IJCRT.ORG ISSN : 2320-2882

INTERNATIONAL JOURNAL OF CREATIVE RESEARCH THOUGHTS (IJCRT)

An International Open Access, Peer-reviewed, Refereed Journal

The effect of dentin pretreatment with polyacrylic acid on the tensile bond strength of self adhesive resin cements. – An in vitro study

Dr. T Sai Prasad[1], Dr. Mahendranadh Reddy[2], Dr. Mahadev Shastry[3], Dr. Venkat Aditya[4]

Prosthodontist and Implantologist[1], Professor[2], Professor[3], Professor[4]

Department of Prosthodontics, Hyderabad, India

Abstract:

Objective: The purpose of this in vitro study was to evaluate the effect of polyacrylic acid as dentin priming agent on tensile bond strength of self adhesive resin cements to dentin.

Materials and method: Extracted premolar which were selected for the study were mounted in self cure acrylic which were trimmed buccally with the help of diamond disc under running water and was smoothened with abrasive paper (180 grit) for flat surface. Zirconium block with 3mm diameter and 5mm height with through and through hole of diameter were designed and fabricated with the help of CAD CAM milling machine.

Teeth were divided into three groups according to dentin surface treatments

1) Group A- Control group (without dentin pre-treatment)

2) Group B- 11.5% polyacrylic acid treated group

3) Group C- 25% polyacrylic acid treated group.

The zirconium blocks were bonded to dentin surfaces with self adhesive resin cements which were tested for tensile bond strength. All the data was statistically analyzed. The comparison between three groups was done by one-way ANOVA test for continuous data and followed by post-hoc multiple comparison test The de-bonded surface were evaluated with scanning electron microscope for assessing mode of failure.

Results: There was a significant difference between the groups and on applying post hoc the difference was found between group C with the group B and group A**.** The mean tensile bond strength of group C was found to be statistically significantly higher statistically than that of group B and group C. ($p<0.05$).

Conclusion: Within the limitations of this in vitro study, group C showed higher tensile bond strength when compared to group A and group B. Dentin pretreatment with group C increased the bond strength of self adhesive resin cement.

I. INTRODUCTION

Resin cements have been widely used for indirect esthetic restorative procedures. With recent advances in adhesive dentistry, resin cements have acquired a very important role in Prosthodontics. Depending on the mode of adhesion, resin cements can be classified as total-etch, self-etch, and self-adhesive resin cements. Self-adhesive resin cements were introduced in 2002[1, 2]. They are also called as universal, all-purpose or multipurpose resin cements. Resin cements present several advantages when compared to conventional bonding agents, that include improved retention, minimal solubility in the oral environment.

The conventional bonding technique includes a 3-step etch, prime and bond protocol. These systems are reported to achieve the highest bond strength values. The superior bonding may be attributed to optimal dentin hybridization and the formation of a complete hybrid layer. However, in clinical practice, techniques with several steps are considered more sensitive and prone to error[3]. Self-adhesive resin cements such as RelyX Unicem 2 (3M ESPE) may eliminate some of the problems with multistep systems. The adhesive properties of these cements are based upon acidic monomers that de-mineralize and infiltrate the tooth substrate, resulting in micromechanical retention.[3]

In an attempt to increase the bond strength between resin cements and tooth surfaces, surface treatments with different conditioning agents have been suggested. Polyacrylic acid is a mild conditioning agent employed for cavity cleansing and surface conditioning in glass ionomer restorations. In these restorations, polyacrylic acid promotes the formation of irregularities on the surface of the substrate, forming an intermediate layer that facilitates ion exchange between the glass ionomer matrix and the calcium and phosphate in the partially demineralized smear layer. Furthermore, the carboxyl ions in the acid increase the cleaning power and wettability of the surface[4].

Mild dentin conditioners have been recommended to modify smear layer and to create surface roughness and expose dentinal tubules which is done by polyacrylic acid. The bond strength of the adhesive cement between the prepared dentin surface and the zirconium is determined by universal testing machine.

II.MATERIALS AND METHOD

2.1 Teeth preparation and Embedding:

Sixty unrestored, non-carious human premolars with complete root formation were selected for this study. The teeth were cleaned with an ultrasonic scaler and stored in 10%formalin solution to prevent dehydration.

