International Journal of Adhesion & Adhesives 62 (2015) 101–106

Contents lists available at ScienceDirect

International Journal of Adhesion & Adhesives

journal homepage: www.elsevier.com/locate/ijadhadh

A novel silane system as a primer for orthodontic bonding—A pilot study

Bangalore H. Durgesh [a,*], Saleh Al Hijji [a], Abdulaziz A Al Kheraif [a], Ravikumar Ramakrishnaiah [a], Santhosh Basavarajappa [a], Mohammed Al Sharawy [b], Jukka Pekka Matinlinna [c]

[a] *Dental Health Department, Dental Biomaterials Research Chair, College of Applied Medical Sciences, King Saud University, P.O Box 10219, Riyadh 11433, Saudi Arabia*

[b] *Dental Biomaterials Research Chair, College of Applied Medical Sciences, King Saud University, P.O Box 10219, Riyadh 11433, Saudi Arabia*

[c] *Dental Materials Science, Faculty of Dentistry, The University of Hong Kong, Hong Kong SAR, China and Visiting Professor Program, King Saud University, Riyadh, Saudi Arabia*

ARTICLE INFO

Article history:
Accepted 21 July 2015
Available online 29 July 2015

Keywords:
Adhesion
3-acryloxypropyltrimethoxysilane
bis-1,2-(triethoxysilyl) ethane
Orthodontic brackets

ABSTRACT

Aim: To determine the adhesion strength (measured as shear bond strength, SBS) of orthodontic brackets using two experimental silane-based primer systems and compare and contrast their effect.

Materials and methods: Sixty acid-etched premolars were randomly assigned into 4 study groups ($n=15$). In group 1, brackets were bonded without primer but using Transbond XT adhesive; in group 2, Transbond XT primer was applied and bracket bonded; in group 3, an experimental silane-based primer of 1.0 vol% of 3-acryloxypropyltrimethoxysilane (ACPS) was applied before bonding; in group 4, an experimental silane-based primer of 1.0 vol% of ACPS+0.5% *bis*-1, 2-(triethoxysilyl) ethane (BTSE) was used. The adhesion strength (measured as shear bond strength) was recorded using a universal testing machine. Failure types were classified according to the Adhesive Remnant Index (ARI). Contact angles of the primers were measured on an enamel slab.

Data was analyzed by ANOVA and Tukey's multiple comparison *post hoc* analysis.

Results: The mean adhesion strength results were high in group 4: 15.8 ± 1.6 MPa followed by group 3: 12.5 ± 1.5 MPa, group 2: 11.9 ± 1.1 MPa and the lowest in group 1: 08.1 ± 0.7 MPa. A significant difference in adhesion strength was observed between all the groups except for group 3 and group 4 ($p < 0.01$).

The ARI score was distributed largely to 0 and 1. One instance of enamel fractures was recorded in group 1. The contact angle measurements suggested that the lowest value with the experimental primer containing (ACPS+BTSE) was, $<5°$ followed by Transbond XT, 41.86 ± 4.56°.

Conclusion: The experimental silane primer systems were hydrophilic in nature and demonstrated higher adhesion strength compared to traditional orthodontic primers.

© 2015 Elsevier Ltd. All rights reserved.

1. Introduction

There is a constant quest in orthodontic profession to improve the techniques of bonding bracket to enamel durably enough [1]. Bond failures of brackets remain as a challenge in clinical practice in spite of the advancement in materials for direct bonding and increase in efficiency of treatment [2]. Bonding brackets using a conventional adhesive requires a completely dry operative field. However, in many instances, a wet environment is unavoidable [3,4]. The retention of the bracket to enamel is influenced by several factors, such as cleaning and conditioning procedures of enamel, adhesive systems used, polymerization chemistry, moisture contamination, occlusal forces and degradation of the adhesive when exposed to saliva [5–7].

Bonding of resin based luting materials to enamel is preferred because of the hydrophobic nature and the higher inorganic mineral content of enamel [8,9]. Acid etched enamel creates a high free energy surface that allows hydrophobic resin adhesives to wet the surface, penetrate and form a strong and durable bond [4]. The acid etch technique facilitates the penetration of the resin into

* Corresponding author. Tel.: +966 537135611.
E-mail addresses: drdurgesh19@gmail.com (B.H. Durgesh), smalhijji@ksu.edu.sa (S.A. Hijji), aalkhuraif@ksu.edu.sa (A.AA. Kheraif), drraviprostho@rediffmail.com (R. Ramakrishnaiah), sbasavarajjappa@ksu.edu.sa (S. Basavarajappa), malsharawy@ksu.edu.sa (M. Al Sharawy).

http://dx.doi.org/10.1016/j.ijadhadh.2015.07.006
0143-7496/© 2015 Elsevier Ltd. All rights reserved.

enamel micropores, providing the mechanism of micromechanical attachment (retention) between the resin and the tooth tissues [10–12]. It is also widely understood that the adhesion of metal brackets to enamel is by mechanical interlock between bracket–resin–enamel [13,14].

