

Effects of silane application on the shear bond strength of ceramic orthodontic brackets to enamel surface

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ABSTRACT

Background: Fixed orthodontic appliances with ceramic brackets are used frequently to fulfill the aesthetic demand of patient through orthodontic treatment. Ceramic brackets have some weaknesses such as bond strength and enamel surface damage. In high bond strength the risk of damage in enamel surfaces increases after debonding. **Purpose:** This study aimed to determine the effect of silane on base of bracket and adhesive to shear bond strength and enamel structure of ceramic bracket. **Method:** Sixteen extracted upper premolars were randomly divided into four groups based on silane or no silane on the bracket base and on the adhesive surface. Design of the base on ceramic bracket in this research was microcrystalline to manage the influence of mechanical interlocking. Samples were tested in shear mode on a universal testing machine after attachment. Following it, adhesive remnant index (ARI) scores were used to assess bond failure site. Statistical analysis was performed using a two-way Anova and the Mann-Whitney test. A scanning electron microscope (SEM) with a magnification of 2000x was used to observe enamel structure after debonding. **Result:** Shear bond strength was increased between group without silane and group with silane on the base of bracket ($p < 0,05$). There was no significance different between group without silane and group with silane on adhesive ($p < 0,05$). **Conclusion:** Application of silane on base of bracket increases shear bond strength, however, application of silane on adhesive site does not increase shear bond strength of ceramic bracket. Most bonding failure occurred at the enamel adhesive interface and damage occurred on enamel structure in group contains silane of ceramic bracket.

Keywords: silane; shear bond strength; ceramic bracket; enamel structure

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INTRODUCTION

Orthodontic treatment is a treatment that aims to improve the aesthetics and function of orofacial region. Tools used in the orthodontic treatment are divided into two, namely removable and fixed orthodontic appliances.¹ The fixed orthodontic appliance with ceramic brackets is widely used to meet the demands of patients related to aesthetic needs. Many researches have already been conducted on the clinical characteristics of the ceramic bracket materials.²

There are some kinds of brackets based on basic materials used, such as acrylic, polycarbonate, and ceramic bracket. The advantages of using ceramic bracket are high-strength material, resistance to change in shape, and good color stability. Meanwhile, the disadvantages of using

ceramic bracket are in terms of adhesion strength and to enamel surface damage. Ceramic bracket also cannot be chemically bound to the acrylic adhesive bonding material due to aluminum oxide contained. Silane is used to improve a chemical bond between the adhesive resin and the ceramic material resulting in a maximum strength.²

Silica element contained in ceramic will be bounded to acrylic derived from composite resin through silanization.³ This chemical element also generates a strong bond between the bracket and the adhesive resin that can trigger a tension on the bond between the enamel and the adhesive resin, leading to enamel surface damage.²

Self adhesive system is the seventh generation of adhesive material, categorized into the group of self etching.⁴ Self adhesive system is also considered as

an adhesive technology with preparations consisted of two pasta, namely basic pasta and catalyst pasta. Self adhesive system is composed of alkaline (basic) fillers, silanated fillers, phosphoric acid modified methacrylate monomers, methacrylate monomers, and initiators. Self adhesive system also contains acidic monomer generating demineralization and infiltration effects on the enamel surface to form a micromechanical retention and a chemical bond with the tooth enamel. Silanated fillers contained then will form a chemical bond between matrix and filler during polymerization.⁵ Therefore, the addition of filler particles in the matrix resin can improve physical and mechanical strength of the composite resin, such as barium glass, silica, apatite, and silane coupling agent.⁶

The minimum strength of the adhesion is 6 to 8 MPa quite capable of holding clinical orthodontic needs. The adhesion strength is considered to be able to tolerate to the mastication and orthodontic forces.¹⁰ Adhesive remnant index (ARI) is used to both assess the rest of the adhesive materials on the surface of the teeth after the release of the ceramic bracket bonding,¹¹ and determine the location of the failure of the bonding between the enamel and the adhesive and basic bracket materials.¹² Bracket can be detached from the tooth surface when receiving a force greater than the adhesive force. The detachment of the ceramic brackets mostly occurs on the bond between enamel and adhesive material. In contrast, the detachment of the metal brackets mostly occurs on the bond between the bracket and the adhesive materials resulting in damage enamel surface.¹¹ Scanning electron microscope (SEM) is a tool that can be used to observe the surface of enamel.¹¹ This study aimed to analyze effects of silanes contained in the bracket base and adhesive materials on the shear bond strength and the enamel surface scars caused by the attachment.

