

## Original Article

# Effects of Artificial Saliva Contamination on the Shear Bond Strength of Different Orthodontic Adhesive Systems

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**Abstract**

The purpose of this study was to compare the shear bond strength of different adhesive systems for bonding orthodontic brackets under non-contaminated and artificial saliva-contaminated conditions. One hundred and sixty maxillary first premolars were divided into eight groups of twenty. Stainless steel brackets were bonded using one of the following adhesive systems: conventional adhesive system (Transbond™ XT), or moisture-resistant adhesive systems (Transbond™ Plus Color Change, Beauty Ortho Bond® and Assure®) on both non-contaminated and artificial saliva-contaminated enamel. Shear bond strength was measured using an Instron® Universal Testing Machine. Analysis of variance and Tukey's test were used to compare the differences in the mean values. The results show that the mean shear bond strength of Transbond™ XT under non-contaminated conditions (11.70±3.14 MPa) was significantly superior to that of the other systems, both under non-contaminated and artificial saliva-contaminated conditions ( $p < .05$ ), whereas the mean shear bond strength of Transbond™ XT under artificial saliva-contaminated conditions (7.24±1.86 MPa), Transbond™ Plus Color Change under non-contaminated and artificial saliva-contaminated conditions (7.37±1.59 and 6.44±1.40 MPa, respectively), Beauty Ortho Bond® under non-contaminated and artificial saliva-contaminated conditions (6.28±2.05 and 6.66±2.01 MPa, respectively), and Assure® under non-contaminated and artificial saliva-contaminated conditions (6.74±1.61 and 7.28±1.06 MPa, respectively) were not significantly different. In conclusion, artificial saliva contamination significantly decreased the mean shear bond strength of Transbond™ XT, but did not affect the mean shear bond strength of the other systems. Nevertheless, the mean shear bond strength of all systems was greater than 6 MPa, which is clinically acceptable for bonding orthodontic brackets.

**Key words:** Artificial saliva contamination; Shear bond strength; Orthodontic adhesive system

**Introduction**

The most common cause of bond failure is contamination during the bonding process. Accordingly, the orthodontist must protect the enamel from contamination by dental wax, dust powder from gloves, remaining aluminum oxide after sand blasting, skin oil, moisture, saliva and blood. These unwanted substances are mostly under control;

however, it is very difficult to control moisture contamination in some areas, for example, partially erupted teeth, posterior teeth, especially second molars, and surgically exposed impacted teeth. It would be advantageous to reduce the chance of bond failure from moisture contamination. Consequently, the use of hydrophilic orthodontic bonding adhesives, which are capable of maintaining proper bond strength in moisture-contaminated conditions has been proposed as the method of choice for orthodontic bonding.

Typically, conventional orthodontic bonding adhesive contains resin monomers, which have hydrophobic properties and requires a completely dry field. Previous studies of a conventional adhesive system under saliva-contaminated conditions demonstrated significantly decreased shear bond strength values, which were not clinically acceptable.<sup>1-5</sup>

Recently, some manufacturers have developed moisture-resistant adhesives, which fulfill the orthodontist's demand by adding hydrophilic monomers, such as HEMA (2-hydroxyethyl methacrylate), PEGDMA (Polyethylene glycol dimethacrylate) or TEGDMA (Triethylene glycol dimethacrylate). HEMA contains two functional groups, which are hydrophobic and hydrophilic components.<sup>6</sup> PEGDMA is a cross-linked monomer and water-soluble enhancer of the adhesive.<sup>7</sup> TEGDMA has low molecular weight with relatively high hydrophilicity.<sup>8</sup> Moisture-resistant orthodontic adhesive, therefore, has become an alternative for bonding in both non-contaminated and moist conditions.

