

Original Article

Effects of ethanol concentrations of acrylate-based dental adhesives on microtensile composite-dentin bond strength and hybrid layer structure of a 10 wt% polyhedral oligomeric silsesquioxane (POSS)-incorporated bonding agent

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ABSTRACT

Background: The aim of this study was to assess for the first time the effects of different amounts of ethanol solvent on the microtensile bond strength of composite bonded to dentin using a polyhedral oligomeric silsesquioxane (POSS)-incorporated adhesive.

Materials and Methods: This experimental study was performed on 120 specimens divided into six groups (in accordance with the ISO TR 11405 standard requiring at least 15 specimens per group). Occlusal dentin of thirty human molar teeth was exposed by removing its enamel. Five teeth were assigned to each of six groups and were converted to 20 microtensile rods (with square cross-sections of 1 mm × 1 mm) per group. The “Prime and Bond NT” (as a common commercial adhesive) was used as the control group. Experimental acrylate-based bonding agents containing 10 wt% POSS were produced with five concentrations of ethanol as solvent (0, 20, 31, 39, and 46 wt%). After application of adhesives on dentin surface, composite cylinders (height = 6 mm) were bonded to dentin surface. The microtensile bond strength of composite to dentin was measured. The fractured surfaces of specimens were evaluated under a scanning electron microscope to assess the morphology of hybrid layer. Data were analyzed using one-sample *t*-test, one-way analysis of variance (ANOVA), and Tukey tests ($\alpha = 0.05$).

Results: the mean bond strength in the groups: “control, ethanol-free, and 20%, 31%, 39%, and 46% ethanol” was, respectively, 46.5 ± 5.6 , 29.4 ± 5.7 , 33.6 ± 4.1 , 59.0 ± 5.5 , 41.9 ± 6.2 , and 18.7 ± 4.6 MPa. Overall difference was significant (ANOVA, $P < 0.0001$). Pairwise differences were all significant (Tukey $P < 0.05$) except those of “ethanol 0% versus 20%” and “20% versus 31%.” All groups except “0% and 46% ethanol” had bond strengths above 30 MPa (*t*-test $P < 0.05$).

Conclusion: Incorporation of 31% ethanol as solvent into a 10 wt% POSS-incorporated experimental dental adhesive might increase the bond strength of composite to dentin and improve the quality and morphology of the hybrid layer. However, higher concentrations of the solvent might not improve the bond strength or quality of the hybrid layer.

Key Words: Dental adhesive, bond strength, polyhedral oligomeric silsesquioxanes, solvent, concentration

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INTRODUCTION

Marginal seal plays a critical role in longevity of composite restorations, reduction of microleakage, and subsequent complications such as tooth sensitivity, discoloration, secondary caries, or pulpitis.^[1-5] Marginal seal remains the main concern of adhesive dentistry, especially when bonding composites to dentin, and there is an ever-increasing effort to improve the marginal seal of dentin adhesives, plus improving their physico-mechanical properties.^[1,6-9]

One of the attempts to improve dentinal marginal seal is to optimize the amount of solvent. Most dentinal adhesives are a mixture of hydrophilic/hydrophobic monomers and a solvent (mostly acetone, ethanol, and water).^[10,11] Some concentrations of solvent would properly reduce the viscosity and enhance the bond between hydrophobic and hydrophilic materials present at the junction of composite-dentin bond. Below that level would be inadequate for proper wettability. On the other hand, above that level would decelerate polymerization and leave remnants of the solvent trapped within the composite-dentin junction, creating porosities within the hybrid layer and weakening the mechanical properties of the adhesive and bond.^[1,12-17]

Two recent approaches for improving chemo-mechanical properties of adhesives are using cyanoacrylates and incorporation of polyhedral oligomeric silsesquioxane (POSS) molecules.^[18-21] Cyanoacrylates are strong synthetic glues recently used in biomaterials due to their superiorities including good adhesion to tissues (especially to wet substrates) and being bacteriostatic.^[18,22-28] Despite their recent application in oral surgery, orthodontics, and periodontics,^[18,25,26] they have not been utilized in restorative dentistry, except in cyanoacrylate modified glass ionomer cements.^[18,24] Therefore, their assessment in restorative dentistry and prosthodontics has valuable clinical implications.

