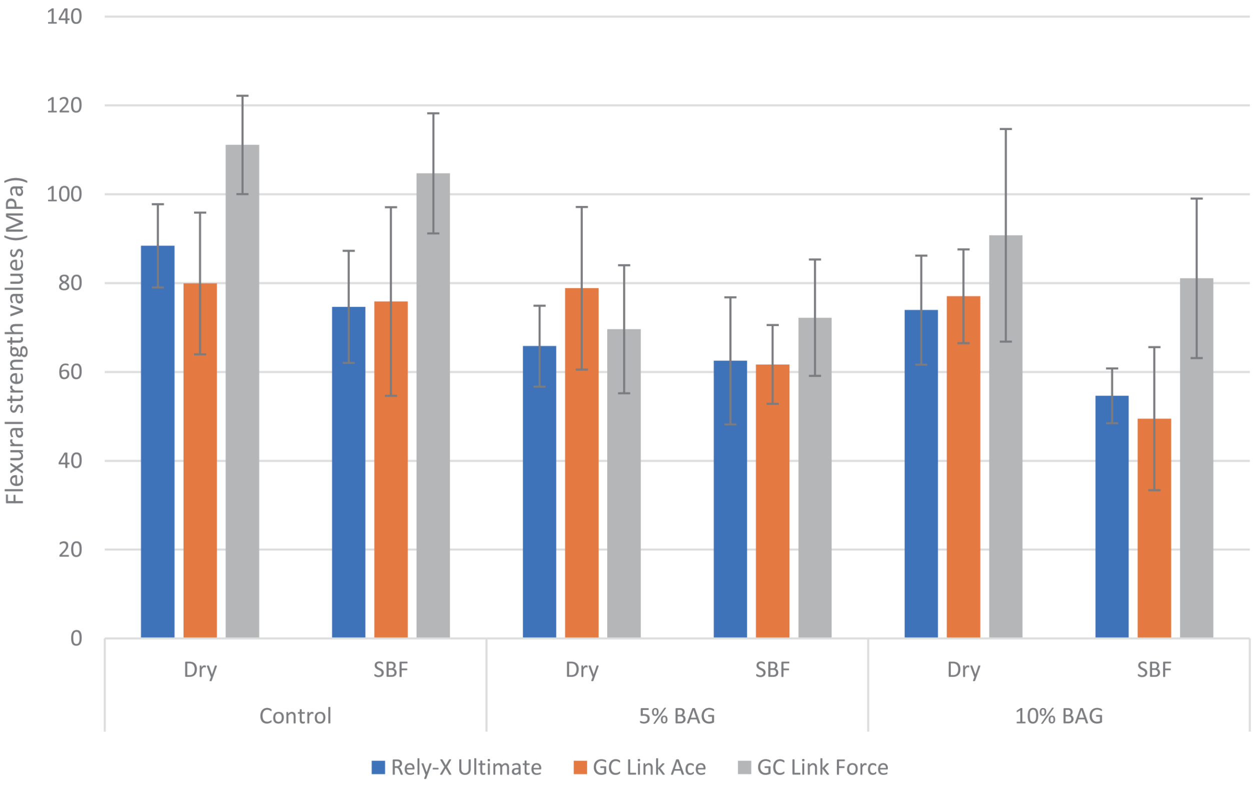

Fig. 1. Flexural strength test values (MPa) and deviations of the tested groups

In the case of the Rely-X Ultimate group, a statistically significant difference was observed between the BAG and control groups ($P < 0.05$). While the 5% BAG group was not affected by the storage conditions, a difference was observed between the 10% BAG groups ($P < 0.05$) that were stored in a dry environment and those stored in SBF. In the case of the Rely-X Ultimate cement group, the addition of 5% and 10% BAG reduced the flexural strength. However, the flexural strength of the 5% BAG group decreased to a greater extent than that of the 10% BAG group.

In the case of the Link-Ace group, there were no statistically significant differences between the groups with and without BAG ($P > 0.05$).

