

Evaluation of shear bond strength of orthodontic molar tubes bonded using hydrophilic primers: An in vitro study

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Keywords

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Hydrophilic primers
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Summary

Objective > To evaluate and compare the shear bond strength (SBS) of orthodontic molar tubes bonded using two hydrophilic primers along with a moisture tolerant adhesive system to dry and saliva-contaminated enamel surfaces; and to assess the mode of their bond failure.

Materials and methods > A total of 60 extracted human mandibular molars were randomly divided into three major groups according to the primer used, each consisting of 20 molars: XT group acts as a control and bonded with the conventional hydrophobic Transbond XT primer, OS group bonded with the hydrophilic Ortho Solo primer, AP group bonded with the hydrophilic Assure Plus all surface bonding resin. Each major group was further divided into two subgroups, of 10 molars each, according to presence or absence of saliva. All the specimens were thermocycled 500 cycles between 5° and 55 °C. Shear forces were applied to the specimens with a universal testing machine at a crosshead speed of 1 mm/min and SBS was measured in megapascals (MPa). The mode of failure was determined using the adhesive remnant index (ARI). Data were analysed using two-way analysis of variance (ANOVA) followed by univariate analysis and Bonferroni post hoc tests.

Results > The three tested primers did not show a significant difference in the mean SBS in dry conditions ($P = 0.137$); the mean SBS of OS and AP primers were 15.60 ± 5.879 MPa and 12.51 ± 2.583 MPa respectively which were comparable to that of the hydrophobic XT primer (12.76 ± 2.952 MPa). In saliva-contaminated conditions, the mean SBS values were 10.41 ± 4.457 MPa and 9.22 ± 3.422 MPa for OS and AP primers respectively, which were significantly higher than that of XT primer (4.82 ± 2.050 MPa) ($P = 0.004$). When comparing the mean SBS for each group according to the bonding condition, it was significantly higher in dry bonding compared to saliva-contaminated bonding for the three primers; XT ($P < 0.001$), OS ($P = 0.003$) and AP ($P = 0.011$). In the dry field, most of the bond failures of the three primers were adhesive (score 3), whereas in the saliva-contaminated field, most of the failures were cohesive (score 1).

Mots clés

Résistance au cisaillement
Tubes molaires
Agents de couplage
hydrophiles
Contamination salivaire
Thermocyclage

Résumé

Évaluation de la résistance au cisaillement des tubes molaires orthodontiques collés à l'aide d'agents de couplage hydrophiles : étude in vitro

Objectif > Évaluer et comparer la résistance au cisaillement (SBS) des tubes molaires orthodontiques collés à l'aide de deux agents de couplage hydrophiles et d'un système adhésif tolérant à l'humidité sur des surfaces émaillées sèches et contaminées par la salive, et évaluer le mode de décollement.

Matériels et méthodes > Un total de 60 molaires mandibulaires humaines extraites ont été réparties au hasard en trois grands groupes selon l'agent de couplage utilisé, chaque groupe était composé de 20 molaires : le groupe XT a servi de témoin et a été collé avec l'agent de couplage hydrophobe conventionnel Transbond XT ; le groupe OS a été collé avec l'agent de couplage hydrophile Ortho Solo ; le groupe AP a été collé avec la résine hydrophile toutes surfaces Assure Plus. Chaque grand groupe a été divisé en deux sous-groupes, de 10 molaires chacun, selon la présence ou l'absence de salive. Tous les échantillons ont été thermocyclés 500 fois entre 5 et 55 °C. Les forces de cisaillement ont été appliquées aux échantillons à l'aide d'une machine d'essais universelle dont la tête se déplace à la vitesse de 1 mm/min et le SBS a été mesuré en mégapascals (MPa). Le mode de décollement a été déterminé à l'aide de l'index des résidus d'adhésifs (ARI). Les données ont été analysées à l'aide d'une analyse de variance bidirectionnelle (ANOVA) suivie d'une analyse univariée et du test post hoc de Bonferroni.

