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Original Article

Hybrid Layer, Shear Bond Strength, and Fracture Patterns of Titanium Dioxide–Doped Phosphate Glass–Filled Universal Dental Adhesives

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Abstract

Objectives The aim of the study was to explore the potential effects of incorporating 5 and 10 wt% of TiO_2-doped phosphate glass powder as fillers into the universal adhesive system.

Materials and Methods Human permanent premolars and molars were used in the study. Five and 10 wt% of TiO_2-doped phosphate glass powder as fillers were added into the universal adhesive system. Unmodified universal adhesive was used as control. The effects of the added filler in the universal adhesive were examined on hybrid layer formation at the resin composite and dentine interface (mesio-occlusal-distal [MOD] cavities) under scanning electron microscope (SEM), shear bond strength (SBS) of resin composite to dentine using shear bond testing machine, and the patterns of fracture at the resin composite–dentin interface, which were examined under stereomicroscope. The SBS analyses were performed with (8 samples per group, $n = 24$) and without (8 samples per group, $n = 24$) 5,000 cycles of thermocycling.

Statistical Analysis One-way analysis of variance (ANOVA) was used to analyze the data of the SBS. For bond strength, the effects of adding fillers into the universal adhesive were analyzed.

Results The SEM images showed that the hybrid layers were similar in all the groups of unmodified and modified adhesives. An ANOVA test revealed that the SBSs of control and modified adhesives were not significantly different before ($p = 0.15$) or after ($p = 0.39$) thermocycling for all the groups. The patterns of fracture revealed various types of fracture in all adhesive groups including composite resin, adhesive, and dentine failure. Composite resin fractures are the most encountered pattern of fracture.

Conclusion Adding 5 and 10 wt% of TiO_2 into universal adhesive did not adversely affect the hybrid layer, SBS, or mode of failure of composite resin to dentine. The

Keywords
- titanium dioxide-doped phosphate glass
- universal dental adhesives
- shear bond strength
- hybrid layer

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pattern of fracture at the resin composite and dentine interface showed a favorable bonding with more cohesive than adhesive failure, particularly with the 5 wt% glass-modified adhesive group.

Introduction

The development of resin composite and adhesion to enamel and dentine has revolutionized the practice of restorative dentistry.[1,2] It removes the dependency on amalgam, which has limitations such as unaesthetic, less conservative cavity preparation, and possible cracks of the tooth due to slow expansion over time.[3] However, bonding resin composite to dentine is not as predictable as its bonding to enamel due to the wet nature of dentine because of dentinal tubules and intratubular fluid. Attempts have been made to improve the bonding of resin composite to dentine by modifying the materials and clinical steps. Originally, the total etch technique was used in which exposed dentine surfaces were etched, primed, and bonded. Unfortunately, the technique had caused posttreatment sensitivity due to opened dentinal tubules after etching. After that, the partial etch technique was introduced to partially remove or modify the smear layer instead of totally removing it with the previous total tech technique.[4,5] The partial etch technique has led to the development of the one-step bonding system, in which all the components are included in one bottle. Many studies had investigated the hybrid layer and bond strength of various bonding systems and techniques. Results have shown comparable bond strengths of these techniques.[6–8] Thus, the one-step technique is popular because it is more user friendly for clinicians. Despite all the efforts to improve the bond strength of resin composite to dentine, limitations still exist such as microleakage due to polymerization shrinkage of the material. Microleakage will lead to bacterial accumulation, toxin penetration, and secondary caries. There were efforts to modify the adhesive by adding fillers to reduce the risk of secondary caries and improve remineralization. A recent review looked at the potential uses of dental glass-ceramics (DGCs), which includes treatment of hypersensitivity.[9] Treatment hypersensitivity may include sealing of potential microgaps at the tooth–restorative material interface. A group of researchers, led by one of the co-authors of this study, looked at the potential use and benefits of titanium dioxide–doped phosphate-based glasses (PBGs).[10] The group had experimented about the potential benefits and applications of 5 and 10% TiO_2. Another study by the same co-author and colleagues investigated the antibacterial effect of titanium dioxide–doped phosphate glass microspheres filled total-etch dental adhesive on *Streptococcus mutans* biofilm and micro-tensile bond strength (MPa).[11] The results of the study showed that all tested adhesives showed higher inhibitory effect on *S. mutans* biofilm growth than the negative control. It was also concluded from the study that glass microspheres modified adhesives further inhibited the biofilm growth. Furthermore, the study also found that incorporation of 5 wt% of glass microspheres produced significantly higher inhibitory effect than 10 wt%. In addition, the micro-tensile bond strength of the modified adhesive was also improved by the addition of glass microspheres. Therefore, the modified adhesive would be promising to reduce the possibility of recurrent caries around restorations due to remineralization potential,[12] increased bond strength of restorations to tooth structures, and minimization of microleakage. However, further studies are needed to investigate the potential effects of titanium dioxide–doped system on other parameters such as shear bond strength (SBS) of composite resin to dentine and the hybrid layer. Positive results from these studies will strengthen the potential clinical applications of the material for benefits of remineralization and antibacterial properties. This study therefore aimed to investigate the effect of incorporation of these titanium dioxide–doped glass powder on hybrid layer and shear bond strength of a universal dental adhesive. The alternative hypothesis was that the "addition of glass microspheres into a universal adhesive would have significant effect on hybrid layer and SBS to dentin."