Silicon mold (putty index) of length 4cm and width 1.5 cm was fabricated **(Fig:1)** . Self-cure acrylic resin was filled in the putty index then the teeth were embedded in the centre of the self-cure acrylic resin such that the roots were embedded till dentino enamel junction in the acrylic and the occlusal surface was at least 5mm above the level of the resin, the assembly was cured in pressure pot**(Fig:2).**The tooth were trimmed buccally with the help of diamond disc under running water removing complete enamel and exposing dentin which was smoothened with abrasive paper for flat surface**(Fig:3).**

Fig:1 putty index

Fig:2 sample moulded

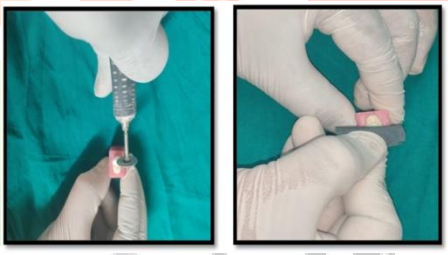


Fig:3 tooth preparation

2.2 Milling of zirconium disc by CAD- CAM Technique:

Using Exo-cad software, a STL (Standard Tessellation language) file of the specimen was designed. The zirconium blank of 98mm diameter and 14mm thickness was inserted in the milling unit and the zirconium block of required dimension of diameter 3mm height 5mm were milled. The zirconium blocks were fabricated with a hole of diameter 1.5mm at 3mm height for holding the block in the UTM machine **(Fig:4,5).** The blocks were then sandblasted using 110µm aluminium oxide particles for 10 seconds with 1.5 bar pressure and 10mm away from vertical distance to improve the bonding strength of zirconium with resin cements.

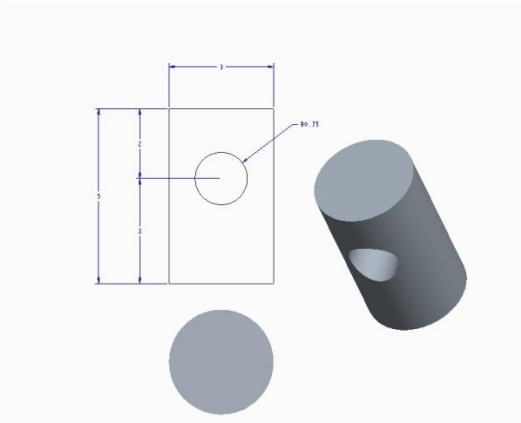


Fig:4 design of block

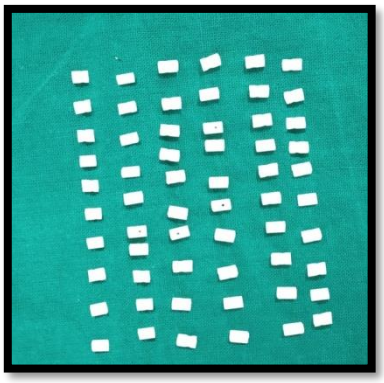


Fig:5 zirconia blocks

2.3 The prepared teeth were divided into three groups based of polyacrylic acid treatment.(Fig:6)

- Group A: control group. with out dentin pretreatment.
- Group B: treated with 11.5% polyacrylic acid for 10 sec.
- Group C: treated with 25% polyacrylic acid for 10 sec.

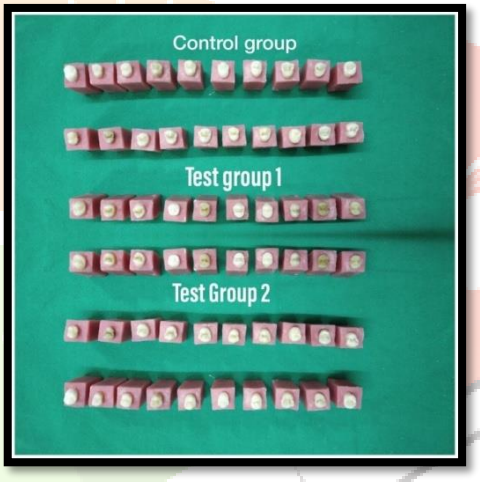


Fig:6 three groups

2.4 Bonding of teeth with zirconium:

The zirconium blocks were bonded to the dentin surfaces of group A(with out dentin pretreatment),group B(with 11.5%polyacrylic acid treatment) and group C with (25% polyacrylic acid treatment) with Rely X Unicem self adhesive universal resin cement.The working time of Rely X Unicem was 2 minutes and the excess cement was removed in gel state with explorer and then the surfaces were light cured for 20 seconds**(Fig7,8).**