In the case of the Link Force group, no significant difference was observed between the BAG groups according to the storage conditions. However, a significant difference was observed between the control and BAG groups ($P < 0.05$).

3.2. *Vickers hardness test results*

The mean values and standard deviations of the Vickers hardness test results are presented in **Figure 2**. The addition of different amounts of BAG and the storage conditions had significant effects on the hardness of the material ($P < 0.05$).

The addition of BAG significantly affected the hardness of the Rely-X Ultimate group ($P < 0.05$). However, no statistically significant difference was found between the 5% and 10% BAG supplementation groups ($P < 0.05$). While the 5% BAG group was not affected by the storage conditions ($P = 0.86$), the 10% BAG group was affected by the storage conditions ($P < 0.05$).

The hardness value for the Link Ace group was affected by the storage conditions ($P < 0.05$). In addition, the hardness values were affected by the addition of BAG ($P < 0.05$). However, there were no significant differences between the 5% BAG and 10% BAG groups stored in SBF ($P = 0.999$).

In the case of the Link Force group, the addition of 5% BAG under dry conditions affected the hardness ($P < 0.05$), but the addition of 10% BAG had no statistically significant effect ($P = 0.991$). However, the addition of BAG (5% and 10%) did not have a statistically significant effect on the groups stored in SBF ($P = 0.990$ and $P = 0.622$, respectively). In addition, all the groups were affected by the storage conditions, except for the group containing 5% BAG ($P = 0.510$).

3.3. *Shear bond strength test results*

The addition of BAG had no statistically significant effect on the bond strength of the cements. The shear bond strength test results are listed in **Table 2**. The addition of 5% and 10% BAG to Rely-X Ultimate did not result in a significant difference between the groups ($P = 0.407$ and $P = 0.463$, respectively). However, a statistically significant difference was observed between Rely-X Ultimate and all the Link Ace and Link Force groups ($P < 0.05$). The addition of BAG (5% and 10%) had no significant effect on the shear bond strength of the Link Ace group ($P = 0.485$ and $P = 0.862$, respectively). Moreover, the addition of BAG (5% and 10%) to the Link Force cement group did not have a statistically significant effect on the bond strength of the