Materials and Methods

Experimental Design

This was an *in vitro* experiment with human extracted teeth looking at the effects of adding titanium dioxide–doped phosphate glass into a universal dental adhesive on a hybrid layer, SBS, and fracture patterns of bonded composite resin to dentin.

Glass Preparation

Titanium dioxide–doped phosphate glass with 50 mol% phosphorus pentoxide (P_2O_5), 30 mol% calcium oxide (CaO), 17 mol% sodium oxide (Na_2O), and 3 mol% titanium dioxides (TiO_2) were prepared using the melt-quenching process.[13] The following precursors were used to prepare the required glass composition: P_2O_5, calcium carbonate ($CaCO_3$), sodium dihydrogen phosphate (NaH_2PO_4), and TiO_2. All precursors have a purity of more than 98%. The required amount of each precursor was mixed and heated in a platinum crucible for 30 minutes at 300°C to remove all gases and water, and then melted at 1,100°C for 1 hour. The melted glass was left to cool overnight at room temperature after being poured on a steel plate. After cooling, the glass powder was prepared by grinding using a ball milling machine[10] and sieved to get particle size in the range of 10 to 60 µm.[13]

Glass-Filled Adhesive

The glass powder was added into the Universal Adhesive (Tetric N-Bond, Ivoclar Vivadent AG, Liechtenstein) at 5 and

10 wt%. Unfilled adhesive was used as a control (named as normal adhesive). The required amount of glass powder was weighted and added to the adhesive and mechanically stirred using a vortex *mixer* (ZX3 Advanced Vortex Mixer, VELP Scientifica SrL, Italy) for 5 minutes. Prior to each use, the filled adhesives were stirred for 2 minutes.[11]