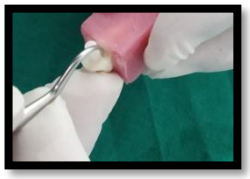


Fig:7, 8 bonding of zirconia to tooth

2.5 Universal Testing Machine:

The bonded specimens were placed on the table and held with clamp from top and below with upper and lower cross heads of the machine such that the bonded specimens base were parallel to the applied force. Specimens were stressed in a bucco-palatal direction at a crosshead speed of 1mm/min. A jig was attached to the load cell and pulled apart by an upper chain. The maximum load necessary to de-bond were recorded in Newton (N) and calculated in MPa **(Fig:9).** De-bonded specimen surfaces were examined with a Scanning Electron Microscope (SEM) **(Fig:10)** to assess the mode of failure, which can be cohesive failure or adhesive failure or due to smear layer.

Fig:9 UTM machine

Fig:10 SEM

III.RESULTS

For testing the tensile bond strength of the specimens, all three groups of 20 samples each were subjected to pull out test on a universal testing machine. Statistical analysis was done using SPSS Statistics 25.0 Descriptive statistics, One way ANOVA, Bonferroni's post hoc test was performed. Confidence interval was set at 95%. P value < 0.05 was considered statistically significant.

Table 1: Tensile bond strength values obtained by group A(in MPa)

1	7.85
2	7.14
3	6.42
4	11.14
5	10
6	10.71
7	10.42
8	8.85
9	9.71
10	9.71
11	6.85
12	8.14
13	9.42
14	9.14
15	7.42
16	8.71
17	10.57
18	8.14
19	9.85
20	10.14

Table 1 shows :Highest tensile bond strength value obtained for group A was **11.14MPa** and lowest was **6.42 MPa**.

Table 2: Tensile bond strength values obtained by group B (in MPa)

1	10
2	8.57
3	11.42
4	5.71
5	7.57
6	9.28
7	10.71
8	10
9	10.42
10	11.14
11	7
12	6
13	7.37
14	9.57
15	10.14
16	9.14
17	9.57
18	8.28
19	8.85
20	10.57

Table 2 shows highest tensile bond strength value obtained for group B was**11.42 MPa** and lowest was **5.71 MPa.**

Table 3: Tensile bond strength values obtained by group C (in MPa)

1	20
2	21.42
3	14
4	14.28
5	17.14
6	19.71
7	24
8	12.82
9	12.57
10	16.42
11	13.57
12	15
13	16.54
14	20
15	21.85
16	19
17	21
18	22.57
19	23.14
20	18.14

Table 3 shows Highest tensile bond strength value obtained for group C was **24 MPa** and lowest was **12.57 MPa**.

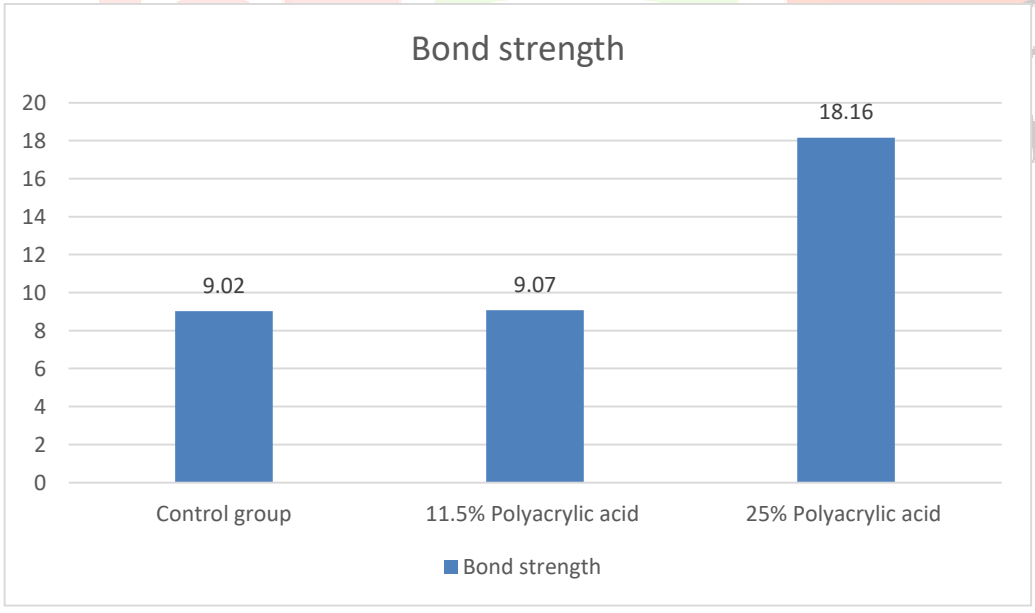
Statistical Analysis:

