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INFLUENCE OF RESIN REMOVER ON MICRO-TENSILE BOND STRENGTH OF RESIN COMPOSITE TO CORONAL DENTIN

Inas Abdel Moneum Elghandour*

ABSTRACT

Aim: This invitro study assessed the effect of resin remover used for removal of resin based endodontic fillings on the microtensile bond strength of resin composite to coronal dentin

Materials and methods; Freshly extracted sound human molars, that were extracted for periodontal reasons not correlated with this study, were disinfected and prepared for microtensile bond strength testing. The occlusal surfaces were ground to expose coronal dentin. In half of the specimens, dentin was treated with resin remover for eight minutes. In the other half, dentin was not treated to serve as control. Tetric N ceram composite blocks were bonded to occlusal surfaces of all specimens using total etch adhesive system; Tetric N bond universal. After storage for 24 hours, specimens were sectioned into beams to provide a total of 40 beams; 20 beams for non-treated dentin group and the other 20 beam for dentin group treated with resin remover. Beam were tested for microtensile bond strength using universal testing machine and failure mode was recorded. The tooth/restoration interface was examined using scanning electron microscope.

Results: resin composite bonded to dentin pretreated using resin remover revealed a statistically higher bond strength than that bonded to untreated dentin. Mixed adhesive cohesive failures and cohesive failure in composite was predominant in resin remover dentin pre-treated groups compared to control group that revealed mostly adhesive failure or cohesive failures in dentin. Scanning Electron microexamination showed more penetration of resin tags and more obturation of dentinal tubules in specimens treated with resin remover.

Conclusion; usage of resin remover showed promising results considering bonding to coronal dentin which requires further research in this field.

* Associate Professor, Conservative Dentistry Department, Faculty of Dentistry, Cairo University

INTRODUCTION

Successful restoration of vital or endodontically treated teeth requires efficient and durable adhesion of restorative materials to the tooth. Bonding to enamel and dentin is challenging as they are different structures with different compositions, water content and chemical and physical characteristics[1]. During endodontic retreatment, solvents are often utilized to facilitate removal of endodontic filling. These solvents included chloroform, xylene, orange oil, eucalyptol, terebinthina oil and Xylol [2,3,4]. Other solvents were used to remove root canal sealer as ethyl acetate, ethanol, acetone, isopropyl alcohol, chlorhexidine and EndoSolv R[3,4]. Resin removers were utilized to facilitate removal of resin based endodontic filling and sealants. During root canal retreatment, the coronal enamel and dentin are subjected to these solvents. In addition, trials to use resin solvents to aid in the removal of failed resin composite restoration have been attempted. It was found that resin solvents have the potential to reduce the flexure strength of resin composite[5]. In another study, it was found that resin solvent stained resin composite which might help in its identification from tooth structure[6]. So, a question arises, would resin remover affect the bonding of subsequently applied resin composite restoration?

In the literature, contradictory findings were found. Solvents was suggested to reduce bond strength due to either chemical modification of tooth structure [7] or if they are oil based, due to physical interference with penetration of subsequent adhesive agent[8]. Alternatively, they might improve it through facilitation of removal of remnants of previous restoration based on their chemical composition.[9] Many studies evaluated effect of endodontic solvents on bonding to root canal dentin. For solvents of endodontic filling, Nasim et al[8] found that xylene and Endosolv E reduced the bond strength of resin-based sealers to radicular dentin. Topçuoğlu et al [10] found that some solvents as chloroform reduced the bond strength of root canal sealer to root canal dentin, while other solvents such as orange oil and Eucalyptol did not influence the bond to root dentin. Guedes et al [11] found that eucalyptol reduced the bond strength of fiber glass post to root dentin. On the other hand, xylene and orange oil did not influence the bond strength. Previous researchers studied the effect of solvents not only on root canal dentin but also on pulp chamber dentin[12]. Considering studies that evaluated bonding of resin solvents to coronal dentin, limited data was found.

