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ORIGINAL ARTICLE

Improving Tensile Bond Strength of Orthodontic Bracket by Applying Papain Gel as an Email Deproteinization Agent

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ABSTRACT

An effort to improve the bonding between bracket and tooth surface is required. **Objective:** The aim of this study was to evaluate the effect of papain gel on tensile bond strength (TBS) and adhesive remnant index (ARI) of the orthodontic brackets. **Methods:** A total of 42 healthy human premolars were randomly divided into six groups. 1) Resin-modified glass ionomer cement (RMGIC) without papain, 2) RMGIC with papain 8%, 3) RMGIC with papain 10%, 4) Composite resin (CR) without papain, 5) CR with papain 8%, 6) CR with papain 10%. The TBS was determined using a universal testing machine. Bond failure was classified according to the ARI. The TBS data were analyzed with Kruskal-Wallis test followed by Mean Whitney tests with 5% of significance level. **Results:** The mean of TBS(MPa) values of RMGIC groups are without papain (5.03 ± 1.52), papain 8% (4.79 ± 2.61), papain 10% ($7.75 \pm 1, 48$). CR groups without papain (5.45 ± 1.23), papain 8% (2.30 ± 0.73), and papain 10% (4.84 ± 1.72). Bond failure was mainly classified as score 1. The TBS values were statistically influenced by the application of papain and adhesive. **Conclusion:** The application of papain 10% before RMGIC cementation improves the tensile bond strength and could decrease the bond failure of the orthodontic bracket.

Key words: enamel, orthodontic bracket, papain, tensile bond strength

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INTRODUCTION

Fixed orthodontics is a type of orthodontic appliances where bracket is bonded to teeth. The purpose of the treatment is to correct malocclusion and malposition of teeth. The bonding of bracket to tooth surface which retains shear and tensile forces plays an important role in succeeding the orthodontic treatment.¹ The strong bonding is needed to ensure there is no bond failure between bracket and teeth. The bond failure of brackets during orthodontic treatment can delay the treatment, thus increasing the treatment cost, and make the patient uncomfortable.² Therefore, an effort to improve the bonding between bracket and tooth surface is essentially required.

One of the factors influencing the failure of bracket bonding to tooth surface is pretreatment of enamel surface. Eliminating organic substances from enamel surface (deproteinization) before enamel etching can increase the shear bond strength of orthodontic bracket.³ For this, enzyme can be employed as organic removal agent. Papain is protease enzyme extracted

from leaves, branches, and fruit of papaya tree (*Carica Papaya* Linn). The enzyme has been employed for antibacterial, antifungal, antiviral, antiinflammation and deproteinization agents.⁴ These useful features can be employed for dental applications. The papain can remove debris from teeth and increase etching process of enamel. It introduces a rougher surface and it is essential to enhance the bond strength between adhesive and tooth enamel.³ Aside the interesting features in the chemical treatment, the papain enzyme has no toxic effects thus showing biocompatibility to tooth surfaces.^{5,6}

The application of papain enzyme in the range of 2-10% to the tooth enamel and investigated the its effect on the shear bond strength of orthodontic brackets.⁵ It was found that there is positive effects when papain was employed as the pretreatment before applying adhesive of resin modified glass ionomer cement (RMGIC) and composite resin (CR) to the teeth. There was an optimum amount of papain which give a high shear stress and the optimum range between 8 and 10% results. However, there is a lack of information of the

effect of papain pretreatment to the tensile strength. For bonding failures due to debonding process, the tensile strength of adhesive and bracket is also an important component, because there is a tensile force to labial and buccal of teeth on orthodontic treatment.^{2,7}

OBJECTIVE

The aim of this study was to evaluate the effect of papain gel as an enamel deproteinization agent on the in vitro tensile bond strength (TBS) and adhesive remnant index (ARI) of the orthodontic brackets.

