A COMPARATIVE EVALUATION OF SHEAR BOND STRENGTH OF IMMEDIATELY REPAIRED COMPOSITE RESIN — AN IN-VITRO STUDY

Rakesh Yadav* MDS; Anil Chandra** MDS; A.P.Tikku** MDS; Hemant Kumar Yadav # MDS

* Assistant Professor, Department of Conservative Dentistry and Endodontics, Faculty of Dental Sciences, King George’s Medical University, Uttar Pradesh, Lucknow. ** Professor, Department of Conservative Dentistry and Endodontics, Faculty of Dental Sciences, King George’s Medical University, Uttar Pradesh, Lucknow. # Lecturer, Department of Conservative Dentistry and Endodontics, Sardar Patel Post Graduate Institute of Dental and Medical Sciences, Uttar Pradesh, Lucknow

Address for correspondence: Dr. Rakesh Yadav, Assistant Professor, Department of Conservative Dentistry and Endodontics, Faculty of Dental Sciences, King George’s Medical University, Uttar Pradesh, Lucknow

Email: rakeshanitali@yahoo.in

Abstract: The aim of this study is to “evaluate the shear bond strength of immediately repaired light cured composite resin using different bonding agents”. Forty recently extracted human incisors were mounted on acrylic resin blocks and cavity (5 × 3mm) was prepared at the middle third of the crown. The prepared cavity was etched, rinsed, dried and restored using Single bond adhesive (3M), composite and visible light cure unit. All the samples were then fractured in Instron machine and randomly divided into four groups (n= 10) depending on the type of bonding agent used during repair of fractured restorations: Control Group A - no bonding agent used, Experimental Group B - Single Bond, Experimental Group C- Prime and Bond NT and Experimental Group D - Xeno III. All the specimens were repaired by composite (Z-250) with application of bonding agent used according to groups. The shear bond strength of all specimens was tested using Instron machine. The result of the study showed that the mean shear bond strength of group D was highest followed by the Group B, group C and the Group A the least. The application of newer bonding systems were found to be more effective in increasing the shear bond strength of the repaired composite restorations.

Keywords: composite, instron machine, prime and bond, single bond.

INTRODUCTION

Esthetic dentistry has been one of the most vital parts of the modern civilization. Composite resin offers a conservative, economical, esthetics treatment alternative for the patient. However, the physical properties of resin composite, offer some limitation in strength and longevity, especially in patients with occlusal habits. Most esthetic restorations provide excellent esthetics at placement time; however, esthetic can be compromised by stresses placed on the restorative materials. These stresses can be due to accidental trauma, occlusal forces generation over a period of the time or incisal interferences on the restorations.

When a composite restoration fails as a result of discoloration, microleakage, ditching at the margins, delamination or simply fracture, it needs to be repaired or replaced (1, 2, 3). The total replacement of the restoration is the most common procedure experienced in daily clinical practice (3). However, when large portions of the restorations are completely removed, significant loss of sound dental tissues occurs (4, 5) because it is often difficult to remove a tooth-colored adhesive restoration without removing an integral
part of the tooth. As a consequence, the dental structure is weakened and pulp injury may occur. In this context, composite repair is considered a minimally invasive protocol with the additional advantages that it is a low cost less costly alternative and demands less chair-side time (1, 6).

Various Restorative materials and techniques are currently available that when correctly used, can provide restoration repairs with excellent esthetics and good longevity.

Buonocore (7) first demonstrated that adhesion of acrylic restorative resins to enamel could be increased significantly by etching the enamel surface with phosphoric acid. This technique primarily provided micro-mechanical bonding but also act as a stimulus for research on the surface preparation of dentin (8, 9).

The chemical treatment by acids to provide a modified surface offers an alternative approach for increasing the adhesion between composite and tooth surface. To increase the bond strength of composite, several types of bonding agents are used. The bonding agents form hybrid layer with dentin and its other side copolymerizes with the matrix phase of dental composite producing strong micro mechanical bonding.

Recently, improvement in bond strength was observed when some hydrophilic monomers were applied to dentin (10, 11). Adhesion of composite to enamel and dentin is inhibited because most of the bonding systems do not completely wet the tooth surface. Poor surface wettability also allows the formation of voids at the interface and adhesion is further greatly reduced because of local stress concentration at these discontinuities. Ideally the bonding system should be hydrophilic to displace the water and thereby wet the surface, permitting it to penetrate into the surface irregularities.