Previous studies,<sup>1-5,9,10</sup> evaluated various factors that might alter the effect of saliva contamination on shear bond strength, factors such as different tooth types, different sources of saliva, different duration of saliva contamination, different curing time or different artificial aging technique. Moreover, some studies<sup>1,10</sup> have compared adhesive systems from the same manufacturer. Therefore, the purposes of this study were to evaluate and compare the shear bond strength of a conventional light-cured orthodontic adhesive system (Transbond™ XT) and three commercial moisture-resistant light-cured orthodontic adhesive systems (Transbond™ Plus Color Change, Beauty Ortho Bond®, and Assure®) in bonding stainless steel brackets on enamel surfaces under either non-contaminated or artificial saliva-contaminated conditions.

The chemical composition of each adhesive, according to the manufacturers are shown in Table 1.

## Materials and methods

One hundred and sixty human maxillary first premolars extracted for orthodontic reasons were included in this study. The inclusion criteria were that the buccal surface of all teeth had sound enamel, with an absence of caries, restorations, fluorosis (Tooth Surface Index of Fluorosis/TSIF score of '0')<sup>11</sup> or other enamel defects. The extracted teeth were stored in 0.1% (weight/volume) thymol solution for one to six months prior to the bonding process. A random numbers table was used to randomly categorize all teeth into eight groups of 20 premolar teeth each. All teeth were sectioned using an IsoMet® 1000 sectioning saw (BUEHLER®, Lake Bluff, Illinois, USA) at 2 to 3 mm. below the cemento-enamel junction. All buccal tooth surfaces were gently polished with fluoride-free pumice and a rubber cup for 10 seconds, rinsed with water for 10 seconds, and dried with an oil-free air source. The specimens were then bonded with 0.022x0.028 inch-slot Mini Masters Series brackets (American Orthodontics, Sheboygan, Wisconsin, USA), whose bases had a total projected surface area of 8.82 square millimeters<sup>12</sup> according to one of the following eight protocols:

Group I: The enamel surfaces were treated with 35% phosphoric acid etching gel (Scotchbond™, 3M Unitek®, Monrovia, California, USA) for 15 seconds, washed for 20 seconds, and dried with an oil-free air stream. Transbond™ XT primer (3M Unitek®) was applied on the etched surfaces, and the brackets were bonded using Transbond™ XT Light Cure Adhesive (3M Unitek®).

Group II: The enamel surfaces were treated with 35% phosphoric acid etching gel for 15 seconds, washed for 20 seconds, and dried with an oil-free air stream. Transbond™ XT primer was applied on the etched surfaces. Artificial saliva (0.02 ml.) (Department of Pharmacy, Faculty of Medicine, Chiang Mai University, Thailand) was dropped onto the primed surface with a Proline® mechanical pipette (Biohit®, Helsinki, Finland), left for 10 seconds and then blown off with an air syringe for five seconds. The brackets were then immediately bonded using Transbond™ XT Light Cure Adhesive.

Group III: Transbond™ Plus Self Etching Primer (3M Unitek®) was applied and rubbed on the enamel surfaces for approximately three seconds. An air spray was gently applied to the enamel, and the brackets were bonded using Transbond™ PLUS Color Change Adhesive (3M Unitek®).

