ISSN: 2581-5989
PubMed - National Library of Medicine - ID: 101738774

International Journal of Dental Science and Innovative Research (IJDSIR)

IJDSIR : Dental Publication Service

Available Online at: www.ijdsir.com

Volume – 2, Issue – 3, May - June- 2019, Page No. : 446 - 455

Effect of Surface Conditioning Methods on Microtensile Bond Strength of Resin Cement to Glass Infiltered Ziriconia-Reinforced Ceramic

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Type of Publication: Original Research Paper

Conflicts of Interest: Nil

Abstract

Aim and objectives: The objective of the study was to evaluate the effect of three surface conditioning methods on the microtensile bond strength of resin cement to a glass infiltrated zirconia reinforced alumina based core ceramic.

Method: 30 blocks of Zirconia In-Ceram were fabricated according to the manufacturer's instructions and duplicated in resin composite. One composite resin block was fabricated for each ceramic block and assigned to one of the three treatment conditions (n=10). Group I (Laboratory Gritblasting): Surface conditioning was done with 110 m aluminum oxide. Group II (Laboratory Silica Coating): Silica coating process was conducted using a laboratory type of air abrasion device in which the specimens were first conditioned by air-abrasion with 110µm grain sized Al_2O_3 particles and with 110µm grain sized SiO_2. Group III (Hydrofluoric Acid-etching): The ceramic substrates were etched with 9.5% hydrofluoric acid gel according to the manufacturer's strict regulations. Following all the three conditioning methods, the blocks were coated with silane coupling agent and the ceramic-composite blocks were then cemented with the resin cement. The samples were subjected to universal testing machine to evaluate the bond strength.

Result: Comparison of the bond strength of the three groups (I, II, III) by one way ANOVA was done. It was seen that there was a statistically significant difference within the groups ($P<0.05$) with Group II that is laboratory silica coating showing the highest mean bond strength (28.23 $\pm$ 1.53 MPa), followed by Group I that is laboratory grit blasting (20.2 $\pm$ 2.33 MPa). Group III that is hydrofluoric acid-etching showed the least mean bond strength (10.41 $\pm$1.46 MPa).

Conclusion: The effect of three surface conditioning methods on the microtensile bond strength of resin cement to a glass infiltrated zirconia reinforced alumina based core ceramic was variable. Roughening the ceramic surfaces with air particle abrasion with 110µm Al_2O_3 followed by silica coating with particle size of 110 µm SiO_2 and silanization prior to cementation provided higher bond strength when compared with air particle abrasion with 110µm Al_2O_3 and silanization. Hydrofluric acid gel used for conditioning the reinforced ceramics showed the least mean shear bond strength.

Keywords: zirconia; surface conditioning, shear bond strength

Introduction

The increased demand for esthetic restorative treatment in dentistry has made dental ceramics an often-used material for both anterior and posterior restorations [1]. Restoring partially destructed teeth using indirect ceramic restorations such as inlays, onlays and laminate veneers has been encouraged by the development of adhesive materials and techniques. Adhesive techniques allow the brittle and "fragile" ceramic to become a reliable tooth-restoration system with an adequate stress distribution to the underlying tooth structure [2, 3]. Chemical etching selectively dissolves the glassy matrix and crystals in ceramics to generate irregular surface topography of microretentive channels [2]. Among the commonly used etchants, hydrofluoric acid seems most effective in creating distinct morphologic changes of deep channels and grooves associated with the increase of bond strength with a composite [10] Ceramics with high crystalline content (aluminum and zirconia oxides) are reported to present more favorable clinical results than feldspathic, leucite, and lithium disilicate ceramics[4]. The increased content of alumina (Al2O3) in feldspathic ceramics led to a significant increase in mechanical properties of these materials, allowing indication for more predictable metal-free restorations in regions where high mechanical strength is needed. Zirconium oxide all-ceramic materials have attractive properties, such as high strength[4, 5] and biocompatibility[6, 7, 8], that permit their use as core materials for all-ceramic crowns[9] and fixed partial dentures (FPDs) [5]. These favorable mechanical properties are due to phase transformation toughening, which increases crack propagation resistance.

