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RESEARCH ARTICLE

COMPARATIVE EVALUATION OF THE SHEAR BOND STRENGTH OF THREE SELF-ETCH DENTIN BONDING AGENTS TO DENTIN: AN IN-VITRO STUDY

Bhalla V.¹, K. Goud M.², Chockattu S.³ and Khera A⁴

1. Dr. Vidhi Kiran Bhalla, Senior Lecturer, ITS Centre for Dental Studies and Research, Ghaziabad, Uttar Pradesh, India.
2. Dr. Mallikarjun Goud K, Professor & Head, Department of Conservative Dentistry, Bapuji Dental College & Hospital, Davangere, India.
3. Dr. Sherin Jose Chockattu, Senior Lecturer, Department of Conservative Dentistry, Bapuji Dental College & Hospital, Davangere, India.
4. Dr. Archie Khera, Consultant Endodontist, Apollo Hospitals.

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Abstract

Background: Dentin bonding is an ever-evolving field in adhesive dentistry. With the introduction of newer systems into the market, there is a crucial need to test their efficiency in terms of bond strength. Dual-cured adhesives in theory may provide for a better degree of conversion as compared to conventional light-cured adhesives. Thus, the aim of this study was to compare the shear bond strength of three different self-etch adhesives namely Clearfil SE bond (Kuraray), Tetric N Bond Universal (Ivoclar Vivadent) and Futura Bond DC (Voco) to dentin.

Materials & Methods: Ninety extracted non-carious, intact human mandibular molar teeth were selected for this study. Each tooth was decoronated using a double-sided diamond disc with water coolant to a depth of 2mm from the cusp tip. The cut dentin surface was then abraded against 600-grit wet silicon carbide papers for 60 seconds to produce a uniform smear layer. The root portion of each tooth was mounted on a plastic ring using cold cure acrylic resin. Specimens were then divided into three adhesive groups of 30 teeth each, Group A: Clearfil SE Bond (Kuraray), Group B: Tetric N Bond Universal (Ivoclar Vivadent), Group C: Futura Bond DC (Voco). All bonding agents were used according to the manufacturers' instructions, in combination with the resin composite Tetric N Ceram (Ivoclar Vivadent). The samples were thermocycled, followed by shear bond strength testing using a Universal testing machine (Hounsfield). Data were subjected to statistical analysis using one-way analysis of variance (ANOVA) ($P < 0.05$) and Post hoc Tukey's test for inter- and intra- group analysis respectively.

Results: Clearfil SE Bond yielded the highest shear bond strength values (30.9 ± 4.66 MPa) which were statistically significant, followed by Tetric N Bond Universal group (29.8 ± 4.34) and the lowest shear bond strength values were recorded for Futura Bond DC (18.2 ± 3.13).

Conclusion: Clearfil SE bond and Tetric N bond Universal can be considered as better options than Futura Bond DC.

Corresponding Author:- Dr. Vidhi Kiran Bhalla

Address:- Senior Lecturer, ITS Centre for Dental Studies and Research, Ghaziabad, Uttar Pradesh, India.

Introduction:-

Patients' demand for aesthetic restorations & the search for restorative materials that can provide long term stability are leading to development of new products in adhesive dentistry at an unprecedented rate. The foundation for adhesive dentistry began with Buonocore in 1955, who reported that acids could be used to alter the surface of enamel to render it more receptive to adhesion.[1] Ever since this landmark discovery, the main challenge for the dental adhesives, to this day, is the need for an effective bond to dental substrates of different nature. Bonding to enamel is more reliable, in contrast dentin contains a significant amount of water and organic material mainly type I collagen which makes bonding to this hard tissue extremely difficult.[2]

Based on the underlying adhesion strategy, i.e., the nature of interaction with enamel and dentin substrates, contemporary adhesive systems can be classified into etch and-rinse and self-etch adhesives. [2] With the etch-and-rinse strategy, the enamel and dentin are treated with an acidic gel (commonly phosphoric acid) to remove the smear layer and demineralize the most superficial hydroxyapatite crystals. Following this chemical etching, a mixture of resin monomers (primer/ adhesive) dissolved in an organic solvent is applied to infiltrate the etched dentin.[2,3] This infiltration results in a hybrid tissue composed of collagen, resin, residual hydroxyapatite with traces of water and is known as the resin- dentin inter diffusion zone (hybrid layer).[4]