Although cyanoacrylates might provide high bond strengths, they are prone to losing a great portion of bond strength and thus can fail in long term.^[18,27,28] This is why POSS can be added to them.^[18,19] These nano-building blocks are recently introduced and showed to improve the physico-mechanical properties of nanostructured materials including recent dental materials.^[1,18-20,29-33] Furthermore, they can considerably reinforce cyanoacrylate adhesives.^[18,19]

Albeit the optimal percentage of ethanol has been previously determined for conventional adhesives, the values are controversial ranging between 5% and 30% ethanol.^[10,34] Regarding newer adhesives, no study has yet evaluated the effect of solvent concentration in cyanoacrylate adhesives or in POSS-reinforced cyanoacrylate adhesives. Hence, this study was conducted to assess ethanol concentration (in an experimental bonding agent incorporating POSS and di/tri-methacrylate) on microtensile bond strength and hybrid layer structure. The weight percentage of POSS in this study was 10 wt% based on our previous study, which found this concentration optimal for shear bond strength.^[18]

MATERIALS AND METHODS

This *in vitro* experimental study was performed on 120 specimens (20 specimens per group, in accordance with the ISO TR11405 standard's requirements [which was at least 15 specimens per group]); it compared the microtensile bond strength of an experimental bonding agent with the composition of di- and tri-methacrylate monomers, 10 wt% POSS,^[18] photoinitiator system, inhibitor, and five different concentrations of ethanol (0, 20, 31, 39, and 46 wt%) with a commercial material as control with the following formula: di- and trimethacrylate resins, dipentaerythritol pentaacrylate monophosphate, nanofillers-amorphous silicon dioxide, photoinitiators, stabilizers, cetyl amine hydrofluoride, and acetone.

Sample

Thirty intact human molar teeth without any caries, restorations, and cracks (inspected macroscopically) were collected and stored in 0.2% thymol solution at 4°C for 1 week maximum. The teeth were stored in double-distilled water for 24 h before the experiment. To expose the dentin, a diamond disc was used to trim down the enamel off the occlusal surface. The trimming was performed carefully and gradually. The crown was checked frequently under light microscopy to see if any enamel still exists on the surface. Trimming was stopped by seeing the first sign of complete enamel removal on the surface. The resulted uneven surface of dentin was polished – under humid conditions – using 600, 800, and 1200 grit silicon carbide abrasive papers, respectively. The teeth were divided into six groups of five each. The first group was bonded using a commercial dentin bonding agent. The rest were bonded with an experimental bonding

agent with five different concentrations of ethanol as their solvents.

Group 1 (control)

In this group, the dentin surface was first etched with 35% phosphoric acid gel (Ultradent, USA) for 15 s followed by rinsing with water spraying for 10 s. It was then rinsed with distilled water at $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$ (room temperature) for 20 s. The excess moisture was removed using a blot drying paper. The bonding agent (Prime and Bond NT, Dentsply/Caulk, UK) was then applied to the dentin surface according to the manufacturer's instructions. The solvent was evaporated using a water- and oil-free air syringe held 10 cm away from the surface. The adhesive was then light cured for 20 s using a light-curing unit (Litex 695c, Taiwan) with a light intensity calibrated at 600 mW/cm^2 . A transparent plastic tube measuring 6 mm in height and 8 mm in diameter was then placed on the prepared surface and filled with three increments of A2 shade light-cure composite resin (Filtek Z350, 3M ESPE, USA). The reason for using these specific bonding agent and composite was their availability and personal clinical experience. Each increment was 2 mm in thickness and separately light cured for 40 s [Figure 1].

Group 2 (experimental bonding agent with 0% ethanol)

In this group, dentin surface conditioning was carried out similar to the Group 1. Afterward, the solvent-free (0%) experimental cyanoacrylate dentin bonding agent was applied to the surface and light cured for 20 s (600 mW/cm^2). Then, a composite cylinder was built up on the conditioned surface as explained above.