For coronal dentin, many researchers investigated the effect of root canal irrigating solutions or antimicrobial agents as chlorhexidine or sodium hypochlorite on bond strength to coronal dentin[13.14]. Other investigators studied the effect of cleaning methods of sealers on bond strength of subsequent composites. They found that cleaning of residual sealer with ethanol improved the bond strength of adhesive agents to coronal dentin [9,15]. The aim of this study is to investigate the effect of resin remover on bond strength of resin composite to coronal dentin. The null hypothesis assumed that resin remover would not affect the subsequent resin/coronal dentin micro- tensile bond strength

MATERIALS AND METHODS

Teeth selection and mounting: Ten recently extracted, sound human molars were collected for the study. They were extracted for periodontal reasons not correlated to the present study. They were cleaned and stored in distilled water at room temperature for no more than 2 months before sample preparation. They were embedded with the occlusal surface facing upward using auto polymerized acrylic resin [acrostone acrylic material, acrostone dental and medical supplies, Egypt] in cylindrical Teflon molds. The mold had dimensions of (15-mm diameter and 40-mm height), with a corresponding metal ring with two opposing screws at its top (Figure 1). The screws were used to hold the tooth in place in a centralized position, parallel to the long axis of the mold, during the setting of acrylic resin. Teeth fixed in acrylic resin blocks were then mounted in an automated diamond saw (Isomet 4000, Buehler Ltd., Lake Bluff, IL, USA) (Figure 2), which was used for all sectioning procedures in this study. Occlusal surfaces were flattened to the level of the dentino-enamel junction (Figure 3) under copious water coolant (Cool 2 water-soluble anticorrosive cooling lubricant, Buehler Ltd., Lake Bluff, IL, USA), with a concentration of 1:30, lubricant: water. Specimens were randomly allocated to two groups. In first group; specimens were not treated by resin remover. In second groups; specimens were treated with resin remover [Produits Dentaires SA, 1800 Vevey, Switzerland] for 8 minutes. The quantity of the resin remover is adjusted to cover the whole surface of exposed dentin.

Specimens' preparation: Application of bonding agent and resin composite was carried out following the directions of manufacturer. A two-step etch and rinse adhesive system; Tetric N bond universal [ivoclar, vivadent AG, Schaan/ Liechtenstein] was used. The dentin surfaces were etched with 37% phosphoric acid for 15 seconds. Dentin was then rinsed with water for 10 seconds and briefly air dried for 2 seconds using oil free air. Adhesive was applied using a brush and agitated at dentin surfaces for 20 seconds. Excess adhesive was removed by gentle air stream. This is followed by light curing for 10 seconds using RTA mini–S LED Light curing unit [Humayun Dental Supplies, China]. Using a transparent hollow cylindrical mould of dimensions [7 mm diameter and 4 mm height], resin composite Tetric N ceram [ivoclar, Vivadent AG, Schaan/Liechtenstein] was applied in the mold onto dentin surface. Two increments [2 mm thick] were used to fill the mould. Light curing was made using blue-light-emitting diode RTA mini-S for 40 seconds. Specimens were removed from the mould and stored in distilled water at room temperature for 24 hours. The procedures were performed by single calibrated operator.

Beam Preparation: The objective of longitudinal sectioning of restored teeth was to obtain composite-dentin beams of (0.9 mm x 0.9 mm) in area. Each beam was composed of composite and dentin with adhesive at the interface. In order to facilitate identification of beam location in restored cavities, hence the type of cavity-bottom dentine; whether peripheral or central, the surfaces of composite restorations were painted with permanent ink so that the end of central beams would have a different color from peripheral ones.

