

**ORIGINAL RESEARCH**

# Measurement of Bond Strength and Contraction Gap of Dentin Bonding Agents

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## ABSTRACT

**Aims and Objective:** The purpose of this study was to evaluate early tensile and shear bond strength of three dentin bonding agents in combination with a common composite restorative resin to human dentin as well as determination of polymerization contraction gap in shallow dentin cavity where bonding interface is loaded by polymerization contraction stress of restorative resin <sup>1</sup>. **Materials and Methods:** The bond strength was measured on the buccal surface of non-carious freshly extracted human first molar using Z100 composite (3M) mediated by bonding agents and cured with the help of visible light cure unit. Bonding agents which was easily available in the local market were chosen. Tensile and shear bond strength were measured by Universal Testing Machine. Restorative margin was inspected in a microscope (×1000). The maximum width of a possible gap was measured in an ocular screw micrometer. **Statistical Analysis Used:** Analysis of variance test was used (ANOVA). **Results and Conclusion:** Of the three bonding agents used (Gluma Comfort Bond, Prim & Bond NT, and Single Bond) Single bond shows maximum shear bond strength and minimum contraction gap. In spite of the high coefficient of correlation found between the of marginal gaps and early shear bond strength, the prediction of shear strength from gap dimension measured and vice versa was not sufficiently relevant.

**Key words:** Dentin bonding agent, composite resin, shear bond strength, visible light curing, contraction gap

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## INTRODUCTION

During the last few years a steadily increasing number of dentin bonding agents or primers have been introduced. While bonding of restorative resin to pretreated enamel is a generally accepted and well-established method in modern restorative dentistry <sup>2</sup>, there is still insufficient clinical evidence for the efficacy of the bond to dentin mediated by the different adhesive available <sup>2</sup>. Although long term clinical testing is the only realistic basis for the definite assessment of a material or a technique, *in-vitro* testing is an indispensable tool for evaluation and prediction of clinical performance.

Determination of early bond strength and contraction gap width is considered suitable to estimate the clinical efficacy of Dentin Bonding Agents <sup>2</sup>. To determine the clinical relevance of bond strength, values the correlation between the strength figures and the width of the marginal contraction gaps were tested.

## MATERIALS AND METHODS

The present study was carried out in the department of Prosthodontics, Faculty of dental sciences, CSMMU (upgraded KGMC) Lucknow with the collaboration of Industrial Toxicology and Research Center (I.T.R.C.) Lucknow and Research Design and Standard

Organization (R.D.S.O.) Lucknow. The sixty extracted teeth were stored in 1% aqueous chloramin solution<sup>3</sup>.

All the samples were divided equally into four major groups on the basis of bonding agents used. Group-A for Gluma Comfort Bond; Group B for Prim & Bond NT, (Fig 1) Group C for Single Bond (Fig-2) and Group D for control group without using any bonding agents.

Each group were further divided into three sub groups on the basis of testing done and each number 1, 2, 3, was suffixed to major group like A1, A2, A3, B1, B2, B3, C1, C2, C3, AND D1, D2, D3. Suffix-1 stand for shear bond strength test, suffix-2 stand for polymerization contraction gap width and suffix-3 stand for tensile bond strength test. Five sample were prepared for each sub group (05x12 subgroup= 60 samples).



Fig 1



Fig 2

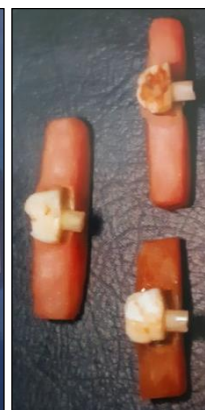


Fig 3

### TESTING OF SHEAR BOND STRENGTH

A flat dentin surface was prepared on the buccal surface of the five teeth for each subgroup by wet grinding on carborundum paper no. 200 and finally on paper no. 1000. Freshly cut dentin surface then were conditioned with Phosphoric acid gel supplied by manufacturer with that particular bonding agent. The bonding agent was applied in two consecutive layers, wait, dried gently for 2 to 5 seconds then irradiation was applied for 10 to 20 second by light cure unit. All the sample were restored with Z100 composite

using cylindrical split mold ( $\phi=4\text{mm}$ ,  $h=5\text{mm}$ ). The light exit window was placed on the Mylar strip and curing time was 80 seconds. (Fig 3)