**Table 1** The chemical composition of each adhesive according to the manufacturers.

Material	Manufacturer	Component	Composition	%
Transbond™ XT	3M Unitek®	Etching gel:	35% Phosphoric acid	
		Primer:	Bisphenol A Diglycidyl Ether Dimethacrylate	45-55
			Triethylene Glycol Dimethacrylate	45-55
			4-(Dimethylamino)-Benzeneethanol	<0.5
			DL-Camphorquinone	<0.3
			Hydroquinone	<0.03
		Paste:	Silane-treated quartz	70-80
			Bisphenol A Diglycidyl Ether Dimethacrylate	10-20
			Bisphenol A Bis(2-Hydroxyethyl Ether) Dimethacrylate	5-10
			Silane-treated silica	<2
Diphenyliodonium Hexafluorophosphate	<0.2			
Transbond™ PLUS Color change	3M Unitek®	Self-etching primer:	Methacrylate Ester Derivative	75-85
		Water	15-25	
			DL-Camphorquinone	<1.0
			Dipotassium Hexafluorotitanate	<0.2
		Paste:	Silane-treated quartz	35-45
			Glass reacted with hydrolyzed silane	35-45
			Polyethylene glycol dimethacrylate	5-15
			Citric acid dimethacrylate oligomer	1-10
			Silane-treated silica	<2
		Bisphenol A diglycidyl ether dimethacrylate	<2	
2,6-Di-Tert-Butyl-P-Cresol	<0.5			
Beauty Ortho Bond®	Shofu®	Primer A:	Water, acetone, initiator	
		Primer B:	Ethanol, phosphoric acid monomer, dye	
		SALIVATECT	Bis-GMA ,TEGDMA, Glass powder, Silane coupling agent	N/A
		Paste:	Bis-GMA ,TEGDMA, Glass powder, Silane coupling agent	
Assure®	Reliance® Orthodontic Products, Inc.	Etching gel:	37% Phosphoric acid	
		Assure® Primer:	Acetone	>40
			Biphenyl Dimethacrylate	>10
		Paste:	Hydroxyethyl methacrylate	>10
			Hydroxyethyl methacrylate	8-30
			Glass Frit	60-99
Sodium Fluoride	1-5			

Group IV: Transbond™ Plus Self Etching Primer was applied and rubbed on the enamel surfaces for approximately three seconds. An air spray was gently applied to the enamel. Artificial saliva (0.02 ml) was dropped onto the primed surface with a micropipette left for 10 seconds and then blown off with an air syringe for five seconds and the brackets were immediately bonded using Transbond™ PLUS Color Change Adhesive.

Group V: Beauty Ortho Bond® (Shofu®, Kyoto, Japan) primers A and B (Self-etching primer) were mixed. The enamel surfaces were rubbed with the solution for approximately three seconds. An air spray was gently applied to the enamel, and the brackets were bonded using Beauty Ortho Bond® Paste.

Group VI: Beauty Ortho Bond® primers A and B were mixed. The enamel surfaces were then rubbed onto the enamel for approximately three seconds. An air spray was briefly applied to the enamel. Then SALIVATECT (Shofu®) was applied on the primed tooth surface according to the manufacturer's directions. Artificial saliva (0.02 ml) was dropped onto the primed surface with a micropipette left for 10 seconds and then blown off with an air syringe for five seconds. The brackets were immediately bonded using Beauty Ortho Bond® Paste.

Group VII: The enamel surfaces were treated with 37% phosphoric acid etching gel for 15 seconds, washed for 20 seconds, and dried with an oil-free air stream. Assure® primer (Reliance Orthodontic Products, Inc., Itasca, Illinois, USA) was applied on the etched surface, and the brackets were bonded using Assure® Adhesive (Reliance Orthodontic Products, Inc.).

Group VIII: The enamel surfaces were treated with 37% phosphoric acid etching gel for 15 seconds, washed for 20 seconds, and dried with an oil-free air stream. Assure® primer was applied on the etched surface. Artificial saliva (0.02 ml) was dropped onto the primed surface with a micropipette left for 10 seconds and then blown off with an air syringe for five seconds. The brackets were then immediately bonded using Assure® Adhesive.

The brackets were each firmly placed at the middle part of the buccal surface by one experienced operator using a Tension and Compression Gauge (DENTAURUM GmbH & Co. KG, Ispringen, Germany) with 300 g of force for three seconds in order to achieve a comparable resin layer thickness.<sup>10</sup> An orthodontic sickle was used to remove any excess adhesives.

All samples were then light cured with a Mini LED™ curing unit (Satelec®, Acteon, Merignac Cedex, France) at 1,250 mW/cm<sup>2</sup> for 10 seconds equally on both mesial and distal aspects of the teeth. The flow chart of the bracket bonding procedures according to the instructions is shown in Figure 1.