Groups 3–6 (Experimental bonding agent with varying concentrations of ethanol)

These groups were similar to the Group 2, only with the difference of having a degree of ethanol as solvent. Ethanol concentrations were 20, 31, 39, and 46 wt% in Groups 3–6, respectively. Hence, there was an additional step (elimination of solvent by air spraying for 3–5 s) after the application of dentin bonding agent.

Preparation of microtensile test rods

Teeth in each group were mounted in self-polymerizing, transparent acrylic resin. They were positioned vertically and completely merged in the acrylic resin. A full-automatic CNC cutting machine (Nemofanavar, Tehran, Iran) was used to

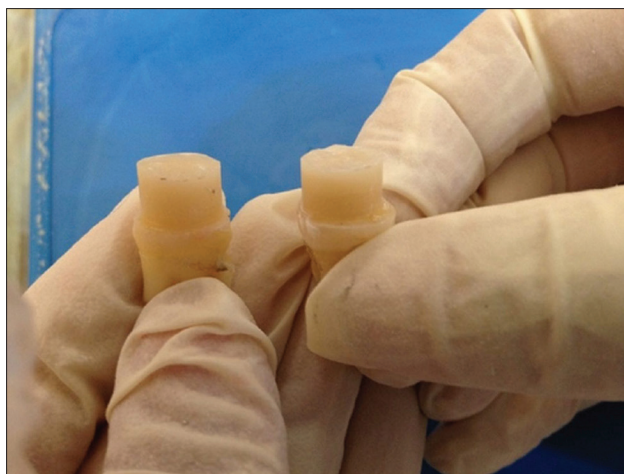


Figure 1: Composite cylinders built upon the dentin surface.

cut the acrylic blocks (and the specimens inside) in a grid pattern with cells of $1 \text{ mm} \times 1 \text{ mm}$. The results were rods with square cross-sections of $1 \text{ mm} \times 1 \text{ mm}$. Rods obtained from the circumference of each composite block might have enamel or acrylic resin in them. However, rods obtained from the center of each tooth were purely made of composite bonded to dentin. At least 20 rods free of any enamel were obtained from each tooth.

Water storage

All sectioned specimens in each group were separately immersed in double-distilled water and incubated at 37°C and 100% humidity for 24 h.

Microtensile testing

From each group, 20 rods were randomly selected for bond strength testing by the Universal Testing Machine (MTD-500 Plus, Germany). They were inspected under light microscopy to ensure the absence of any defects or enamel remnants.

The distance between two parts of the machine was adjusted to a fixed value for all specimens. Each specimen was mounted on the jig in a way that the dentin-composite interface was in the midway and parallel to the two horizontal plates keeping the rod. The two ends of each specimen were fixed to the two plates using cyanoacrylate adhesive (Akfix, Turkey). The tensile force was applied at a crosshead speed of 0.5 mm/min . The failure load was recorded. The length and width of each rod were accurately measured by a digital caliper with 0.01 mm accuracy level (Mitutoyo, Sakado, Japan). The surface area of each rod was calculated from its length and width. The microtensile bond strength (MPa) was calculated for each rod by

dividing the failure load (N) by the surface area of the dentin-composite interface (mm²).

Scanning electron microscope

From each group, three other rods were immersed in 5N hydrochloric acid for 5 s for better visualization of the hybrid layer and resin tags. They were then rinsed with water for 20 s. After 24 h of drying, rods were gold-coated and evaluated under a scanning electron microscope (SEM) unit (JXA-840, JEOL, Japan). SEM images of each specimen were recorded.

Statistical analysis

Descriptive statistics as well as 95% confidence intervals were calculated for the tensile bond strengths. One-way analysis of variance (ANOVA) was applied to compare the bond strength values among different groups. Tukey test was used for pairwise comparison of groups. One-sample *t*-test was used to compare bond strengths with the value 30 MPa, as the ceiling of the range of recommended minimum bond strengths representing clinical success.^[35,36] Significant level was predetermined as 0.05.