Résultats > Les trois agents de couplage testés n'ont pas montré de différence significative dans le SBS moyen dans des conditions sèches ($p = 0,137$) ; le SBS moyen des agents de couplage OS et AP était respectivement de $15,60 \pm 5,879$ MPa et $12,51 \pm 2,583$ MPa et était comparable à celui de l'agent de couplage hydrophobe XT ($12,76 \pm 2,952$ MPa). Dans des conditions de contamination salivaire, les valeurs SBS moyennes étaient de $10,41 \pm 4,457$ MPa et de $9,22 \pm 3,422$ MPa respectivement pour les agents de couplage OS et AP, elles étaient significativement supérieures à celles des agents de couplage XT ($4,82 \pm 2,050$ MPa) ($p = 0,004$). Lorsque l'on compare le SBS moyen pour chaque groupe selon les conditions de collage, il était significativement plus élevé pour le collage à sec que pour le collage contaminé par la salive pour les trois agents de couplage : XT ($p < 0,001$), OS ($p = 0,003$) et AP ($p = 0,011$). En milieu sec, la plupart des décollements des trois agents de couplage étaient adhésifs (score 3), tandis qu'en milieu contaminé par la salive, la plupart des décollements étaient cohésifs (score 1).

Conclusion > Le collage à sec a donné le SBS le plus élevé pour les trois agents de couplage. La contamination salivaire a réduit de façon significative l'adhérence des deux agents de couplage hydrophiles ; toutefois, les valeurs étaient supérieures à la limite acceptable sur le plan clinique. Les agents de couplage hydrophiles testés dans la présente étude peuvent être utilisés avec succès pour le collage des tubes molaires orthodontiques dans des conditions de surface d'émail sèche et contaminée par la salive.

Introduction

Orthodontic bonding is an essential step that greatly impacts the success of the treatment. The classical procedure for bonding orthodontic attachments to enamel surface requires the use of

three different agents: an enamel etchant, a primer solution, and an adhesive resin [1]. Because of their hydrophobic nature, these products require completely dry and isolated fields to achieve clinically acceptable bond strengths [2-5]. Moisture

contamination with water, saliva or gingival fluid has been reported as the most common cause for bond failure [6]. This happens particularly in hard-to-reach areas such as posterior teeth, partially erupted teeth, surgically exposed impacted teeth, lingual bonding of brackets or retainers. Bond failure during treatment is inconvenient and costly to both the orthodontist and the patient.

When etched enamel is contaminated by saliva, most of the porosities are occluded and resin penetration is impaired. As a result, resin tags of insufficient number and length are formed and bond strength is reduced [7-10].

To address this problem, manufacturers introduced hydrophilic bonding materials, suggesting the possibility of achieving successful and efficient orthodontic bonding to a moisture-contaminated enamel surface [11]. These novel bonding materials contain hydrophilic components such as Hydroxyethyl Methacrylate (HEMA), which act as a wetting agent, allowing a lower contact angle and rapid extension of the molecule which bonds easily to the resin composite. Also, they contain alcohol which acts as a drying agent that seeks out moisture, evaporates it from the bonding field, and brings the resin in; thus, ensuring an efficient bonding [3,11].

Several studies have tested the bond strength of hydrophilic primers; however, the results were contradictory. While some researchers have claimed acceptable performance for hydrophilic primers in a wet environment, others have suggested the opposite [4,12-15]. Besides, most in vitro studies on bond strength after saliva contamination did not use an artificial ageing procedure before testing, though thermocycling of the specimens has been recommended to consider the durability of the bond [16,17]. Up to our knowledge, there is no available evidence on bonding molar tubes using hydrophilic primers and moisture tolerant adhesive systems.

Accordingly, the purpose of this in vitro study is to evaluate and compare the SBS of orthodontic molar tubes bonded using two hydrophilic primers, along with a moisture tolerant adhesive system to dry and saliva-contaminated enamel following thermocycling.

The study, therefore, will address the following null hypothesis: there is no difference in the SBS of molar tubes bonded with hydrophilic primers under dry and saliva-contaminated conditions.

Materials and methods

Sample manipulation

A total of 60 extracted human mandibular molars with no caries, cracks, restored or damaged buccal surface or treated with chemical agents were collected and stored in a 0.1% thymol solution for 1 week before the experiment to inhibit bacterial growth. The mandibular molars were randomly divided into 3 major groups according to the primer used, each consisting of 20 molars. Each major group was further divided into two

subgroups, of 10 molars each, according to presence or absence of saliva.

Before bonding, the buccal surface of each molar was cleaned for 10 seconds with a mixture of water and non-fluoridated pumice in a rubber-polishing cup with a slow-speed hand piece and then thoroughly rinsed with water and an oil-free air stream.