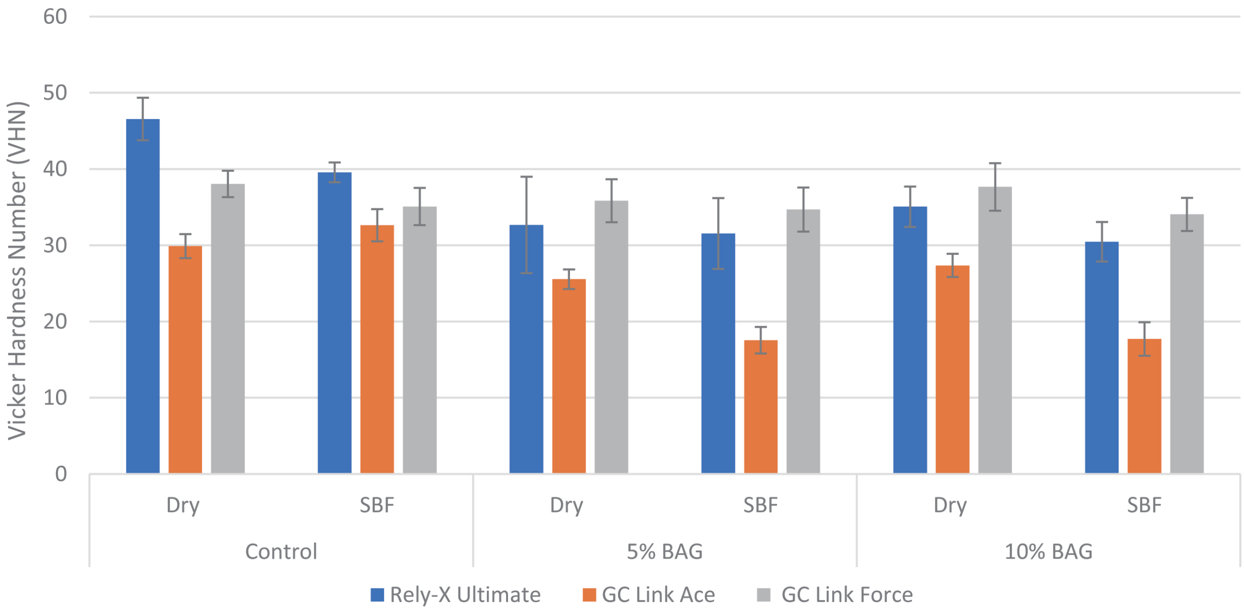

Fig. 2. Vickers hardness test values (VHN) and deviations of the tested groups

Table 2. Shear bond strength test results

		Shear Bond Strength Test	
		Mean	Std. Deviation
Rely-X Ultimate	Control ^{a}	21,1	4,8
	5% BAG ^{a}	17,8	4,5
	10% BAG ^{a}	17,6	4,2
GC Link Ace	Control ^{bcd}	7,1	3,1
	5% BAG ^{d}	3,7	1,5
	10% BAG ^{cd}	4,6	3,6
GC Link Force	Control ^{bc}	9,2	3,1
	5% BAG ^{bcd}	8,7	2,0
	10% BAG ^{b}	10,4	3,1

Table 3. Failure modes of the groups

		Adhesive	Cohesive	Mixed
Rely-X Ultimate	Control	1	7	2
	5% BAG	1	4	5
	10% BAG	1	6	3
GC Link Ace	Control	9	0	1
	5% BAG	8	0	2
	10% BAG	10	0	0
GC Link Force	Control	9	0	1
	5% BAG	9	0	1
	10% BAG	7	0	3

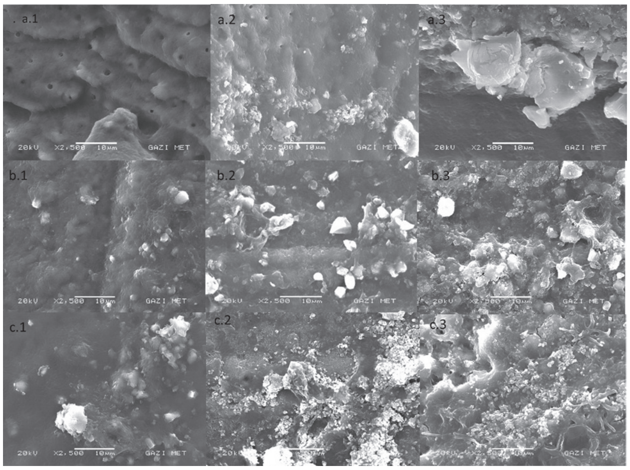

Fig. 3. SEM images obtained after shear bond strength test: (a.1) Rely-X Ultimate Control; (a.2) Rely-X Ultimate 5% BAG; (a.3) Rely-X Ultimate 10% BAG; (b.1) GC Link Ace Control; (b.2) Link Ace 5% BAG; (b.3) Link Ace 10% BAG; (c.1) Link Force Control; (c.2) Link Force 5% BAG; (c.3) Link Force 10% BAG (While irregular mineralized deposits were not observed in the control groups, they were observed in the groups to which 5% and 10% BAG were added. The observed mineralized deposits are more intense in the groups containing 10% BAG).

cement to dentin ($P = 1.000$ and $P = 0.998$, respectively).

3.4. *Analysis of failure modes*

The patterns of the failure modes are listed in **Table 3**. While the dominant failure mode for Rely-X Ultimate was a cohesive fracture in the dentin, the dominant failure mode for the other cements was an adhesive fracture between the cement and dentin.

