



Original Article

# Effect of Fluoride Releasing Bonding Materials on Shear Bond Strength of Orthodontic Brackets

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## ABSTRACT

**Objective:** The aim of the present study was to compare the shear bond strength (SBS) of three different fluoride-releasing bonding agents with a conventional adhesive system.

**Methods:** Eighty-four extracted human premolar teeth were separated into four groups and embedded in acrylic molds consisting of 21 teeth in each group. Brackets were bonded with Transbond XT in group 1, Clearfil SE Protect Bond in group 2, LED Proseal in group 3, and Opalseal in group 4. After bracket bonding, the teeth were thermocycled 1000 times. SBS test was performed, and Adhesive Remnant Index (ARI) scores of the groups were assessed.

**Results:** One-way analysis of variance test was used to compare the significant differences between the groups. Chi-square and Fisher's exact tests were used to evaluate ARI scores. The Opalseal group showed the highest bond strength, but there was no statistically significant difference between the groups in SBS values ( $p=0.067$ ). The results of ARI scores were statistically significant.

**Conclusions:** All bonding materials used in the study showed clinically sufficient bond strengths.

**Keywords:** ARI, bonding, fluoride, orthodontic brackets, shear bond strength

## INTRODUCTION

It is difficult to maintain oral hygiene when the fixed orthodontic appliances are placed on the teeth. Residual adhesive and rough surfaces of brackets, arch wires, or ligatures may increase bacterial colonization, and tooth demineralization may occur (1). During fixed orthodontic treatment, demineralization is one of the major problems, especially in patients with poor oral hygiene (2-4). The first step of demineralization is white spot lesions (WSLs), and these lesions could be seen clinically exactly 4 weeks after the beginning of orthodontic treatment (5). The frequencies of WSL were reported to be between 2% and 96% in orthodontic patients and 25% in non-orthodontic patients (2-6).

It has been reported that the use of fluoride during orthodontic treatment reduces demineralization (4-6). The uses of fluoride-containing toothpastes, mouthwashes, and gels require patient cooperation, but applications of fluoride-releasing glass ionomer cements, fluoride-added composites, fluoride-releasing bonding agents, fluoroelastomeric ligatures, or fluoride lacquers need no cooperation. The use of fluoride-containing bonding agents during orthodontic treatment is a non-patient-dependent protective action.

In orthodontic direct bonding, acid etchant is used to remove prismatic and interprismatic enamels, and after that a primer (bonding agent) is applied to the enamel to form resin tags. Orthodontic adhesive can penetrate the enamel surface by the aid of a bonding agent (7, 8).

Conventional or fluoride-releasing bonding agents can be used in orthodontic bonding. Fluoride-releasing bonding supplies fluoride ions by the aid of the aqueous oral environment, and these ions penetrate the enam-

el prisms. Fluoride ions transform the hydroxyapatite crystals to fluorohydroxyapatite, and the structure of the enamel becomes more resistant to acid attacks and caries. Therefore, this strong fluorohydroxyapatite barrier may have different effects on the bond strength of orthodontic brackets (9).

The effect of bonding agents on bracket bond strength was previously reported in several studies (10, 11). Various brands of bonding agents are present in the market, and bond strength of these agents is critical for orthodontists. Therefore, the aim of the present study was to compare the shear bond strength (SBS) of three different fluoride-releasing bonding agents with a conventional bonding agent. The null hypothesis of the present study was that the fluoride-releasing bonding materials do not have any effect on the SBS of orthodontic brackets.

## METHODS

Sample size estimation was performed prior to the study using the G\*Power 3.0.10 software with a 95% confidence interval (CI) and  $\alpha$  of 0.05 to detect a significant difference of 1 MPa in SBS value, and it was determined that to have a power of 80%, there should be 19 teeth in each group (12). According to sample size estimation, 84 human first premolar teeth were used in the present study, meaning that 21 teeth were included in each group. Inclusion criteria were as follows: teeth

were not extracted for periodontal purpose and teeth with no caries, no filling or restoration, no crack on the surface of the enamel, and no malformation on the vestibule surface. The study was approved by the Research Ethics Committee of Istanbul Medipol University. (protocol no. 10840098-604.01.01-E.5731, 21/04/2016).