Comparing data from the literature seems to be difficult because various adhesive systems and materials under various conditions are tested. Since the mode of action of adhesives is complex and may be different for each of them; so the effect of these factor is not easy to determine, and additional research is needed on this subject. Several bonding agent claiming superior bonding properties are now available. Hence, the purpose of this study is to “evaluate the shear bond strength of immediately repaired light cured composite resin using different bonding agents”.

**MATERIALS AND METHOD**

Preparation of samples: For this study 40 extracted human incisors were collected, washed thoroughly and stored in normal saline. After removing from normal saline, all the teeth were mounted on acrylic resin blocks vertically with the crowns above the blocks. Then, cavity preparation was done at the middle of the crown with a diamond bur with the help of contra angle hand piece and micro motor. The dimension of cavity was 5mm in length and 3mm in width in rectangular form. Residual dentin debris was removed carefully. The prepared cavity was then etched by etchant (37% phosphoric acid) for 15 second followed by thorough rinsing with water to remove all the etchant applied. Surface was then dried using blot – dry technique. Single Bond adhesive (3M) was then applied and cured for 20 second; Z-250 composite resin was then applied and cured with visible light cure unit for 40 second (Figure 1 to 4). All the samples were then fractured in Instron universal testing instrument.

Distribution of samples: All the samples were divided into four groups of 10 teeth in each group depending on the type of bonding agent used during repair of fractured restorations as given below: (Figure 5 to 9)

<table>
<thead>
<tr>
<th>Groups</th>
<th>No. of samples (n)</th>
<th>Bonding Agent</th>
<th>Composition of Bonding Agents</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control Group A</td>
<td>10</td>
<td>No bonding agent used</td>
<td>BisGMA, HEMA, dimethacrylate, polyalkenoic acid copolymer, initiator, water, ethanol</td>
</tr>
<tr>
<td>Experiment 1 Group B</td>
<td>10</td>
<td>Single bond (3M)</td>
<td>PENTA, UDMA + T-resin (cross-linking agent) + D-resin( small hydrophilic molecule), Butylated Hydroxytoluene, 4-ethylidimethylaminobenzoxazol, cetylamine, hydroxypropyl aceton, silica nanofiller</td>
</tr>
<tr>
<td>Experiment 1 Group C</td>
<td>10</td>
<td>prime and bond (Dentsply, Caulk)</td>
<td>HEMA, UDMA, butylated hydroxytoluene, camphorquione, ethyl-4-dimethylaminobenzoxazol, ethanol, water, silica, fluoride complexes ( pyro-EMA, PEM-F)</td>
</tr>
<tr>
<td>Experiment 1 Group D</td>
<td>10</td>
<td>Xeno III (Dentsply)</td>
<td>HEMA, UDMA, butylated hydroxytoluene, camphorquione, ethyl-4-dimethylaminobenzoxazol, ethanol, water, silica, fluoride complexes ( pyro-EMA, PEM-F)</td>
</tr>
</tbody>
</table>
All the samples were grind of fractured restoration by Diamond Stone to roughen the surface then acid etched by 37% phosphoric acid followed by thorough rinsing with water to remove all the etchant applied. The surface was then dried using blot dry technique and repaired by composite resin (Z – 250) with application of bonding agent according to groups except in control group in which no bonding agent was used. The samples were subjected to Instron Universal Testing machine to test shear bond strength.
Testing of the bond strength with Instron Universal Testing Machine:

First the instrument was calibrated with the standard dead load, the block of sample was tightened in the one end of machine and the samples were subjected to the load. The speed of the load was kept at 5mm/min when the restoration broke reading was recorded from the scale.

All the samples were subjected to the Instron in the same manner. The bond strength was obtained by:

\[ \text{Strength} = \frac{\text{force}}{\text{area}} \times \text{Load} \times 9.8 / \text{area} \]

Statistical Analysis:

Data were collected and subjected for statistical analysis using analysis of variance, Student’s t test and level of significant ‘p’.

RESULTS:

The mean of shear bond strength in these groups (Group A, B, C and D) ranged from 13.72 - 18.29 Mpa, 20.92 - 31.36 Mpa, 20.92-29.40 Mpa and 26.13-33.97 Mpa respectively with mean (+ SD) 16.33±1.51 Mpa, 25.48±3.44 Mpa, 24.02±2.89 Mpa and 30.12±2.41 Mpa respectively.

The shear bond strength varied among the groups and was evident highest in Group D followed by Group B, Group C and Group A the least.