This explains that the use of some experimental reactive monomers in experimental primer solutions have significantly proven to increase the bond strength of adhesives to both metal and tooth substrates [4]. Silanes bond dissimilar materials. Such a primer is usually an unfilled bonding agent used to improve the effectiveness of the final bond. In orthodontic bonding system it could be used to protect the enamel from consequent demineralization by the acid-etch procedures, enhancing bond strength, increasing the retention of etched enamel and to reduce marginal leakage [15–18].

In general, numerous reactive silane monomers are used as coatings, surface modifiers and adhesion promoters and they are considered harmless and interestingly, possess no intrinsic toxicity [19]. For dental use in prosthodontics and in dental laboratories, silane coupling agents (silanes) are usually supplied as 1–2 vol% in a activated (pre-hydrolyzed) form diluted in 90–95% ethanol, and, they may have varying functional groups and formulations [20,21]. In general, prior to the use of reactive silanes, they need to be activated by hydrolysis before they can react with the substrate, through the formed Si–OH groups with the –OH groups on the substrate surface, to form durable chemical bonds [19]. In dentistry, functional silane coupling agents, have found many applications in conjunction with luting cements, resin composite restorative filling materials, and as pretreatment to silica-coated substrates before cementation or veneering [22–24].

Some *in vitro* studies have shown that 3-acryloxypropyltrimethoxysilane (ACPS) which has an acrylate group as the functional entity can provide significantly enhanced adhesion onto silica-coated titanium and zirconia surfaces when compared with methacrylate silane [25–27]. ACPS is a highly reactive silane monomer due to the higher reactivity of the unprotected acrylate group in it compared to methacrylate group as in the case of ACPS the acrylate group doesn't have the protecting methyl group in the vicinity of the reactive C=C, thus it is understood that it would be more prone to hydrolytic effects [26]. Adding and using a cross-linker silane adds drastically the hydrolytic stability of the bonded systems. This concept is called a novel silane system. Silanes bond to Ca-minerals [25,26].

Such experimental silane-based primers have not been evaluated in orthodontic use and they might incorporate enhanced wetting and durable bonding. The aim of the study was to:

a. Evaluate the effect of two experimental silane based primers on the adhesion strength of orthodontic brackets.
b. Measure the contact angle of the experimental silane primers.
c. Assess the failure mode at the site of debonding.

In this pilot study we aimed at testing the immediate adhesion strength with our silane-based primers. The hypothesis was that silane based primers may increase the adhesion strength of orthodontic brackets to enamel. This hypothesis could aim to produce high adhesion (shear bond) strength values thereby improving the stability of the brackets in clinical use.

2. Materials and methods

2.1. Specimen preparation

A total of 60 human premolars were extracted recently for orthodontic purposes from young subjects and used in this study. Teeth with any enamel defect were excluded. Teeth were stored in thymol solution to prevent bacterial growth till the start of the study. Each tooth was mounted vertically in a self-curing acrylic resin to have the buccal bond surface parallel to the force applied. The teeth were cleaned and polished for 10 s using slurry of pumice and a rubber prophylactic cup and then rinsed with stream of water for 10 s and dried. The dried enamel surfaces were etched with 37% orthophosphoric acid for 15 s, rinsed and gently air-dried for the same length of time. The acid-etched teeth were randomly assigned to 4 study groups ($n=15$). The test procedure was in accordance with the DIN 13990-2 standard governing test methods for the entire attachment–adhesive–enamel system [28]. The project has an ethical approval by the Bio-ethical committee of the College of Applied Medical Sciences Research Center, King Saud University (in 2015).

The materials used in the study are summarized in Table 1.

2.2. A novel silane primer system

Two experimental silane primers consisting of 1.0 vol% of 3-acryloxypropyltrimethoxysilane (ACPS) in 95.0%/5.0% ethanol/water, with a pH of 4.5 (adjusted with 1 M acetic acid), were prepared according to literature [23] (Table 1). Next, a cross-linking silane monomer *bis*-1,2-(triethoxysilyl)ethane (BTSE) was added to one of the experimental silane blends, corresponding to a final 1.5 vol% of silane. A conventional commercially available orthodontic primer (Transbond XT) was used as the other study group.

Chemical formulas of the constituents of the primer solutions are given in Fig. 1.