The enamel surfaces were assessed before the experiment by using a stereomicroscope (SZX10; Olympus, Japan) at 10 $\times$  magnification, and the teeth that did not meet the criteria were excluded from the study. The teeth were washed to remove organic debris and were kept in a 0.1% thymol solution to prevent degradation of the enamel structure and bacterial colonization.

### Preparation of Acrylic Molds

The teeth were removed from the thymol solution, washed, and dried, and grooves were opened using a diamond bur on the root surface to provide retention before embedding to acrylic blocks. The teeth were embedded in acrylic blocks vertically to the ground, and the long axis of the teeth up to 1 mm apical of the cement–enamel junction was exposed (Figure 1). Plastic cylindrical molds with a 25 mm inner diameter and 30 mm height were used to prepare acrylic blocks. The vestibule surfaces of the teeth were brushed with a micromotor for 15 s using a soft brush and a fluoride-free pumice, washed for 15 s, and then dried. The teeth were treated with 37% phosphoric acid, each bonding material was applied in accordance with the manufacturer's instruction, and then all of the brackets were bonded with Transbond XT adhesive (3M Unitek, Monrovia, CA, USA). Light curing of the adhesive was performed for 20 s using 3M Espe Elipar S10 (3M ESPE, Seefeld, Germany). Brackets were bonded with Transbond XT (3M Unitek) in group 1, with Clearfil SE Protect Bond (Kuraray Medical Inc., Tokyo, Japan) in group 2, with LED Proseal (Reliance Orthodontics, IL, USA) in group 3, and with Opalseal (Opal Orthodontic; Ultradent, South Jordan, UT, USA) in group 4. After bonding of the brackets, all groups were kept in distilled water at room temperature for 24 hours and then subjected to thermocycling with a thermal cycler (SD Mechatronik Thermocycler THE-1100; Feldkirchen-Westerham, Germany). The samples were immersed in water baths at temperatures between 5 °C and 55 °C for 1000 times. The samples were set to have a waiting time of 30 s and a transfer time of 5 s in each bath.

### SBS Test

SBS tests were performed by a Universal Test Machine (Shimadzu Autograph AGS-X, Japan) at a crosshead speed of 1 mm/min loading on bracket–tooth interface by using a 0.5 mm thickness blade (60° cut end face, Shimadzu toothed pushrod B, Japan) (Figure 2). The specimens were placed as their long axis was vertical to the ground and fixed in the mesiodistal direction by using two screw plates to avoid their rotational movement. The force at debonding of the bracket was recorded in Newton (N); thereafter, the results were converted to megapascals (MPa) by dividing the force value (N) into the bracket base area (mm<sup>2</sup>). The bracket surface area was 11.98 mm<sup>2</sup> according to the manufacturer's instruction. The buccal surfaces of the teeth were assessed using a camera of a stereomicroscope (SZX10; Olympus) at 20 $\times$  magnification. Residual adhesive on the teeth surface was classified using the Adhesive Remnant Index (ARI) (13). The ARI scores were as follows: 0: no adhesive residue on the tooth, 1: <50% of