Hybrid Layer Under SEM

Nine (9) extracted permanent human premolars were used after getting an ethical approval from the Research Ethics Committee of the University of Sharjah (Reference number: REC-22–06–15) and randomly distributed into three groups (normal adhesive, 5wt% glass-filled adhesive, and 10 wt% glass-filled adhesive). A mesio-occlusal-distal (MOD) cavity preparation was done using straight fissure diamond round burs (Diaswiss SA, Nyon, Switzerland) with a high-speed handpiece (Sirona, Germany). The preparation dimensions were 3 mm proximal depth, 1.5 to 2 mm occlusal depth, and isthmus width was no more than 2 mm. Selective enamel etching was done using 37% phosphoric acid (Cica Etching Gel, Promedica, Neumünster, Germany) for 10 seconds, then rinsed with tap water and dried for 5 seconds with gentle blow of air from the air syringe, and dapping with dry cotton palettes. Care was taken not to blow air too hard to avoid desiccation of dentin. After this, the application of the adhesive was done according to the manufacturer's instructions for 20 seconds, then dried for 5 seconds, and cured for 20 seconds. Curing was performed using a light-curing unit (BLUEDENT LED smart, BG Light Ltd) at 1,400 mW/cm^2 and calibrated by a Led Radiometer (SDI). The MOD cavities were filled using packable composite shade A3 (Tetric N-Ceram, Ivoclar Vivadent AG). Teeth were then sectioned mesiodistally using a precision saw (IsoMet 1000 TechCut, Buehler, Lake Bluff, Illinois, United States).

For scanning electron microscope (SEM) analysis, the teeth were immersed in 6 N HCl acid for 3 minutes, then rinsed with tap water for 5 minutes. This was followed by deproteinization using 2.5% sodium hypochlorite for 5 minutes, then rinsing with tap water for another 5 minutes. After that, samples were fixed with 2.5% (v/v) glutaraldehyde (Sigma Aldrich, St. Louis, Missouri, United States) for 1 hour[14] followed by rinsing with Dulbecco's phosphate-buffered saline (Sigma Aldrich) two to three times. Finally, the samples were dehydrated using 50, 70, 90, and 100% ethanol 10 minutes each before being gold coated and viewed under the SEM (Tescan VEGA XM, Czech Republic).

Shear Bond Strength without Thermocycling

For this part of the study, 24 human permanent molars were used (ethical approval from the Research Ethics Committee, University of Sharjah [Reference number: REC-22–06–15]). The teeth were randomly assigned into three groups (unmodified universal adhesive, 5 wt% glass-filled adhesive, and 10 wt% glass-filled adhesive) and sectioned mesiodistally exposing the buccal mid-coronal dentin using a precision saw (IsoMet 1000 TechCut, Buehler). After this, the mid-coronal dentin was etched using 37% phosphoric acid (Cica Etching Gel, Promedica) for 10 seconds, followed by rinsing and drying for 5 seconds and application of adhesive according to the manufacturer's instructions. The area was then air dried for 5 seconds and cured for 20 seconds. A small 2-mm-diameter straw was used as matrix; the straw was marked and cut at a length of 5 mm, then placed on the bonded surface. Packable composite shade A3 (Tetric N-Ceram, Ivoclar Vivadent AG) was placed in 2-mm increments and cured to produce composite cylinders. The teeth were then mounted on self-cure acrylic resin (Vertex Self-Curing, Vertex-Dental, Soesterberg, the Netherlands) and kept in humidity in room temperature for 24 hours. After which, the operator mounted the acrylic-mounted samples on Shear Bond Tester (Bisco, United States) and force was applied using a notched arm at a crosshead speed of 0.5 mm/min until failure.[15] Load at failure was recorded for each sample.

Shear Bond Strength after Thermocycling

Twenty-four extracted human permanent molar teeth (ethical approval from the Research Ethics Committee, University of Sharjah [Reference number: REC-22–06–15]) were randomly assigned to three groups (normal adhesive, 5% adhesive, and 10% adhesive). Each tooth was prepared following the same procedures as mentioned earlier to produce 2×5 mm composite cylinders. The acrylic resin in which the teeth were mounted was marked with 1, 2, and 3 dots pertaining to normal adhesive, 5% adhesives, and 10% adhesives, respectively. This was done to mark which adhesive group each tooth belonged to. The thermocycling test was performed using Thermocycler THE 1100 (SD Mechatronik GMBH, Feldkirchen-Westerham, Germany) for 5,000 cycles, the equivalent of 6 months. The specimens were placed in the basket and immersed in hot (55°C) and cold water (5°C) for 30 seconds alternatively, with 5 seconds for transfer in between.[16] The teeth were then taken out, loaded onto the SBS testing machine, and subjected to force using a notched arm at a crosshead speed of 0.5 mm/min until failure.[17]