2.3. Bracket bonding

a. Group 1 (the negative control): standard premolar metal bracket (Lancer Orthodontics, Milano, Italy) with a surface mesh area of 11.86 mm^2 were used. A thin layer of adhesive (Transbond XT, 3M Unitek, Monrovia, CA, USA) was applied on the bracket surface and pressed firmly on the labial surfaces of the tooth. The excess resin was removed using a scaler. The medial and distal surfaces of the bracket edges were irradiated using a hand held light curing unit (Elipar Free Light 2, 3M ESPE, Seefeld, Germany) with a light radiance power of 1505 mW/cm^2 and a wavelength maximum between 420-540 nm (Managing Accurate Resin Curing [MARC] System;

Table 1
Materials used in the study.

Name	Composition	Batch No.	Manufacturer
Transbond XT	Primer: *bis*-GMA,TEGDMA Adhesive paste: silane treated quartz, silane, *bis*-GMA	N575782	3M Unitek, Monrovia, USA
ACPS	3-acryloxypropyltrimethoxysilane (95%)	9E-14566	Gelest, Morrisville, PA, USA
BTSE	*bis*-1,2- (triethoxysilyl) ethane	4R-4325	Gelest, Morrisville, PA, USA

Key: *bis*-GMA=*bis*-phenol A-glycidyl dimethacrylate; TEGDMA=triethylene glycol dimethacrylate.

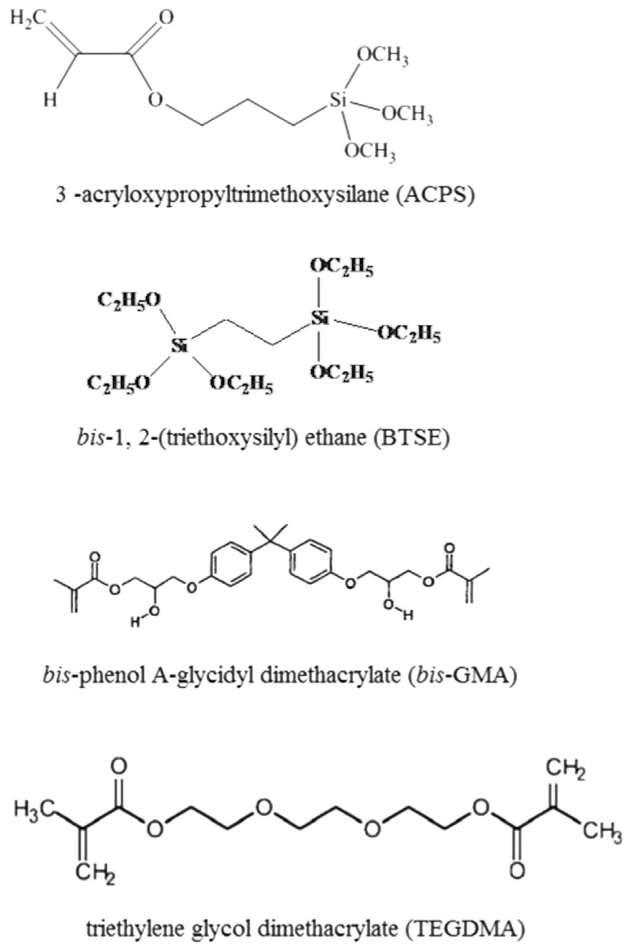

Fig. 1. The molecular structures of the primers used in the current study.

Table 2
Mean and standard deviation of the adhesion strength (MPa) values of tested samples.

Groups ($n=15$)	Adhesion strength (MPa) Mean $\pm$SD	[a]*Post hoc* analysis
Group 1	08.1 $\pm$ 0.7	A
Group 2	11.9 $\pm$ 1.1	B
Group 3	12.5 $\pm$ 1.5	C
Group 4	15.8 $\pm$ 1.6	D

[a] Same letters in a column indicates no significant differences between the groups ($p<0.01$).

Blue Light Analytics, Halifax, Canada). Light-curing was carried out for 20 s on each side of the bracket.

b. Group 2 (the positive control): in this group a thin layer of primer (Transbond XT Primer, 3M Unitek, Monrovia, CA, USA) was applied on the enamel surface with a fine disposable brush and sprayed with oil-free air to promote the complete penetration of the resin. After photo-polymerization for 10 s, brackets with a thin layer of adhesive (Transbond XT) were placed and pressed firmly on the labial surfaces of the teeth. The excess resin was removed using a scaler. The medial and distal surface of the bracket edges was irradiated for 20 s on each side.
c. Group 3: the procedure of bonding for this group was similar to group 2 except for the primer application. An experimental silane-based primer 1.0 vol% 3-acryloxypropyltrimethoxysilane (ACPS) primer was used. A single thin layer of the primer was applied with a fine disposable brush onto the enamel surface and the bracket was bonded using Transbond XT adhesive.
d. Group 4: in this group 1.0 vol% of 3-acryloxypropyltrimethoxysilane (ACPS)+0.5% *bis*-1,2-(triethoxysilyl)ethane (BTSE) primer was used. A single thin layer of the primer was applied with a fine disposable brush onto the enamel surface and bracket was bonded using Transbond XT adhesive.