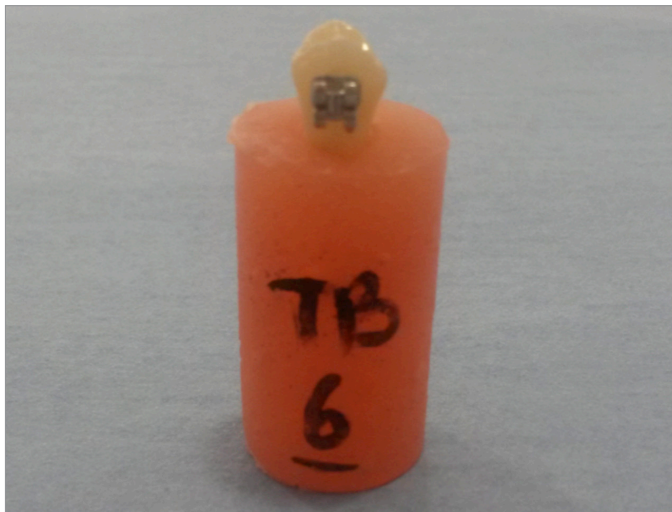


Figure 1. Plastic cylindrical molds

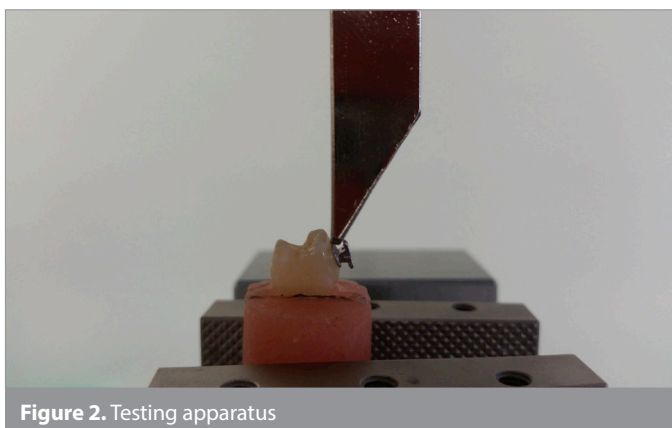
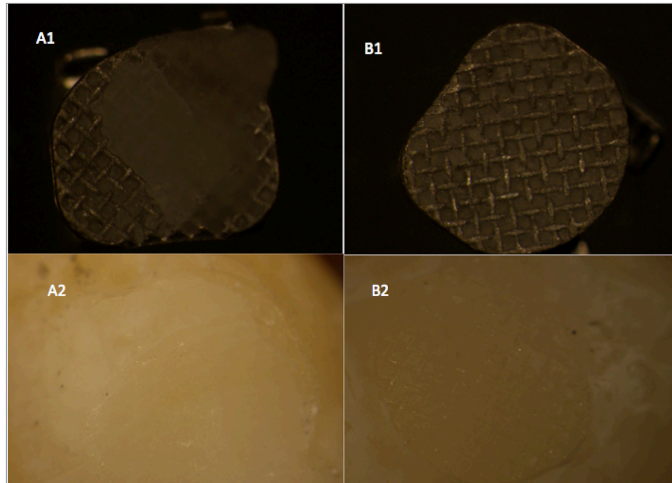


Figure 2. Testing apparatus

adhesive remains on the tooth, 2: >50% of adhesive remains on the tooth, and 3: all the adhesive remains on the tooth (Figure 3).

Two samples from each group were examined at 40x and 250x magnification by using a scanning electron microscope (Zeiss EVO LS 10; Carl Zeiss, Oberkochen, Germany) (Figure 4-7).



**Figure 3. a, b.** ARI views of the sample brackets and teeth.

(A1) ARI 1 score bracket view. (A2) ARI 1 score tooth view. (B1) ARI 2 score bracket view. (B2) ARI 2 score tooth view

**Statistical Analysis**

Data were evaluated by Statistical Package for Social Sciences version 22.0 (IBM Corp.; Armonk, NY, USA). Shapiro–Wilk test was used to evaluate the normality of the data. One-way analysis of variance (ANOVA) test was used to compare group differences. Tukey HSD (Honestly Significant Difference) test was used for post-hoc comparisons. Chi-square and Fisher’s exact tests were used to evaluate qualitative data. A p value <0.05 was considered as significant.

