

In Vitro Evaluation of Shear Bond Strength of Nanocomposites to Dentin

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ABSTRACT

Aims: To compare the shear bond strength of nanocomposites to dentin using three different types of adhesive systems; and to test few specimens under Scanning Electron Microscope (SEM) for analysing whether the bond failure is adhesive or cohesive.

Materials and Methods: Sixty human premolar teeth were selected and were randomly grouped, with 20 specimens in each group: group 1 - fluoride releasing dentin bonding agent; group 2 - antibacterial containing dentin bonding agent; and group 3 - one step conventional self etch adhesive. Each group was treated with its respective bonding agents, composite resin build up was done, and shear bond strengths were tested using Instron Universal testing machine. Few of the specimens were tested under SEM.

Results: The results were statistically analysed using One-way ANOVA and paired t-test. It was observed that group 3 has the highest shear bond strength followed by group 2, and then group 1. Adhesive failures and mixed failures were most frequent types of failures as seen under SEM.

Conclusion: Addition of antimicrobial agent decreases the bond strength of dentin bonding agent and addition of fluoride further decreases the bond strength. From SEM results it can be concluded that the zone of failure could not be defined and also that the failure mode was independent of the dentin bonding agent used.

Keywords: Antibacterial, Dentin bonding agents, Fluorides, Nanocomposites, Self etch adhesives, Shear bond strength, Total etch adhesives

INTRODUCTION

Adhesive dentistry is a rapidly evolving discipline. For many years, the dental profession has strived to achieve good adhesion of resin composite to tooth substrate [1]. In 1955, Bunocore introduced the concept of acid etching of the enamel which later on gave way to total etch techniques [2]. The total etch adhesives provide good bond strength yet newer concepts of self etching system have been developed and have proven to be good clinically [3]. Self etching primers combine both dentin conditioning and bonding in a single step [4].

The introduction of antibacterial properties into the bonding agents is a new concept. For dentin bonding agents, either fluoride can be incorporated in them [5], or monomers by themselves may produce antibacterial effects. Methacryloyldodecylpyridiniumbromide (MDPB), a new monomer, shows antibacterial activity against bacterial growth and plaque accumulation [6].

The replacement of composite resin restorations due to secondary caries at restoration-tooth interface is still one of the greatest problems [7]. The decreased incidence of secondary caries along the margins of silicate restorations led to the development of fluoride releasing composite resins [8]. However, the bonding agent applied prior to the placement of resin may block the passing of fluoride to the exposed dentin. This led to the introduction of fluoridated bonding agents [9].

Thus, with this background, the present study was undertaken to compare the shear bond strength of nanocomposites to dentin using fluoride releasing 7th generation dentin bonding agent, antibacterial (MDPB) containing dentin bonding agent, and conventional one step self etch system; to test whether these additional agents have any effect on the shear bond strength of these adhesives; and to test some of the specimens under Scanning Electron Microscope (SEM) for analysing whether the bond failure is adhesive or cohesive. The present study assesses the shear bond strength as it has been

proven to be useful as a screening tool to help understand and predict the clinical behaviour of adhesives [10].

MATERIALS AND METHODS

The study was conducted in the Department of Conservative Dentistry and Endodontics, Krishnadevaraya College of Dental Sciences & Hospital, Bangalore, Karnataka, India. The study was started in August 2012 and was conducted over a time period of eight months. The institutional ethical committee approved this study.

Study sample, inclusion and exclusion criteria: Sixty freshly extracted human pre-molars were collected. Teeth extracted within six month period were included as study sample. Teeth with caries, fracture or cracks, teeth with anatomical variations, teeth with pre existing restorations, and teeth affected with flourosis were not included in the present study.