Table 3
Adhesive remnant index (ARI) scores of the tested groups, for scoring see text.

Groups ($n=15$)	ARI Score				
	0	1	2	3	Enamel fracture
Group 1	7	4	3	1	1
Group 2	9	2	2	1	0
Group 3	12	3	0	0	0
Group 4	14	1	0	0	0

After bonding, the specimens were stored in distilled water at 37 °C for 24 h to assess the hydrothermal stability and resistance. The samples were then dried at ambient air for 30 min and mounted in a jig in a universal testing machine (Instron Corporation, Canton, MA, USA) with the enamel surface parallel to the blunt shear force blade for adhesion strength measurements. The specimens were subjected at stress at the bracket–tooth interface in an occlusogingival direction at a crosshead speed of 1 mm/min. The results of each test were obtained in Newtons (N) and calculated then and presented in megapascals (MPa).

2.4. Analysis of bond failure

After debonding the bracket bases (adhesive surfaces) and the tested enamel surfaces were inspected and assessed to determine the predominant bond failure site using a light stereomicroscope (Nikon SM2-10, Tokyo, Japan) at x20 magnification to establish the character of the debonded surface. The failure sites were classified according to the Adhesive Remnant Index (ARI), 0-3, as follows [29]:

0: no adhesive remaining.
1: less than 50% of the adhesive remaining.
2: more than 50% of the adhesive remaining.
3: all adhesive remaining on the enamel with a distinct impression of the bracket mesh.

Representative failure types after debonding were further examined using scanning electron microscopy (SEM) (Jeol JSM-5900 LV SEM, Tokyo, Japan) operated at 10 kV, in vacuum and with x100 magnification.

2.5. Contact angle measurement

Contact angle measurements of the experimental primer were performed using a camera based optical tensiometer (Theta Lite, Dyne Technology, Staffordshire, UK). A sessile drop of the test primers was applied on the enamel slab prepared using extracted molars. In Transbond XT specimen, a layer of Transbond XT primer was applied on the enamel surface and light cured for 5 s; for the experimental primer layers, a thin layer of the respective primer was applied on the enamel surface and dried for 3 min before contact angle measurements. Next, 1.0 μl drop of distilled water was applied toward the enamel specimen located on a movable table by using a syringe tip until the droplet made contact with the specimen surface. The contact angle was measured after 30 s, when the droplet was stabilized. The illuminated drop was cap-