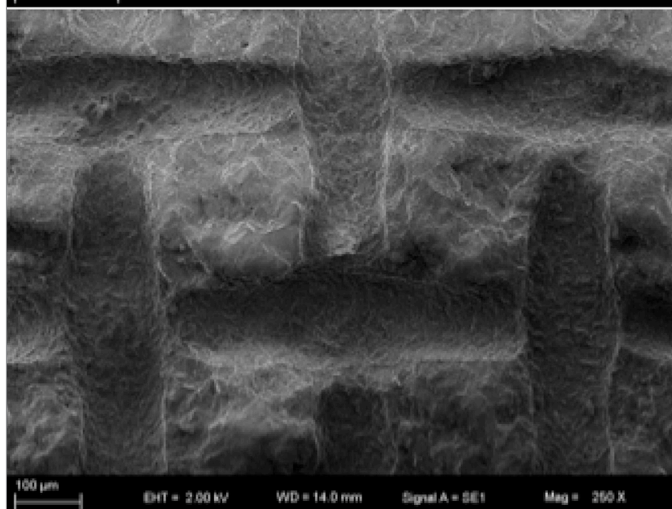
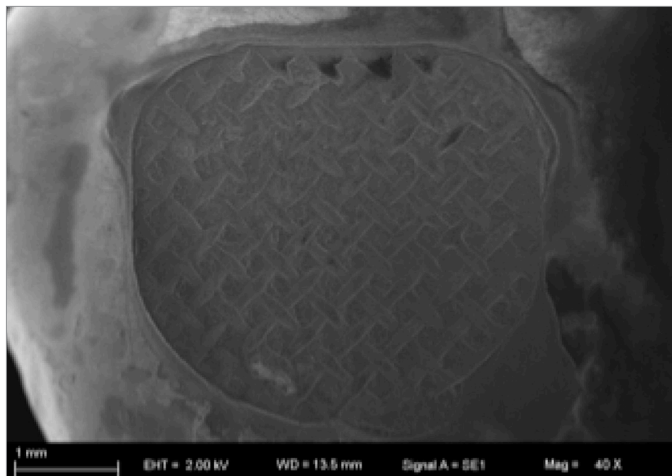
**RESULTS**

**SBS Test Results**

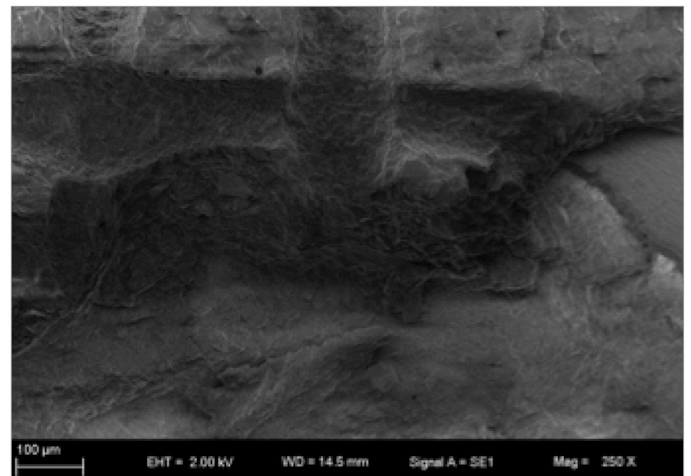
The results of SBS tests are given in Table 1. There was no statistically significant difference in SBS between the groups (p=0.067). The Opalseal group showed the highest bond strength (12.56±2.32). The lowest bond strength was measured in the Proseal group (10.66±2.06).

**ARI Scores**

The results of ARI scores are given in Table 2. There was a statistically significant difference between the groups with respect to ARI scores (p=0.016 and p<0.05). There were no ARI scores of 0 and 3.