Pattern of Fracture

After SBS testing, the samples were examined under a stereomicroscope (Stereo Discovery V20; Carl Zeiss Ltda., Rio de Janeiro, RJ, Brazil) at 200× magnification to evaluate the pattern of fracture and classify it into dentinal, composite, or adhesive layer fractures.[18] The examination was done by one operator, then repeated after 5 days to ensure reliability. Cohen's kappa values showed that intra-evaluator reliability was good (0.92).

Sample Size Calculation

Sample size was calculated using a standard deviation and comparing two means from a previous study[15] and the following formula[19]:

$$n = [2(Z\alpha + Z\beta)2\sigma 2]/\Delta 2,$$

where $n =$ required sample size, $Z\alpha = 1.96$ (a constant), $Z1 - \beta = 0.8416$ (a constant), $\sigma =$ standard deviation, and

Δ = the difference in effect of two interventions (means difference between control and test from a previous study). So, eight samples per group can reject the null hypothesis that the means of the experimental and control groups were equal with a probability (power) of 0.85. The type I error probability (α) associated with this test of this null hypothesis was 0.05.

Statistical Analysis

One-way analysis of variance (ANOVA) and post hoc test were used for SBS analysis. The level of significant was set at $p < 0.05$.

Results

Hybrid Layer under SEM

SEM images showed resin tag formation in all samples (►**Figs. 1**–**3**). The tags were of similar size among all adhesives. The images also showed good penetration of adhesive into the tubules that formed good length of resin tags. Incorporation of 5 and 10 wt% titanium dioxide-doped phosphate glass did not seem to affect the formation of resin tags.

Shear Bond Strength

The mean SBS and standard deviations of all adhesive groups are shown in ►**Fig. 4**. ANOVA showed no statistically significance difference before ($p = 0.15$) and after thermocycling ($p = 0.39$) in the SBSs within and among all the adhesive groups. The SBS among unmodified adhesive, 5 wt% glass-modified adhesive, and 10 wt% glass-modified adhesives were not statistically significant different with or without thermocycling.

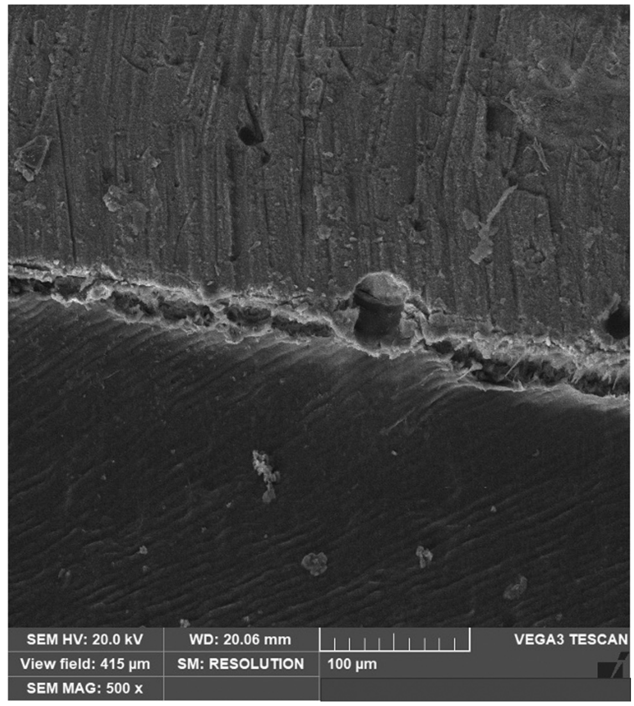

Fig. 2 Representative image of hybrid layer with resin tags of 5 wt% titanium-doped phosphate glass–modified adhesive.