After the bonding procedure, each tooth was embedded in a cylindrical PVC ring block. Self-cured acrylic resin was used to fill the space in the PVC ring to allow exposure of only the surface of the tooth-bracket assembly surface.

All samples were incubated in distilled water at 37°C for 24 hours. Then a thermocycling procedure was performed, using a thermocycling machine (Medical and Environmental Equipment Research Laboratory, King Mongkut's Institute of Technology Ladkrabang, Bangkok, Thailand) in a cold-water bath at 5°C and a hot-water bath at 55°C for 30 seconds in each bath and with a transferring time of 10 seconds, for 2,000 cycles.

The brackets were then de-bonded with an Instron® universal testing machine (model number 5566, Instron Ltd., High Wycombe, England). The de-bonding plate was installed and fixed into the upper pneumatic grip, while the mounting jig was attached to the lower pneumatic grip. The PVC ring was attached to the mounting jig. The de-bonding plate was vertically adjusted and the force was applied to the ligature groove between bracket base and wings at the time of testing (Figure 2). Brackets were de-bonded from the tooth surfaces at a cross head speed of 0.5 mm per minute with a load cell of 500 Newtons. The occluso-gingival force was provided parallel to the buccal tooth surface until the bracket was dislodged from the tooth surface. The force values in Newtons were divided by the area of the bracket base, which is 8.82 square millimeters.<sup>12</sup> The bond strength for removing brackets was recorded in units of Megapascals (MPa).

The SPSS for Windows Release 17.0 program was used to calculate the following analysis:

1. The shear bond strength values in each sample group were described by means, standard deviations, and ranges.

2. Analysis of variance (ANOVA) was used to compare the means shear bond strength values among eight groups followed by multiple comparisons (Tukey's test).