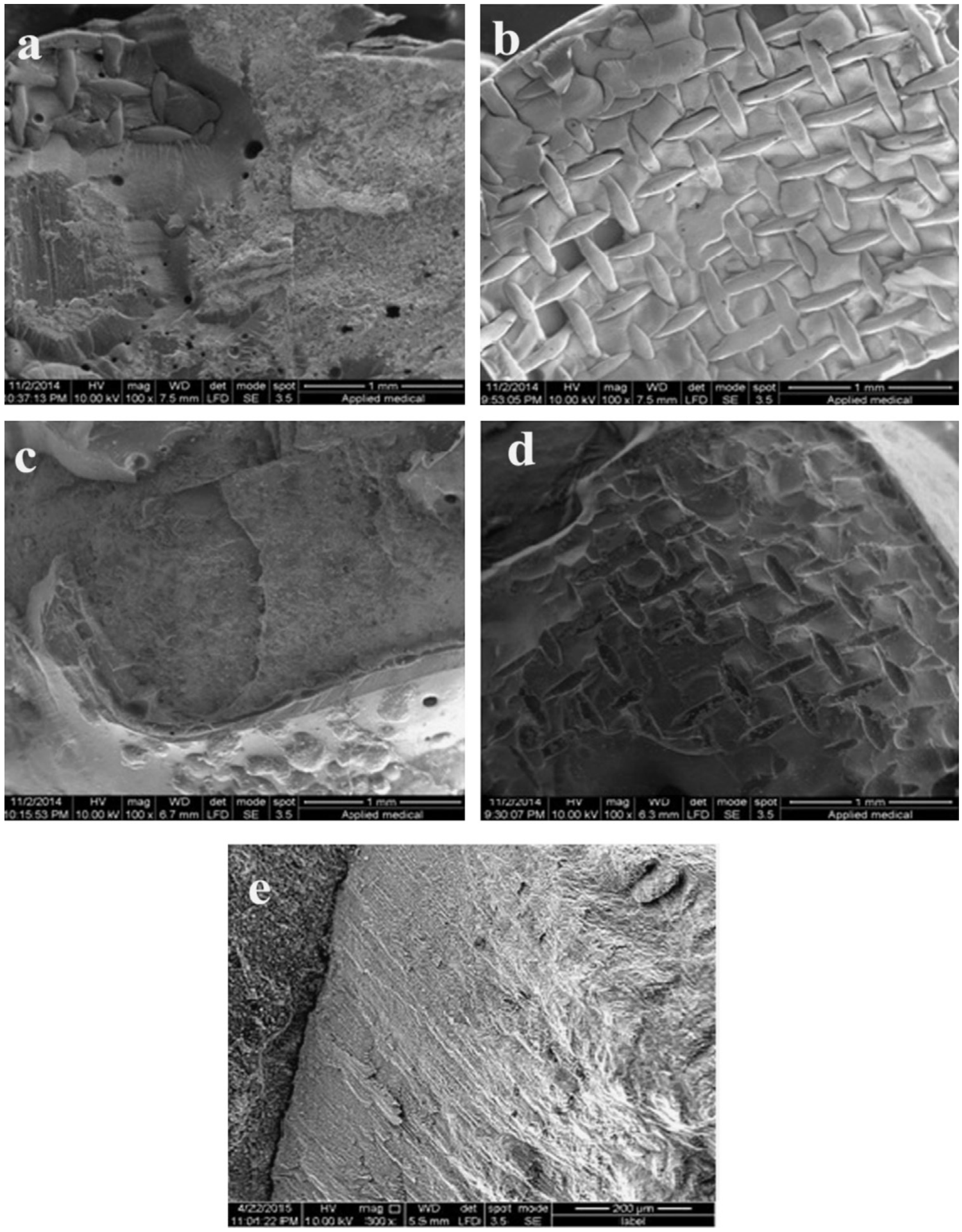

Fig. 2. Scanning electron photomicrographs of the debonded surface representing ARI score and one instance of enamel fracture: (a) ARI score 0, (b) ARI score 1, (c) ARI score 2, (d) ARI score 3 and (e) enamel fracture in group 1 at X500, for scoring see text.

tured from the opposite side by the camera. The contact angle was then automatically calculated by the computer connected to the optical tensiometer.

2.6. Statistical analysis

The statistical software Statistical Package for Social Sciences (SPSS) v 22.0 (SPSS, Chicago, IL) was used to analyze the data. One-way ANOVA and multiple comparisons of means were tested with Tukey's multiple comparisons *post hoc* analysis at a significance level of $p < 0.01$.

3. Results

The mean bond strengths and standard deviations of the groups are presented in Table 2. The mean adhesion strength results (MPa) were relatively high in groups containing the experimental primers, *i.e.*, group 4 (15.8 ± 1.6) followed by group 3 (12.5 ± 1.5), group 2 (11.9 ± 1.1) and group 1 (08.1 ± 0.7). Significant difference was observed among the groups except for group 3 and group 4 ($p < 0.01$) as demonstrated by Tukey's *post hoc* analysis.

The ARI score was distributed to 0 and 1, in groups 3 and 4, respectively and in group 1 and 2; the scores of 2 and 3 were also observed. One instance of enamel fractures were recorded in group 1 when a load of 94.2 N was applied to debond the bracket (Table 3).

SEM images of the representative ARI score and the only instance of enamel fracture among group 1 specimens are presented in Fig. 2.

The contact angle measurements suggested the lowest values with primer containing (ACPS) and (ACPS+BTSE), $< 5°$, followed by Transbond XT, (41.9 ± 4.56) degrees (Fig. 3).