RESULTS

Increasing the concentration of ethanol solvent up to 31% increased the microtensile bond strength of composite to dentin. However, further increase in the concentration of ethanol solvent decreased the bond strength of composite to dentin [Table 1]. The ANOVA showed a significant difference between tested groups ($P < 0.0001$). Tukey *post hoc* test showed significant pair-wise differences between bond strengths in all groups ($P < 0.001$), except for the differences between 0% and 20% ethanol groups and between the control and 39% ethanol group [$P > 0.05$, Table 2]. According to the one-sample *t*-test, all groups except the experimental groups with 0% and 46% ethanol concentrations had bond strengths above 30 MPa the *P* value is calculated by comparing mean bond strength with the value 30 megapascal using one-sample *t*-test [Table 1].

The SEM showed improved hybrid layers when 20% and 31% ethanol concentrations were used as solvent [Figure 2].

DISCUSSION

The findings of this study indicated that the bond strength could be the highest when the ethanol

Table 1: The mean microtensile bond strength (megapascal) of composite to dentin in the understudy groups

Group	n	Mean±SD	CV (%)	95% CI	P
Control	20	46.5±5.6	12.0	43.88-49.12	<0.001
0% ethanol	20	29.4±5.7	19.4	26.73-32.07	0.643
20% ethanol	20	33.6±4.1	12.2	31.68-35.52	<0.001
31% ethanol	20	59.0±5.5	9.3	56.43-61.57	<0.001
39% ethanol	20	41.9±6.2	14.8	39.00-44.80	<0.001
46% ethanol	20	18.7±4.6	24.6	16.55-20.85	<0.001

n: Number; SD: Standard deviation; CV: Coefficient of variation; CI: Confidence interval for the mean

Table 2: The results of Tukey *post hoc* test comparing each group with all other ones

Groups	Mean difference	P	95% CI
Control versus 0%	17.10	<0.001	12.21-21.99
Control versus 20%	12.90	<0.001	8.009-17.79
Control versus 31%	-12.50	<0.001	-17.39--7.609
Control versus 39%	4.600	>0.05	-0.2905-9.491
Control versus 46%	27.80	<0.001	22.91-32.69
0% versus 20%	-4.200	>0.05	-9.091-0.6905
0% versus 31%	-29.60	<0.001	-34.49--24.71
0% versus 39%	-12.50	<0.001	-17.39--7.609
0% versus 46%	10.70	<0.001	5.809-15.59
20% versus 31%	-25.40	<0.001	-30.29--20.51
20% versus 39%	-8.300	<0.001	-13.19--3.409
20% versus 46%	14.90	<0.001	10.01-19.79
31% versus 39%	17.10	<0.001	12.21-21.99
31% versus 46%	40.30	<0.001	35.41-45.19
39% versus 46%	23.20	<0.001	18.31-28.09

CI: Confidence interval

percentage is about 31%; at this concentration of ethanol, bond strength of the experimental 10% POSS-incorporated material might be greater than that of a successful commercial bonding agent. Proper resin-to-enamel bond strengths have been estimated to be between 15 and 30 MPa by some authors, while being estimated as over 18 MPa, or between 20 and 25 MPa at least by other authors.^[35-39] In this study, the bond strength of all groups except the one lacking ethanol and the one with 46% ethanol was significantly above 30 MPa. These results were in line with some studies reporting an increase in bond strength by adding about 30% ethanol to the solvent.^[10] However, it was in contrast to the study of Mirzaee *et al.*,^[34] who evaluated the effect of addition of ethanol as solvent to solvent-free James-2 dental adhesive on microshear bond strength of composite to dentin and demonstrated that addition of 5% ethanol increased the bond strength; however, higher amounts of solvent could not increase the bond strength