Shear bond strength of all samples were tested with the help of universal testing machine (Fig. 4). Shear force was applied with the help of blade perpendicular to the vertical axis of the composite cylinder at a distance of 0.2 mm from the bond interface. The cross-head speed was 1 mm/min (Fig 5). Loads applied were recorded after De- bonded sample(Fig 6).

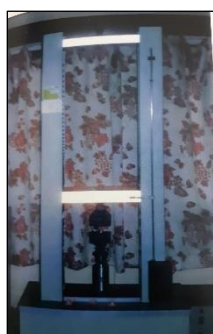


Fig 5



Fig 6



Fig 7

### TESTING OF TENSILE BOND STRENGTH

For tensile bond strength test, all the samples prepared were in same way as were in shear bond strength test. For tensile bond strength testing, a round contra angle latch type bur was inserted head side down in the composite resin within the cylindrical mold. The free

end of the bur was later used for clamping in the universal testing machine (Fig 7). Now the specimens loaded with the 1 mm/min cross head speed of clamp. Tensile Loads applied were recorded after De- bonded sample. (Fig 8)



Fig 7

### MEASURING OF CONTRACTION GAP

3mm diameter cavity was prepared on buccal surface of the teeth with the help of fissure bur approximately at 4000 RPM under water cooling. The depth of the cavity was approximately 1.5 mm and the cavosurface angle was about 90°.

Now cavities of all the samples were treated with bonding agent and filled with Z100 composite and then cured as earlier. The excess was removed by wet grinding by carborundum paper no. 1000 followed by polishing. After through rinsing with water and drying by a gentle air blast, a drop of dye methylene blue was placed and left for 5 minutes. After these dye samples were washed away in tap water, restorative margin was inspected in a microscope (×1000). The maximum width of a possible gap was measured via an ocular screw micrometer. The maximum time for microscopic inspection used was 10 minute to ensure that the gaps measured were contraction gap only but not desiccation gap<sup>2</sup>. Five reading at different sites were taken for each sample to measure margin gap.

All the above three test was done within 20 minutes after the end of polymerization.

### DISCUSSION

When we are in the way of using composite restoration, patients frequently ask the doctor to how much time he should spent without eating and drinking. Thus, early determination of bond strength and gap width is considered suitable to estimate the clinical efficacy of dentin bonding agents as shown in Table 1 mean shear bond strength of C1 and B1 was higher followed by A<sub>1</sub> & D<sub>1</sub>.

A<sub>1</sub>, of being third generation product have much lower shear bond strength than single bond (C1) and prime and bond NT (B1) of fifth generation product. It may be presumed that the third-generation dentin bonding agent based upon formation of resin tags penetrating into tubules of conditioned dentin, formation of precipitate on pretreated dentin surface followed by chemical and mechanical bonding to the resin and chemical union to either inorganic or organic components of the dentin. On the other hand recent concept dentin bonding agents rely on the concept proposed by Nakabayashi 1991 in which diffusion and impregnation of the resin into partially



Fig 8

decalcified dentin followed by polymerization created a resin reinforced layer or the **Hybrid** layer. The fifth-generation bonding system rely on this hybridization or attaining attachment<sup>4,5</sup>.

As mentioned in Table 3, C<sub>1</sub> had higher shear bond strength than B<sub>1</sub> but it was not statistically significant. It can be explained by both being fifth generation product. The difference may be presumed by the factor (being acetone based) Prime Bond NT is very sensitive to even mild desiccation. If the dentin is dried or exposed to air, water evaporates leaving collagen in a collapsed stiffened state because of surface tension forces. This reduces the ability of the subsequently applied agents to penetrate the collagen web. Also, when the collagen fibrils are brought, closer secondary forces start becoming active between adjacent peptide chains in the collagen triple helix, which is not possible when water is present, thereby increasing stiffness or modulus of elasticity for collagen. On the other hand, C<sub>1</sub> is water based, this water is responsible for maintaining the collagen in an expanded state and thereby preserving the spaces needed for infiltration of resin.