Accent Mini 0.22" slot molar tubes from Ormco, Orange, California, USA, with a base surface area of 19.99 mm² were used [18]. All molar tubes were bonded by the same operator and placed in their ideal position at the center of the buccal surface of each molar. The adhesive used in the three groups was the moisture tolerant Transbond Plus Color Change Adhesive (TPCCA) from 3M Unitek, Monrovia, California, USA, and the artificial saliva was prepared according to Macknight-Hanes and Whitford formula [19].

Sample bonding procedure

Groups and subgroups

XT Group: Molar tubes bonded with Transbond XT primer used as a control

- XT/Dry Subgroup: The buccal surface of each molar was etched with 37% phosphoric acid gel from Reliance, Itasca, IL, USA, for 30 seconds and then rinsed with water spray for 20 seconds and gently dried with oil/moisture free compressed air until enamel becomes frosted in appearance. A uniform coat of XT primer was applied, gently air thinned and light cured for 10 seconds. TPCCA was applied to the tube base, the tube was positioned on the buccal surface of the molar and pressed firmly into its proper place to expel the excess adhesive which was then removed by a dental probe. After that, the adhesive was light cured for 20 seconds on the occlusal side of the tube and 20 seconds on the gingival side.
- XT/Saliva Subgroup: A thin coat of artificial saliva was then applied with a brush to the etched and dried enamel surface just before the application of the primer and left for 10 seconds to ensure full hydration of the surface. The excess was blotted out leaving the surface moist. XT primer and TPCCA were applied, and the tube was placed and bonded the same way as in XT/Dry subgroup.

OS Group: Molar tubes bonded with Ortho Solo primer

- OS/Dry Subgroup: After etching and drying, a thin coat of OS primer was applied, left undisturbed for 10 seconds, gently air dried and light cured for 10 seconds. Then TPCCA was applied to the tube base, and the tube was bonded according to the control protocol.
- OS/Saliva Subgroup: A thin coat of artificial saliva was applied to the etched and dried enamel surface, left for 10 seconds, the excess was blotted out and a moist enamel surface was maintained. Then, OS primer and TPCCA were applied and the tubes were bonded as in OS/Dry subgroup.

AP Group: Molar tubes bonded with Assure Plus primer

- AP/Dry Subgroup: After etching and drying, two coats of AP primer were applied, left undisturbed for 10 seconds, gently air dried and light cured for 10 seconds. TPCCA was applied and the tubes were bonded according to the control protocol.
- AP/Saliva Subgroup: A thin coat of saliva was applied after etching and drying the enamel surface and left for 10 seconds before blotting it out, leaving the enamel surface moist. Then, two coats of AP primer were applied, left undisturbed for 10 seconds, gently air dried and light cured for 10 seconds. TPCCA was applied and the tubes were bonded according to the control protocol.

The molars were mounted into acrylic cubes up to 1 mm apical to the cement-enamel junction. For standardization purposes and to ensure that all the molars were mounted in the same orientation relative to the acrylic cubes, a segment of .021 × .025" straight stainless steel rectangular wire from Ormco, Orange, California, USA, was inserted into the molar tubes slot. This provides a means for orientation and leveling of the buccal surface parallel to the direction of the applied force. After that, all the samples were stored in distilled water at room temperature for 24 hours.

To simulate temperature changes and the moisture in the oral environment, all samples were exposed to thermocycling 500 cycles between 5 and 55 °C with a dwell time of 30 seconds.

Each molar was oriented so that the buccal surface remains parallel to the applied de-bonding force by a Universal Testing Machine (YL-UTM YLE/Germany) connected to a computer. A knife-edge shearing rod at a crosshead speed of 1 mm/min was used to deliver the shear force at the molar tube base-enamel interface. The SBS was calculated in Megapascals (1 MPa = 1 N/mm²) by dividing the failure load in Newton (N) by the surface area of the molar tube base in mm².

After bond failure, the tube base and buccal surface of each molar were examined by the same operator at ×10 magnification under a dental operating microscope. The ARI was used to assess the amount of adhesive that remained on the enamel surface after de-bonding. This scale ranges from 0 to 3 [20]:

- 0 = no amount of adhesive material remained on the tooth;
- 1 = less than half of the adhesive material remained on the tooth;
- 2 = more than half of the adhesive material remained on the tooth and;
- 3 = all adhesive material remained on the tooth, with a distinct impression of the tube mesh.