3.5. *SEM and EDS analysis results*

In the SEM analysis of the samples wherein BAG was added to the cements under testing, a clustered non-proliferated mineral texture was observed on the dentin surface. In parallel with the SEM analysis, a high silicium ratio was observed in the EDS analysis in the regions comprising mineralized sediments (**Figs. 3 and 4**). This silicium is thought to be released from the SiO_2 in the BAG content. Dentin tubules, thought to be related to the cohesive fracture type,

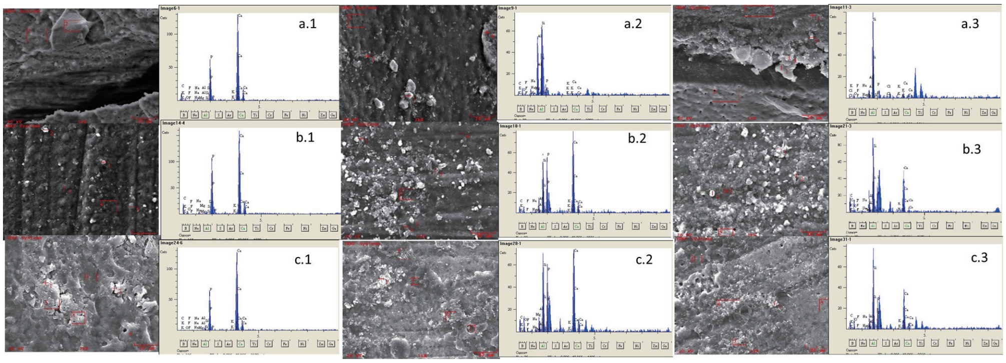

Fig. 4. EDS images obtained after shear bond strength test: (a.1) Rely-X Ultimate Control; (a.2) Rely-X Ultimate 5% BAG; (a.3) Rely-X Ultimate 10% BAG; (b.1) GC Link Ace Control; (b.2) Link Ace 5% BAG; (b.3) Link Ace 10% BAG; (c.1) Link Force Control; (c.2) Link Force 5% BAG; (c.3) Link Force 10% BAG. In the samples prepared with BAG-added resin cements, a high Si ratio was observed owing to the dissolution of SiO_2.

were observed in the sample selected from the Rely-X Ultimate cement group, in which that fracture type was dominant.

4. Discussion

The mechanical and adhesive properties of resin cements have continued to improve through various modifications and content changes in materials[6,27]. In this study, 5% and 10% BAG were added to three resin cements in order to improve the mechanical and adhesive properties of resin cements. And its mechanical and adhesive properties were evaluated and compared. Because self-etch adhesive cements and adhesives in the market have different filler ratios, pH values, and chemical compositions, one self-adhesive and two self-etch cements were tested in this study. The addition of BAG weakened the mechanical properties of all the resin cements, and the first null hypothesis was rejected.

Bending forces are the result of forces produced in the mouth in clinical situations. Dental materials must be able to withstand repeated bending, twisting, and stretching. A high bending strength is desired when these materials are under the influence of chewing stress, which can cause a permanent set[28]. Studies have been conducted to examine the mechanical effects of BAG addition to dental cements, but the majority of them have comprised glass ionomer cements and resin-modified glass ionomer cements (RMGICs) [16,29–31]. In a study conducted by Valanezhad *et al.*, the addition of 3% and 5% sol-gel derivative BAG nanoparticles increased the flexural strength of RMGICs by 5%–70%[16]. However, studies on the addition of BAG to resin cements are limited[6]. As in the study conducted by Assad *et al.*, the addition of BAG reduced the flexural strength in the current study[6]. Similar results were also observed in the study conducted by Yang *et al*. The authors attributed this result to the weak attachment of the BAG particles to the resin matrix because they were not silanized[6,31,32]. In Yang *et al.*'s study, silanized glass and BAG were added to strengthen the mechanical strength of the pit and fissure sealants. The flexural strength decreased with an increase in the BAG proportion[32].