Zirconia, a high strength ceramic has been recently introduced as a core material for complete coverage crowns and bridges[6.] An increasing number of all-ceramic materials and systems are currently available for clinical use[9]. Chemical etching selectively dissolves the glassy matrix and crystals in ceramics to generate irregular surface topography of micro retentive channels[2]. Among the commonly used etchants, hydrofluoric acid seems most effective in creating distinct morphologic changes of deep channels and grooves associated with the increase of bond strength with a composite [10] Multiple clinical studies document excellent long-term success of resin-bonded restorations, such as porcelain laminate veneers, ceramic inlays and onlays, resin-bonded fixed partial dentures, and all-ceramic crowns. A strong, durable resin bond provides high retention improves marginal adaptation and prevents micro leakage and increases fracture resistance of the restored tooth and the restoration. Adhesive bonding techniques and modern all-ceramic systems offer a wide range of highly esthetic treatment options.

Bonding of ceramic to dental tissue is based on the adhesion of luting cement to the ceramic substrate, together with the adhesion of luting cement to enamel and/or dentin[9]. Zirconia restorations can be cemented by conventional methods but use of resin-luting agent enhances retention and provides better marginal seal[6]. Establishing a strong and stable bond of Zirconia with resin-luting agent has always proven to be difficult as the material is acid resistant and does not respond to common etching and silanization procedures used for other glass containing ceramics. Therefore, resin-luting cements could not be used for Zirconia based restorations. With surface conditioning increased adhesion between Zirconia and resin-luting agent could be achieved.

Airborne- particle abrasion using fine alumina oxides under pressure is also used to modify the ceramic surface as there is a risk of tissue damage when using hydrofluoric acid. The procedure removes relatively weaker phases to create an irregular rough surface and to increase surface area for bonding. Meanwhile, it has been suggested that a combination of airborne-particle abrasion followed by etching would provide a better surface for composite-ceramic bonding because neither etching or airborne-particle abrasion alone generates enough mechanical retentive characteristics on ceramic surface for clinical longevity[11].However, there is limited knowledge as to whether micromechanical retention using large or small particle size increases resin bond to high-strength ceramics of different microstructures and chemical compositions[12]. There are very few studies on the bond strength of resin cements to the zirconium based ceramics in combination with conditioning methods. The aim of this study was to evaluate the effect of three surface conditioning methods on the micro tensile bond strength of resin cement to a glass infiltrated zirconia reinforced alumina based core ceramic.

Material and methods

30 blocks of Zirconia In-Ceram (5mm×5mm×4mm) (Vita Zahnfabrik, Bad Saeckingen, Germany) were fabricated using Silicon Carbide Abrasive (3M, St Paul, USA) in a polishing machine (Labpol 8-12,Extec, USA) . The specimens were polished and cleaned in ultrasonic bath (Sidilu Ultrasonics) for 10 min and air dried. Each ceramic block was duplicated in composite resin (3M/ESPE, St. Paul,USA) using a mould made out of silicon impression material (Express, 3M/ESPE, St. Paul, USA). Composite resin layers were incrementally condensed into the mould to fill up the mould and each layer light polymerized with Light curing unit (SELECTOR LA500, Apoza Enterprise Co). One composite resin block was fabricated for each ceramic block and assigned to three different treatment conditions (n=10).

Groups

1. Laboratory Grit blasting: Here air-borne particle abrasion was performed using air abrasive device (Carlo de giorgi, Italy) at mean particle size 110um Al_2O_3 at a pressure of 2.8 bars from a distance of 10mm for 20 second in circling movements.
2. Laboratory Silica Coating (LSC): Silica coating process was conducted using a laboratory type of air abrasion device (3M ESPE AG, Seefeld, Germany)in which the specimens were first conditioned by air-abrasion with 110um grain sized Al_2O_3 particles at a pressure of 2.8 bars. Then the specimens were air-abraded with 110um grain sized SiO_2, at 2.8 bars from a distance of approx. 10mm, for 20s in circling movements.
3. Hydrofluoric Acid-etching: The ceramic substrates were etched with 9.5% hydrofluoric acid gel 9.5% hydrofluoric acid (Ultra dent) for 90 s according to the manufacturer's strict regulations. The ceramic surfaces were etched in the laboratory under ventilation, wearing acid-resistant gloves and protective glasses. The etching gel was then rinsed in a polyethylene cup and the diluted solution neutralized using the neutralizing powder (calcium carbonate, $CaCo_3$ and sodium carbonate, Na_2Co_3) for 5 min and washed thoroughly for 20 s, the etched substrates were then washed and rinsed thoroughly to remove the residual acid after etching and air-dried.

