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Original Article

Comparative evaluation of shear bond strength of metallic brackets bonded with two different bonding agents under dry conditions and with saliva contamination

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Abstract

Background: This study compared the shear bond strength of metallic brackets bonded with Single Bond and Assure bonding agents under dry and saliva-contamination conditions.

Methods: Sixty sound premolar teeth were selected, and stainless-steel brackets were bonded on enamel surfaces with Single Bond and Assure bonding agents under dry condition or with saliva contamination. Shear bond strength values of brackets were measured in a universal testing machine. The adhesive remnant index scores were determined after debonding of the brackets under a stereomicroscope. One-way analysis of variance (ANOVA) was used to analyze bond strength. Two-by-two comparisons were made with *post hoc* Tukey tests ($p < 0.001$). Frequencies of adhesive remnant index scores were analyzed by Kruskal−Wallis test.

Results: Bond strength values of brackets to tooth structure were 9.29 ± 8.56 MPa and 21.25 ± 8.93 MPa with the use of Assure resin bonding agent under saliva-contamination and dry conditions, respectively. These values were 10.13 ± 6.69 MPa and 14.09 ± 6.6 MPa, respectively, under the same conditions with the use of Single Bond adhesive. Contamination with saliva resulted in a significant decrease in the bond strength of brackets to tooth structure with the application of Assure adhesive resin ($p < 0.001$). There were no significant differences in the adhesive remnant index scores between the study groups.

Conclusion: Application of Single Bond and Assure bonding agents resulted in adequate bond strength of brackets to tooth structures. Contamination with saliva significantly decreased the bond strength of Assure bonding agent compared with dry conditions.

Keywords: Assure universal bonding resin; shear bond strength; Single Bond adhesive resin

1. Introduction

A proper bond between a bracket and the enamel is necessary for orthodontic treatment.[1] Favorable shear bond strength is in a range to withstand oral and occlusal forces during treatment. At the same time, it should be easy to debond the bracket at the end of treatment without inflicting any damages on the enamel. During the bonding process, there is always the risk of contamination of the etched surfaces with saliva. Contamination of enamel surfaces with saliva has been reported as one of the etiologic factors for bond failure.[2] Conventional composite resins require a dry and contamination-free surface to achieve adequate bond strength; however, under clinical conditions, it is difficult to completely isolate the area in question against moisture during the bracket-bonding procedure,[3] and it is possible for the enamel surfaces to become contaminated during etching and after the application

Conflicts of interest: The authors declare that they have no conflicts of interest related to the subject matter or materials discussed in this article.

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of primer.[4] If the enamel surfaces are contaminated before the application of primer, porosities produced due to the effect of the acid etching procedure will become occluded and surface energy of the enamel will decrease, interfering with the penetration of resin tags, which will result in a decrease in micromechanical retention and finally in a decrease in the bond strength between the resin and the etched enamel.[5,6]

Assure universal bonding resin is a relatively new product with fluoride-releasing properties. This bonding agent has been reinforced with a resin cement,[7] has hydrophilic properties, does not need to be photoactivated, and has the capacity to bond to light-cured or dual-cured adhesives. The Assure hydrophilic resin system (Reliance Orthodontic Products, Inc., Itasca, Illinois, USA) has been evaluated under wet conditions in some cases, and proper bond strength values have been reported under such conditions.[3,4,8] It has been claimed that the bond strength of Assure adhesive agent is not affected by contamination with saliva.[9] Therefore, the present study was undertaken to compare the shear bond strength values of metallic brackets bonded with the use of Single Bond and Assure bonding agents in order to determine a more reliable technique for bonding under dry conditions and contamination with saliva.