**Figure 4.** SEM view of the Transbond XT sample



**Figure 5.** SEM view of the Clearfil sample



**DISCUSSION**

Fluoride-containing materials are widely used to prevent WSLs in orthodontic practice. It is possible to measure the bond strength of brackets in in vivo and in vitro conditions. Murray and Hobson reported that there is a difference in force values between these two conditions in their study (14). The mean bond strength values of brackets were found to be 9.78 MPa in vitro and 14.34

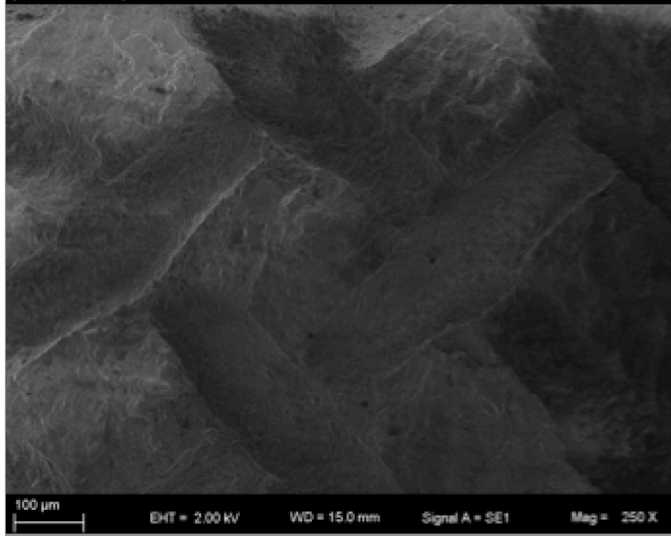
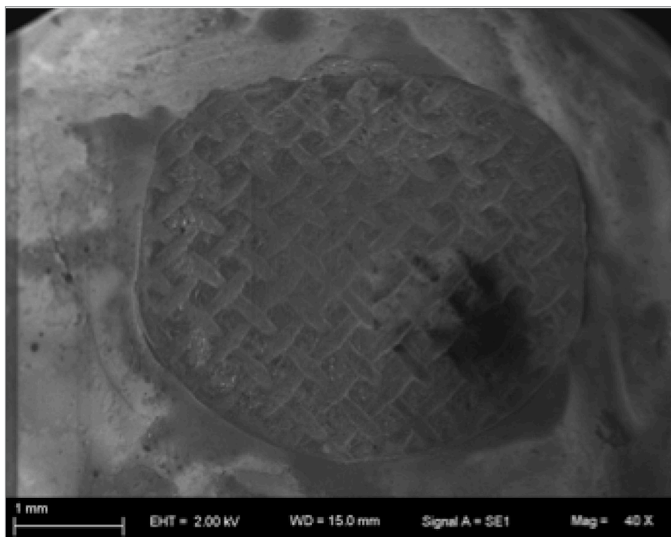
MPa in vivo. Researchers have usually preferred to perform in vitro bond strength tests instead of in vivo ones because of the difficulty of intraoral measurements of bond strength (10-12). Therefore, bond strength values were measured in vitro in the present study.

The incisor, premolar, and molar teeth can be used in SBS tests (10-12, 15). Human premolar teeth were used in the present