Overall pattern of shear bond strength was:

$$C_1 \geq B_1 \gg A_1 \gg \gg D_1$$

Single bond (C2) showed both highest bond strength and best marginal seal in table no. 4. Although Prime Bond NT (B2) showed high bond strength than Gluma (A2) but marginal gap was more in Prime Bond NT group. Gluma showed adequate bond strength (less than Prim and Bond NT) and less marginal gap than Prime Bond NT. Mean marginal gap for control group was significantly higher.

As shown in Fig. 1 Overall, a negative correlation ( $r=-0.607$ ;  $p=0.004$ ) was seen between mean marginal gap and shear bond strength signifying that as the mean marginal gap increased the shear bond strength decreased and vice versa.

However, in Group A, a mild negative correlation ( $r=-0.035$ ;  $p=0.956$ ) between the shear bond strength and mean marginal gap was seen but it was not stastically significant. In Group B too a negative ( $r=-0.393$ ;  $p=0.512$ ) correlation was seen which was also not statistically significant. In Group C a positive

correlation ( $r=0.457$ ;  $p=0.439$ ) and in Group D ( $r=-0.303$ ;  $p=0.621$ ) negative correlation was seen but it was also not statistically significant.

As the number of samples in each group was few it was not possible to draw a correlation individually and hence overall negative correlation between was seen.

Most of the cylinders were fractured from within composite before de-bonding so that actual tensile bond strength cannot be measured in that situation. However, in control group cylinder was de-bonded from tooth surface at much lower strength there was no intermediate adhesive agent was used.

### CONCLUSIONS AND RESULT

From the observations made, statistically analyzed and duly discussed, following conclusions were drawn:

- Fifth generation bonding agent have better shear bond strength than previous third generation bonding system <sup>6</sup>.
- Resin reinforced Hybrid layer formation play significant role in increasing the bond strength.
- Adhesive based on wet bonding technique showed higher bond strength than based on dry bonding technique.
- Contraction gap & shear bond strength not showed a linear correlation.
- Single bond show highest bond strength and least marginal gap in the entire adhesive used in this study.
- Controls group showed very less amount of shear bond strength thus indicating adhesive is important for attaining adequate bond strength.
- Controls group showed bminimum bond strength and maximum margin gap.
- Tensile bond strength of bonding agent had more adhesive strength than cohesive strength of composite cylinder.
- Based on above study these tensile measurements are only suitable for the evaluation of the bond strength between resin and dentin on the condition that the bonding agent to be tested mediates a bond weaker than approximately 8-10 MPa. With more effective adhesives under tensile loading the fracture often occur in the resin cylinder.
- With the shear testing method used in this study however the fracture will always be located at or at least very close to dentin resin interface.
- In spite of the high coefficient of correlation found between the of marginal gaps and early shear bond strengths the prediction of shear strengths from gap dimensions measured and vice versa is not sufficiently relevant.

**Table 1: Showing Shear Bond Strength**

S. N.	Sub group	No. of samples	Load applied (in Newton)	Strength (MPa)	Mean shear strength (MPa)
1	A1	5	92.5 to 152.50	7.4 to 12.14	9.396
2	B1	5	175 to 247.50	13.52 to 19.70	17.924
3	C1	5	210 to 260	16.70 to 20.68	18.212
4	D1	5	20 to 50	2.36 to 3.90	2.520

**Table 2: Showing Analysis of variance of shear bond strength in subgroups**

	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	851.666	3	283.889	102.278	.000
Within Groups	44.411	16	2.776		
Total	896.077	19			