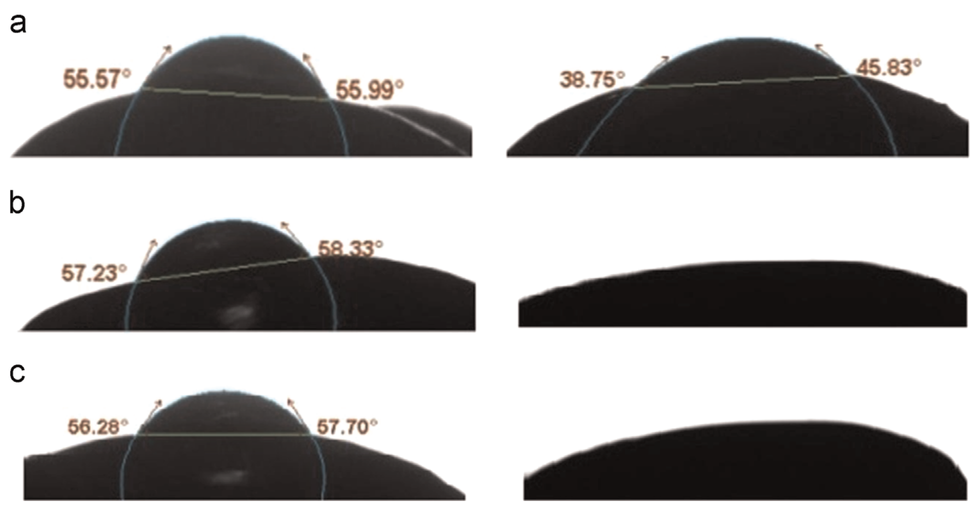

Fig. 3. Contact angle measurements without primer application and with primer application. (a) Transbond XT (b) ACPS and (c) ACPS + BTSE. After primer application, b and c showed 0° water contact angles indicating a hydrophilic surface.

4. Discussion

There is always a constant quest in the orthodontic field to overcome the short comings of clinical orthodontic bonding. To minimize these problems, application of primer has been advocated by some researchers [30].

Adhesion strength of the bracket adhesives to tooth substrates can be effectively increased by primer application [31,32]. This said, the current novel experimental silane primer system has been considered a successful treatment option in non-etchable ceramic materials [23–33]. This outcome encouraged the authors to test the current novel primers also for orthodontic bonding to promote good adhesion between the bracket and tooth enamel as this has been so far unreported. Based on the result of the present study the hypothesis was accepted.

The experimental silane blend of ACPS and BTSE in group 4 produced the highest adhesion strength among the study groups. This result also answered the question 'Do we need primers for orthodontic bonding?' raised by a few studies [34–36]. Definitely, the application of primers in the present test setup showed promising enhanced adhesion strength (measured and reported as shear bond strength) as compared to the group without experimental primers. Thus, both the experimental primers with reactive acrylate silane monomers have shown greater adhesion strength values compared to the conventional orthodontic primer in the current study. It is not clear whether the impact of silane-based experimental primers is of physical or chemical interactions – or both. This needs to be assessed in the near future. Fig. 1 is exhibiting the molecular structures of the adhesive reactive monomers in the primers used. The exact composition of the commercial primer Transbond XT is not disclosed, but it contains both *bis*-GMA and TEGDMA, with reactive vinylic methacrylate groups. Both of them are bigger as molecules and have higher molecular weight (*bis*-GMA = 512,59 g/mol; TEGDMA = 286,33 g/mol) than the silanes used. It is unclear if this had any effect in adhesion strength *per se*.

Adhesion (bond) strength of *ca.* 6–8 MPa is reported to be enough for clinical orthodontic needs [37,38]. This bond strength value was exceeded in all of the study groups in the present study. However, these reference values have been criticized [39–41] because of the factors influencing the outcome of bond test such as the bonding area, substrate surface topography, placement of bracket and direction of the debonding force [42]. It is also noteworthy that the *in vitro* bond strength differs considerably from that of *in vivo* because of the oral environment conditions in which moisture contamination greatly reduces adhesion [13]. However, the maximum adhesion strength should be inferior to the tensile strength of enamel, which ranges between 11 and 25 MPa, depending on the prismatic orientation [43]. Bonding with the experimental silane based primers showed low ARI scores. The only instance of enamel fracture was seen in group 1 with no primers and low adhesion strength of 9 MPa. This suggests that high bond strength will not necessarily fracture the enamel as shown in the present study. Newman et al. stated that adhesion strength should be less than 21 MPa to avoid damage to enamel [44]. Enamel fractures could be a possibility during the clinical process of debonding especially if the tooth is nonvital [45]. The hydrophilic nature of the primers might be a good choice during clinical orthodontic bonding where complete dry enamel surface cannot be maintained [5–46].

5. Conclusion

Within the limitations of the present *in vitro* study, it might be concluded that application of silane based primers prior to bonding increased the adhesion strength of orthodontic brackets.

Conflict of interest

None declared.

Acknowledgments

The project was financially supported by Vice Deanship of Research Chairs, King Saud University.