When microhardness was examined after 24 h, similar values were found between the control and BAG groups. This is attributed to the poor attachment of non-silanized particles to the matrix[33]. As a result, it can be considered that the matrix rather than the filler ratio is the most influential factor on the microhardness. However, interesting observations were made in this study on the effect of the hardness of resin cements on adding BAG to them. In all resin cements, the addition of 10% BAG slightly increased the hardness after 24 h compared to the case of the addition of 5% BAG. The highest degree of hardness was observed in the control group. In the groups stored in SBF for 2 weeks, the degree of hardness decreased significantly compared to the values after 24 h.

In the present study, the mechanical properties were affected by storage conditions; thus, the second null hypothesis was rejected. A previous study recommended that bone bonding materials should be stored in SBF for 4 weeks to observe the apatites on the surface[26]. In addition, it has been reported that the most appropriate storage period for dentin remineralization is 8 weeks; however, this period is relatively long. Furthermore, Jang *et al.* demonstrated partial remineralization *in vitro* after 2 weeks with experimental composite resins to which they added BAG[34]. Therefore, a storage period of 2 weeks was used in the present study.

In accordance with a previous study conducted by Valanezhad *et al.*, storing the samples in SBF decreased the Vickers hardness value in the current study[16]. In a previous study, this result was attributed to the surface cracks on the sample stored in SBF[16]. In previous studies, it was observed that the degradation of BAG in BAG-doped composite materials in an aqueous medium reduced their mechanical properties[23,35]. In addition, commercial dental restorative composites show a similar strength loss after aging in aqueous media owing to the plasticization of the resin matrix or degradation of the matrix–filler interface[36,37]. In a study conducted by Ferracane *et al.*, the aging of dental composites in an aqueous medium was surveyed, and it was hypothesized that water absorption causes softening of the polymer resin ingredient by decreasing the frictional forces and swelling the mesh between the polymer chains[38]. One of the purposes of developing composites containing BAG is the leaching of ions from BAG. Furthermore, there is a concern that such ion leakage may result in deterioration of the filler and reduced mechanical properties[21]. Furthermore, the results of our study justify this concern.

It was found that the addition of BAG weakened the bond strength in all resin cements except one, and the third null hypothesis was partially rejected. The most important purpose of any dental cement system is to create a strong and durable bond between the restorative material and dental tissue. Shear bond strength testing is one method of assessing the bond strength in dental research because it is easy to perform and requires minimal equipment and sample preparation[39]. In the groups prepared using 3M Rely-X Ultimate cement, the bond strength of the control group was found to be higher than that of the groups containing 5% and 10% BAG. In the groups prepared using GC Link Ace Cement, the highest bond strength values were observed in the control group. In addition, the 10% BAG group exhibited lower bond strength values than the control group but values higher than the 5% BAG group. In the groups prepared with the GC Link Force cement, the highest bond strength values were observed in the group containing 10% BAG. The lowest bonding value was observed for the group containing 5% BAG. Souza *et al.* evaluated the bond strength of five different resin cements to dentin. It was reported that Rely-X Ultimate, a self-etch cement, had a higher bond strength than the GC Link Ace, a self-adhesive resin cement[40] which is in line with the results of our study. When the bond strength of the two different self-etch resin cements used in our study was evaluated, a statistically significant difference was observed ($P < 0.05$). This may be owing to the different pH values or different solvents used in the adhesive systems. The Scotchbond Universal Adhesive used for 3M Rely-X Ultimate cement is a slightly acidic (pH 2.7) self-etch system for which ethanol is used as a solvent. In the case of the G-Primo Bond used for the GC Link Force cement, acetone is used as a solvent (pH = 1.5), and it is a medium acidic self-etch system. In a study by Ozmen *et al.*, the bond strength of self-etch adhesives of different pH values to the dentin of permanent and primary teeth was evaluated. A weak acidic self-etch adhesive system exhibited the highest bonding values in permanent teeth[41]. These results are similar to the results of our study. Weakly acidic self-etch adhesives form a thin layer of HA, leaving a significant hybrid layer around the collagen fibrils. More aggressive adhesives produce a thick hybrid layer[42,43]. The reason 3M Rely-X Ultimate cement exhibits a higher bond strength than the GC Link Force in the present study could be owing to the weakly acidic primer used.