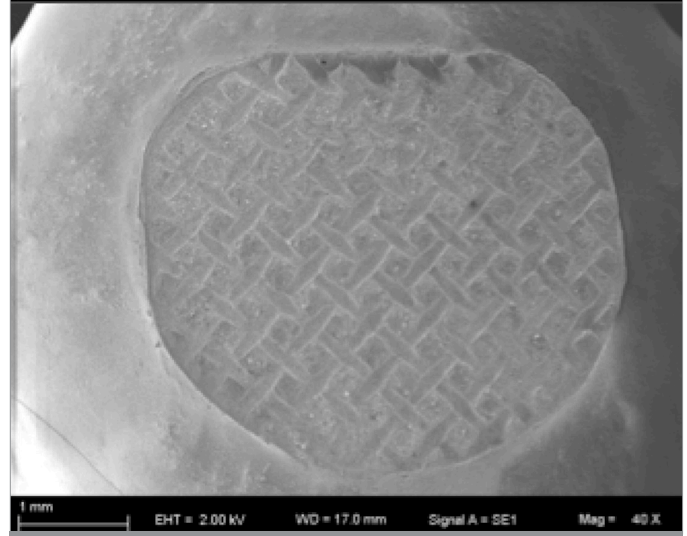
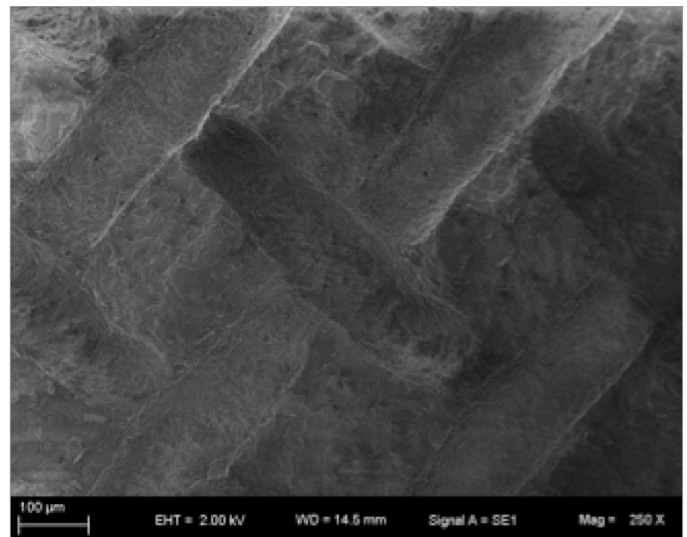
**Table 1.** Shear bond strength values of the compared groups stratified by one-way ANOVA test and Tukey HSD test results

	One-way ANOVA		Tukey HSD			
	Shear bond strength (MPa) Mean±SD		Mean difference	95% CI (min)	95% CI (max)	p
Transbond XT	11.55±3.06	Transbond XT–Clearfil	0.800	-2.875	1.275	0.743 (NS)
Clearfil	10.75±2.70	Transbond XT–Opalseal	1.010	-1.065	3.085	0.580 (NS)
Opalseal	12.56±2.32	Transbond XT–Proseal	0.890	-2.965	1.185	0.675 (NS)
Proseal	10.66±2.06	Clearfil–Opalseal	1.810	-0.265	3.885	0.109 (NS)
p	0.067 (NS)	Clearfil–Proseal	0.090	-2.165	1.985	0.999 (NS)
		Opalseal–Proseal	1.900	-3.975	0.175	0.084 (NS)

\*p<0.05.  
NS, non-significant.



**Figure 6.** SEM view of the Opalseal sample



**Figure 7.** SEM view of the Proseal sample

**Table 2.** Comparison of ARI scores between the groups based on Fisher's exact and chi-square tests

	ARI scores n (%)			
	0	1	2	3
Transbond XT	0 (0)	5 (23.8)	16 (76.2)	0 (0)
Clearfil	0 (0)	0 (0)	21 (100)	0 (0)
Opalseal	0 (0)	0 (0)	21 (100)	0 (0)
Proseal	0 (0)	4 (19)	17 (81)	0 (0)

\*p<0.05.

study because they can be easily obtained by orthodontic extractions.

Dental materials are subjected to thermal, mechanical, and chemical stresses in the mouth. Applying thermal cycle or water retention process to the dental materials allows to simulate oral conditions. Application of thermal cycling was previously reported to make a decrease in SBS values (16-18). The thermal cycling was usually conducted between 5 °C and 55 °C in different numbers of cycle, such as 500, 1000, 2000, 10,000, and 20,000. Bishara et al. (19) demonstrated that there is a significant reduction in bond strength of a cyanoacrylate-containing adhesive up to 80% after 500 cycles of thermal cycling. However, Hasegawa et al. (20) suggested that the effect of 500 rounds of thermal cycling will not be sufficient to change the bond strength. In light of this knowledge, 1000 rounds of thermal cycle between the temperatures of 5 °C and 55 °C were applied to the specimens in the present study.