2. Methods

The present *in vitro* study was carried out on 60 sound human premolar teeth extracted for orthodontic reasons. The teeth had no carious lesions, fractures, cracks, or abrasion. The teeth were stored in 0.2% thymol solution at room temperature before initiation of the study and between the various study procedures.[10] The samples were randomly divided into the following four groups ($n = 15$):

(1) Single Bond (3M ESPE, St. Paul, MN, USA) group under dry conditions
(2) Single Bond group under contamination with natural saliva
(3) Assure universal bonding resin (Reliance Orthodontic Products, Inc.) group under dry conditions
(4) Assure universal bonding resin group under contamination with natural saliva

In all the groups, the coronal buccal surfaces of the teeth were polished with fluoride-free pumice for 10 seconds, rinsed for 30 seconds, and dried.[10]

Ortho Organizer Company (San Marcos, Calif, USA) 0.22 standard metallic stainless-steel brackets, with a base surface area of 11.8 mm^2, were bonded to tooth structures using different bonding protocols as follows:

(1) In Group 1, the buccal enamel surfaces of the teeth were etched with 37% phosphoric acid (3M Unitek, Monrovia, Calif, USA) for 15 seconds, rinsed for 30 seconds,[11] and dried with oil-free air stream so that a white chalky appearance of enamel was achieved. Then, the Single Bond bonding agent (3M ESPE) was applied to the buccal surface in two layers, left undisturbed for 10 seconds to dry gradually, and light cured for 10 seconds using a Woodpecker light-curing unit (Foshan, Guangdong, China). Then 3M Unitek composite resin was applied to the base of the brackets, followed by determination of the exact position of the brackets. The brackets were pressed on the tooth surface to extrude extra composite resin from underneath the brackets. Extra composite resin was removed from the periphery of the bracket bases using a small dental explorer. Then, the brackets were irradiated from the mesial and distal aspects for 20 seconds each. All the procedures were carried out according to the manufacturers' instructions.
(2) In Group 2, all the etching, rinsing, and drying steps were carried out based on the Single Bond protocol; however, before the application of bonding, a thin layer of natural saliva was applied on the enamel surface.[3] The saliva sample had been collected by the operator after cleaning the teeth of the persons abstaining from eating for 1 hour. All other procedures were similar to those in Group 1.
(3) In Group 3, Assure universal bonding resin was used. All the etching, rinsing and drying procedures conformed to the Assure bonding agent application protocol. The bonding agent was applied in two layers on the buccal surface, left undisturbed for 10 seconds, and dried gently. Then, the composite resin was applied to the bracket bases, and their positions on the enamel surfaces were determined carefully. The brackets were pressed on the enamel surfaces to extrude the extra composite resin to leave a minimum thickness of composite resin under the bracket. Extra composite resin was removed from the periphery of the brackets, followed by light curing from the mesial and distal aspects for 20 seconds each.
(4) In Group 4, the teeth were etched, rinsed, and dried. Before application of the Assure bonding agent, a thin layer of natural saliva was applied on the surface of the etched enamel. Then two coats of the Assure adhesive resin were applied on the buccal surface and left undisturbed for 10 seconds. The rest of the procedures were similar to those carried out and explained for Group 3.

After the bonding procedures, all of the samples were incubated at 37°C for 1 week. The samples were then subjected to a 100-round thermocycling procedure at 5–50°C, consisting of 30 seconds of dwell time and 15 seconds for transfer between water baths. In the next stage, a surveyor was used to mount the samples in a way that brackets were placed in the highest buccal surfaces of the teeth in an identical position so that the debonding force would be applied perpendicular to the tooth−bracket interface. An elecromechanical universal testing machine (K-21046; Walter+bai, Löhningen, Switzerland) was used to apply shearing force with a preload force of 0.5 N at a crosshead speed of 1 mm/min to debond the bracket from the tooth surface. The debonding force was measured in Newtons. Then the shear bond strength values were calculated in MPa by dividing force (N) by the cross-sectional surface area (mm).