Pattern of Fracture

The types of failure at the resin composite–dentin interface are shown in ►**Table 1**. The 5 wt% glass-modified adhesive group had more fractures in the composite resin (6 fractures in the composite resin and 2 fractures at adhesives). The control adhesive had more fractures at adhesive (5 fractures

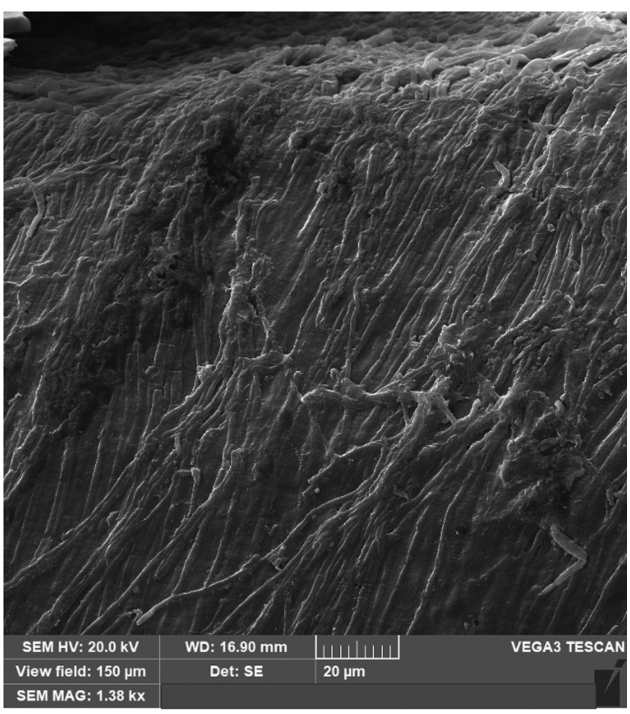

Fig. 1 Representative image of hybrid layer with resin tags of unmodified universal adhesive.

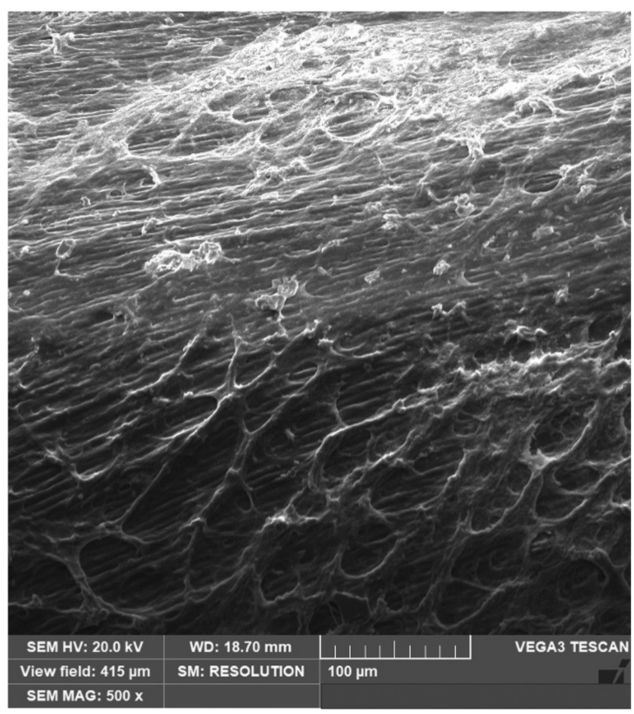

Fig. 3 Representative image of hybrid layer with resin tags of 10 wt% titanium-doped phosphate glass–modified adhesive.