Statistical analysis

Descriptive statistics of SBS (mean, standard deviation, minimum, maximum, and significance) were calculated for all groups (table I). Two-way analysis of variance (ANOVA) followed by univariate analysis and Bonferroni post hoc tests were used to compare the SBS among different groups and subgroups. Chi² and Fisher Exact tests were used to compare the ARI among different groups. The significance level was set at $P \leq 0.05$.

Results

In dry conditions, no statistically significant difference was found in the mean SBS among the primers XT, OS and AP ($P = 0.137$) (table II). In saliva-contaminated conditions, the mean SBS was significantly different among the groups where the $P = 0.004$ ($P < 0.05$); a significant difference was found between XT and OS ($P = 0.005$) and between XT and AP ($P = 0.036$). However, no significant difference was found between the mean SBS of OS and AP ($P = 1.000$) (table II). The mean SBS was significantly higher in dry conditions compared to saliva-contaminated conditions for XT ($P < 0.001$), OS ($P = 0.003$) and AP ($P = 0.011$) primers (figure 1, table III). The ARI was not significantly

TABLE I
Mean SBS of different study groups.

Groups	n	Mean SBS (MPa)	Standard deviation	95% confidence interval		Minimum	Maximum
				Lower	Upper		
XT/Dry	10	12.76	2.952	10.64	14.87	7.80	19.26
XT/Saliva	10	4.82	2.050	3.36	6.29	2.00	8.05
OS/Dry	10	15.60	5.879	11.39	19.80	7.30	23.26
OS/Saliva	10	10.41	4.457	7.22	13.59	2.05	15.06
AP/Dry	10	12.51	2.583	10.66	14.35	9.35	16.56
AP/Saliva	10	9.22	3.422	6.77	11.67	5.70	17.81

n: number of teeth in each subgroup.

Evaluation of shear bond strength of orthodontic molar tubes bonded using hydrophilic primers: An in vitro study

TABLE II
Bonferroni post hoc tests for comparison of SBS among primer groups.

Groups		Mean difference of SBS		P value	95% confidence interval of the difference	
					Lower	Upper
Dry <i>P</i> < 0.137	XT	OS	-2.841	0.296	-7.018	1.335
		AP	0.250	1.000	-3.927	4.427
	OS	XT	2.841	0.296	-1.335	7.018
		AP	3.092	0.219	-1.085	7.268
Saliva <i>P</i> < 0.004*	XT	OS	-5.583	0.005*	-9.760	-1.406
		AP	-4.397	0.036*	-8.574	-0.220
	OS	XT	5.583	0.005*	1.406	9.760
		AP	1.186	1.000	-2.991	5.362

**P* < 0.05 (significant difference).

different among XT, OS and AP primers bonded under dry (*P* = 0.879) and saliva-contaminated conditions (*P* = 0.185) (table IV).

Discussion

Many studies have investigated methods to enhance the bond strength of molar tubes to withstand the orthodontic and heavy masticatory forces in the posterior region [21]. Since molar teeth are at high risk of saliva contamination because of their limited accessibility, the purpose of this study was to evaluate the effect of the presence and absence of saliva on the SBS of orthodontic molar tubes bonded using two

hydrophilic primers in comparison with a conventional primer and to assess the mode of their bond failure.

Shear Bond Strength

From a statistical point of view, the null hypothesis of this study has been rejected. In the present investigation, each hydrophilic primer (OS and AP) revealed a significant drop in the SBS in saliva-contaminated conditions when compared to dry bonding conditions. Clinically, acceptable bond strengths have been reported by Reynolds to range from 6 to 8 MPa [2]. This bond strength range is considered favorable so that to be able to withstand masticatory and orthodontic forces from one side, and to allow easy removal of the orthodontic attachment at the conclusion of the

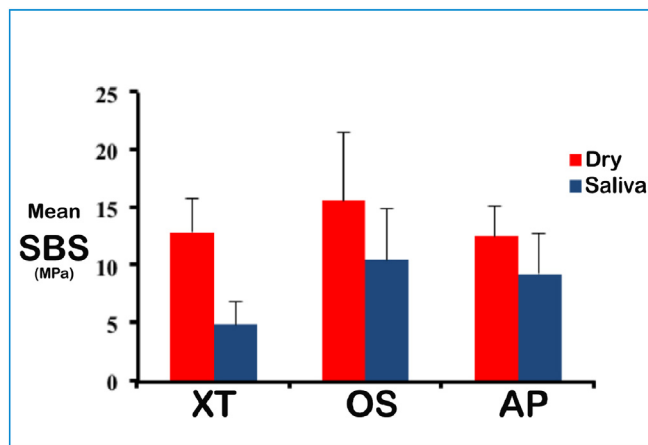


FIGURE 1
Column chart of the mean Shear Bond Strength (SBS) of primer groups and subgroups
XT: hydrophobic Transbond XT group; OS: hydrophilic Ortho Solo primer; AP: hydrophilic Assure Plus.