The mean shear bond strength in control group A was the least followed by Group C, group B and group D was the most. Pair wise mean comparison by Student ‘t’ test showed that the mean shear bond strength in group B, group C and Group D were significantly higher as compare to group A. When comparison was made within experimental groups, it was found that the mean shear bond strength in group B and group D were higher than in group C. The mean shear bond strength in group B was statistically lower than Group D. Furthermore, the mean shear bond strength in group C was also found to be statistically lower than Group D. However, the mean shear bond strength did not differ between group B and group C i.e. found to be statistically the same (p<0.005). The following sequence of mean shear bond strength was found;

Group D > Group B > Group C > Group A

DISCUSSION:

Early dentin bonding agents were hydrophobic and were bonded directly to the dentin smear layer. Therefore the bond strength was less than 6 Mpa. These bonding systems do not completely wet the tooth surface. Poor surface wettability also allows the formation of voids at the interface and adhesion is further greatly reduced due to the local stress concentration present in these interfacial areas.

Later dentin bonding agents based on hydrophilic monomer such as 2-hydroxy ethyl methacrylate (2-HEMA or HEMA) or pyromellitic dihydride and hydroxyethylmethacrylate (PMDM) to displace the water by penetrating into the porosities of the dentin surfaces, results in its interaction strongly with the tooth structure. This dramatically improved the bond strength to the level of significance (12).

Newer generation bonding agents are now more hydrophilic and use Solvent Systems that allow better wetting of the tooth surface, resulting in high bond strength (13).

In an effort to simplify dentin bonding agents, manufacturers have recently introduced system that can combine the primer and adhesive agent. These systems have generally been referred to as ‘one component’ systems. Most dentin bonding resins are light cured.

In this study, bonding agents used contained both primer and adhesive in a single bottle. Single Bond (3M) consists of BISGMA, HEMA, water ethanol, photo initiator and poly alkenoic acid copolymer. According to the clinical research associates, 3M single bond dental adhesive is a faster bonding agent. The presence of polyalkenoic acid copolymer has an important role in increasing its bond strength.

Prime and Bond NT Dental adhesive has traditional fillers incorporated in the composite or
compomers, which increase the bond strength of the restorative material. This bonding agent has nanofillers with a particle size of about 7 nanometers, 100 times smaller than the fillers in composite resin. These nanofillers therefore are capable to penetrate the typical key-hole etch pattern of enamel as well as the smallest dentinal tubules and even the hybrid layer to achieve nano-retention between tooth structure and the restorative material resulting in best sealing, higher adhesion, best marginal integrity and minimum postoperative sensitivity.

Xeno-III, self-etching adhesive system offered integrated, precise etching/pretreatment of enamel and dentine with water/ethanol as a solvent. Xeno-III contains two unique adhesion promoters:

• The polymerizable PEM-F release fluoride, which is bonding to calcium ions and thus enhancing the decalcification of the enamel/dentin.

• Pyro-EMA, which forms phosphoric acid group after hydrolysis.

This bonding agent was used because:

• High etching efficacy with PH value below-1.

• High adhesive strength.

• In one controllable step.

The introduction of the filled resin materials in 1962 became the basis for restorative material that is generically termed as composite. A composite is a physical mixture of materials. Composite typically involves a dispersed phase of filler particles that are distributed within a continuous phase (matrix phase). A dental composite consist of a mixture of Silicon glass particles with an acrylic monomer that is polymerized during application.

For this study Z-250 composite material was used. Z-250 is a visible light activated, radiopaque, restorative composite designed for use in both anterior and posterior restorations. The filler in filtek Z-250 restorative is zirconia/silica. The inorganic filler loading is 60% by volume with a particle size range of 0.01 to 3.5 microns. It contains Bis-GMA, UDMA and BIS-EMA resins, as matrix.

Choice of restorative materials is dependent on the extent of the fracture and anticipated functional loading. If a large fracture is present, an intermediate layer of hybrid composite should be placed to provide greater opacity and mechanical properties. The intermediate hybrid layer can then be veneered with a more highly polishable microfilled in nonfunctional areas. A microfill, such as Durafill VS, allows the facial contours to be finesse with an artist’s brush moistened with alcohol or bonding agent. The sculpted composite repair requires minimal finishing after polymerization (2).

In a preliminary experiment it was confirmed that blood contamination after primer application remarkably decreased bond strength when the contamination was not rinsed away. If the blood is not rinsed, the adhesion is inhibited severely regardless of the step at which contamination occur (14, 15).

The chemical composition of the adhesive agents and the condition of the tooth structure affect their bond strength (16).

Because of the complex chemical composition of the adhesive and the complex and varying structure of dentin, bond strength is affected by various factors such as blood, saliva, handpiece lubricant and especially liners, bases and temporary materials (14). Since indirect techniques require at least a temporary restoration, provisional materials are used for sealing the cavity or as luting cement. An adverse effect of temporary materials may either be due to changes in wettability and reactivity of the dentin or due to remnants of the material on the surface (17).