MATERIALS AND METHOD

This research was a laboratory experimental study. This research was conducted on 16 premolars randomly divided into four groups, namely group IA (bracket base and adhesive material), group IB (bracket base and adhesive material coated by silane agent), group IIA (bracket base coated silanes agent and adhesive materials), and group IIB (bracket base and adhesive materials coated by silane agents). The classification of A and B was based on the variable of adhesive materials. Group A was a group using only adhesive materials, while Group B was a group using adhesive materials coated by silane agent. On the other hand, the classification of I and II was based on the variable of bracket base. Group I was a group using bracket base without silane agent, while group II was a group using bracket base containing silanes.

The design of bracket base used in this research, moreover, was in the form of microcrystalline in order to control the effects of mechanical retention on the base of

the brackets. Adhesive materials containing no silanes used in this research, on the other hand, were Transbond plus and pasta Z250 composed of methacrylated phosphoric acid ester, Tri ethylene glycol dimethacrylate (UDMA), Bisphenol-polyethylene glycol dimethacrylate (Bis-EMA), and silica/ zirconia.^{13,14} Meanwhile, adhesive materials containing silanes used in this research was Relyx 200 consisted of two preparations, namely basic pasta and catalyst pasta, composed of methacrylated phosphoric acid esters, methacrylated monomer, silanated fillers, alkaline basic fillers, initiators, stabilizers, and staining substance.⁵ Shear bond strength of ceramic brackets in each group then was measured, and both the failure of the bonding as well as the surface of the tooth enamel after debonding were measured.

This research was conducted in several places. This research was performed at the Research Laboratory of the Faculty of Dentistry, Universitas Gadjah Mada from the first phase to soaking the teeth in saliva in an incubator. To measure the shear bond strength using a Universal Testing Machine (UTM), this research was carried out at the Laboratory of Materials of the Faculty of Mechanical Engineering, Universitas Gadjah Mada. After that, to observe ARI using a stereo-microscope with a magnification of 10x, this research was conducted at the Laboratory of Structural Animal Development of Faculty of Biology, Universitas Gadjah Mada. To observe the structure of enamel using a SEM with a magnification of 2,000x, this research was performed at LPPT Unit I, Universitas Gadjah Mada.

Anova analysis test was carried out to compare the shear bond strengths between the groups with a significance α level of 0.05. The failure of the bracket ceramic attachment based on ARI then was analyzed with nonparametric Mann-Whitney test. The structure of enamel was observed using a SEM presented descriptively.

RESULTS

In this research, the effects of silane agents on the shear bond strengths of the ceramic brackets were observed on 16 samples divided into four groups, namely group IA (bracket base and adhesive materials), group IB (bracket base containing no silanes, while adhesive materials containing silanes), group IIA (bracket base containing silanes, while adhesive materials containing no silanes), and group IIB (bracket base and adhesive materials containing silanes). The results then showed the following means and standard deviations of the shear bond strengths of the four groups as shown in Table 1. The mean values of the shear bond strength of the ceramic brackets were shown in Table 2.

Moreover, the means and standard deviations of the shear bond strengths of the ceramic brackets based on the variable of bracket base between the groups using the bracket base containing no silanes and the groups using adhesive material, either containing silanes or not (group

Table 1. Means and standard deviations of the shear bond strengths of the ceramic brackets on the groups IA, IB, IIA, and IIB

Group	Number of samples	Mean (MPa)	Standard Deviation
IA: Bracket base and adhesive materials containing no silanes	4	12.0175	1.44126
IB: Bracket base containing no silanes, while adhesive materials containing silanes	4	5.6775	.35762
IIA: Bracket base containing silanes, while adhesive materials containing no silanes	4	15.5275	.98290
IIB: Bracket base and adhesive materials containing silanes	4	7.7300	.76123