After debonding, the samples were evaluated under a stereomicroscope at 10× magnification to determine adhesive remnant index (ARI) scores as follows:

0: no adhesive resin remaining on the composite resin
1: less than 50% of the adhesive resin remaining on the composite resin surface
2: more than 50% of the adhesive resin remaining on the composite resin surface
3: 100% of the adhesive resin remaining on the composite resin surface

Finally, four samples were randomly selected from each group for Scanning Electron Microscopy (SEM) evaluations. To this end, the samples were bisected using a diamond saw after measuring the shear bond strength values. One-half was selected for the visualization of the contact surface. Sample surfaces were sputter coated and evaluated using SEM to determine the bond failure modes and the quality of enamel destruction.

Two-way analysis of variance (ANOVA) was used to determine the effect of bonding agent and bonding conditions on the shear bond strength. One-way ANOVA was used to analyze differences in bond strength values with the use of two different bonding agents under dry and saliva-contamination conditions. *Post hoc* Tukey tests were used for two-by-two comparisons. Nonparametric Kruskal−Wallis test was used to compare the frequencies of different ARI scores between the four study groups. Statistical significance was set at $p < 0.0001$.

3. Results

Two-way ANOVA did not reveal any significant differences between the effects of bonding agent type on the shear bond strength of metallic brackets to tooth structures ($p = 0.12$). However, the effects of dry condition and saliva contamination on the shear bond strengths of brackets were significant ($p < 0.0001$). Table 1 presents the results of two-way ANOVA.

One-way ANOVA showed significant differences in the shear bond strength values of metallic brackets bonded to tooth structures with Single Bond and Assure bonding agents under dry and wet (contamination with natural saliva) conditions ($p < 0.0001$), with Assure bonding agent providing the highest bond strength under dry conditions and the lowest bond strength under contamination with saliva.

The results of *post hoc* Tukey tests showed significant differences in the bond strength values of brackets to tooth structures between Single Bond bonding agent under saliva-contamination conditions and Assure adhesive resin under dry and saliva-contamination conditions ($p < 0.001$). However, in other cases there were no significant differences between the groups. In general, the shear bond strength of metallic brackets under saliva-contamination conditions was less than that under dry conditions.

Table 2 presents the ARI scores in different study groups. Kruskal−Wallis test did not demonstrate any significant differences in the frequencies of ARI scores between the different study groups ($n = 15$; $p = 0.29$).

Figures 1−4 present the SEM photomicrographs of the effects of different bonding agents and bonding conditions on the quality of bracket bonds to enamel. As shown by the photomicrographs, contamination with saliva prevented complete penetration of resin tags into the enamel surface porosities and their obturation with the use of both bonding agents, resulting in a decrease in bond strength when contamination with saliva occurred (Table 1).

4. Discussion

One of the prerequisites for bonding of brackets to tooth structures is the provision of a dry environment by careful isolation of the tooth surface. Unfortunately, such isolation is difficult, especially in the posterior area, and is considered a clinical challenge for clinicians. Several methods have been suggested to solve this problem, including the use of hydrophilic materials, the bonding of which is either not influenced or influenced minimally by environmental moisture.[2,12]

Based on the results of the present study, effects of bonding agent type (Single Bond vs. Assure universal bonding resin) on the shear bond strength of metallic brackets to tooth structure were not significant ($p = 0.12$); however, the effects of bonding conditions (dry and wet) on the bond strength of brackets were significant ($p < 0.0001$). Bond strength values of stainless-steel brackets bonded to enamel with the use of Single Bond adhesive (14.09 MPa in dry condition and 10.13 MPa with saliva contamination) and Assure resin

Table 1
Shear bond strength of metallic brackets to tooth structures with the use of different bonding systems and conditions (MPa).

Group	Mean	SD	Std error	95% Confidence interval		Min MPa	Max MPa
				Lower bound	Upper bound		
Dry; Single Bond	14.09	6.6	1.7	10.43	17.74	4.11	25.26
Wet; Single Bond	10.13	6.69	1.7	6.43	13.84	2.43	20.7
Dry; Assure	21.25	8.93	2.3	16.3	26.19	7.02	33.84
Wet; Assure	9.29	8.56	2.2	4.55	14.02	1.63	29.1

$p < 0.001$.
SD = standard deviation.