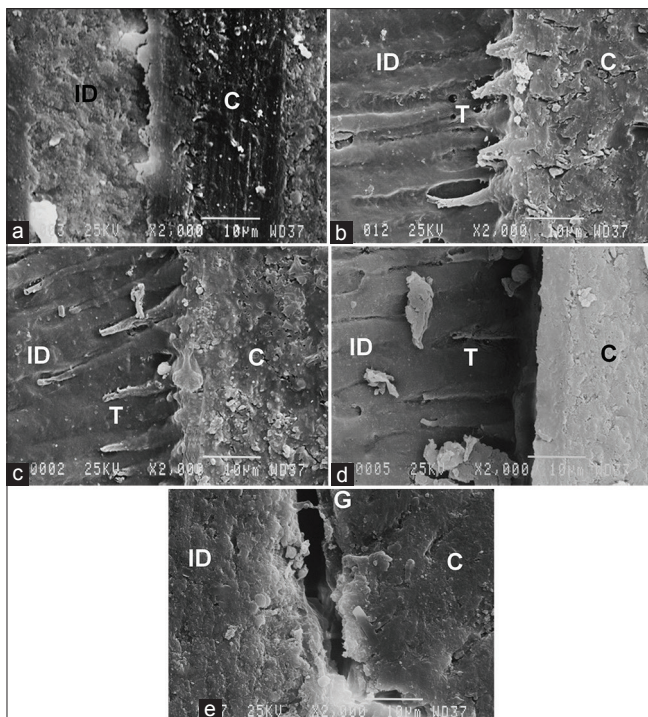


Figure 2: Electron micrographs (bar = 10 µm) of the interface of composite (right) and intertubular dentin (left) in the case of different composites ($\times 2000$): (a) Solvent-free experimental bonding agent; (b) experimental bonding agent containing 20% solvent; (c) 31% solvent; (d) 39% solvent; and (e) 46% solvent. C: Composite; ID: Intertubular dentin; T: Resin tags; G: Gap between composite and dentin indicating no resin penetration.

more.^[34] This issue is probably attributed to the residual solvent that was not completely eliminated and interfered with adequate polymerization and resulted in porosities in the adhesive layer. In a study by Cho and Dickens,^[40] further increase in the solvent up to 67 wt % decreased the bond strength to 38 MPa. Incorporation of lower amounts of solvent in the adhesive resulted in more uniform bonding of the adhesive. By increasing the solvent content, the frequency of cracks increased within the adhesive layer, which subsequently decreased the bond strength.^[40] Adhesive layers thinner than 25 µm are not adequately polymerized due to the inhibitory effect of oxygen. Thus, there is a possibility that the poor quality of bond in groups with a thin layer of adhesive (67 wt %) does not resist polymerization shrinkage stresses; therefore, gaps form at the adhesive–composite interface.^[40] Solvent evaporation can also cause defects such as porosities, phase separation, and uneven shrinkage of the adhesive layer. Such defects may decrease the fracture strength since they can initiate crack formation. In brief, polymerization shrinkage stresses may concentrate

in these defects and lead to early cohesive failure of the low-strength adhesive layer.^[10-17,40,41] Zheng *et al.*^[41] reported that higher thickness of adhesive layer increases the microtensile bond strength probably because of more uniform distribution of stress and resistance of the adhesive layer to the applied load.^[41] However, in bonding agents containing ethanol/water, increased thickness of adhesive layer decreases the microtensile bond strength. This may be due to the entrapment of solvent and interference with complete polymerization.^[10-17,40-42] Hence, solvents must be completely eliminated from the adhesive before composite resin polymerization.^[15,16] Solvents are eliminated by air spraying which makes the adhesive layer thinner, reducing the risk of cohesive failure in this layer.^[43] Moosavi *et al.*^[44] reported a decrease in the microleakage following a more effective solvent evaporation, but the decrease depended on the type and generation of adhesive. However, they did not find significant differences in bond strengths of bonding agents to dentin, after different durations of solvent evaporation^[42] which could be due to methodological differences. Compared to acetone, ethanol solvent is more likely to remain in the adhesive due to its lower evaporation rate as compared to acetone.^[45,46] Due to the high boiling point and forming hydrogen bonds with the residual water in dentin, ethanol is less volatile than acetone; however, it has a higher evaporation rate compared to water. Thus, in the current study, ethanol was chosen as the solvent.^[34,46]