Self-etch adhesives do not require a separate acid-etch step as they condition and prime enamel and dentin simultaneously by infiltrating and dissolving the smear layer and hydroxyapatite to generate a hybrid zone that incorporates minerals and smear layer.[5] With the self-etching adhesive systems, the smear layer becomes part of the bonding substrate since the acidic primer is not rinsed from the surface and the demineralized smear layer may be incorporated into the hybrid layer. New dental adhesives are continuously being developed and the newer self-etching adhesives are marketed as being less technique sensitive, less time consuming, and as effective as older total-etch adhesives.[5,6]

A new bonding agent FUTURA BOND DC (Voco) dual-cured, self-etching bonding agent reinforced with nanofillers has been recently introduced in the market. The manufacturer claims that it can create strong durable bonds with composite restorative materials. Tetric N Bond Universal (Ivoclar Vivadent) is a single component, light curing universal adhesive suitable for all etching techniques: self-etch, selective enamel-etch and total-etch. Clearfil SE Bond (Kuraray) is a mild two step self-etch adhesive considered as the gold standard for self-etch adhesives.

Thus, the aim of the present in-vitro study was to evaluate the shear bond strength of three different self-etch adhesives namely Clearfil SE bond (Kuraray), Tetric N Bond Universal (Ivoclar Vivadent) and Futura Bond DC (Voco) to dentin. The null Hypothesis was that there will be no difference in the shear bond strengths of the three self-etch dentin bonding agents to dentin.

Calculation of sample size:

The sample size was determined scientifically. Considering :

μ_A :36.6

μ_B : 25.1

Alpha : 0.05

Power of the study : 0.02

Number of the groups: 3

Therefore estimated sample size for the study is 30 for each group. Hence the total sample size will be 90. This is calculated using the formula

$$n = 2 \left(\frac{\sigma \left(z_{1-\alpha/(2\tau)} + z_{1-\beta} \right)}{\mu_A - \mu_B} \right)^2$$

Materials & Methods:-

Ninety freshly extracted human mandibular molars were included in this study. Carious, previously restored, fractured tooth and teeth with attrition, abrasion, erosion were excluded.

The teeth were debrided by washing under running water and cleaned with ultrasonic tip followed by storage in isotonic saline. Each tooth was decoronated using a double sided diamond disc with water coolant to a depth of 2mm from the cusp tip. The cut dentin surface was then abraded against 600-grit wet silicon carbide papers for 60s to produce a uniform smear layer. The root portion of each tooth was mounted on a plastic ring using cold cure acrylic resin. The specimens were then divided into three adhesive groups of 30 teeth each (n=30). Manufacturer's instructions were followed in the application of the bonding agents to dentin (Table 1).

All bonding agents were used in combination with resin composite Tetric N Ceram Bulk Fill (Ivoclar Vivadent). Following application of adhesive, a teflon mold was used to build composite resin cylinder on the dentinal surface of all samples, measuring 2mm in diameter & 3mm in height in a single increment followed by light curing for 20s.

After 24 hr storage in distilled water at 37°C, the samples were thermocycled between 50°-55°C with a dwell time of 5s for 500 thermocycles. The shear bond strength was determined using a knifeedge blade in a universal testing machine at a crosshead speed of 1 mm/min and readings were recorded in kgF.

Data were subjected to statistical analysis using one-way analysis of variance (ANOVA) ($P < 0.05$) and Post hoc Tukey's test for inter- and intra-group analysis, respectively.

Table 1:- Manufacturer's instructions for the 3 experimental groups.

Bonding Agent (Manufacturer)	Manufacturer's instructions
Group A: Clearfil SE Bond (Kuraray)	Primer was applied to the entire tooth with a brush and will be left for 20 seconds. It was then dried with mild air flow. Bond was applied with a brush, dispersed with very mild stream of air and then light cured for 10 seconds.
Group B: Tetric N Bond Universal (Ivoclar Vivadent)	One layer of Adhesive coat was applied and agitated for 20 seconds and then dispersed with compressed air till a glossy, film layer forms. It was then light cured for 10 seconds.
Group C: Futura Bond DC (Voco)	Supplied as a 2-bottle system. One drop of Liquid 1 and one drop of Liquid 2 was dispensed and mixed with an applicator tip for 2 seconds. It was then applied with the same applicator tip onto the prepared cavity with a rubbing motion for 15 seconds, gently air dried for 5 seconds, and then light cured for 10 seconds.