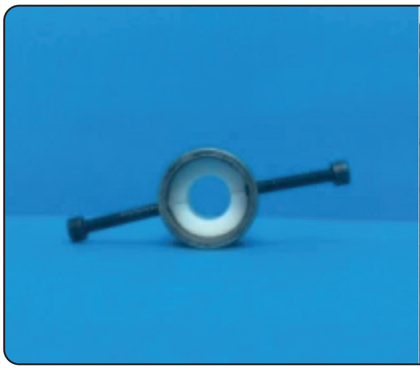

Fig. (1): Teflon mold with corresponding metal ring and paralleling screws (Top view)

Fig. (2): Isomet 4000

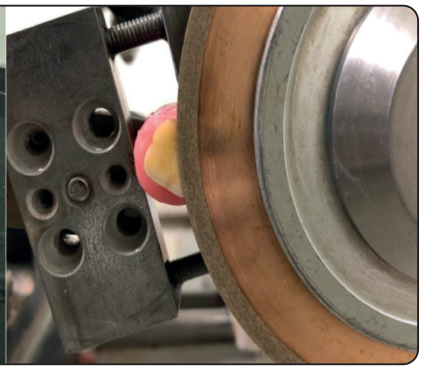

Fig. (3): flattening of occlusal surface

For the longitudinal sectioning to be perpendicular to the flat occlusal surface of restored teeth, a specially designed gripping attachment was used to hold acrylic blocks with mounted teeth firm in place, parallel to the sectioning direction, thus maintaining the perpendicular relation between the cutting disc and the occlusal surface. The L-shaped attachment (figure 1) is composed of a cylindrical metal ring (16-mm in diameter, 3-mm height, 2-m thickness) soldered at its base to a metal rod, which is used to mount the attachment into the diamond saw machine. Two axial grooves, perpendicular to each other, were made on top surface of metal ring to facilitate accurate positioning and rotation of acrylic blocks inside the gripping attachment. The final components are two 5-cm long screws in-line with each other in order to fix acrylic blocks in place with minimal movement during sectioning. After mounting in the gripping attachment, restored teeth were serially sectioned, using a 0.3-mm thick diamond coated disc (Buehler, IL, USA), at 2050 rpm; 8.8 mm/min feeding rate; under copious coolant. Serial sectioning was done in bucco-lingual direction then rotated 90° clockwise and sectioned in mesio-distal A final horizontal cut at level of cemento-enamel junction was done to obtain beams (Figure 4). Resultant beams were 0.9±0.1 mm in thickness and 5.5±1 mm in length (Figure 5). A digital caliper (Mitutoyo, Tokyo, Japan) was used to check the thickness and length of all beams. Each beam was stored in distilled water at room temperature in a tight-seal plastic cone labeled according to subgroup and tooth of origin

Micro-tensile bond strength measurements: For each tested subgroup, 20 beams were tested. Geraldeli's jig[2] was used to mount beams onto the universal testing machine (*Instron, MA, USA)*. Each beam was aligned in the central groove of the jig and glued in place by its ends using cyanoacrylate-based glue (Zapit, DVA Inc, USA) (Figure 6). Zapit accelerator was used to accelerate hardening of the glue. The jig was in turn mounted into the universal testing machine *(Instron, MA, USA)* with a load cell of 500 N. Tensile load was applied, at a cross-head speed of 0.5 mm/min, until bonding failure of the specimen occurred. Bond strength was calculated in MegaPascal *(Bluehill Lite software, Instron, MA, USA)*. Specimen fragments were carefully removed from the jig with a scalpel and stored in their corresponding labelled plastic cones until examination of failure mode.

Statistical Analysis: Data management and statistical analysis were performed using the Statistical Package for Social Sciences (SPSS) version 24. Bond strength data were summarized using means and standard deviations Data were explored for normality using Kolmogrov-Smirnov