The comparison of tensile bond strength between three groups was done by one-way ANOVA test and followed by Bonferroni's post-hoc multiple comparisons test. All p-values less than 0.05 were considered as statistically significant.

Table 4 shows the comparison of mean bond strengths between Group A,Group B and Group C.One way ANOVA and Bonferroni's post hoc test were used for analysis.

Test groups	N	Mean	SD	Std. Error	95% Confidence Interval for Mean		Min	Max	F	P value	Post hoc analysis
					Lower Bound	Upper Bound					
Group A	20	9.02	1.38	0.31	8.37	9.66	6.42	11.14	93.645	0.000	3>2,1
Group B: 11.5% Polyacrylic acid	20	9.07	1.64	0.37	8.30	9.83	5.71	11.42			
Group C: 25% Polyacrylic acid	20	18.16	3.63	0.81	16.46	19.86	12.57	24.00			
Total	60	12.08	4.95	0.64	10.80	13.36	5.71	24.00			

The above table 4 represents the comparison between group A, group B and group C on tensile bond strength. The maximum value and the minimum values for group A, was **11.14** and**6.42** respectively with a standard deviation **of1.38**. For group B the maximum value was **11.42** and lowest being **5.71** with standard deviation of**1.64** and group C the maximum value was **24** and lowest being **12.57** with standard deviation of**3.63**. There was a significant difference between the groups and on applying post hoc the difference was found between group C with the other two groups**.** The mean tensile bond strength of group C was found to be statistically significantly higher than that of group A and B.



Graph 1: Graphical representation showing mean tensile bong strength values

Graph representation depicts that mean values for group A is **9.02**

For group B is **9.07** and for group C is **18.16.**

It shows that tensile bond strength of group C is highest than compared to A and B.

For the characterization of mode of failure, The specimens were allowed to air-dry overnight, after which they were sputter-coated with gold and evaluated by SEM at a magnification of 1.00 KX at 8.00K V.

SEM images of specimens of group A:

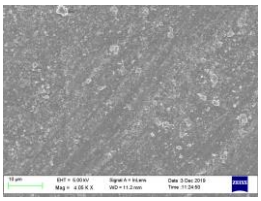


Fig11: the above image shows the tooth interface showing adhesive failure.

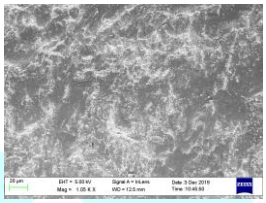


Fig12: the above image shows the zirconium interface showing mixed failure.

Table 5 failure pattern for group A

Group A	Failure pattern
Cohesive	1
Adhesive	14
Mixed	5

Table 5 shows number of fracture pattern cohesive =1,adhesive=14, mixed=5

SEM images of specimens of group B:

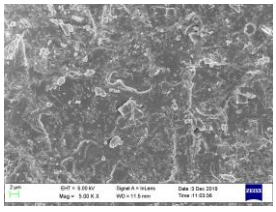


Fig13: the above image shows the adhesive failure of tooth surface

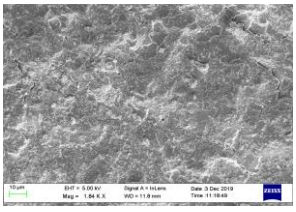


Fig14: the above image shows the adhesive failure at zirconium interface

Table 6: failure pattern in group B

Group B	Failure pattern
Cohesive	2
Adhesive	14
Mixed	4

Table 6; shows number of fracture pattern cohesive =2 adhesive=14, mixed=4.