METHODS

Materials used in the study was papain enzyme (P3125) from papaya latex which was purchased from Sigma Aldrich (Singapore). Adhesive materials used resin modified glass ionomer cement (RMGIC) were from GC Corporation (Tokyo, Japan) and composite resin (CR) were obtained from Ortho Technology, Inc. (Tampa, USA). For etching the enamel, 36% phosphoric acid was obtained from PPH Cerkamed (Polska), and 10% polyacrylate acid from GC Corporation (Tokyo, Japan). Orthodontics metal brackets were purchased from Alpha Drive, (McMinnville, USA). The research samples consist of 42 maxillary premolar teeth with a careful selection to meet requirements of free of fracture, crack and caries. The teeth were firstly cleaned from calculus and stored in distilled water for 24 hours. Approval to conduct clinical tests was obtained from Ethical Commission of Dentistry Faculty at the Universitas Gadjah Mada (No 00296/KKEP/FKG-UGM/EC/2015).

Teeth selection and papain gel preparation

The crowns were cut until cement-enamel junction and embedded in polymethyl methacrylate (PMMA) in aluminium moldings (dimension of 15mm x 15mm x 15mm). The buccal surface of the teeth projected above and parallel to the surface of PMMA. Buccal tooth surfaces were flatted and smoothed by carbide paper (P600 type) in the water flooding (ISO 11405: 2013). After that, the buccal surfaces were polished with water and pumice (without flour content) by applying prophylaxis brush. Papain gel was made by heating distilled water prior to temperature 70°C. Nipagin, nipasol and papain enzyme incorporated and homogenized at 1000rpm stirrer. CMC-Na gradually incorporated to the mixture until the gel was formed.

Classification of sample group

The teeth were randomly divided into six groups which each category consists of 7 samples, according to the type of adhesive and surface treatment method. Details for each group are described as follow. Group 1 (control); the sample was etched with 10% polyacrylic acid, fixed with RMGIC (GC Corporation, Tokyo,

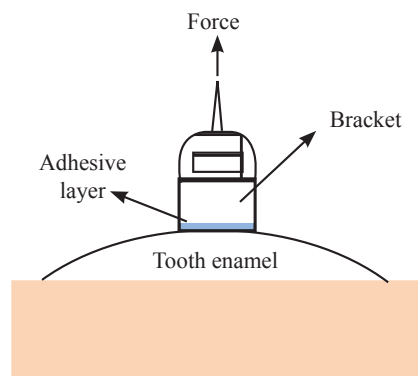


Figure 1. Schematic of tensile bond strength determination

Japan); Group 2; the sample was deproteinized with 8% papain gel for 30 s, etched with 10% polyacrylic acid, fixed RMGIC; Group 3; the sample was deproteinized with 10% papain gel, etched with 10% polyacrylic acid, fixed RMGIC; Group 4 (control); the sample was etched with 36% phosphoric acid, followed by primer application, fixed with composite resin (Ortho Technology, Inc., Tampa, Florida); Group 5; the sample was deproteinized with 8% papain gel, etched with 36% phosphoric acid, followed by primer application, fixed with a composite resin; Group 6; teeth were polished with 10% papain gel, etched with 36% phosphoric acid, followed by primer application, fixed with a composite resin.

The etching of enamel with 10% polyacrylic acid (Dentin conditioner, GC, Japan) and 36% phosphoric acid (Blue etch, PPH cerkamed, Polska) are for 20s with an applicator brush according to the manufacture's recommendation. Deproteinization of enamel with 8% and 10% papain was performed in 30s. Orthodontics metal brackets (Ortho Classic, NE Alpha Drive, McMinnville, USA) were bonded and activated with light curing unit (Woodpecker Co, Cina) for 20s on each mesial and distal bracket. All samples were stored in distilled water at 37°C for 24 hours (ISO 11405, 2003).

Tensile bond strength testing and analysis methods

The tensile strength of subject was tested using the universal testing machine (Universe, USA) (Figure 1). After debonding, the surface morphology of the teeth was observed by stereomicroscope and scored by adhesive remnant index (ARI). Figure 2 displays sample scoring using ARI.

Statistical analysis

The data obtained was evaluated using Kruskal-Wallis non-parametric analysis, because data were not normally distributed by Kolmogorov-Smirnov test. The analysis was continued by post hoc test to differentiate between the means. All evaluations were performed using SPSS 16 software with a significant level of $p < 0.05$.