Different brands of universal testing machines were previously used for SBS tests (10-12). The angle and the speed of the blade can change the reliability of SBS tests. As the angle of the applied force changes, the SBS is also affected. Klocke and Kahl-Nieke (21) reported that as the blade angle changes from +15° to -45°, the connection force values decrease from 22.9 MPa to 6.65 MPa. In our study, the parallelism of the blade to the long axis of the bracket was checked before each force application. Bishara et al. (22) stated that if the blade speed decreases from 5 to 0.5 mm/min, the bond strength increases from 7 to 12.2 MPa. The test reliability of in vitro studies decreases as the blade speed increases; therefore, we used a blade with a crosshead speed of 1 mm/min (10-12, 23).

Reynolds (2) stated that the bond strength values of the brackets should be in the range of 5.9–7.8 MPa or above in clinical and 4.9 MPa in laboratory conditions. Enamel fracture strength was known as 14 MPa, and it was reported that an increase in the risk of enamel fracture can be seen over the value of 10 MPa. The desired mean values of SBS were criticized in previous studies, but no consensus was present in the literature (25-27). All groups in our study provided sufficient SBS values.

Artun and Bergland (13) defined the ARI score to assess the adhesive remnants, which is still widely used today, and in our study, the original ARI score was performed (12, 23, 28, 29). The failure type is not only related with the applied debonding force but also related with the type of the adhesive and the bracket

base design (29). In our study, although statistically significant differences were observed in ARI scores between the groups, the results were generally concentrated in ARI 2 score. Although bonding materials were different, the use of the same bracket and adhesive might have an effect on the similarity of ARI scores. The ARI 2 score shows that debonding occurred at the bracket–adhesive interface. Bishara et al. (30) advocated the failure that occurred in the bracket–adhesive interface and stated that this type of debonding can reduce enamel fractures.

The results of our study were compared with other similar studies in the literature. However, the lack of standardization in many factors, such as the type of teeth, storage conditions, preferred acid type, type of the adhesive, bracket type, light curing device, and light curing time, whether thermal cycle is applied or not, and the crosshead speed of the test device prevented us to perform the precise comparison. Korbmacher et al. (23) compared the SBS values of a conventional bonding agent (Transbond XT) with fluoride-releasing self-etching primer (Clearfil Protect Bond, CPB) and found that SBS results and ARI scores of their study were consistent with our study. Arhun et al. (31) evaluated the SBS values of Adper Prompt L-Pop (3M ESPE, St. Paul, MN, USA) self-etch adhesive, CPB, and Transbond plus self-etching primer (3M Unitek) in their study and found a significant difference between the groups. CPB showed the highest SBS value of 13.85±4.32 MPa. Although the etching process was not performed before application, the SBS values of CPB was higher than that of our study. Application of thermal cycle might have decreased the bond strength in this study. Tuncer et al. (10) assessed the SBS values of Transbond Self-etching Primer (3M Unitek) and Ortho-Coat, CPB, and CPB+Ortho-Coat. The mean SBS value of the CPB group was 13.48±1.78 MPa, which was higher than that of our study, and this result can be attributed to the absence of thermal cycle in their study. No significant difference was observed in ARI scores between the groups, and the majority of the failures were in the enamel–adhesive interface in contrast to our study. Minick et al. (28) used Aegis Ortho (Bosworth Co., IL, USA), CPB, iBond, Clearfil S3 Bond (Kuraray, USA), and Transbond XT (3M Unitek) combined with metal brackets and bovine teeth in their study. Transbond XT showed 10.05±0.84 MPa, and CPB showed a 7.5±0.79 MPa bond strength exactly after bonding. The specimens that were tested after 24 h showed 10.11±1.02 MPa and 6.09±0.56 MPa SBS values, respectively. Lower SBS values of samples may be related to the use of bovine teeth in that study. On the other hand, CPB showed clinically sufficient bond strength values, and the ARI scores were similar to our study. Raji et al. (32) assessed the SBS values of Transbond XT and CPB, and they could not find a significant difference between the groups. The SBS values and ARI scores of their study were consistent with our study. Soake et al. (33) evaluated the SBS values of Clearfil SE, CPB, Prompt L-Pop, and Reliance self-etching primer and found that the mean SBS value of CBP is 11.94±2.74 MPa, which was similar to our study.