Results:-

The results of the study are given in Table 2. The inter-group and intra-group comparisons are given in Table 3 and 4, respectively. Clearfil SE bond yielded the highest shear bond strength (SBS) values (30.9 ± 4.66 MPa) followed by Tetric N bond Universal group (29.8 ± 4.34 MPa) and the lowest shear bond strength values were recorded for Futura Bond DC (18.2 ± 3.13 MPa). Mean SBS value of Group C (Futura Bond DC) is found to be significantly lower from Group A (Clearfil SE Bond) ($P < 0.001$) and from Group B (Tetric N bond Universal) ($P = 0.012$). Mean SBS value of Group B (Tetric N bond Universal) is not found to be statistically significant from Group A (Clearfil SE Bond) ($P = 0.076$) but was significantly higher than Group C (Futura Bond DC) ($P < 0.001$). Mean SBS value of Group A (Clearfil SE Bond) is not found to be statistically significant from Group B (Tetric N bond Universal) ($P = 0.076$) but was significantly higher than Group C (Futura Bond DC) ($p < 0.001$).

Table 2:- Shear bond strength (SBS) values of the three groups.

Shear Bond Strength (MPa)						
Group	N	Minimum	Maximum	Median	Mean	SD
A	30	11.9	30.9	16.2	17.0	4.66
B	30	8.4	29.8	14.5	14.6	4.34
C	30	5.4	18.2	11.2	11.5	3.13
Total	90	5.4	30.9	13.9	14.4	4.63

Table 3:- Comparison of groups with respect to shear bond strength by ANOVA.

ANOVA					
Shear Bond Strength (M Pa)					
	Sum of Squares	df	Mean Square	F	p value
Between Groups	446.2	2	223.1	13.28	<0.001
Within Groups	1461.6	87	16.8		
TOTAL	1907.8	89			

Table 4:- Pairwise comparison of shear bond strength values in three study groups by post hoc test.

Multiple Comparisons						
Shear Bond Strength (MPa)						
Tukey HSD						
(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
A	B	2.3	1.1	0.076	-0.19	4.86
	C	5.4	1.1	< 0.001	2.91	7.96
B	A	-2.3	1.1	0.076	-4.86	0.19
	C	3.1	1.1	0.012	0.58	5.63
C	A	-5.4	1.1	< 0.001	-7.96	-2.91
	B	-3.1	1.1	0.012	-5.63	-0.58

Discussion:-

A major goal of modern restorative materials research is the development of dental restorative materials that form strong bonds to tooth structures. The durability of adhesive bond between resin and the tooth structure is of significant importance for longevity of adhesive restorations.[7] Kirematiciet al. recommends a minimum bond strength of 17-21 MPa to withstand this stress of polymerization shrinkage of the composite restoration.[8] Hence measuring bond strength is important in the evaluation of mechanical fracture and therefore the prognosis of dental treatment.

Under the influence of customer-oriented strategic marketing, research chemists have been forced to alter existing bonding strategies and formulate adhesives that are simpler, speedier and more user-friendly. The bonding performance attained by self-etch adhesives varies a great deal, depending not only on the actual class of self-etch adhesives, but also on the actual composition, more specifically on the functional monomer included in the adhesive formulation.[6] The bonding agents used in the present study were Clearfil SE Bond (Kuraray), Futura Bond DC (Voco) and Tetric N bond Universal (Ivoclar Vivadent).

For the present study, 90 freshly extracted human mandibular molars were taken which were then stored in distilled water. ISO 11405 recommends bond strength should be measured immediately post extraction but not more than six months.[9]

Extracted mandibular molars were trimmed with a double sided diamond disc to expose the superficial dentin and then finished with a 600 grit Si-C abrasive paper. The Current ISO standards for bond strength testing recommends surface preparation with 600-grit silicon carbide (SiC) abrasive paper. This method produces a relatively smooth surface that does not reproduce the surface topography and smear layer thickness obtained with rotary cutting instruments.[10]

All the three self-etch adhesives were used as per the manufacturers' instructions. Following which, a teflon mold was used to build composite resin cylinder on the dentinal surface of all samples, measuring 2mm in diameter & 3mm in height in a single increment.