References

[1] Fitzgerald I, Bradley GT, Bosio JA, Hefti AF, Berzins DW. Bonding with self-etching primers–pumice or pre-etch? An in vitro study Eur J Orthod 2012;34(2):257–61.
[2] Northrup RG, Berzins DW, Bradley TG, Schuckit W. Shear bond strength comparison between two orthodontic adhesives and self-ligating and conventional brackets. Angle Orthod 2007;77(4):701–6.
[3] Vicente A, Toledano M, Bravo LA, Romeo A, de la Higuera B, Osorio R. Effect of water contamination on the shear bond strength of five orthodontic adhesives. Med Oral Patol Oral Cir Bucal 2010;15(5):e820–6.

[4] Mustafa AA, Matinlinna JP, Razak AA, Hussin AS. Effect of experimental silane-based primers with various contents of 2-hydroxyethyl methacrylate on the bond strength of orthodontic adhesives. J Investig Clin Dent 2014;5:1–9.
[5] Bishara SE, Oonsombat C, Ajlouni R, Denehy G. The effect of saliva contamination on shear bond strength of orthodontic brackets when using a selfetch primer. Angle Orthod 2002;72(6):554–7.
[6] Wang WN, Li CH, Chou TH, Wang DD, Lin LH, Lin CT. Bond strength of various bracket base designs. Am J Orthod Dentofac Orthop 2004;125(1):65–70.
[7] Bishara SE, Soliman M, Laffoon J, Warren JJ. Effect of changing a test parameter on the shear bond strength of orthodontic brackets. Angle Orthod 2005;75(5):832–5.
[8] Pashley DH. Dentin: a dynamic substrate–a review. Scanning Microsc 1989;3(1):161–74 Discussion 174-6. 18.
[9] Soderholm KJ. Correlation of in vivo and in vitro performance of adhesive restorative materials: a report of the ASC MD156 task group on test methods for the adhesion of restorative materials. Dent Mater 1991;7(2):74–83.
[10] Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. J Dent Res 1955;34(6):849–53.
[11] Jou GL, Leung RL, White SN, Zernik JH. Bonding ceramic brackets with light-cured glass ionomer cements. J Clin Orthod 1995;29(3):184–7.
[12] Watanabe T, Tsubota K, Takamizawa T, Kurokawa H, Rikuta A, Ando S, et al. Effect of prior acid etching on bonding durability of single-step adhesives. Oper Dent 2008;33(4):426–33.
[13] Cozza P, Martucci L, De Toffol L, Penco SI. Shear bond strength of metal brackets on enamel. Angle Orthod 2006;76(5):851–6.
[14] Zhu JJ, Tang AT, Matinlinna JP, Hagg U. Acid etching of human enamel in clinical applications: a systematic review. J Prosthet Dent 2014;112(2):122–35.
[15] Coreil MN, McInnes-Ledoux P, Ledoux WR, Weinberg R. Shear bond strength of four orthodontic bonding systems. Am J Orthod Dentofac Orthop 1990;97(2):126–9.
[16] Ghiz MA, Ngan P, Kao E, Martin C, Gunel E. Effects of sealant and selfetching primer on enamel decalcification. Part II: an in-vivo study. Am J Orthod Dentofac Orthop 2009;135(2):206–13.
[17] Lowder PD, Foley T, Banting DW. Bond strength of 4 orthodontic adhesives used with a caries-protective resin sealant. Am J Orthod Dentofac Orthop 2008;134(2):291–5.
[18] Paschos E, Okuka S, Ilie N, Huth KC, Hickel R, Rudzki-Janson I. Investigation of shear-peel bond strength of orthodontic brackets on enamel after using Pro Seal. J Orofac Orthop 2006;67(3):196–206.
[19] Lung YK, Matinlinna JP. Silanes for Adhesion Promotion and Surface Modification. Hauppauge: Nova Science Publishers; 2013. p. 87.
[20] Matinlinna JP, Lassila LV, Vallittu PK. The effect of three silane coupling agents and their blends with a cross-linker silane on bonding a bis-GMA resin to silicatized titanium (a novel silane system). J Dent 2006;34(10):740–6.
[21] Matinlinna JP, Lassila LV, Vallittu PK. Evaluation of five dental silanes on bonding a luting cement onto silica-coated titanium. J Dent 2006;34(9):721–6.
[22] Lung CY, Matinlinna JP. Aspects of silane coupling agents and surface conditioning in dentistry: an overview. Dent Mater 2012;28(5):467–77.
[23] Matinlinna JP, Lassila LV, Kangasniemi I, Vallittu PK. Isocyanato- and methacryloxysilanes promote Bis-GMA adhesion to titanium. J Dent Res 2005;84(4):360–4.