Table 2
Frequencies of ARI scores in different study groups.

Group	ARI			
	0	1	2	3
Dry; Single Bond	0 (0%)	8 (53.3%)	4 (26.7%)	3 (20.0%)
Wet; Single Bond	5 (33.3%)	10 (66.7%)	0 (0%)	0 (0%)
Dry; Assure	2 (13.3%)	8 (53.3%)	3 (20.0%)	2 (13.3%)
Wet; Assure	5 (33.3%)	8 (53.3%)	1 (6.7%)	1 (6.7%)
Total ($n = 60$)	12 (20.0%)	34 (56.7%)	8 (53.3%)	6 (10.0%)

$p = 0.29$.
ARI = adhesive remnant index.

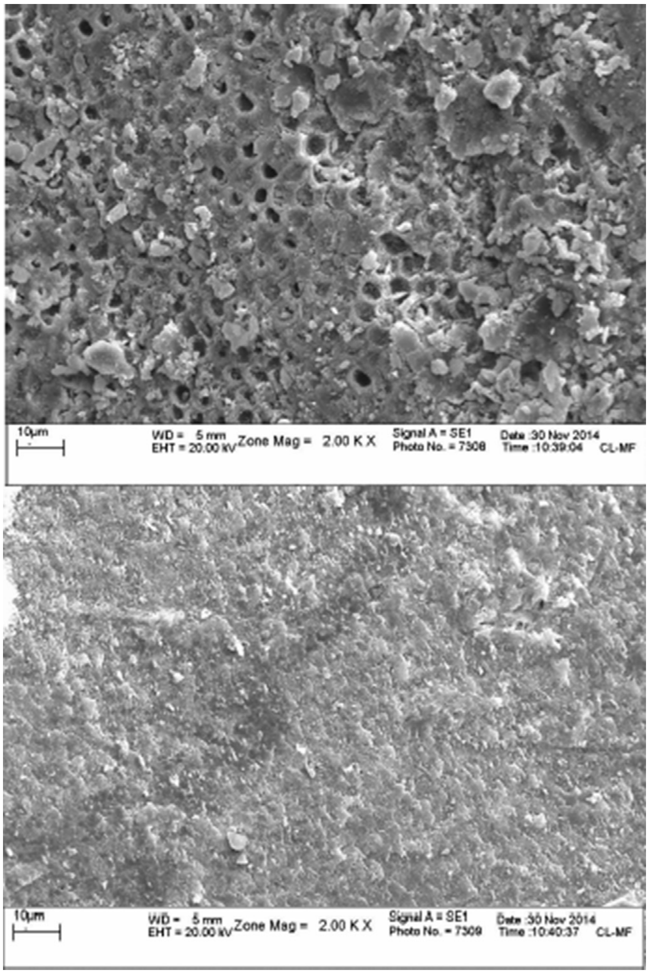

Fig. 1. SEM photomicrographs in the Single Bond group under dry conditions; penetration of resin tags into enamel porosities and their complete obturation. SEM = scanning electron microscopy.