Bishara et al. (34) investigated the effects of Proseal on the bond strength of orthodontic brackets comparing with conventional bonding agent, and no significant difference was found between the groups. Furthermore, the mean SBS value of the Proseal group was found to be 4.8±2.3 MPa. Although the SBS value of Proseal

was clinically sufficient, it was quite low compared with the SBS value in this study. This difference may have been related to the application of SBS tests exactly 30 min after bonding of the brackets and use of molar teeth in that study. Paschos et al. (35) assessed conventional and self-etch adhesive systems whether they affect the bond strength. As a result, they found that the use of Proseal had no negative effect on the bond strength. The bond strength of Proseal after 500 cycles of thermal cycling showed a very close result ( $10.8 \pm 2.9$  MPa) to our findings. Similar to our study, the ARI scores were concentrated in 2 scores. Varlik and Ulusoy (36) reported that Proseal does not have a significant effect on the SBS values of any group in their study. The Proseal-metal bracket combination group presented a mean value of  $6.65 \pm 1.01$  MPa, which was lower than that of our results. This difference may have originated from the use of different types of bracket and adhesive.

Hofmann et al. (37) combined three different kinds of fluoride-releasing bonding materials and a conventional bonding agent (Transbond XT) with four different kinds of orthodontic brackets. Similar to our study, they stated that all bonding materials presented adequate SBS values for clinical application. Furthermore, Transbond XT showed the highest SBS values among the other fluoride-releasing agents.

Kirschneck 2019 et al. (38) used Proseal in their prospective split-mouth study, and they stated that the use of enamel sealant before bracket bonding may increase the probability of bond failure especially in the lower jaw. They concluded that it is more suitable to use fluoride-releasing materials adjacent to the brackets after bracket bonding.

The nature of the present study was a limitation, and in vivo studies would provide more precise knowledge about this issue.

The study would be more valuable if the calcium and fluoride mass of the enamel could be measured with energy dispersive X-ray microanalysis.

## CONCLUSION

- There was no statistically significant difference between mean SBS values of the Transbond XT, Clearfil SE Bond, Opalseal, and LED Proseal groups. The null hypothesis was accepted.
- The highest SBS values were measured in the Opalseal group, followed by the Transbond XT, Clearfil SE Protect Bond, and Proseal groups, respectively.
- The bond strength of all groups were above the desired SBS value of 6-8 MPa.

**Ethics Committee Approval:** Ethics committee approval was received for this study from the Research Ethics Committee of Istanbul Medipol University (protocol no. 10840098-604.01.01-E.5731, 21/04/2016).

**Informed Consent:** N/A.

**Peer-review:** Externally peer-reviewed.

**Author Contributions:** Concept - G.S.; Design - G.S.; Supervision - G.S.; Resources - D.Y.; Materials - D.Y.; G.S.; Data Collection and/or Processing - G.S., D.Y.; Analysis and/or Interpretation - G.S.; Literature Search - D.Y.; Writing Manuscript - G.S.; Critical Review - G.S.

**Conflict of Interest:** The authors have no conflict of interest to declare.

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