

## ORIGINAL ARTICLE

## SHEAR BOND STRENGTH OF ETCH AND RINSE ADHESIVES TO DENTIN: COMPARISON OF BOND STRENGTH AFTER ACID AND PAPACARIE PRE-TREATMENT

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**Background:** Papacarie pre-treatment of dentin surface has been reported to exhibit good bond strength values between dentin and composites. Its desirable properties like high pH, sites specificity and anti-inflammatory response makes it a future material in place acid etching pre-treatment. Acid etching may cause sensitivity and collapse of dentinal matrix. **Methods:** This in-vitro study involved, 60 caries free extracted premolars, randomly divided into three groups. Control Group (Group A) No pre-treatment of dentin prior adhesive application. Experimental Group B acid etchant was applied before adhesive application. Experimental Group C Papacarie was used as a pre-treatment agent. All these specimens were tested for shear bond strength with the help of Universal Testing Machine. All the collected data was entered in SPSS version 20.0. ANOVA was used to determine the mean SBS (Shear Bond strength) values of control and experimental groups. **Results** The mean shear bond strength of material was  $7.74 \pm 0.47$  in group A,  $17.80 \pm 1.43$  in group B and  $15.11 \pm 0.70$  in group C. Group B showing better strength than other two groups. **Conclusion:** The study provides information about longevity of composite restorations and may help in extending the clinical usage of papacarie to avoid harmful effects of acid etching on dentin and pulpal tissue.

**Keywords:** Adhesive; Shear Bond strength; Tensile strength; Acid etching

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### INTRODUCTION

Dental adhesives are used to bond composite resins with tooth structure. A strong bond between dentin and resin is required to prevent microleakage, recurrent caries, and pulpal irritation to give good marginal seal at the tooth restoration interface.<sup>1</sup> A strong bond between the hydrophilic dentin and hydrophobic composite is difficult without use of dentin adhesives.<sup>2</sup> Adhesives are classified as etch and rinse adhesives and self-etch adhesives based on adhesion strategy. In etch and rinse adhesive, separate acid etching and conditioning is required to demineralize the tooth substrate. Adhesive flow into the porosities to form resin tags. These resin tags facilitate the mechanical bonding of adhesive to the dentin. In self-etch adhesives, acid etchant and bonding agent is suspended in the same bottle. Demineralization and infiltration occur simultaneously to save the clinician's time and number of steps.<sup>3</sup>

Acid etching enhances micromechanical bond. It demineralises and makes tooth substrate porous, removes the contaminants from the surface hence making the surface more reactive to form a strong bond.<sup>4</sup> Acid etching to dentine is more challenging than acid etching to enamel. It is due to the heterogeneous structure of dentine, comprising of 50% by volume of

hydroxyapatite crystals, 30% organic components and 20% water.<sup>5</sup> Several measures adapted, to improve the bond strength include, Ethanol wet bonding, extending adhesive application time, use of warm air to accelerate solvent evaporation, use of protease enzyme inhibitors, use of collagen cross linkers, rubbing action during the adhesive application are some of clinical procedures suggested to improve bond durability with dentin.<sup>6</sup>

Papacarie pre-treatment of dentin has been reported to exhibit strong adhesive bond. It enhances the marginal seal and act in a similar way as acid etchant.<sup>7</sup> The main advantage of using Papacarie is its neutral pH in contrast to 37% Phosphoric acid as acid etchant.<sup>8</sup> It was introduced as Chemomechanical agent for caries removal in 2003. Chemomechanical method of caries removal is based on biological principle as it removes the infected dentin making it softer so that it can be easily removed resulting in rough dentinal surface suitable for strong bonding.<sup>9</sup> Drilling techniques can cause pain and discomfort in patients due to heat generation, pressure desiccation and vibration of the instruments. Drilling also removes healthy tooth tissue along with decayed part. The concept of conserving healthy tooth tissue has gained popularity with the introduction of chemomechanical methods of caries removal.<sup>10</sup> The papacarie gel composed of papain, a proteolytic cysteine enzyme having anti-

inflammatory and antibacterial properties. Papain is site specific and acts against infected tissue only and does not do any harm to healthy tissue.<sup>11</sup> Chloramine, chlorinate and softens the carious dentin so that it can be easily removed with the help of blunt instrument.<sup>12,13,9</sup>

Tensile analysis and shear analysis are the two common modes to determine the bond strength values. Shear mode of analysis determine local bonding conditions and provide accurate results.<sup>14</sup> These bond strength values determine and predict the longevity of dental restorations.<sup>15</sup> Bond strength is the initial mechanical load to fracture divided by geometrically cross-sectional area of the bond.