[24] Heikkinen T, Matinlinna J, Vallittu P, Lassila LJ. Dental zirconia adhesion with silicon compounds using some experimental and conventional surface conditioning methods. Silicon 2009;1(3):199–202.
[25] Matinlinna JP, Choi AH, Tsoi JK. Bonding promotion of resin composite to silica-coated zirconia implant surface using a novel silane system. Clin Oral Implant Res 2013;24(3):290–6.
[26] Matinlinna JP, Tsoi JK, de Vries J, Busscher HJ. Characterization of novel silane coatings on titanium implant surfaces. Clin Oral Implant Res 2013;24(6):688–97.
[27] Matinlinna J, Ozcan M, Lassila L, Kalk W, Vallittu P. Effect of the crosslinking silane concentration in a novel silane system on bonding resin-composite cement. Acta Odontol Scand 2008;66(4):250–5.
[28] 13990-2 D. Zahnheilkunde-Prüfverfahren für die Scherhaftfestigkeit von Adhäsiven für Kieferorthopädische Befestigungselemente. Gesamtverbund Befestigungselement-Adhäsiv-Zahnschmelz. Berlin: Beuth; 2009.
[29] Artun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. Am J Orthod 1984;85(4):333–40.
[30] Ozcan M, Vallittu PK, Peltomaki T, Huysmans MC, Kalk W. Bonding polycarbonate brackets to ceramic: effects of substrate treatment on bond strength. Am J Orthod Dentofac Orthop 2004;126(2):220–7.
[31] Bishara SE, Soliman M, Laffoon J, Warren JJ. Effect of antimicrobial monomer-containing adhesive on shear bond strength of orthodontic brackets. Angle Orthod 2005;75(3):397–9.
[32.] Shinya M, Shinya A, Lassila LV, Gomi H, Varrela J, Vallittu PK, et al. Treated enamel surface patterns associated with five orthodontic adhesive systems – surface morphology and shear bond strength. Dent Mater J 2008;27(1):1–6.
[33] Lung CY, Botelho MG, Heinonen M, Matinlinna JP. Resin zirconia bonding promotion with some novel coupling agents. Dent Mater 2012;28(8):863–72.
[34] O'Brien KD, Watts DC, Read MJ. Light cured direct bonding–is it necessary to use a primer? Eur J Orthod 1991;13(1):22–6.
[35] Tang AT, Bjorkman L, Adamczak E, Andlin-Sobocki A, Ekstrand J. *In vitro* shear bond strength of orthodontic bondings without liquid resin. Acta Odontol Scand 2000;58(1):44–8.
[36] Wang WN, Tarng TH. Evaluation of the sealant in orthodontic bonding. Am J Orthod Dentofac Orthop 1991;100(3):209–11.
[37] Reynolds IR. A review of direct orthodontic bonding. Br J Orthodont 1975;2:171–8.
[38] Turgut MD, Attar N, Korkmaz Y, Gokcelik A. Comparison of shear bond strengths of orthodontic brackets bonded with flowable composites. Dent Mater J 2011;30(1):66–71.
[39] Eliades T. Comparing bond strengths. Am J Orthod Dentofac Orthop 2002;122(6):13A–5A Author reply 15A.
[40] Eliades T, Brantley WA. The inappropriateness of conventional orthodontic bond strength assessment protocols. Eur J Orthod 2000;22(1):13–23.
[41] Finnema KJ, Ozcan M, Post WJ, Ren Y, Dijkstra PU. In-vitro orthodontic bond strength testing: a systematic review and meta-analysis. Am J Orthod Dentofac Orthop 2010;137(5):615–22 e3.
[42] Yetkiner E, Ozcan M, Wegehaupt FJ, Wiegand A, Eden E, Attin T. Effect of a low-viscosity adhesive resin on the adhesion of metal brackets to enamel etched with hydrochloric or phosphoric acid combined with conventional adhesives. J Adhes Dent 2013;15(6):575–81.
[43] Carvalho RM, Santiago SL, Fernandes CA, Suh BI, Pashley DH. Effects of prism orientation on tensile strength of enamel. J Adhes Dent 2000;2(4):251–7.
[44] Newman GV, Newman RA, Sun BI, Ha JL, Ozsoylu SA. Adhesion promoters, their effect on the bond strength of metal brackets. Am J Orthod Dentofac Orthop 1995;108(3):237–41.
[45] Joseph VP, Rossouw E. The shear bond strengths of stainless steel and ceramic brackets used with chemically and light-activated composite resins. Am J Orthod Dentofac Orthop 1990;97(2):121–5.
[46] Campoy MD, Vicente A, Bravo LA. Effect of saliva contamination on the shear bond strength of orthodontic brackets bonded with a self-etching primer. Angle Orthod 2005;75(5):865–9.