Before subjecting the specimen to experimental procedure, selected specimens were stored in distilled water at 37°C for 24 hr. This was done to ensure complete and uniform polymerization of all composite specimens.[11] After this, the selected specimens were subjected to thermocycling procedure. Thermocycling allows the bonded specimens to be subjected to extreme temperatures, which mimics intraoral temperature variations. So in the present

study, thermocycling of specimens of each group was carried out in accordance with the ISO-11405 standards i.e. 500 cycles at 5°C and 55°C, dwell time of 30s and transfer time of 15s.[11]

All the forces acting on an adhesive bond *in vivo* can be resolved as components acting at right angles and parallel to the interface. It is therefore important to measure the shear strength in order to evaluate a bond adequately.[7]

The results of the present study reported that shear bond strength of Clearfil SE bond (30.9 ± 4.66 MPa) was the highest when compared to Tetric N bond Universal (29.8 ± 4.34 MPa) and Futura Bond DC (18.2 ± 3.13 MPa) (Table 2). The highest shear bond strength was reported with Clearfil SE bond. It has been shown in multiple studies to have consistently stronger bond strengths than other two-step self-etch bonding agents and similar to those of the three-step etch-and-rinse with superior clinical longevity.[12] The higher clinical and *in vitro* performance and durability are related to the following factors – being a mild self-etch, CSE slightly demineralizes dentin, allowing some residual hydroxyapatite to evolve and protect collagen fibrils; presence of the functional monomer 10-methacryloyldecyl dihydrogen phosphate (10-MDP) component of the CSE primer; and the presence of an hydrophobic coat, as a 2-step self-etch, in the second bottle of the CSE instructions for use. The monomer 10-MDP can ionically interact with calcium in Hydroxyapatite and form hydrolytically stable 10-MDP-calcium salts, through a self-assembled nano-layered interaction which makes the adhesive interface more resistant to degradation over time.[13]

In the present study, No statistically significant differences were reported between Clearfil SE Bond (Kuraray) and Tetric N Bond Universal (Ivoclar Vivadent). In regard to Tetric N Bond Universal, this material has a methacrylate carboxylic acid polymer (MCAP). A previous study by Yoshihara et al., showed that MCAP formed hydrolysis-resistant monomer-calcium salts that remained attached to the dentin surface even after being washed. It is believed that the chemical stability of monomer-Ca salts produced by MDP and MCAP contributes to the higher durability.[14] Also, Tetric N bond Universal was applied on the tooth surface and agitated for a minimum of 20s, as recommended by the manufacturers of the adhesive. Active agitation is essential for self-etch adhesives as it may assist in smear layer removal and improve the contact of the acidic monomers with the tooth surface.[15]

However, in the present study Futura Bond DC presented the lowest shear bond strength values which were significantly lower than Clearfil SE Bond and Tetric N Bond Universal. The addition of fillers to the adhesives has been intended to increase their viscosity to prevent the occurrence of too thin adhesive layer, to increase the elastic modulus for providing a flexible intermediate shock-absorber layer that can resist the polymerization shrinkage stress, and distribute the occlusal loads, and accordingly to increase the dentin bond strength.¹⁶ Adding fillers that are larger than the interfibrillar spaces (15-20 nm) of the etched dentin increases the viscosity of the adhesive and also causes filler accumulation occurrence on the top of the etched dentin substrate. Therefore, it might reduce the adhesive penetration into the dentin and produce a defective hybrid layer.[16,17] This could be the reason for the lowest shear bond strength values reported in the present study.

The clinical implication of this study is that both Clearfil SE bond and Tetric N bond Universal in self-etch mode can be considered as better options than Futura Bond DC. Among Clearfil SE and Tetric N Bond Universal in self-etch mode, both can be considered for bonding to dentin. Further studies evaluating the longevity of newer bonding agents conducted *in-vivo* would be relevant.

Conclusion:-

Within the limitations of the present study, we can conclude that Tetric N Bond Universal in self-etch mode showed comparable bond strength to Clearfil SE bond. With regard to Futura Bond DC, lower bond strength values have been reported.

Conflicts Of Interest:

No potential conflict of interest relevant to this article was reported.

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