Table 2. Mean values of the shear bond strengths of the ceramic brackets in a unit of MPa

	Bracket base containing no silanes	Bracket base containing silanes	Mean
Adhesive materials containing no silanes	12.0175	15.5275	13.7725
Adhesive materials containing silanes	5.6775	7.7300	6.7038
Mean	8.8475	11.6287	

Table 3. Means and standard deviations of the shear bond strengths of the ceramic brackets based on the variable of bracket base (group I and group II)

Group	Bracket base	Mean (MPa)	Standard Deviation
I	Containing no silanes	8.8475	3.5625
II	Containing silanes	11.6287	4.2466

Table 4. Means and standard deviations of the shear bond strengths of the ceramic brackets based on the variable of adhesive materials (group A and group B)

Group	Adhesive materials	Mean (MPa)	Standard Deviation
A	Containing no silanes	13.7725	2.1694
B	Containing silanes	6.7038	1.2275

Table 5. Results of the two-way Anova test on the shear bond strengths of the ceramic brackets with the variables of bracket base and adhesive materials

Variables	Total of Multiplication Results	df	Multiplication Results of Mean	F	p
Bracket base	30.941	1	30.941	30.071	.000*
Adhesive materials	199.868	1	199.868	194.246	.000*

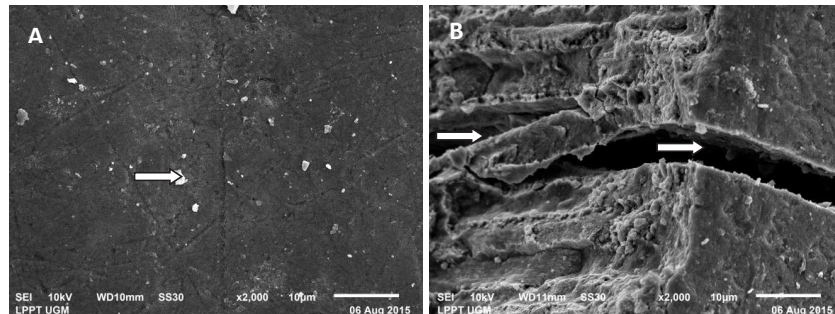
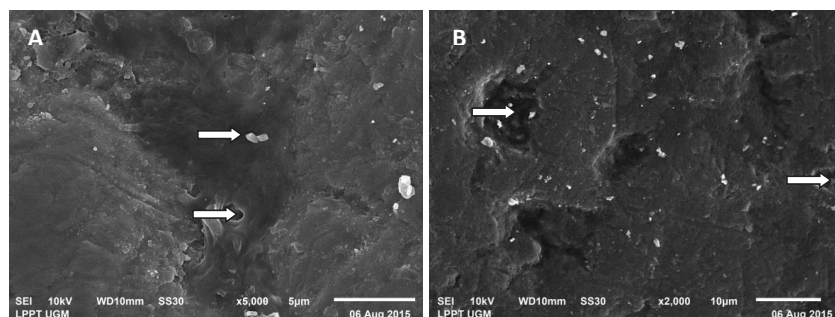
Note: *: Significantly different ($p < 0.05$)

Table 6. Distribution of data on the failure of the bracket ceramic attachments based on the variable of bracket base (group I and group II)

Group	ARI values							
	Value 0		Value 1		Value 2		Value 3	
	N	%	N	%	N	%	N	%
I	0	0	5	62.5%	2	25%	1	12.5
II	4	50%	4	50%	0	0	0	0

Table 7. Distribution of data on the failure of the bracket ceramic attachments based on the variable of adhesive materials (group A and group B)

Group	ARI Values							
	Value 0		Value 1		Value 2		Value 3	
	N	%	N	%	N	%	N	%
A	2	25%	3	37.5%	2	25%	1	12.5%
B	2	25%	6	75%	0	0	0	0

**Figure 5.** Photomicrographs of enamels in group IA and group IIA using SEM with a magnification of 2,000x. A) after debonding on group IA; B) after debonding on group IIA.**Figure 6.** Photomicrographs of enamels in group IB and group IIB using SEM with a magnification of 2,000x. A) after debonding on group IB; B) after debonding on group IIB.