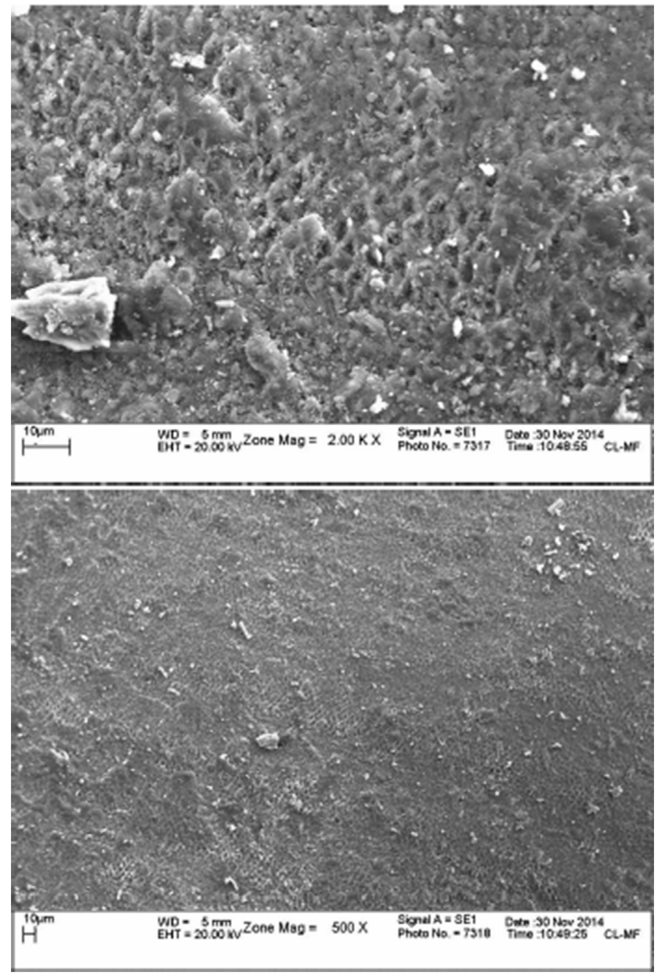

Fig. 2. SEM photomicrographs in the Single Bond group in the presence of saliva contamination; partial penetration of resin tags into enamel porosities. SEM = scanning electron microscopy.

bonding agent (21.25 MPa in dry condition and 9.29 with saliva contamination) were in the favorable range of bond strength to enamel. However, contamination with saliva resulted in a significant decrease in the shear bond strength values of metallic brackets bonded to enamel with the use of Assure adhesive resin ($p < 0.001$), but such a decrease was not significant with the application of Single Bond adhesive agent. Although the bond strength with the application of Assure adhesive resin was significant with saliva contamination, the bond strength was in the favorable range.

Previous studies on the effects of contamination with saliva on the bond strengths of brackets have yielded different and in some cases contradictory results. While some researchers have reported an increase in bond strength after contamination with saliva,[13–15] some others have reported either no decreases [15] or significant decreases in bond strength after contamination with saliva.[16] Differences in these study results might be attributed to the use of artificial or natural saliva or the amount of saliva used. Moreover, composition of saliva might be different based on the conditions of the test.[17] In addition, bonding technique, too, might affect the results of the bond strength test.

Assure adhesive resin is composed of biphenyl dimethacrylate (<35%), hydroxyethyl methacrylate (<20%), and acetone (<80%). It has been formulated to improve adhesion to normal and abnormal enamel surfaces, hypocalcified dentin, and surfaces with fluorosis and carious lesions, and can bond to rough metallic surfaces and composite resin restorations without any need for the application of extra primers. With its application, contamination of enamel surfaces with saliva has no important role in decreasing the bond strength and it does not need photoactivation during the bonding procedure (except for dentin)[7]; however, the results of the present study did not show any increase in the bond strength under contamination with saliva.

Conversely, in a study by Rix et al,[9] no clinically significant differences were observed in the shear bond strength values of brackets bonded to enamel with the use of Assure adhesive resin under saliva-contamination conditions. In a study by Eslami et al,[18] application of Assure adhesive resin to bond stainless-steel brackets to enamel yielded adequate bond strength under dry conditions (mean = 14.18 MPa) and under contamination with saliva (mean = 13.32 MPa). Bond strength values of the brackets bonded to enamel with the use of Assure