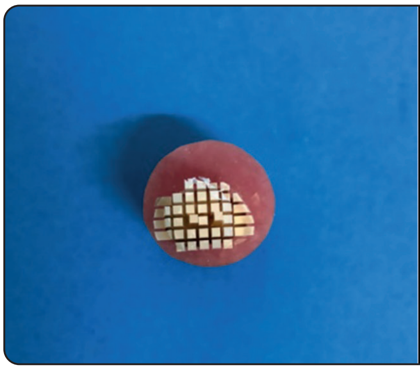

Fig. (4): Sectioned specimen

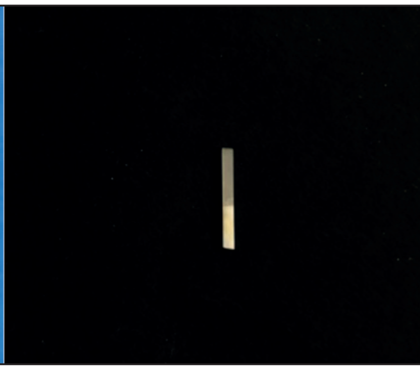

Fig. (5): Beam for microtensile strength testing

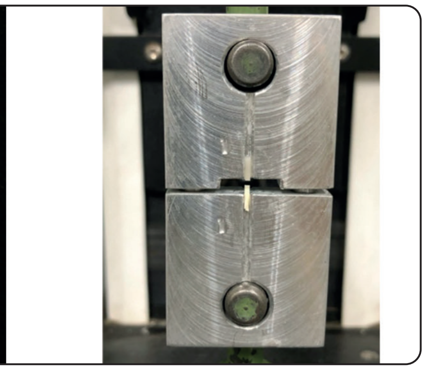

Fig. (6): Microtensile strength testing

test and Shapiro-Wilk test. Comparison between the 2 groups was done using the independent t-test at p-values of < 0.05 significant level. The type of failure was examined after debonding under magnification and at right-angle position. The type of failure was categorized as follows:(1) adhesive failure, when fractures were recorded at tooth adhesive interface; (2) cohesive failure in the resin composite; (3) cohesive failure in dentin; and (4) mixed cohesive adhesive failure.

Electron microscope Examination:

A specimen selected randomly from each group was used for Scanning Electron micro examination. 37% phosphoric acid was used for 5 seconds to clean the tooth restoration interface followed by rinsing with water. Specimens were briefly dried and mounted on aluminum stubs using a double-sided adhesive tape. They were sputter coated with gold and examined using high resolution scanning electron microscope [SEM Quanta FEG 250 with field emission gun, FEI Company – Netherlands] at an accelerated voltage of 20 kv. Pictures were captured at a magnification of 1500x.

RESULTS

Statistical analysis of data showed that the microtensile bond strength of resin composite bonded to dentin treated with resin remover was higher than that of resin composite bonded to non-treated dentin surfaces. The difference was statistically significant at p value of 0.046 [Table 1]. Examination of fractured sites revealed that for resin remover treated dentin group, most of the fractures were either cohesive in composite [40%] or mixed adhesive cohesive fracture [40%]. The least fracture mode recorded was 10% cohesive in dentin and 10% adhesive failue. Meanwhile, for non-treated dentin group, most of the fractures were either adhesive [30%] or cohesive in dentin [30%]. The remaining failures in non treated dentin group were 20% mixed cohesive- adhesive and 20% cohesive in composite. However, all types of fractures were revealed in both groups.

TABLE (1): Microtensile bond strength values [mean and SD] of composite resin bonded to dentin with or without resin remover prior treatment

	Treated dentin(n=20)		NonTreated Dentin(n=20)		
	Mean	**SD**	**Mean**	**SD**	**p value**
Maximum Load(N)	28.8	9.3	22.8	9.1	0.046

P<0.05 is statistically significant, SD: standard deviation, analysis done by independent t test

Scanning electron microscopic observations:

The ultrastructural observations of the resin composite/dentine interfaces are illustrated in the scanning electron micrographs shown in (Figures 7a and 7b, and figures 8a and 8b). In case of pretreatment of dentin with resin remover, gap free interface was revealed as shown in figure 7a and 7b. True hybridization was evident. Long slender resin tags were shown penetrating dentinal tubules. The hybrid layer was thicker for treated dentin group compared to control group. Meanwhile, for the control group, true hybridization was evident as shown in figures 8a and 8b. However, the resin tags were shorter, more funnel shaped with partial obturation of the dentinal tubules and not penetrating so long compared to those for dentin pretreated with resin remover. Open dentinal tubules without resin tags penetration were revealed. In both groups, no voids were shown at adhesive dentin interface.