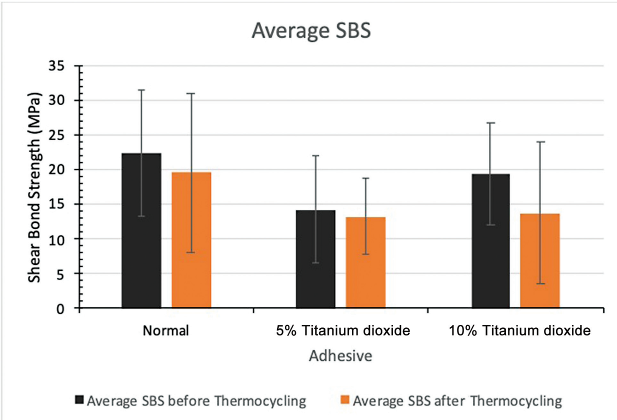

Fig. 4 Averages shear bond strength (SBS) values of the three groups before and after thermocycling.

at adhesive and 3 fractures in the composite resin). The 10 wt % glass-modified adhesive group had four fractures in the composite resin, three fractures in adhesives, and one fracture in dentine.

Discussion

A hybrid layer is formed at the interface of composite resin and dentine surface.[14] It is composed of adhesive around the collagen matrix of demineralized dentine and resin tags in the dentinal tubules. Application of acid onto the surface of dentine either through the total-etch or self-etch technique will completely or partially remove the smear layer, which will facilitate the formation of resin tags and a hybrid layer. Removal of the smear layer will expose dentinal tubules that will facilitate the penetration of adhesive into dentinal tubules forming resin tags. In addition, etching will remove hydroxyapatite crystals around the collagen matrix of dentine. The removal of minerals around the collagen will also facilitate the penetration of adhesive around it forming the hybrid layer. The formation of resin tags and a hybrid layer gives bond strength of composite resin to dentinal surfaces. It was reported that a strong correlation exists between hybrid layer thickness and bond strength.[20] Therefore, it is critical to examine the effects of adding a new material into the adhesive, which may affect the hybrid layer and resin tag formation.

PBGs are degradable, and their degradation can vary widely according to the type of metallic oxides included in the composition. As observed in a previous study, the degradation of PBGs can be reduced by two orders of magnitude with the incorporation of 5 mol% iron oxides due to the formation of more hydration-resistant bonds.[21] In another study, the incorporation of 5 mol% of titanium oxides reduced the degradation rate by one order of magnitude[22] and going beyond 5 mol% did not produce a significant change in the degradation rate.[23] Generally, any change in the degradation rate is associated with a change in the level of ion release from these glasses and hence the pH in the surrounding environment. Since these glasses can accommodate a variety of metallic oxides, they can be potentially used for a wide spectrum of applications including remineralization of hard tissues,[12] therapeutic ion release,[24] stem cell research,[25] and tissue engineering.[26]

The SEM images of this study did not show any adverse effects of adding 5 and 10% by weight of titanium dioxide–doped phosphate glass into the universal adhesive on hybrid layer formation at the interface of composite resin and dentine. Resin tag formation appears similar among the three adhesives (control, 5 wt%, and 10 wt% glass-modified adhesives). A study reported different thickness of hybrid layer among various adhesive systems.[14] However, its appearance under SEM is similar in this study. The absence of different characteristics of the hybrid layer in this study could be due to the homogenous distribution of the glass powder in the adhesives and its noneffectiveness or nonreactiveness to the surrounding tissues and materials. During stirring of the adhesive after adding the glass powder, small-sized particles were well dispersed and blended in. That was confirmed by a previous work.[11] As the material did not adversely affect the formation of the hybrid layer, the beneficial properties of the material such as remineralization potential, antibacterial, and antirecurrent caries can be fully utilized. These beneficial properties of the materials were already reported in a previous publication.[23]

A strong bond to hard tooth tissue is a criterion used to select a dental material in restorative dentistry. This strong bond can minimize microleakage and withstand the forces during mastication.[27] In line with the results of hybrid layer from SEM images, the SBS for all groups with and without thermocycling did not show any statistically significant