Based on the results of SEM analysis in the current study, in the group containing 0% solvent, the quality of hybrid layer was very poor and no resin penetration or resin tags were seen. A thick adhesive layer was noted in this group. These factors are responsible for the low bond strength in this group. By increasing the amount of solvent to 20% and 31%, the quality of the hybrid layer improved in terms of resin penetration and length or number of resin tags; resin penetration depth and the number and length of resin tags were the highest in the group containing 31% ethanol. Our results in this respect were similar to those of Wang *et al.*,^[10] who evaluated the effects of different concentrations of ethanol solvent on the quality of the hybrid layer and stated that 30% concentration of the solvent was the most efficient among 10%, 30%, and 50% concentrations.^[10] In 46% concentration of solvent, no resin penetration or resin tags were seen in this study. Instead, separation of adhesive layer from dentin was noticed in most

parts; this explains the lower bond strength in this group even in comparison with the 0% ethanol group. This result is in line with that of Wang *et al.*^[10] In their study, 50% concentration of ethanol significantly decreased the penetration of monomers.^[10] They explained this reduction to be due to component dilution or severe chemical dehydration causing the collapse of nanochannels between collagen fibrils.^[10] The 50% concentration of ethanol was found not to be a suitable concentration due to severe evaporation of solvent and forming a porous hybrid layer.^[10] Thus, if the concentration of solvent exceeds the optimal threshold, it prevents the polymerization of adhesive monomers and increases the percentage of unreacted monomers due to the oxygen inhibition effect, which *per se* decreases the mechanical properties of the bonding agent.^[10] Solvent-free adhesives form a thicker layer that provides better thermal protection and decreases the shear stresses. However, if this layer becomes too thick, it may compromise the bond strength.^[47]

Use of nanostructured materials is becoming increasingly popular in dental materials science.^[30,33] Considering the fact that incorporation of different nano-scaled materials may improve the properties of dental adhesives,^[48] in the current study, 10 wt% POSS was added to the experimental dental adhesive and it might have influenced the bond strength results. Sadat-Shojae *et al.*^[49] demonstrated that incorporation of 0.5 and 0.2 wt% nano-hydroxyapatite fillers improved the microshear bond strength and mechanical properties of the bonding agent.^[49] Different concentrations of nanoclay fillers were added to an acrylate-based experimental dental adhesive, and it was revealed that 0.5 wt% poly (methacrylic acid)-grafted-nanoclay significantly increased the microshear bond strength of composite to dentin.^[50]

This study was limited by some factors. It was better to include a larger sample with more concentrations of ethanol, to find a more accurate optimum for ethanol solvent. Furthermore, it was better to use different percentages of POSS as well; however, due to limited budget and time of this study, we preferred to find the optimal percentage of POSS in a separate study.^[18] However, the number of specimens per group was sufficient and even above the ISO TR11405 standard.^[51] In addition, the use of microtensile testing was advantageous in terms of improved stress distribution, possibility of measuring regional differences in bond strength at the dentin-resin

interface, prevention of cohesive fracture of dentin, and ability to measure higher bond strength values, which are attributed to the decreased concentration of defects and the small bonding surface area.^[52] Both shear and microtensile forces can be used to analyze bond strength of composites. Shear tests need simpler sample preparation procedures, but microtensile method is more reliable because of rather smaller adhesive interfaces.^[51,53-55] As another limitation, because the solvent of experimental bonding agents was ethanol, a more suitable commercial bonding agent with ethanol solvent should have been included in this study instead of the current material that was acetone-based. Moreover, compatibility between the adhesive and composite from the same manufacturer is recommended to achieve effective bonding to dentin.[†] Future studies should verify lack of cytotoxicity of this new adhesive on pulp cells. Other important factors in applying adhesives are adequate mechanical properties, resistance to solubility, and adequate copolymerization with composite resin. Longer durations of aging and use of thermocycling would improve the reliability of findings.

CONCLUSION

Incorporation of 31 wt% ethanol as solvent to an experimental POSS-containing bonding agent increased the bond strength to levels beyond those of the commercial bonding agent and resulted in more distinct morphology of hybrid layer.

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Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or non-financial in this article.

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