TABLE III
Univariate analysis for comparison of SBS between subgroups.

Groups			Mean difference of mean SBS	Significance	95% confidence interval of the difference	
					Lower	Upper
XT	Dry	Saliva	7.934	0.000*	4.545	11.323
OS	Dry	Saliva	5.193	0.003*	1.803	8.582
AP	Dry	Saliva	3.287	0.011*	0.053	6.576

* $P < 0.05$ (significant difference).

treatment without damaging the enamel surface from the other side. In the current study, the mean SBS of OS and AP primers bonded under dry conditions were 15.60 ± 5.879 MPa and 12.51 ± 2.583 MPa respectively; which were comparable to that of the hydrophobic XT primer (12.76 ± 2.952 MPa).

On the other hand, in saliva-contaminated bonding, the mean SBS values were 10.41 ± 4.457 MPa and 9.22 ± 3.422 MPa for OS and AP primers respectively; which were significantly higher than that of XT primer (4.82 ± 2.050 MPa).

Based on this, the two hydrophilic primers used in this study (OS and AP) yielded clinically acceptable bond strengths in both dry and saliva-contaminated conditions and this may be owing to the inherent hydrophilic properties of the primers mentioned before. However, the conventional primer XT revealed low bond strength values in saliva-contaminated bonding, which were below the ideal clinical bond strength. This finding is probably due to the fact that they contain Bis-GMA as a main constituent, which reveals a hydrophobic characteristic that makes them unable to penetrate the saliva on the etched enamel.

Ousehal et al. compared the efficacy of the hydrophilic primer Ortho Solo with a self-etching primer in terms of bond failure rate. It was noted that OS is as effective as the SEP with an added advantage of cost saving [22].

Previous studies on the effect of saliva contamination on the bond strength of orthodontic attachments have yielded different and in some cases contradictory results. Littlewood et al. stated that the bond strength achieved with the hydrophilic primer was significantly lower than that achieved with the conventional

primer in dry conditions and they did not recommend it for clinical use [12]. Whereas Webster et al. and Grandhi et al. reported no significant differences between the two primers in dry conditions, as was confirmed in the present study [3,8]. Kula et al., Cacciafesta et al., Zeppieri et al., and Rajagopal et al. found that wetting the enamel surface with saliva yielded a significant drop in the bond strength of moisture-insensitive primer [4,13,23,24]. However, they stated that wet conditions did not decrease the bond strength below the clinically acceptable level, which is also in agreement with our findings.

In a study by Schaneveldt and Foley, the mean SBS of the hydrophilic primers used (MIP and Assure bonding resin) were not influenced by contamination with saliva [17]; however, such observation was not obtained in the present study. This might be accredited to the use of natural saliva in their study while using artificial saliva in the present study.

Also, Oztoprak et al. evaluated the effect 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 the hydrophilic Assure adhesive resin [5]; consistent with the results of the present study.

Besides, Nirupama et al. assessed the SBS of hydrophilic bonding materials (Transbond MIP, Enhance LC, Prime and Bond NT) after contamination with artificial saliva in comparison with the conventional Transbond XT primer. It was stated that non-contaminated bonding yielded the highest bond strength for both hydrophilic and hydrophobic materials, which is in agreement with the findings of the current study [25].

TABLE IV
Statistical significance for ARI scores among primer groups and subgroups.

Groups				P-value
Dry	XT Dry	OS Dry	AP Dry	0.879
Saliva	XT Saliva	OS Saliva	AP Saliva	0.185
P-value	0.001*	0.300	0.001*	

* $P < 0.05$ (significant difference).

In a study by Eslami Amiabadi et al., the application of the hydrophilic primer Assure resin under dry and saliva contaminated conditions did not result in significant changes in the SBS values of orthodontic brackets to enamel (14.18 ± 4.78 MPa and 13.32 ± 4.74 MPa respectively) [14]. However, in the present study, the SBS of molar tubes decreased significantly with the application of hydrophilic primers under saliva-contaminated conditions (OS: 10.41 ± 4.457 MPa; AP: 9.22 ± 3.422 MPa). Bond strength values of the hydrophilic primers used under dry conditions in the present study were higher for OS (15.60 ± 5.879 MPa) and lower for AP (12.51 ± 2.583 MPa) than those in Eslami's study, and lower with saliva contamination for both primers. This might be justified by the different bonding agents used in both studies. Besides, Eslami's study used brackets instead of molar tubes, so the different attachment base designs may affect the SBS values.