I), as well as between the groups using the bracket base containing silanes and the groups using adhesive material, either containing silanes or not (group II) were shown in Table 3.

Furthermore, the means and standard deviations of the shear bond strengths of the ceramic brackets based on the variable of adhesive materials between the groups using the adhesive materials containing no silanes and the groups using bracket base, either containing silanes or not (group A), as well as between the groups using the adhesive materials containing silanes and the groups using bracket base, either containing silanes or not (group B) were shown in Table 4.

Normality and homogeneity tests were performed on the entire data of the shear bond strengths as seen in appendix 1 before a two-way Anova test was performed. Results of the normality test using the Shapiro-Wilk test showed that the data of all groups were normally distributed ($p > 0.05$). Meanwhile, results of the homogeneity test using the Levene test indicated that the data obtained were

homogeneous ($p > 0.05$) as shown in appendix 2. Therefore, the two-way Anova test then was performed.

The results of the two-way Anova test showed that there were significant differences in the shear bond strengths on the variables of bracket base and adhesive material ($p < 0.05$). The failure of the bracket ceramic attachment was examined based on the ARI using the stereo-microscope, 10x magnification. Distribution of the data based on the bracket base used, namely group I and group II was shown in Table 6, while the data distribution based on the adhesive materials used, namely group A and group B, was demonstrated in Table 7.

The nonparametric Mann-Whitney test was performed to compare between group I and group II based on the variable of bracket base and between group A and group B based on the variable of adhesive materials. The results showed that there was no significant difference between the groups ($p < 0.05$). Nevertheless, the results showed that there was a statistically significant difference in the values of ARI ($p = 0.01$) between group I and group II based on the

variable of bracket base. There was no significant difference in the value of ARI ($p=0.293$) between group A and group B based on the variable of adhesive material.

DISCUSSION

Ceramic bracket is mostly used for orthodontic treatment due to aesthetic demand of patients.¹⁵ Ceramic bracket, nevertheless, has some weaknesses. For instance, ceramic bracket cannot chemically bind to acrylic adhesive materials due to aluminum oxide contained. Therefore, silanes are added as a coupling agent to overcome this weakness.² Silanes are chemical elements that will make a bond between two materials, inorganic and organic ones.¹⁵ One part of silane molecules on the base of ceramic brackets will bind to an inorganic cluster, aluminum oxide, while the other part of the molecules will bind to an organic group, namely acrylic resin contained in the adhesive materials.

The results of this research showed that the mean value of the shear bond strengths of the ceramic brackets in group II (the bracket base containing silanes) increased significantly compared to group I (the bracket base containing no silanes) as shown in Table 1 and Table 3 ($p<0.05$). Therefore, it can be said that silane contained in the bracket base could increase the shear bond strengths of the ceramic brackets.

Ceramic brackets actually have some weaknesses in terms of adhesion strength and damage to enamel surface.² The optimal shear bond strength required in clinical orthodontic treatment is from 6 MPa to 10 MPa.²⁰ Meanwhile, the mean shear bond strength of the ceramic brackets containing silanes on the brackets base (group II) was 11.6287 MPa. On the other hand, the mean shear bond strength of the ceramic brackets containing no silanes on the bracket bases (group I) was 8.8475 MPa. Therefore, it can be said that the shear bond strengths in group I and group II were in line with the shear bond strength required. The shear bond strength in group I containing no silanes on the bracket base even still was in line with the shear bond strength recommended. This indicates that the mechanical retention of microcrystalline without chemical retention is still able to produce good shear bond strength.