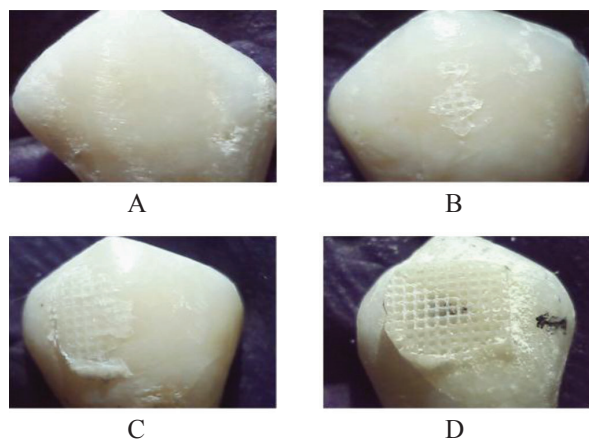


Figure 2. Adhesive Remnant Index (ARI) score: 0, there is no adhesive residual on the teeth (A); 1: less than half of the adhesive residual on the teeth (B); 2: more than half of the adhesive residual on the teeth (C); 3: all the adhesive residual on the teeth (D)

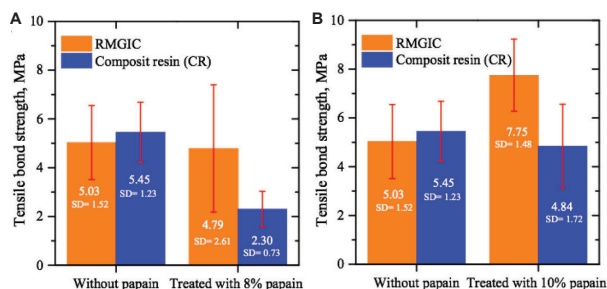


Figure 3. Tensile bond strength (MPa) values of bracket orthodontics without or with application of 8% papain (A) and with application of 10% papain (B)

RESULTS

The present study showed that the groups of 10% papain before cementation with RMGIC had the highest tensile bond strength value. On the RMGIC groups, the enhancement of tensile bond strength by application of 10% papain gel was 54%, while the use of 8% papain gel decreased the tensile bond strength 4.7%. In otherwise, the tensile bond strength of CR groups showed the different result. The highest tensile bond strength was showed by the groups without papain application. The use of 8% and 10% papain decreased 57% and 11% tensile bond strength in respect to the property without applying papain. On the groups without papain, the tensile bond strength of bracket orthodontic with CR was higher than RMGIC groups. However, application of papain gel before RMGIC increased the tensile bond strength significantly.

Normality of tensile bond strength data was analyzed use Kolmogorov-Smirnov test, while homogeneity of data was analyzed with a Lavene test. The result of that test showed that data were not homogeneity with

Table 1. Distribution of ARI score of the groups

Groups	n	ARI score				
		0	1	2	3	
RMGIC	Without papain	7	0	6	0	1
	8% Papain	7	1	3	3	0
	10% Papain	7	1	6	0	0
Composite Resin	Without papain	7	2	4	0	1
	8% Papain	7	7	0	0	0
	10% Papain	7	5	0	0	2

significance level of $p < 0.05$. Therefore, data were analyzed with Kruskal-Wallis nonparametric test to analyze differences the tensile bond strength between the groups.

The result of Kruskal-Wallis test showed that application papain gel gives significantly effect on the tensile bond strength of bracket orthodontic with value 10.98 ($p < 0.05$). In the other hand, the adhesive also significantly give effect on the tensile bond strength of orthodontic bracket with value 5.24 ($p < 0.05$). According to that result, it can be concluded that papain gel and adhesive material had significantly effect on the improvement of the tensile bond strength of orthodontic bracket. The research data were further tested using a post hoc Mann Whitney test. Mann-Whitney post hoc test showed the application of 10% papain gel increases the tensile bond strength of bracket orthodontic on RMGIC groups significantly, while 8% papain gel decrease the tensile bond strength of bracket orthodontic on RMGIC groups significantly. ARI value distribution is presented in Table 1 which showed that 90% of the value of ARI in RMGIC groups distributed over a range of 1-3. The different results obtained in composite resin group showed 95% of ARI values distributed over a range of 0-1.