Khanehmasjedi et al. compared the SBS of two hydrophilic primers (Single Bond and Assure primers) and suggested that both bonding agents gave adequate bond strength for bonding orthodontic brackets; however, their bond strength decreased significantly upon saliva contamination compared to dry conditions [15]. These findings are compatible with the results of this study. The results were justified by the scanning electron microscope assessment which revealed that contamination with saliva prevented complete penetration of resin tags into the enamel surface porosities resulting in a drop in bond strength when saliva contamination occurred.

Bond failure assessment

In orthodontic practice, the achievement of adequate bond strength for safe bonding is more favorable than obtaining the maximum possible bond strength [26]. Therefore, ARI scores

are used in different studies to determine the site of bond failure between the enamel, the adhesive, and the tube base by examining the amount of the remaining composite on the enamel surface.

Bond failures are commonly said to be either cohesive failures or adhesive failures. A cohesive failure is a failure in the bulk layer of the adhesive and is usually the desired mode of failure to prevent fractures or cracks on enamel surfaces; however, it increases the necessity for cleaning up the remaining adhesive [2]. On the other hand, an adhesive failure occurs at the interface between the adhesive and the substrate.

In the present study, the three primers used under dry conditions had a higher frequency of bond failures at the tube-adhesive interface, with all the adhesive remaining on the tooth surface (score 3) (table V). On the other hand, saliva contamination in all the primer groups resulted in higher frequency of bond failures occurring within the adhesive, with less than half of the adhesive left on the tooth (score 1) (table V). This offers the advantage that the risk of damage to the tooth surface decreases. However, the orthodontist will take more time to remove leftovers of the bonding material when more adhesive remains on the tooth. These ARI findings may be explained by the scanning electron microscope results in the studies of Eslami Amirabadi et al. and Khanehmasjedi et al., which suggested that saliva contamination prevented the complete penetration of resin tags into the porosities of the etched enamel surface [14,15]. One note to be added, that the ARI scores for all groups showed the absence of enamel fracture, which means that even the highest SBS values were not able to damage the enamel surface.

The present in vitro study provided promising clinical possibilities for the use of hydrophilic primers under dry and saliva-contaminated conditions. However, clinical conditions may

TABLE V
The frequencies of ARI scores in different study groups.

	ARI Score	Groups			Total
		XT	OS	AP	
Dry	0	0 (00.0%)	0 (00.0%)	0 (00.0%)	0 (00.0%)
	1	2 (20.0%)	4 (40.0%)	2 (20.0%)	8 (26.7%)
	2	3 (30.0%)	2 (20.0%)	3 (30.0%)	8 (26.7%)
	3	5 (50.0%)	4 (40.0%)	5 (50.0%)	14 (46.7%)
Saliva	0	4 (40.0%)	2 (20.0%)	1 (10.0%)	7 (23.3%)
	1	6 (60.0%)	5 (50.0%)	9 (90.0%)	20 (66.7%)
	2	0 (00.0%)	2 (20.0%)	0 (00.0%)	2 (6.7%)
	3	0 (00.0%)	1 (10.0%)	0 (00.0%)	1 (3.3%)

significantly differ from an in vitro setting. Hence, clinical trials of these novel materials are recommended to obtain more evident data about their clinical performance.

Conclusion

Within the limitations of this in vitro study, the following could be concluded:

- bonding orthodontic molar tubes under dry conditions yielded the highest SBS for the three primers tested;
- saliva contamination resulted in a significant reduction in SBS of all the primers, with OS primer being the least influenced and the conventional XT primer the most influenced;

- both hydrophilic primers, unlike the conventional one, yielded clinically acceptable bond strength values in both dry and saliva-contaminated conditions;
- in dry bonding, most of the bond failures of the three primers were adhesive, whereas in saliva-contaminated bonding, most of the failures were cohesive;
- accordingly, the hydrophilic primers tested in the present study can be successfully used for bonding orthodontic molar tubes under dry and saliva-contaminated enamel surface conditions.

Disclosure of interest: The authors declare that they have no competing interest.

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