Group B, moreover, was a group using the adhesive materials containing silanes. Silanes contained in the adhesive materials were in the form of silanated fillers. Silanated fillers are derived from the addition of silanes as coupling agents in inorganic fillers of the adhesive materials that binds chemically to monomer as organic group, and then forms a matrix when polymerized.⁵ Adhesive materials containing no silanes used in this research were Transbond plus and Z250 paste composed of methacrylated phosphoric acid ester, Tri ethylene glycol dimethacrylate (UDMA), Bisphenol-polyethylene glycol dimethacrylate (Bis-EMA), and silica/zirconia.^{13,14} Meanwhile, adhesive material containing silanes used in this research was Relyx 200 consisted of two preparations, namely basic pasta and

catalyst paste, composed of methacrylated phosphoric acid esters, methacrylated monomer, silanated fillers, alkaline basic fillers, initiators, stabilizers, and staining substance.⁵ The mean shear bond strength of the ceramic brackets in group B (using adhesive materials containing silanes) significantly decreased compared to group A (using adhesive materials containing no silanes) (Table 1 and Table 3).

Therefore, the hypothesis stating that the adhesive materials containing silanes will increase the shear bond strength of the bracket ceramics was rejected. De Munck *et al.*¹⁶ suggested that in an experimental study, a decrease in the shear bond strength of Relyx Unicem as an adhesive material is due to high viscosity factor and short penetration time, resulting in reducing of the adaptation ability of the materials on the surface after applied. The morphological examination, furthermore, will show the porosity of enamel surface and dentin after the use of Relyx Unicem, illustrating superficial interactions.¹⁶ The lowest shear bond strength then may occur at the thinnest area of the adhesive materials due to the loss of homogeneity of the material and the increased pressure on the thinnest area.¹⁷

The failure of the ceramic bracket attachment, can be examined using the ARI of the residual adhesive materials on the surface of the enamel using the 4-point scale.¹¹ The location of the failure the ceramic bracket attachment actually then can provide important information about the shear bond.¹⁸ Table 6 illustrates that the value of ARI was 0. It means that there were no residual adhesive materials on the surface of the enamels in group II compared to group I. There was even a significant difference (Table 8). This finding indicates that the location of the failure of the ceramic bracket attachment using the bracket base applied silanes occurred between the enamel surface and the adhesive materials.

In addition, results of the observation on the values of the ARI in group A and group B are shown in Table 7. The results demonstrate that there was no significant difference in the location of the failure of the ceramic bracket attachment between the groups based on the variable of the adhesive materials (Table 8). This is likely due to air ingestion in group B that affects bonding in the adhesive materials. This finding is in line with a research conducted by Setiarini²¹ showing that air trap formed will lead to the formation of the thinnest area of the bond between the teeth, the adhesive materials, and the bracket. Consequently, the location of the failure of the bracket attachment cannot be predicted, depending on the location of the air trap. Ideally, the strong shear bond strength will have a better failure of the attachment if located between the enamel surface and the adhesive materials since it will simplify the polishing process of the surface of the teeth after debonding.¹⁸

The shear bond strength above 13 MPa actually can cause damage in the form of tear out.²⁰ The morphology of the enamels in group IA and group IIA then was observed using a SEM (Figure 5). Figure 5-A illustrates the morphology of enamels in group IA with the mean shear

bond strength of 12.0175 MPa after debonding, indicating the adhesive materials remained on the surface of the enamels. Figure 5-B, on the other hand, demonstrates the morphology of the enamels in group IIA with the mean shear bond strength of 15.5275 MPa, showing damages in the enamel surfaces, such as cracks and open dentinal tubules.

Enamels on preparations in group IB and group IIB were observed using SEM as shown in Figure 6. Figure 6-A illustrates enamel morphology in group IB (the mean shear bond strength of 5.6775 MPa) after debonding. This picture indicates porosity in the enamel surface not filled by the adhesive materials. Figure 6-B, on the other hand, demonstrates enamel morphology in group IIB (the mean shear bond strength of 7.7300 MPa) after debonding. This picture also shows porosity in the enamel surfaces not filled by the adhesive materials. The ceramic bracket containing silanes on the base and the adhesive materials, therefore, is hypothesized to damage the surface of the enamel structure on which the bracket attached.

Finally, it can be concluded that there are some effects of silanes contained in ceramic bracket on shear bond strength and enamel structure. For instance, silane applied on the base of a ceramic bracket can increase the shear bond strength of the bracket. Silane applied on the ceramic bracket, nevertheless, can also make a failure of the bonding between enamel surface and adhesive material. Silane application can influence the structure of enamel surface on which the ceramic bracket is attached.

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