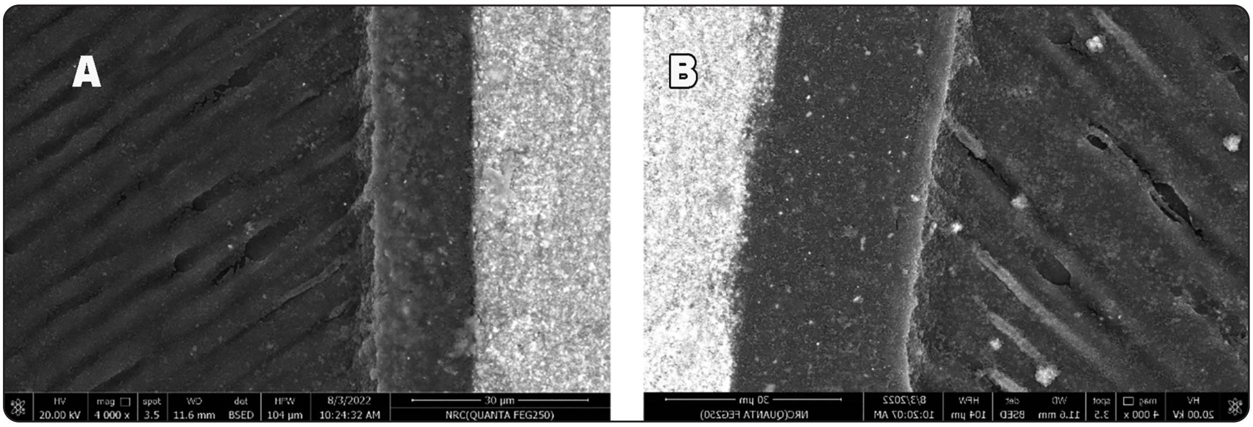

Fig. (7) a and b. Tooth restoration interface for dentin pretreated with resin remover at x 1500 magnification

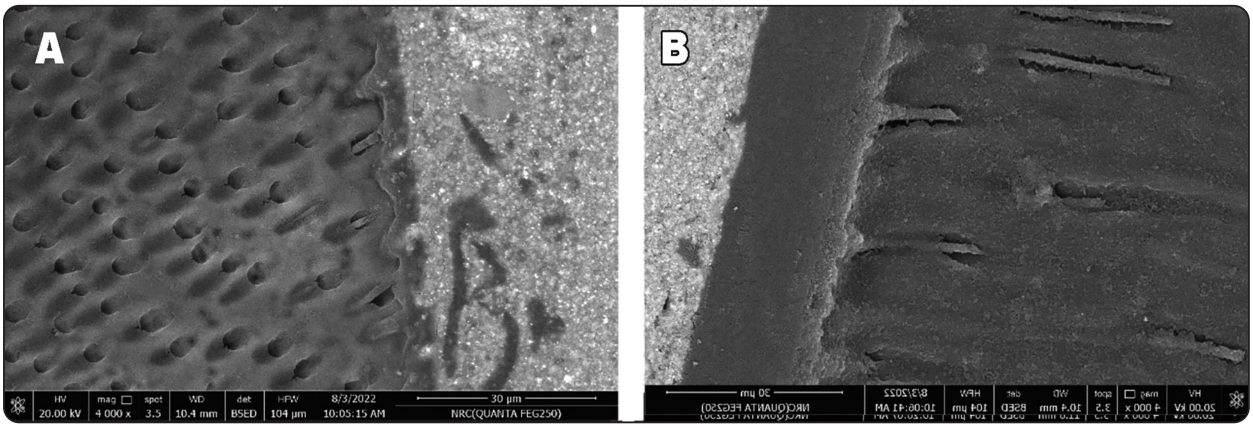

Fig. (8) and b; tooth restoration interface for non treated dentin at x 1500 magnification

DISCUSSION

This study evaluated the microtensile bond strength of resin composite bonded to coronal dentin treated with resin solvents that are used in removal of resin based endodontic fillings. Microtensile bond strength was measured due to general agreement of most authors for its importance to evaluate the bonding effectiveness[16]. It allows improved control over regional differences and better distribution of stress at the tooth material interface during testing. It also helps in conservation of number of teeth used to undergo testing[17]. In the present study, universal adhesive was used in etch and rinse form. This was selected to aid in removal of remnants of resin solvent from dentin. This was in accordance with Chen et al, 2022 [18].