Table 1 Patterns of fractures for each group

Sample no.	Control adhesive	5 wt% glass-modified adhesive	10 wt% glass-modified adhesive
1	Composite	Composite	Composite
2	Adhesive	Composite	Adhesive
3	Adhesive	Adhesive	Composite
4	Adhesive	Composite	Adhesive
5	Adhesive	Composite	Composite
6	Composite	Adhesive	Composite
7	Adhesive	Composite	Dentine
8	Composite	Composite	Adhesive

differences. The SBS values of this study range between 14 and 22 MPa (►**Fig. 4**), which are almost similar to the ones reported by a previous study.[15] The study reported SBS values of 15 to 22 MPa. Mortazavi et al also concluded that there was a statistically significant difference in the SBS between filled and unfilled adhesives in the enamel; however, the opposite was true in the dentin, which is echoing the results of this study.[28] Thermocycling as aging-induced changes in bonding of an adhesive to human teeth was also part of this study. It is an important investigation done to mimic the oral environment.[17] Miljkovic et al reported that a decrease in what was an initially high SBS was observed in certain adhesives, which is why thermocycling is important.[29] The finding was supported by a previous study that described reduced bond strengths after a thermocycling process of 5,000 cycles.[14] However, the results of the current study were different. It could be due to the use of different adhesives in the previous study. Furthermore, it could be related to the release of ions such as calcium and phosphorus from the glass-modified groups; these ions could enhance the stability of the formed bond and therefore, a nonsignificant change in SBS was observed after thermocycling. In the thermocycling process, samples can be subjected to thermal aging ranging from 100 to 10,000 cycles. According to the ISO/TR 11405,[30] 500 thermocycles in water at temperatures between 5 and 55°C is an appropriate test for aging of dental materials. In this study, 5,000 thermal cycles were selected to correspond to approximately 6 months of *in vivo* functioning.[31] Aging-induced changes through thermocycling may affect the smear layer and bond strength. However, the average bond strength in all the groups in this study, regardless of thermocycling, was not statistically significant different. That indicates there were no significant effects from thermocycling on the bond strength in this study. However, longer cycles, such as 10,000, may produce different outcomes. The average SBS for all groups in this study is also comparable with previous studies,[14,15] indicating that the methodology was valid. As bond strength is affected by the formation of the hybrid layer at the composite resin and dentine interface,[14,20] the results of this study corroborate the findings from SEM images. The results of no statistically significant different in SBS in this study are important because good bond strength is crucial in minimizing potential microleakage due to polymerization shrinkage of composite resin. In addition, thermocycling also did not significantly affect the SBS. This is beneficial for potential clinical application as the mouth temperature varies during intake of hot food and drinks.

In terms of the patterns of fracture after SBS test, the 5 wt% glass-modified group seems to have more fracture in composite resin than within the control adhesive. That indicates a stronger bond of 5 wt% glass-modified groups. This finding is similar to that recorded by a previous study, which observed cohesive fracture patterns in dentin or composite in adhesives with under 20 wt% filler content.[14] This finding also relates to the condition of the hybrid layer. A good hybrid layer will produce stronger SBS and result in cohesive fractures as supported by an earlier investigation.[29] The fracture patterns of adhesives and cohesive are similar to those reported in a previous study.[18] The most prominent type of fracture seemed to be cohesive fracture in composite; the same finding was recorded by other studies.[14,17]

Within limitation of this *in vitro* study, incorporation of 5% and 10% titanium dioxide-doped phosphate glass into universal dental adhesive had no significant effect on the hybrid layer and SBS of resin composite to dentin. Therefore, the null hypothesis was rejected. In addition, the pattern of fracture at resin composite and dentine interface showed favorable bonding with more cohesive than adhesive failure. The results of this study indicate that 5% and 10% titanium dioxide–doped phosphate glass–filled universal dental adhesive has potential in terms of other properties of the material such as remineralization and antibacterial properties, which was proven by a pervious study as mentioned earlier.

Authors' Contributions

A.B.H., H.M.E., and E.A.A.N. conceptualized and designed the study. S.M.F.S. and I.M.A. performed acquisition of data. All the authors revised the manuscript for submission.

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Conflict of Interest

None declared.

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