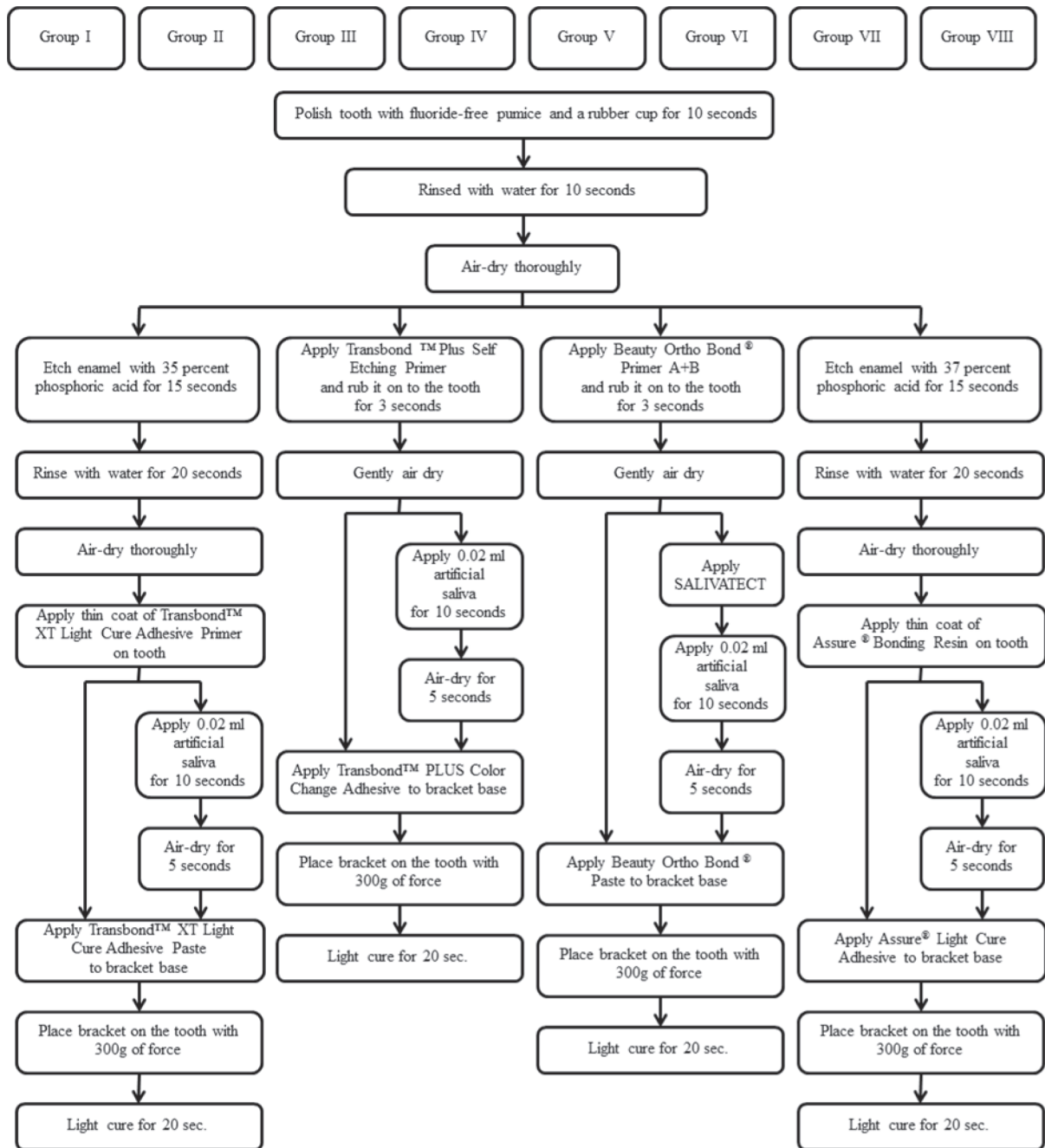


Fig. 1 Flow chart of bracket bonding procedures



**Fig. 2** The apparatus assembled for testing shear bond strength

## Results

The results of this study show that Transbond™ XT under non-contaminated conditions (Group I) provided the highest mean shear bond strength values of  $11.70 \pm 3.14$  MPa. The values, in descending order, were as follows: Groups III, VIII, II, VII, VI, IV

and V provided mean shear bond strength values of  $7.37 \pm 1.59$ ,  $7.28 \pm 1.06$ ,  $7.24 \pm 1.86$ ,  $6.74 \pm 1.61$ ,  $6.66 \pm 2.01$ ,  $6.44 \pm 1.40$  and  $6.28 \pm 2.05$  MPa, respectively. (Table 2)

The Shapiro-Wilk test showed normal distributions of the data within each group. Also, Levene's test showed the same variance data for each group. In order to compare the means of shear bond strength among the eight groups, one-way analysis of variance was applied. This test showed significant differences in the mean shear bond strength values among the eight groups at  $p < .05$ . Transbond™ XT under non-contaminated conditions showed the highest statistically significant mean shear bond strength value at  $p < .05$ . No significant difference was found between the shear bond strength values for adhesives bonded under artificial saliva-contaminated conditions. For each adhesive system, no significant difference in the shear bond strength values was detected between non-contaminated and artificial saliva-contaminated conditions, with the exception of Transbond™ XT, whose shear bond strength under non-contaminated conditions was significantly higher than that under artificial saliva-contaminated conditions ( $p < .05$ ).

**Table 2** Means ( $\bar{x}$ ), standard deviations (SD), and ranges (Min, Max) of shear bond strength in each adhesive system (by group) under non-contaminated and saliva-contaminated conditions.