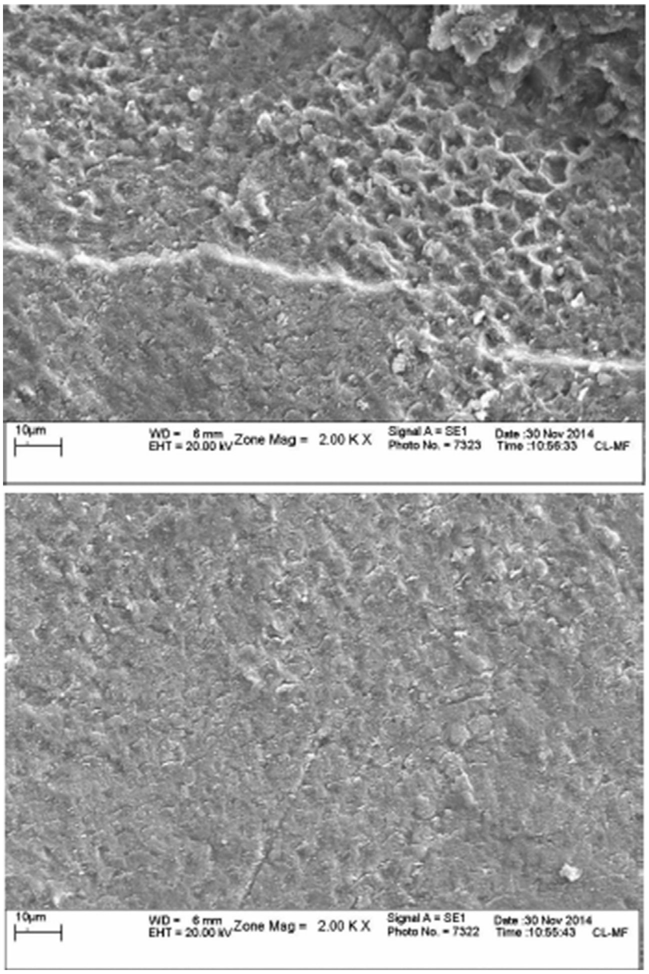

Fig. 3. SEM photomicrographs in the Assure group under dry conditions; penetration of resin tags into enamel porosities and their complete obturation. SEM = scanning electron microscopy.

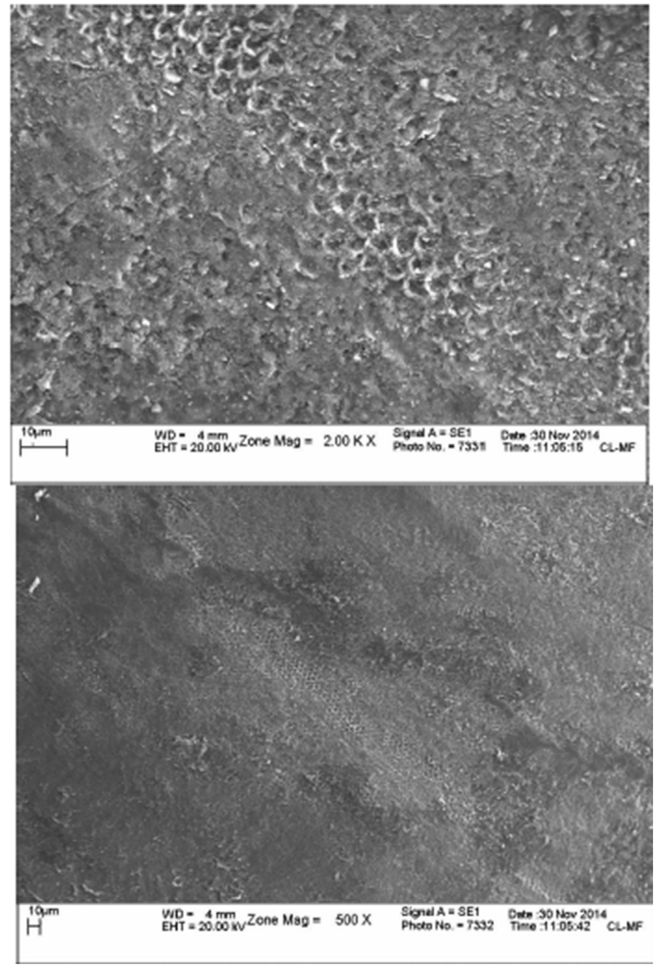

Fig. 4. SEM photomicrographs in the Assure group in the presence of saliva contamination; partial penetration of resin tags into enamel porosities in some areas and complete penetration in some other areas. SEM = scanning electron microscopy.

adhesive resin under dry conditions in the present study (21.25 MPa) were higher than those in Eslami et al's study, but lower with saliva contamination (9.29 MPa). In addition, in a study by Schaneveldt and Foley,[4] too, the mean shear bond strength values of Assure adhesive resin were not influenced by contamination with saliva; however, such an observation was not made in the present study.