In a previous study, the bond strength was investigated by adding different ratios of BAG to commercially available composite resins. An increase in the amount of BAG decreased the bond strength proportionally[44]. The authors reported that unsilanized BAG may cause behavior similar to that observed if there are structural defects in the material. The lower bond strength values can be explained by the increase in the amount of BAG. Similarly, in a recent study, the Rely-X Ultimate cement group with BAG displayed lower bond strength values than the control groups. However, this contradicts the results obtained for the other two cement groups. In addition, we did not encounter studies examining the effect of BAG addition on the bond strength of cement to dentin.

It is known that BAG added to resin cement is glass that precipitates hydroxyapatite in aqueous solutions[16]. In a study by Sauro *et al.*, significant resin degradation was observed during SBF storage to promote ion exchange and mineral precipitation at the dentin-resin interface[45]. This can be considered the reason for the decrease in the bond strength values as a result of the long storage period in SBF. Yang *et al.* found that the bond strength of a resin material containing silanized BAG was significantly higher than that of a material containing non-silanized BAG[32]. As the bond strength to enamel was evaluated in this study, it is incorrect to directly compare it with our study. There are limited studies in the literature on the bond strength of BAG-containing restorative materials to dentin. In these studies, it has been reported that the addition of BAG to resin-containing materials will cause reactivity with glass particles, especially in the presence of acidic monomers. For this reason, it is thought that it will give controversial results in terms of bond strength[46][47]. However, Bauer *et al.* reported that the bond strength of dentin conditioned with bioactive glass suspension increased as the amount of bioactive glass increased[48].

In a previous study, experimental glass ionomer cement materials prepared via the addition of BAG were reported to be bioactive under simulated physiological conditions and were found to mineralize human dentin *in vitro*[31]. In the current study, non-proliferated, irregular mineral precipitates were observed in the SEM images of the BAG-containing groups, except for the control groups. At the same time, the EDS analyses showed results consistent with the SEM analysis. Jang *et al.* showed that the addition of BAG particles to experimental composites increased the microhardness of dentin and the mineral composition accumulated in the dentinal tubules. This result was supported by the SEM analysis[34]. Similar to our study, a previous study reported that BAG accumulated on the dentin surface[49]. Aleesa *et al.* added BAG to orthodontic adhesives and observed apatite precipitation using SEM images. In addition, they stated that the rate of apatite precipitation increases over time[50]. However, to evaluate the contribution of BAG addition to the mineralization of resin composites, Yun *et al.* compared alternative solutions other than the standard SBF solution. In this study, it was concluded that SBF is not a good alternative to evaluate mineralization in resin composites containing low BAG[51].

One of the limitations of this study is that aging methods such as mechanical loading or thermal cycling were not used when evaluating the mechanical properties of cements containing BAG. Another limitation is that the storage period of the materials tested in this study was limited. However, the longevity of these materials in clinical use is a crucial factor. Therefore, this is an *in vitro* study, and clinical studies are needed to mimic the dynamic environment of the oral cavity.

5. Conclusions

Within the limitations of this *in vitro* study, the following conclusions were drawn:

1. The flexural strengths of the Rely-X Ultimate and Link Force cements were affected by the addition of different amounts of BAG.
2. While the hardness of the cements was not affected by the addition of BAG, it was affected by the storage conditions.
3. It was observed that the addition of 10% BAG to the Link Force resin cement had a positive effect on the bond strength. In the cases of the other two cements, the addition of BAG decreased the bond strength compared to that of the control group.

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Conflict of interest

There are no conflicts of interest to declare regarding this study.

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