Group	Adhesive system	Condition	Shear bond strength (MPa)			
			$\bar{x}$	SD	Min	Max
I	Transbond™ XT	Non-contaminated	11.70	3.14	6.35	18.10
II	Transbond™ XT	Saliva-contaminated	7.24*	1.86	4.24	10.58
III	Transbond™ Plus Color Change	Non-contaminated	7.37*	1.59	4.47	11.16
IV	Transbond™ Plus Color Change	Saliva-contaminated	6.44*	1.40	4.04	9.34
V	Beauty Ortho Bond®	Non-contaminated	6.28*	2.05	1.81	11.48
VI	Beauty Ortho Bond®	Saliva-contaminated	6.66*	2.01	4.10	11.46
VII	Assure®	Non-contaminated	6.74*	1.61	4.32	9.88
VIII	Assure®	Saliva-contaminated	7.28*	1.06	5.33	9.11

\*Values with the same superscript for each group are not significantly different ( $p > .05$ )

## Discussion

The results indicate that under artificial saliva-contaminated conditions, Transbond™ XT adhesive system showed significantly decreased mean shear bond strength. This finding may be due in part to the loss of mechanical retention when the etched surface was contaminated. Likewise, the previous studies demonstrated that human saliva causes plugging of a biofilm,<sup>13,14</sup> It can be postulated that a high degree of moisture retention from artificial saliva might decrease the penetration of adhesive resins into the enamel surface. This finding was consistent with those of other studies,<sup>1-5,9</sup> in which Transbond™ XT was bonded under saliva-contaminated conditions, resulting in reduction of bond strength with statistical significance.

In the present study, moisture-resistant adhesive systems did not show any significant alterations in the shear bond strength for bonding orthodontic brackets under artificial saliva-contaminated conditions. This is in agreement with previous studies.<sup>1,10</sup> An explanation for these findings might be the presence of PEGDMA in Transbond™ Plus Color Change, TEGDMA in Beauty Ortho Bond®, and HEMA in Assure®. Some studies<sup>4,5,9</sup> found significant difference between conventional and moisture-resistant adhesive systems under the same conditions. These differences might result from the different tooth types and durations of light-curing.<sup>4</sup>

Under non-contaminated conditions, Transbond™ XT showed a significantly greater mean shear bond strength than did the other adhesive systems. In agreement with previous studies,<sup>10,15</sup> the decreased mean shear bond strength values achieved under non-contaminated conditions were found for self-etching adhesive systems (Transbond™ Plus Color Change and Beauty Ortho Bond®) in comparison to conventional adhesive system. This might be explained by the fact that Transbond™ XT, which is a total-etching adhesive system, dissolves hydroxyapatite crystals, and enhances the superior penetration of resin into the etched enamel,<sup>16</sup> whereas, self-etching adhesive systems show more conservative etch patterns and have fewer adhesive penetrations, leading to lower bond strength.<sup>15</sup>

Even though both Assure® and Transbond™ XT are total-etching adhesive systems, Assure® provided significantly lower bond strength than did Transbond™ XT under non-contaminated conditions. The rationale for this lower bond strength might be that Assure® is a compomer, which contains conventional glass ionomer cement (GIC). The physical properties of GIC have an effect on the weaker and lower strength compared with resin adhesives.<sup>17</sup>

In this study, the mean shear bond strengths of all adhesive systems were greater than 6 MPa, a value suggested as adequate for most clinical orthodontic needs,<sup>18</sup> because it can withstand the forces of orthodontic mechanotherapy and of mastication.

## Conclusion

Artificial saliva contamination had the effect of a significant decrease in the mean shear bond strength values of a conventional adhesive system (Transbond™ XT), but did not affect the mean shear bond strength values of moisture-resistant adhesive systems (Transbond™ Plus Color Change, Beauty Ortho Bond®, and Assure®). Nevertheless, the mean shear bond strength values of all adhesive systems were greater than 6 MPa, which is clinically acceptable for bonding orthodontic brackets.

## Conflict of interest

The corresponding author states that there are no conflicts of interest.