SEM images of specimens of group C:

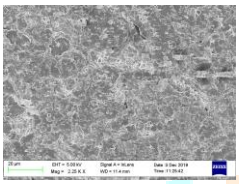


Fig15: the above image shows the tooth interface showing cohesive failure.

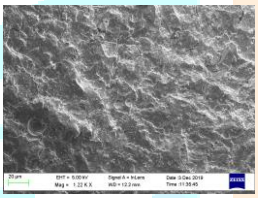


Fig16: the above image shows the zirconium interface showing adhesive failure.

Table 7: failure pattern in group C

Group C	Failure pattern
Cohesive	8
Adhesive	6
Mixed	6

Table 7; shows the number of fracture pattern cohesive =8, adhesive=6, mixed=6.

IV.DISCUSSION

Self-adhesive cements were introduced with the aim of simplifying clinical procedures and replacing the sensitive, multistep procedures. The material is applied directly on the dentin surface, without the necessity for any pretreatment. The smear layer is partially removed or incorporated by acidic monomers that promote micromechanical retention to the tooth structure. Chemical retention may also be achieved by the reaction between phosphoric acid monomers and the hydroxyl-apatite of dentin. Additional advantages to the 1-step resin-based luting systems are decreased postoperative sensitivity and less susceptibility to moisture.

Though these cements have many advantages compared to conventional cements they have less bond strength as conventional cements relay on surface pretreatment with acids before bonding procedure.

Mechanism of polyacrylic acid treatment on dentin:

This mild acid removes the smear layer partially but leaves smear plugs in the tubules. In addition, it leaves free calcium and phosphate ions on the dentin surface to promote a better chemical reaction with some of the restoration materials (SELF ADHESIVE RESIN CEMENT, GIC etc).The functional carboxyl ion groups present in PAA formulation can form a multiplicity of hydrogen bonds, With dentin substrate. They promote demineralization with in dentin which helps in penetration of resinous compounds into the dentin surface irregularities.

Tanumiharja M et al (2000)[5] stated that polyacrylic acid contains numerous carboxyl ion groups that can form a variety of hydrogen bonds and promote cleaning and wetting of the substrates. Marshall GW et al (1997)[6] also concluded by saying that the wetting ability and the extent to which the adhesive penetrates the dentin play a major role in determining the quality of bonding. The film of acid applied wets the entire dentin surface and increases the surface area which in turn improves the adhesion of resin to dentin. Kanca, J. (1992)[7] stated that bonding of dentin is increased in wet condition than in dry condition as the natural state of dentin in vital teeth includes fluid in the tubules. This tends to create a moist surface on dentin.
Pisani-Proença.j et al (2011)[8] conducted a study and stated that self-adhesive cement requires more water in its interaction on the dental surface in order to achieve higher bond strength values. Derand et al (2005)[9], Atsu et al (2006)[10] stated that although cementation of zirconia restorations with traditional cements (such as zinc phosphate or glass ionomer cements) may provide adequate clinical fixation, adhesive cementation is preferable to ensure better retention and marginal adaptation. As resin cements have methacrylate which bond chemically with the zirconium.

Statistical analysis was done using SPSS Statistics 25.0 Descriptive statistics, One way ANOVA, Bonferroni's post hoc test was performed. Confidence interval was set at 95%. P value < 0.05 was considered statistically significant. The maximum value and the minimum values for group A, was **11.14** and **6.42** respectively with a standard deviation **of 1.38**. For group B the maximum value was **11.42** and lowest being **5.71** with standard deviation of**1.64** and group C the maximum value was **24** and lowest being **12.57** with standard deviation of**3.63**. There was a significant difference between the groups and on applying post hoc the difference was found between group C with the other two groups**.** The mean tensile bond strength of group C was found to be statistically significantly higher than that of group A and B.