The study may elaborate the efficacy of Papacarie as pre-treatment agent in comparison to acid etchant. There is a need for further studies to be conducted to elaborate this subject.

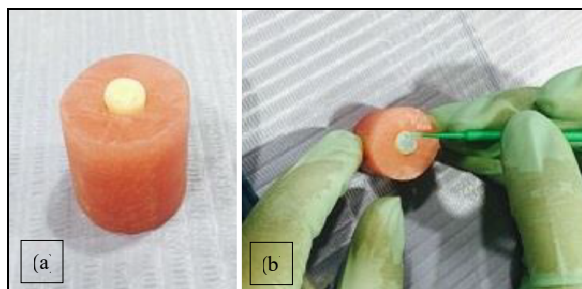
## MATERIAL AND METHODS

This experimental in-vitro study was conducted at de' Montmorency College of Dentistry, Lahore in collaboration with PCSIR lab Lahore.

Sixty healthy caries and fracture free human premolars indicated for extraction as a part of orthodontic treatment were used for this study and stored in 0.2 % thymol solution. Sample size was calculated by formula

$$n = (Z1-\beta + Z1-\alpha/2)^2 (\sigma1^2 + \sigma2^2) / (\mu1 - \mu2)$$

They were polished with slurry of pumice and water. Occlusal one third of teeth were reduced to obtain flat dentin surface with the help of slow speed diamond bur and their axial surfaces were reduced to form round cylinders, attaining an occlusal diameter of 5mm and height of acrylic resin. (Figure-1 (a,b))



**Figure-1: (a) Mounted samples in acrylic blocks. (b) Application of papacarie**

Teeth were randomly divided into three groups; Group A: Control group: Adhesive was applied directly. No pre-treatment with conditioning agent was performed. Adper single bond plus was applied

according to manufacturer instructions followed by light cured composite resin (Z100) restoration.

Group B: Pre-treatment with 35% phosphoric acid (15 sec) was performed and then Adper single bond plus was applied according to manufacturer instructions followed by light cured composite resin restoration.

Group C: Pre-treatment with papacarie was performed. Papacarie was applied to the dentin surface for 60 seconds then gel was removed with excavator. Dentin surface was then cleaned with wet cotton pellet and Adper single bond plus was applied according to manufacturer instructions followed by light cured composite resin restoration.

The details of the material used in this study are mentioned in the table-1.

Composite placed in a two-layer increment using plastic mould and light cured for 40 seconds. Each composite cylinder was cured again for 40 s after removal of mould. All the specimens were stored for 24 hours before shear bond testing. Each acrylic embedded premolar tooth with its bonded composite resin was mounted on universal testing machine (Shimadzu Corporation, Tokyo, Japan). Force applied by the machine on each specimen was at a cross head speed of 1mm/min in a compression mode using a blade parallel to the adhesive dentin interface. The bonded composite cylinder was positioned horizontally so that the shearing blade is perpendicular at composite dentin interface. Each specimen was loaded until failure occurs. Shear force required to debond the specimen were recorded. The shear forces were recorded in MPa and were obtained directly from Instron computer software.

## RESULTS

Data was entered and analysed through SPSS version 20.0. Mean±S.D were calculated for quantitative variables, e.g., shear bond strength. ANOVA test was used to see the significant difference between the shear bond strengths. For pair wise comparison (A vs. B, A vs. C, B vs. C) Tukey's test applied. Level of significance was set at  $p=0.05$  or  $p<0.0$ . The mean Shear Bond Strength in group A was  $7.74\pm0.47$ MPa, in group B was  $17.80\pm1.43$  MPa, in group C was  $15.11\pm0.70$ MPa. There was significant difference observed in all groups regarding mean shear bond strength ( $p$ -value  $<0.001$ ). Group B showed highest shear bond strength while group A showed least shear bond strength. Table-2

For multiple comparisons, Post Hoc Tukey test was used that showed that the mean shear bond strength of group B was significantly higher as compared to groups A and C ( $p$ -value  $<0.001$ ). There was also significant difference observed among groups A & C ( $p$ -value  $<0.001$ ). Table-3

**Table-1: Material used in the study**

| Product name                             | Composition   | Manufacturer                          |
|--|---|---------------------------------------|
| Papacarie                                | Papain, chloramine, toluidineblue, salts, preservative and thickener  | Formula & Acao, Sao Paulo, SP, Brazil |
| 3M ESPE Scotchbond™ Etchant              | 35% phosphoric acid by weight.  | 3M/ESPE                               |
| 3M ESPE Adper single bond plus Adhesive. | BisGMA, HEMA, dimethacrylates, ethanol, water, Photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids. | 3M/ESPE                               |
| Composite resin(Z100)                    | Bis-GMA, TEGDMA, Zirconia, Silica, Filler volume :66% Filler weight :84.5%  | 3M/ESPE                               |