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## ผลกระทบของการปนเปื้อนน้ำลายเทียมต่อกำลังยึดติดแบบเนียน ของระบบสารยึดติดทางทันตกรรมจัดฟันต่างชนิด

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### บทคัดย่อ

วัตถุประสงค์ของการศึกษาค้นคว้าครั้งนี้เพื่อเปรียบเทียบกำลังยึดติดแบบเนียนของระบบสารยึดติดต่างชนิดในการยึดติดแบร็กเกตจัดฟันภายใต้สภาวะปราศจากการปนเปื้อนและสภาวะที่ปนเปื้อนน้ำลายเทียม โดยใช้ฟันกรามน้อยบนซี่ที่หนึ่งจำนวน 160 ซี่ สุ่มเป็น 8 กลุ่ม กลุ่มละ 20 ซี่ นำมายึดติดกับแบร็กเกตจัดฟันเหล็กกล้าไร้สนิมด้วยระบบสารยึดติดดังต่อไปนี้ ระบบสารยึดติดธรรมดา (ทรานส์บอนด์เอ็กซ์ซีที) หรือระบบสารยึดติดที่ต้านทานความชื้น (ทรานส์บอนด์พลัสคัลเลอร์เซนจ์ บิวตี้โอโรบอนด์ และแอสซัวร์) ทั้งในสภาวะที่เคลือบฟันปราศจากการปนเปื้อนและที่ปนเปื้อนน้ำลายเทียม วัดค่าเฉลี่ยกำลังยึดติดแบบเนียนโดยใช้เครื่องทดสอบวัสดุเอนกประสงค์บริษัทอินสตรอนจากนั้นทำการเปรียบเทียบความแตกต่างของค่าเฉลี่ยโดยการวิเคราะห์ความแปรปรวนและการวิเคราะห์หัตถุ์กร ผลการศึกษาพบว่า ค่าเฉลี่ยกำลังยึดติดแบบเนียนของทรานส์บอนด์เอ็กซ์ซีทีภายใต้สภาวะปราศจากการปนเปื้อนมีค่าเท่ากับ  $11.70 \pm 3.14$  เมกะพาสคาล ซึ่งสูงกว่าระบบอื่นทั้งในสภาวะปราศจากการปนเปื้อนและสภาวะที่ปนเปื้อนน้ำลายเทียมอย่างมีนัยสำคัญทางสถิติ ( $p < .05$ ) ค่าเฉลี่ยกำลังยึดติดแบบเนียนของระบบอื่น ๆ ไม่มีความแตกต่างกันอย่างมีนัยสำคัญทางสถิติ ไม่ว่าจะเป็นทรานส์บอนด์เอ็กซ์ซีทีภายใต้สภาวะที่ปนเปื้อนน้ำลายเทียม ( $7.24 \pm 1.86$  เมกะพาสคาล) ทรานส์บอนด์พลัสคัลเลอร์เซนจ์ในสภาวะปราศจากการปนเปื้อนและสภาวะที่ปนเปื้อนน้ำลายเทียม ( $7.37 \pm 1.59$  และ  $6.44 \pm 1.40$  เมกะพาสคาล ตามลำดับ) บิวตี้โอโรบอนด์ในสภาวะปราศจากการปนเปื้อนและสภาวะที่ปนเปื้อนน้ำลายเทียม ( $6.28 \pm 2.05$  และ  $6.66 \pm 2.01$  เมกะพาสคาล ตามลำดับ) แอสซัวร์ในสภาวะปราศจากการปนเปื้อนและสภาวะที่ปนเปื้อนน้ำลายเทียม ( $6.74 \pm 1.61$  และ  $7.28 \pm 1.06$  เมกะพาสคาล ตามลำดับ) สรุปได้ว่าการปนเปื้อนน้ำลายเทียมทำให้กำลังยึดติดแบบเนียนของทรานส์บอนด์เอ็กซ์ซีทีลดลงอย่างมีนัยสำคัญ แต่ไม่ได้ส่งผลต่อกำลังยึดติดแบบเนียนของระบบสารยึดติดอื่น อย่างไรก็ตาม ค่าเฉลี่ยกำลังยึดติดแบบเนียนของสารยึดติดทุกระบบมีค่ามากกว่า 6 เมกะพาสคาล ซึ่งเป็นค่าที่ยอมรับได้ในทางคลินิกสำหรับการยึดติดแบร็กเกตจัดฟัน