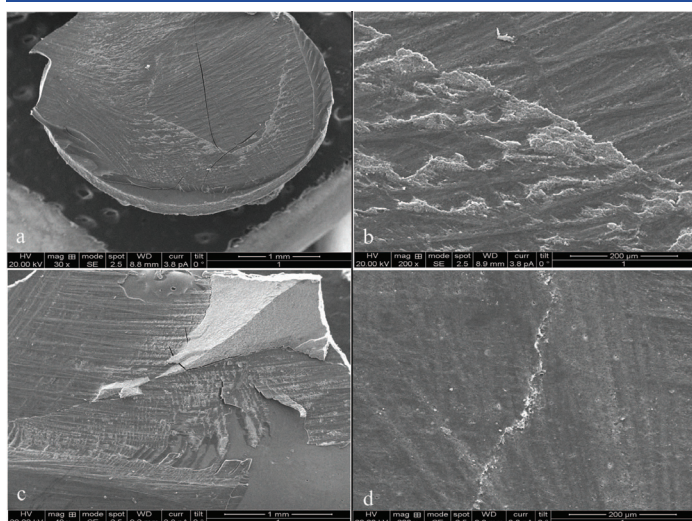
Preparation and grouping of the specimens for shear bond strength: Based on the dentin bonding agent used teeth were divided into three groups of 20 teeth each: group 1 - fluoride releasing 7th generation dentin bonding agent (Bond Force Tokuyama Dental Corp); group 2 - antibacterial (MDPB) containing dentin bonding agent (Clearfil Protect Bond- Kuraray); and group 3 - one step conventional self etch adhesive (Xeno-V Dentsply). Teeth were mounted in stainless steel holders with the help of self cure acrylic resin (DPI) to embed the root portion. These stainless steel holders were colour coded according to the groups i.e. red colour for group 1, yellow colour for group 2, and green colour for group 3. The occlusal surfaces of teeth were grounded with the help of a diamond disk (DFS Germany) mounted on a straight micromotor hand piece (NSK Japan) to prepare flat surfaces at a depth of 1.5 mm from the cuspal tip of the tooth. Bonding agents were applied to all the specimens as per manufacturer's instructions and then light cured accordingly. Filtek Z350 XT (3M ESPE) composite was

	N	Mean	SD	Min	Max	t-value	p-value
Group 1	20	18.280	1.1696	16.6	20.2	806.331	<0.001
Group 2	20	22.820	1.4185	19.9	25.6		
Group 3	20	33.015	.9258	30.8	34.6		

[Table/Fig-1]: The results averaged (mean + standard deviation) for each group (Shear bond strength values in MPa)

	N	Mean	SD	Mean Difference	p-value
Group 1	20	18.280	1.1696	-4.5400	<0.001
Group 2	20	22.820	1.4185		
Group 1	20	18.280	1.1696	-14.7350	<0.001
Group 3	20	33.015	.9258		
Group 2	20	22.820	1.4185	-10.1950	<0.001
Group 3	20	33.015	.9258		

[Table/Fig-2]: Comparison of the results between the three Groups (Shear bond strength values in MPa)



[Table/Fig-3]: a) SEM picture of adhesive failure at 30x, b) SEM picture of adhesive failure at 200x, c) SEM picture of mixed failure at 40x, d) SEM picture of cohesive failure at 100x

then placed in 2mm increment, using a thermoforming sheet which was indented in the shape of a circle producing discs of composites measuring 2 mm in diameter and cured for 60 sec on all the 60 specimens. The outer surface of the composite pellet was marked with the help of a permanent marker to identify the surface during scanning electron microscopic study of the sheared pellet.

Shear bond strength analysis: All the specimens were then transferred to the Instron universal machine individually and subjected to shear bond strength analysis at cross head speed of 1.0 mm/min. The values obtained were calculated in Mega Pascal (MPa) peak load at failure divided by the specimen surface area.

SEM analysis: Few of the specimens were tested under SEM for analysing whether the bond failure is adhesive i.e. in between the bonding agent & the dentin or in between the bonding agent and composite, or it is cohesive i.e. in between the bonding agent itself or within the composite.

RESULTS

The One-way ANOVA and paired t-test were used for statistical analysis. The results were averaged for each group and are presented in [Table/Fig-1]. The results calculated using ANOVA showed a statistical difference between the groups with respect to mean scores (<0.001); group 1 having the least mean score of 18.280 MPa compared to the group 2 (22.820 MPa) and group 3 with the highest mean score of 33.015 MPa.

Group 1 versus Group 2

The comparison of the results between Group 1 and Group 2 showed a statistically significant difference in the shear bond strength to dentin ($p < 0.001$) which leads to the inference that antibacterial

releasing bonding agent has comparatively high bond strength to dentin than fluoride releasing dentin bonding agent [Table/Fig-2].

Group 1 versus Group 3: The comparison of the results between Group 1 and Group 3 showed a statistically significant difference in the shear bond strength to dentin ($p < 0.001$) which leads to the inference that Xeno V- bonding agent has comparatively high bond strength to dentin than fluoride releasing dentin bonding agent [Table/Fig-2].

Group 2 versus Group 3: The comparison of the results between Group 2 and Group 3 showed a statistically significant difference in the shear bond strength to dentin ($p < 0.001$) which leads to the inference that Xeno V- bonding agent has comparatively higher shear bond strength to dentin than antibacterial releasing dentin bonding agent [Table/Fig-2].

SEM RESULTS

The sheared samples obtained after the shear bond strength testing were sent for the scanning electron microscopic analysis to check the mode of failure i.e. cohesive failure in dentin, cohesive failure in composite resin, adhesive failure or mixed failure. All the four types of failures were seen in variable frequencies but most frequent were adhesive and mixed failures [Table/Fig-3a-d].