The null hypothesis was rejected as it was found that dentin treatment with resin remover enhanced bonding to resin composite restorative material. Bonding between restorative materials and tooth structure must mimic connection between enamel and dentin. Adhesive materials should be in intimate contact with adherend for chemical or micromechanical bonding to occur. Wetting ability of the adhesive is important factor for this process. This means that its surface tension should be lower than surface energy of adherend. Also, lower contact angle of adhesive with adherend ensures good wettability. This mean that proper bonding depends on physiochemical characteristics of tooth structure and restorative materials[1]. The findings of the present study suggests that resin remover provided a dentine surface that was more prone to bonding agent penetration. Treatment with resin remover or solvent exhibit thicker hybrid layer which indicated that treating dentin with resin solvent permit a larger area of resinous interdiffusion and greater infiltration of the bonding agent into the dentin collagen fibrils. This was in accordance with Manzoli et al, 2022[19].

The resin remover is oil based, and the universal adhesive was used in etch and rinse mode. Tetric N-Bond Universal is ethanol based adhesive system. It has pH of 3.52 and considered as ultra-mild etchant. The lower bond strength to untreated dentin might be due to excessive etching by etchant and acidic monomer of universal adhesive which was minimized by presence of remnant of resin solvent in treated dentin group. This was in accordance with Chen et al, 2022[18]. In demineralized dentin, ethanol could substitute water, fill in spaces between collagen fibers and increase the collagen matrix hydrophobicity in addition to being a solvent to most hydrophobic resin monomers. Accordingly, the penetration of such monomers into dentin collagen fibers and dentinal tubules is improved aiding the infiltration of hydrophobic resin to form a hybrid layer that is dense and homogenous for superior bonding[20]. MORAIS et al[20] mentioned that ethanol is a polar solvent that solubilizes apolar materials as resins. This led to less residues on dentin and hence did not influence bond strength. Nasim et al[8] found that affection of bond strength by resin solvents depend on type of sealer used where the bond strength the epoxy based sealer to dentin was more reduced than that to methacrylate based sealer. Tetric N bond is a methacrylate based bonding agent. So, bonding was not negatively affected by treatment with resin remover.

The finding of the present study was contradictory to several researchers[9,10,11] who found that solvents either reduced or did not affect bonding to dentin. Roberts et al[3] found that usage of EndoSolv R to remove remnants of Ah Plus filled root canal results in bond strength of adhesive resin to coronal dentin equivalent to controls where solvent was not used. Palhais et al [21] found that xylol and orange oil did not reduce the bond strength of resin based sealer to root dentin. Meanwhile, eucalyptol was found to reduce bond strength compared to retreatments without using solvent. Carvalho et al[22] found that endodontic irrigation protocols [5% NaOCl+ 17% EDTA or 2% chlorhexidine followed by saline and 17% EDTA] did not influence the bond strength of self-etch or total etch adhesive systems to coronal dentin or enamel. However, their studies examined different solvents than that used in the present study and bonding was tested to radicular or pulp chamber dentin not coronal dentin. In addition, differences in amount of solvent used ,the time of contact between dentin and the solvent and different methodologies in the present and previous studies also might account for different results. [23]

Limitation of the study

It should be noted that no previous restoration was removed but virgin dentin is used which is considered a limitation of this study. Also, the teeth were non carious. Caries affected dentin is characterized by occlusion of dentinal tubules which might affect bond strength[24]. No thermal or mechanical cycling was made which might further affect bond strength which necessitate recommendation for further investigation in this field. Also, bond strength testing should not be relied on solely to predict the clinical behavior of an adhesive protocol. So other laboratory testing is needed to fully explore the adhesive technique[24]

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