**Table-2: Comparison of shear bond strength among groups.**

| Groups  | Mean  | S.D  | p-value <sup>a</sup> |
|---------|-------|------|----------------------|
| Group A | 7.74  | 0.47 | <0.001*              |
| Group B | 17.80 | 1.43 |                      |
| Group C | 15.11 | 0.70 |                      |

a One way ANOVA. \*The mean difference is significant at the 0.05 level.

**Table-3: Pair wise comparison of shear bond strength among groups.**

| Group | Groups | Mean Difference | Std. Error | p-value <sup>a</sup> |
|-------|--------|-----------------|------------|----------------------|
| A     | B      | - 10.055        | 0.3027     | < 0.001*             |
|       | C      | - 7.360         | 0.3027     | < 0.001*             |
| B     | C      | 2.690           | 0.3027     | < 0.001*             |

a Post hoc Tukey test. \*The mean difference is significant at the 0.05 level.

## DISCUSSION

This study showed mean Shear Bond Strength in group A was  $7.74 \pm 0.47$  MPa, in group B was  $17.80 \pm 1.43$  MPa and in group C was  $15.11 \pm 0.70$  MPa. Thus, group B showed significantly highest shear bond strength than other two groups ( $p < 0.001$ ). These finding are found parallel with the study conducted in 2012 by Khattab and Omar. Where the mean shear bond strength value were  $17.855 \pm 4.729$  MPa in accordance with our study. They concluded that if papacarie is applied before placement of composite restoration it will increase the shear bond strength of composite with dentin. They stated that papacarie acts in a similar way as total etch technique and papacarie induces certain morphological changes in the residual dentin that are in favour of strong bonding and gives good marginal sealing when used in absence of acid etchant.<sup>7</sup>

Arora *et al.*, found that Papacarie has minimal smear layer and open dentinal tubules. Caries removal with rotary instruments showed a typical smear layer and occluded dentinal tubules. Resin tags obtained with Papacarie were the longest and significantly superior to those obtained with rotary method of caries removal. Researchers concluded that Papacarie exhibited better bonding characteristics of residual dentine than rotary method of caries removal. Thus, Papacarie is a suitable alternative to conventional method of caries removal.<sup>16</sup>

Kotb *et al.*, 2016 reported in their study that when papacarie is applied on dentin it makes the residual dentin more porous and irregular. So, in this

way residual dentin enhance the bond strength and also the dentin is free of smear layer that hinder the bonding capacity of the resin. They observed open dentinal tubules with patent orifices. All these features reported in the study are in favour of increasing shear bond strength.<sup>17</sup> In our study Group B pre-treated with acid etchant showed highest shear bond strength than other. Group C pre-treated with papacarie also showed results better than that of control group.

Pithon *et al.*, revealed far better shear bond strength values of  $50.88 \pm 17.75$  MPa than this study. This difference might be due to enamel deproteinized with 10% papain gel followed by etching with 37% phosphoric acid and underwent primer application prior bonding.<sup>18</sup> Current study was conducted on dentine and only Papacarie was used prior bonding application.

Acid etching technique makes the dental hard tissues porous, adhesive flow into these pores to form a mechanical bond. Sometime demineralization zone is not completely filled by resin due to smear layer formed between resin and tooth substrate. This gap formation leads to microleakage. Another challenge is, Acid etching to dentin is not easy as compare to etching enamel because dentin comprises of complex structure making it a difficult substrate for bonding.<sup>19</sup> Due to these reasons new techniques are under investigations to produce better clinical effects than acids.

## CONCLUSION

Adhesive dentistry has undergone great progress in the last decades and dental professionals are working

on to produce good adhesion with tooth substrate. Different strategies have been proposed to improve the durability of resin dentin interface.

In the study, Pre-treatment of dentinal surface in both the groups increases the shear bond strength of composite to dentin. Acid etching with phosphoric acid exhibited higher shear bond strength compared to papacarie treated teeth.

In future papacarie can be used to remove caries as well as to increase bond strength of composite restorations to dentin. It can be used in place of acid etchants to avoid harmful effects of acid on dentin and pulpal tissue.