**Table 3: Showing inter group comparison of Shear Bond Strength Between subgroups**

Comparison	't'	'p'
A <sub>1</sub> vs. B <sub>1</sub>	-6.748	<0.001
A <sub>1</sub> vs. C <sub>1</sub>	-8.052	<0.001
A <sub>1</sub> vs. D <sub>1</sub>	7.278	<0.001
B <sub>1</sub> vs. C <sub>1</sub>	-0.250	0.809
B <sub>1</sub> vs. D <sub>1</sub>	15.240	<0.001
C <sub>1</sub> vs. D <sub>1</sub>	19.874	<0.001

**Table 4: Showing Mean Marginal Gap**

S. No.	Sub groups	Diameter of cavity (mm)	No. of samples	No. of sites looked for contraction gaps	Mean marginal gap ( $\mu$ m)	Mean of mean marginal gaps ( $\mu$ m)
1.	A2	3	5	5	4.48 to 4.66	4.596
2.	B2	3	5	5	5.36 to 5.52	5.480
3.	C2	3	5	5	4.20 to 4.36	4.272
4.	D2	3	5	5	6.12 to 66.52	6.288

**Table 5: Showing inter group comparison of Mean of Mean Marginal Gaps Between subgroups**

Comparison	't'	'p'
A <sub>2</sub> vs. B <sub>2</sub>	19.309	<0.001
A <sub>2</sub> vs. C <sub>2</sub>	6.306	<0.001
A <sub>2</sub> vs. D <sub>2</sub>	21.283	<0.001
B <sub>2</sub> vs. C <sub>2</sub>	24.336	<0.001
B <sub>2</sub> vs. D <sub>2</sub>	10.308	<0.001
C <sub>2</sub> vs. D <sub>2</sub>	24.651	<0.001

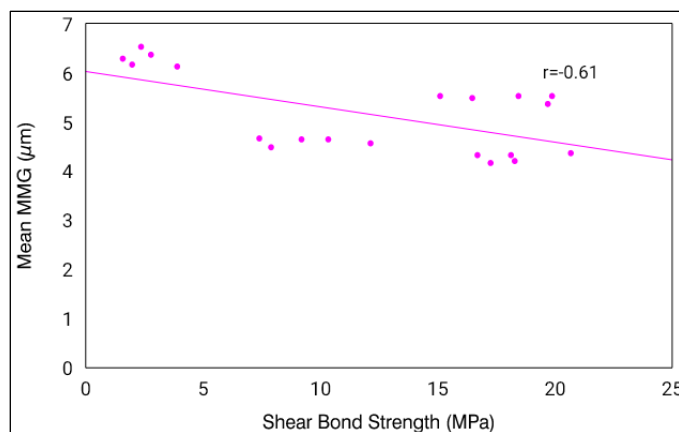
**Table 6: Showing Tensile Bond Strength of subgroups**

S. N.	Radius (mm)	Sub groups	No. of samples	Load (Newton)	Strength (MPa)	No of samples* before debonding
1.	2	A3	5	115 to 140	9.14 to 11.17	4
2.	2	B3	5	100 to 147.5	7.45 to 11.74	4
3.	2	C3	5	62.5 to 170	4.79 to 13.53	4
4.	2	D3	5	38 to 55.50	3.02 to 4.41	0

\*Indicate fracture within composite cylinder before de bonding.

**Table 7: Showing inter group comparison of Tensile Bond Strength Between subgroups**

Comparison	't'	'p'
A <sub>3</sub> vs. B <sub>3</sub>	0.073	0.944
A <sub>3</sub> vs. C <sub>3</sub>	0.191	0.853
A <sub>3</sub> vs. D <sub>3</sub>	13.361	<0.001
B <sub>3</sub> vs. C <sub>3</sub>	0.215	0.835
B <sub>3</sub> vs. D <sub>3</sub>	8.554	<0.001
C <sub>3</sub> vs. D <sub>3</sub>	4.069	0.004

**Figure 1: Showing Correlation between Shear Bond Strength and Mean Marginal gap (overall)**

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