DISCUSSION

This work aimed to study the effect of papain on the resulting composite of bracket orthodontic. Therefore, the subject treated with papain gel was compared to the groups without papain gel treatment. The glass ionomer cement and resin composite were employed as the test subject. The result of this study showed that the application of papain gel and adhesive material significantly affect the tensile bond strength of orthodontic brackets. The sample treated with 10% papain for email deproteinization before the RMGIC application shows the highest the tensile bond strength among the others. The application of 10% papain influences on tensile bond strength enhancement, especially if papain was combined with glass ionomer

cement as adhesive. That combination produces the highest tensile bond strength than the other groups. Pithon et al. (2013) reported that 10% papain give the best result to improve the tensile bond strength of bracket orthodontic.⁵

Papain is a proteolytic enzyme which is extracted from papaya (*Carica papaya* Linn) latex and has ability on enamel deproteinization.⁸ Deproteinization is a method to remove the organic element and acquired pellicle from email, therefore improve the orthodontic bracket bonding and prevent white spot formation.^{3,5} Deproteinization before etching can improve quality etching pattern, therefore enamel surface able to bind to the adhesive material.⁹

In groups without papain application, the tensile bond strength of composite resin group was higher than RMGIC group. Before application of composite resin, the teeth previously were etched by 37% phosphate acid, while the teeth were etched by 10% polyacrylate acid before RMGIC application. Enamel etching with phosphate acid gives etching pattern better than polyacrylate etching. Therefore, phosphate etching can increase the bonding between the orthodontic bracket and enamel.³ The enhancement of orthodontic bracket bonding with phosphate acid has also been reported that shear bond strength with 37% acid phosphate acid gives higher value than polyacrylate acid etching.¹⁰ Application of 10% papain gel has no significant effect on the enhancement of the tensile bond strength of composite resin groups. In otherwise, has a significant effect on RMGIC groups.

The enhancement of tensile bond strength of RMGIC groups showed that deproteinization with papain gel before cementation with RMGIC would be effective in clinical practice. Without deproteinization, glass ionomer cement resin only chemical binding through bonding between polyacrylate acid and enamel.³ The result of this study, as well as previous study that conclude combination between deproteinization material and phosphate acid decreased the shear bond strength of orthodontic bracket,¹¹ while combination between deproteinization material and polyacrylate acid increased the shear bond strength of orthodontic bracket. Therefore, deproteinization gives effect on enhancement tensile bond strength if combined with RMGIC. The result of tensile bond strength of bracket orthodontic on RMGIC groups with papain application was 7.75 MPa. The tensile bond strength has been needed by an orthodontic bracket to retain oral cavity, and orthodontic force is 5-8 Mpa.¹² Therefore, application of papain gel before RMGIC as adhesive material on orthodontic bracket attachment can be recommended.

Distribution of ARI value in RMGIC and composite resin groups without application of papain showed the result of attachment failure caused due to failure of the

bond between the bracket and the adhesive material while failure of attachment to the composite resin group combined with papain application caused due to failure of the bond between enamels and adhesive, since more than 50% of adhesive material attached to the bracket. These results are consistent with the results of research showing that the failure of the composite resin adhesions due to weak bonding between the adhesive material and email.¹³ In the composite resin group that preceded the application of papain 8% indicated that 100% adhesion failure between email and adhesive material, because there is no residual adhesive material was left on the teeth after the tensile test belt. Values lower tensile testing would decrease the value of ARI with no or at least an indication of the material remaining in the email.

The use of materials previously RMGIC combined with papain gel to deproteinization email can be an option for improving the attachment of orthodontic brackets. RMGIC can be a good binding to either the email so that the tensile strength of the resulting belt able to withstand the force of orthodontic and oral cavity. RMGIC is also able to release fluoride which can prevent the risk of white spot lesions are easily formed on email attached by a bracket.¹⁴

CONCLUSION

The effect of enzyme deproteinization of papain on the tensile bond strength of orthodontic bracket was studied. In the case of glass ionomer cement, application of 10% papain gel before cementation of the adhesive could improve the tensile bond strength of orthodontics bracket, hence decreasing the attachment failure of orthodontics bracket. Another case, when using composite resin as adhesive there is no positive effect of application of papain gel in the attachment of orthodontic bracket.

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