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### AUTHORS' CONTRIBUTION

MS: Literature Search and execution of study plan.  
 NM: Conceptualization of study design, Organization of Data Scientifically, Corresponding author.  
 NI: Collection of result and compilation of data.  
 AQ: Data Collection and Data Interpretation.  
 MW: Conceptualization of study design and Data Collection. MWK: Proof reading and Literature search.

### REFERENCES

1. Nair M, Paul J, Kumar S, Chakravarthy Y, Krishna V, Shivaprasad. Comparative evaluation of the bonding efficacy of sixth and seventh generation bonding agents: An in vitro study. *J Conserve Dent* 2014;17(1):27-30.
2. Hoferichter C, Rüttermann S, Gerhardt-Szep S. Influence of three different dentin bonding agents on the adhesion of composite resin to dentine-An in vitro study. *J Dent Probl Solut* 2019;6(2):056-60.
3. Poggio C, Scribante A, Della Zoppa F, Colombo M, Beltrami R, Chiesa M. Shear bond strength of one step self-etch adhesives to enamel: effect of acid pretreatment. *Dental Traumatology* 2013;30(1):43-8.
4. Gateva N, Gusyiska A, Stanimirov P, Kabaktchieva R, Raichev I. Effect of etching time and acid concentration on micromorphological changes in dentin of both dentitions. *J IMAB* 2016;22(2):1099-1110.
5. Adhesive restorative materials: bonding of resin-based materials. In: McCabe JF, Walls AW, editors. *Applied Dental Materials*. 11 ed. Cambridge: Blackwell Publishing Ltd. 2011; p.230.

6. Carvalho RM, Manso AP, Geraldini S, Tay FR, Pashley DH. Durability of bonds and clinical success of adhesive restorations. *Dent Mater* 2012;28(1):72-86.
7. Khattab NM, Omar OA. Papain-based gel for chemo-mechanical caries removal: Influence on Microleakage and Microshear bond strength of esthetic restorative materials. *J Am Sci* 2012;8(3):391-9.
8. Gianini RJ, do Amaral FL, Flório FM, Basting RT. Microtensile bond strength of etch and rinse and self etch adhesive systems to demineralized dentin after the use of a papain based chemomechanical method. *Am J Dent* 2010;23(1):23-8.
9. Jain K, Bardia A, Geetha S, Goel A. Papacarie: A chemomechanical caries removal agent. *IJSS Case Rep Rev* 2015;1(9):57-60.
10. Goomer P, Jain RL, Kaur H, Sood R. Comparison of the efficacy of chemomechanical caries removal with conventional methods- A clinical study. *J Int Oral Health* 2013;5(3):42-7.
11. Bussadori SK, Castro LC, Galvao AC. Papain Gel: A new chemo-mechanical caries removal agent. *J Clin Paediatr Dent* 2005;30(2):115-20.
12. Cecchin D, Farina AP, Brusco EH, Carlini-Júnior B. Effect of cariosolv and papacarie on the resin dentin bond strength in sound and caries affected primary molars. *Braz J Oral Sci* 2010;9(1):25-9.
13. Hamama HH, Yiu CKY, Burrow MF, King NM. Chemical morphological and microhardness changes of dentine after chemomechanical caries removal. *Aust Dent J* 2013;58(3):283-92.
14. Munir N, Inayat N, Qaisar A, Khan SA, Rana MH. Evaluation of integrity of Amalgam-composite interface with two resin based intermediate materials. *J Bahria Univ Med Dent* 2017;7(2):119-24.
15. El Mourad AM. Assessment of Bonding Effectiveness of Adhesive Materials to Tooth Structure using Bond Strength Test Methods: A Review of Literature. *Open Dent J* 2018;12:664-78.
16. Arora R, Goswami M, Chaudhary S, Chaitra TR, Kishor A, Rallan M. Comparative evaluation of effects of chemo-mechanical and conventional caries removal on dentinal morphology and its bonding characteristics-An SEM study. *Eur Arch Paediatr Dent* 2012;13:179-84.
17. Kotb RM, Elkateb MA, Ahmed AM, Kawana KY, El Meligy OA. Dentin Topographic features following chemomechanical caries removal in primary teeth. *J Peadiatr Dent* 2016;40(6):472-9.
18. Pithon MM, Ferraz Cde S, de Oliveira Gdo C, Pereira TB, Oliveira DD, de Souza RA, *et al.* Effect of 10% papain gel on enamel deproteinization before bonding procedure. *Angle Orthod* 2012;82(3):541-5.
19. Davari A, Sadeghi M, Bakhshi H. Shear bond strength of an etch and rinse adhesive to Er : YAG Laser and for phosphoric Acid treated Dentin. *J Dent Res Dent Clin Dent Prospects* 2013;7(2):67-73.

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