DISCUSSION

Modern dental practice has an increasing demand for esthetic restorations that lead to the extensive use of adhesive dental materials like composites and bonding agents. The clinical success of composite restoration depends on the adhesive system [11]. The adhesion of dental materials to dentin has been extensively investigated in the last decades in order to make it effective and durable. Different mechanical tests have been proposed to assess the bonding performance of restorative materials. Although it suffers criticism, shear testing has been widely used to evaluate the bonding ability of adhesive materials to dental structure [12]. Shear bond strength test is a simple evaluation procedure used to test the adhesion of dental adhesives [13]. Thus, in our study shear bond strength testing was done with a universal testing machine, Instron, which is conventionally popular for evaluating the adhesive ability of adhesive/restorative materials [14].

All the three adhesive systems used in the present study achieved the optimal bond strength values for dentin. However, the self etch adhesive system showed better bond strength as compared to antibacterial dentin bonding agent; antibacterial dentin bonding agent showed better bonding than fluoride releasing dentin bonding agent. The results of our study are in accordance with the studies done by Kiremitci et al., who concluded that self etching adhesive systems produced higher bond strength than conventional total etch systems, especially the all-in-one system, which produced the highest bond strength [15]. On the contrary, Sensi et al., stated that self etch and total etch primer showed comparable dentin bond strength [16].

In the present study, Xeno V, a one-step self etch adhesive demonstrated good bond strength values with dentin. Meerbeek Van B et al., attributed the good bond strength values obtained with Xeno V to it being an intermediate strong self etch adhesive, with an acidic pH of 1.4 [17]. This acidic nature results in better micromechanical interlocking to enamel and dentin, as compared to mild self etch adhesives [18].

Clearfil protect bond, which is antibacterial self etching adhesive, has bond strength comparable with other self etch adhesives, even though it showed lower bond strength when compared with Xeno V. Imazato and Mc Cabe demonstrated that a small improvement in the curing behavior of a Bis-GMA based resin was caused by incorporation of MDPB [19]. Achievement of strong micromechanical bonding depends upon the depth of monomer penetration into demineralized dentin [20]. It is possible that MDPB aids monomer penetration and that this results in greater bond strengths [21].

The incorporation of fluorides in the composition of dentin bonding systems is an attempt to mainly augment the demineralization protective effect of these materials. The three main microorganism-growth inhibitory mechanisms of fluoride are direct binding of F-/HF to enzymes and other bacterial proteins; binding of metal F complexes; and action as a transmembrane proton carrier [22]. However, fluoride release from resinous materials has been proven inferior to that from glass-ionomer cements, and results in a compromised physical properties of the material like shear bond strength as demonstrated in this study that the bond force shows the lowest shear bond strength to dentin [23,24]. The pH level of bond force immediately after dispensing is approximately 2.3 which make it mild self etch adhesive resulting in inferior micromechanical bonding to dentin resulting in decreased bond strength.

The high standard deviation and wide ranges obtained in the present study may be attributed to several factors that may influence in vitro bond strength to dentin, such as the type and age of the teeth, the degree of dentin mineralization, the dentin surface being bonded, the type of bond strength test (shear or tensile), the storage media, and the environmental relative humidity in substrates and testing conditions [25].

SEM evaluation of the debonded composite pellets showed all the three types of failures i.e. adhesive, cohesive and mixed failures for all the three groups undertaken in the study. But the most frequent were the adhesive failures followed by the mixed failures for any type of the bonding agent. Thus it can be concluded that the type of the bonding agent used, its composition and applied load does not cause any change in the failure mode of the bond between the resin and the tooth. The type and amount of solvents, the content and percentage of monomers, and diluents in the mixture influence the bond strength. The filler load or percent mass load differs between products according to manufacturers' technology and is not well described in the adhesives' composition. Nevertheless, there is little information about the shrinkage and stiffness of these filled adhesives after polymerization. These are some of the factors that could affect the shear bond strength significantly, but is not listed by the manufacturers' since the final formulation is a proprietary secret [26].

CONCLUSION

From the results of the present study, it can be concluded that addition of antimicrobial agent like MDPB decreases the bond strength of dentin bonding agent and addition of fluoride further decreases the bond strength more than MDPB. SEM evaluation leads to the inference that the zone of failure could not be concluded. Furthermore, this in vitro study needs further in vivo implementation, because this study was conducted using extracted teeth without regarding the pulpal pressure and presence of dentinal fluid under realistic physiological conditions, which may adversely affect dentin bonding. In extracted teeth, the collagen fibrillar network of dentin may collapse and prevent proper resin penetration in dentin. So, long-term clinical studies are required to evaluate the efficacy and durability of these self-etching bonding systems.

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