In SEM analysis group A prominent failure were adhesive as the teeth surface with smear layer hydroxy apatite crystal can be seen on the bonded surface group B adhesive failure pattern are prominent when viewed in SEM the teeth surface with smear layer hydroxy apatite crystal seen. In group C the prominent failure were cohesive failure as the remnants of cements can be seen on tooth structure indicating that fracture occurred between the cements indicating increased strength of the bond. Limitations of this study include that the pre-treatment of dentin was evaluated under specific in vitro conditions; therefore, this investigation does not substitute for clinical trials. Tooth tissue may show varying thickness of dentin and it may become brittle due to storage conditions. As a result, standardization was not achieved.

V.CONCLUSION

Within the limitations of this in vitro study, following conclusions were drawn:

1. 25%polyacrylic acid treated group showed higher tensile bond strength compared to control group and 11.5% polyacrylic acid treated group.
2. Dentin pretreatment with 25%polyacrylic acid increased the bond strength of self adhesive resin cement.
3. In the present study only one acid was used for dentin pre-treatment. The effect of other acids on dentin pretreatment should be evaluated.

Further studies with larger sample size have to be done to come to a definitive conclusion

Acknowledgment

I offer my fervent thanks to the Almighty for the blessings showered on me and guiding me through every step. I am indebted to individuals too numerous to mention separately. I take this opportunity to thank everyone who has contributed in completing this manuscript.

I express my deep sense of gratitude to my respected teacher and guide, **Dr. K. Mahendranadh Reddy,** Professor and HOD for his tolerant nature and suggestions. His untiring enthusiasm and constructive criticism have made this study possible. He taught me how to question thoughts and express ideas. His patience and support helped me overcome many crisis situations and finish this dissertation.

I am thankful to **Dr. Y. Mahadev Shastry,** Professor, **Dr. N. Venkat Aditya,** professor, for their thoughtfulness and encouragement. I am grateful to them for holding me to a high research standard and enforcing strict validations for each research result and thus teaching me how to do research. I am also thankful to them for encouraging the use of correct grammar and consistent notation in my writings and for carefully reading and commenting on countless revisions of this manuscript.

I would like to thank my parents, **T Anjaneyulu & T Kotamma** for their lifelong support, wisdom and direction. The sacrifices that they have made allowed me to pursue the aspirations and desires for my life and my career.

REFERENCES

1) Pavan S, Dos Santos PH, Berger S, Bedran-Russo AK. The effect of dentin pretreatment on the microtensile bond strength of self-adhesive resin cements. *J Prosthet Dent 2010;104(4):258-64.*
2) Sekhri S, Mittal S, Garg S. Tensile bond strength of self adhesive resin cement after various surface treatment of enamel. *J Clin Diagn Res 2016;10(1):ZC01*
3) Brigagao VC, Barreto LF, Gonçalves KA, Amaral M, Vitti RP, Neves AC, Silva-Concílio LR. Effect of interim cement application on bond strength between resin cements and dentin: Immediate and delayed dentin sealing.*J ProsthetDent2016;117(6):792-8.*
4) Mushashe AM, Gonzaga CC, Cunha LF, Furuse AY, Moro A, Correr GM. Effect of enamel and dentin surface treatment on the self-adhesive resin cement bond strength. *BrazDentJ 2016 Oct;27(5):537-42.*
5) Tanumiharja M, Burrow MF, Tyas MJ. Microtensile bond strengths of glass ionomer (polyalkenoate) cements to dentine using four conditioners. *J Dent 2000;28:361-6.*
6) Marshall GW Jr, Marshall SJ, Kinney JH, Balooch M. The dentin substrate: structure and properties related to bonding. *J Dent 1997;25:441-58.*
7) Kanca, J., Improving bond strengths through acid etching of dentin and bonding to wet dentin surfaces. *J Am Dent Ass 1992; 123: 35-43.*
8) Pisani-Proença J, Erhardt MCG, Amaral R, Valandro LF, Bottino MA, Castillo-Salmerón RD. Influence of different surface conditioning protocols on microtensile bond strength of self-adhesive resin cements to dentin. *J Prosthet Dent 2011;105:227-35.*
9) Derand T, Molin M, Kvam K 2005. Bond strength of composite luting cement to zirconia ceramic surfaces. *Dent Mater, 2005;21(12): 1158–62.*
10) Atsu SS, Kilicarslan MA, Kucukesmen HC, Aka PS. Effect of zirconium-oxide ceramic surface treatments on the bond strength to adhesive resin. *J Prosthet Dent 2006; 95(6): 430–36.*