Bonding to cut composite resin surfaces was promoted by using unfilled liquid resin as a bonding agent. The greatest strength, over 80% of the strength of the control specimen, was obtained using a thin layer of the unfilled catalyst resin. Strength also developed more rapidly when using...
the catalyst and universal liquid resins. It is important to use a very thin layer of the liquid resin; a thick layer of liquid apparently prevents good adaptation of the new to the old composite resin during placement. The liquid resin probably wets the micropores of the cut surface as well as the filler (18).

Factors, which are most likely to influence the bond between composite and its repair, include the ability to wet the surface of the material, the amount and chemical activity of available resin for bonding and the effect of surface treatment prior to repair (19).

The three possible mechanisms during composite repair with the use of intermediate unfilled resins are:

1. chemical bond formation to the matrix,
2. chemical bonds to the exposed filler particles
3. Micromechanical retention caused by penetration of the monomer components to micro cracks in the matrix.

Previous studies have shown that mechanical interlocking is the most significant factor establishing a bond between old and repair composites and most likely dominates chemical bonds to the resin matrix or to exposed filler particles (20, 21). In fact, it is confirmed by the lack of improvements in terms of repair strength when silane is applied to aged composite, with the aim to improve chemical bond with the repair composite (22, 23), is a further evidence that micromechanical interlocking may be the main bonding mechanism underlying composite repair.

Bonding between two composite layers is achieved in the presence of an oxygen inhibited layer of unpolymerized resin. Aged restoration do not contain unpolymerized surface layer. Several techniques have been suggested to improve the composite to composite bond. One of the techniques is roughening the surface and the others are based on attempts to improve adhesion of new resin to cross-linked polymer matrix or filler particles of the composite. Recently, adhesion primers have been introduced for improving bonding to composites (24).

Group-A (Control group) had lowest bond strength because no any bonding agent was used. So the bonding between fractured restoration and repaired composite resin was weak.

Group-C (Prime Bond NT) had lower bond strength than group-B (Single Bond) and Group-D (Xeno-III) because ethanol based bonding agents had higher bond strength than acetone based bonding agents.

Acetone has a relatively high vapor pressure value (184 mmHg at 200C) compared to ethanol (43.mm Hg at 200C) and water (175.mm Hg at 200C). A higher vapor pressure will allow the solvent to evaporate more easily. As the solvent evaporates, the viscosity of the dentin bonding agent increase, which decreases the ability of the bonding system to penetrate around the expanded collagen fibers and the opened dentinal tubules, subsequently, inhibits the proper bond formation. (10, 11)

Ethanol based bonding agents had the following advantages:

- Closest surface energy to enamel and dentin so bond strength was more.
- Less volatile than acetone
- Less technique sensitive.

Swift EJ, Perdigao J et al., (25) studied the shear bond strength of ethanol, acetone and aqueous based adhesives they found that ethanol based (Single Bond) had (26.5 MPa), acetone based Prime Bond NT (25.5 MPa) and aqueous based Scotch Bond Multiple Purpose (22.0 MPa).

Group-D (Xeno-III) had greater bond strength than Group-B and Group-C. Both Group-B and Group-D were Ethanol based bonding agent, but Group-D (Xeno-III) had some other advantages:

It contains two unique adhesion promoters:

- The polymerizable PEM-F releases fluoride, which is bonding to calcium ions and thus enhancing the decalcification of the enamel/dentine.
Pyro-EMA, which forms phosphoric acid groups after hydrolysis it contributes to:

- Etching of the dentine/enamel, forming noticeable etching pattern on enamel.
- Partial dissolution of the smear layer and smear plug.
- Neutralization by dissolving calcium hydroxy apatite on the tooth surface.
- Co-polymerization via the methacrylate groups upon light-curing.

The comparative shear bond strength of composite as obtained in this study is as follows:

- Group D (Xeno-III) > Group B (Single Bond) > Group C (Prime and Bond NT) > Group A (without bonding agent).

Clinical trials take considerable time to evaluate efficacy of new bonding systems or techniques. More clinical trials with larger sample size may be necessary to establish a more appropriate and conclusive data, this study is by no means a conclusive one, but nearly suggests that future of adhesive dentistry is like a silver lining in horizon.

**CONCLUSIONS:**

The results of this study suggest the application of bonding agent increased the shear bond strength and those newly introduced bonding systems increased greater shear bond strength.

**REFERENCES:**


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