Based on the results of some studies, the clinically acceptable range of shear bond strength for bonding of orthodontic brackets is 5.9–7.8 MPa.[19–21] Therefore, both Single Bond and Assure bonding agents yielded adequate strengths of bonding to tooth structures under dry and wet conditions.

In a study by Eslami et al,[18] application of Assure adhesive resin under dry and wet (contamination with saliva) conditions did not result in significant changes in the shear bond strength values of orthodontic brackets to enamel. However, in the present study, the shear bond strength of stainless-steel brackets decreased significantly with the application of Assure adhesive resin under saliva-contamination condition. However, the bond strength (9.29 MPa) was higher than the minimum bond strength necessary for bonding orthodontic brackets to enamel (5.9 MPa). Oztoprak et al[13] evaluated the effects of contamination with saliva on the bond strength of adhesive resins and reported that contamination with saliva resulted in a significant decrease in the bond strength of Assure adhesive resin, consistent with the results of the present study.

Bond strength values are under the influence of variables such as the tool used to measure bond strength, type of the force applied to debond the brackets, speed of the blade of the tool, type of the bracket, and variations in materials and methods.[22]

In the present study, the bond strength values of stainless-steel brackets with the use of Single Bond adhesive were at acceptable levels (14.9 MPa under dry conditions and 10.13 MPa with saliva contamination). SEM evaluations in the present study showed penetration of resin tags into the enamel porosities, and their complete obturation with the application of both Single Bond and Assure bonding agents under dry bonding conditions; however, with saliva contamination in the Assure group there was complete penetration in some areas and partial penetration in some other areas. In the Single Bond group, partial penetration of resin tags into enamel surface porosities was evident under saliva contamination.

Kanca[23] showed comparable bond strength with the application of a dentin-bonding agent on dry and wet enamel surfaces, with the bond strength for dry enamel being slightly higher than for wet enamel. Wakefield et al[24] showed that moisture on the enamel surface did not decrease the bond strength with the use of dentin-bonding agents. In a study by Woronko et al,[25] absence or presence of moisture did not increase or decrease the bond strength to enamel surfaces. Yasini and Malekan[26] did not report any significant differences in bond strength values with dry and wet enamel, which is not consistent with the results of the present study.

In routine orthodontic procedures, it is important to achieve adequate bond strength for safe debonding rather than achieving maximum bond strength.[27] ARI scores have been used in various studies in order to determine the bond failure location in enamel, adhesive, and bracket base by evaluating the amount of composite resin remaining on enamel surfaces. In the present study, no significant differences were observed in the frequencies of ARI scores between different study groups.

To prevent fractures or cracks on enamel surfaces, it is favorable that failures occur within the resin[28]; however, removal of the adhesive resin after debonding from tooth surfaces might be difficult and time consuming, resulting in defects on the enamel surface. The adhesive should provide adequate bond strength and withstand orthodontic and masticatory forces; however, at the end of treatment, it should be removed easily so that the enamel is not damaged. It appears that other factors, too, might have a significant role in the ARI scores, including the bracket retention mechanism.[29] Based on a report by O'Brien et al,[30] ARI scores depend on different factors, including the design of the bracket base and the type of the adhesive, and only the bond strength values do not affect ARI scores. By contrast, ARI scores are determined visually, which might influence the results of studies in association with differences in the conditions of bond strength tests.

Application of Single Bond and Assure bonding agents may provide adequate bond strength during bonding of brackets to enamel surfaces. Bond strength of Assure adhesive resin decreased significantly in the presence of saliva contamination compared with dry bonding conditions.

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