Following all the three conditioning methods, the remnants of sand particles were gently air blown and coated with silane coupling agent (3M ESPE AG, Seefeld, Germany) and waited for its evaporation for 5 min. The ceramic-composite blocks were then cemented with the resin cement (3M Relyx) and stored at $37^{o}C$ in distilled water for 7 days prior to bond tests. The blocks were then bonded with cyanoacrylate glue to a metal base that is coupled to a cutting machine. Slices were obtained using a slow-speed diamond wheel saw under cooling. The peripheral slices were disregarded in case the results could be influenced by either the excess or insufficient amount of resin cement at the interface. Three slices were obtained per block initially. The slices were then rotated 90 degrees and bonded onto the metal base again. The peripheral bar specimens were also disregarded for the same reasons described above. Other 3 sectioning were also carried out. The blocks were then cut under coolant water to produce bar specimens with a bonding area of approximately 0.6 mm^2.

Testing the shear bond strength

The specimens were held horizontally in the lower member of shear chuck available especially for testing the shear in Universal Testing Machine (LR 50K: Lloyd Instrument ltd, U.K). The inner surface configuration of the chuck was such that it could firmly grip the specimens once placed and tightened. A knife edge shearing chisel was employed to debond the prepared specimens. The chisel was then mounted on to the upper member of the shear chuck. The cutting edge of the chisel was then engaged at zirconia-composite interface and force applied perpendicular to the long axis of the specimen. The equipment was operated at a crosshead speed of $1min^{-1}$ and the maximum load to debond the specimens was recorded in Newton. Shear bond strength is calculated in Mega Pascals by the ratio of maximum load in Newton to the cross-sectional area of the bonded interface in millimeters.

The statistical method employed in the present study was Analysis of Variance. The One-Way ANOVA procedure produces a one way analysis of variance for a quantitative dependent variable by a single factor (independent) variable. Analysis of variance is use to test the hypothesis that several means are equal.

Statistical analysis

Statistical analysis was performed using two way analysis of variance (ANOVA) with micro tensile bond strength as the dependent variable and surface conditioning methods and the storage condition as the independent factors (statistics 8.0 for windows, Analytical software Inc., Tallahassee, FL, USA). Multiple comparisons were made by Turkey's adjustment test. Furthermore, one-way ANOVA was used to determine the significant differences between surface conditioning methods.

Decision Criterion: We compare the P-Value with the level of significance. If $P<0.05$, we reject the null hypothesis and accept the alternate hypothesis. If $P\geq0.05$, we accept the null hypothesis.

Results

30 blocks of Zirconia In-Ceram were fabricated according to the manufacturer's instructions and duplicated in resin composite. One composite resin block was fabricated for each ceramic block and assigned to one of the three treatment conditions (n=10).

The data obtained from this study was evaluated and comparisons were made within the group and between the groups I, II and III.

Means, standard deviations, coefficient of variation of shear bond strength and level of significance in three groups (I, II, III) were determined. ANOVA test was done to find the significance of study parameters within groups and between groups. Pair wise comparison of three groups (I, II, III) with respect to mean bond strength was done by Turkey's multiple post hoc procedure. P value < 0.05 indicated significance.

The mean bond strength and standard deviations in each group. Group II that is laboratory silica coating showed the highest mean bond strength (28.23 ± 1.53 MPa), followed by

Group I that is laboratory gritblasting (20.2 ± 2.33 MPa).

Group III that is hydrofluoric acid-etching showed the least mean bond strength (10.41 ±1.46 MPa).Comparison of mean bond strength of the three groups (I, II, III) by one way ANOVA was also done.

It was seen that there was a statistically significant difference within the groups ($P<0.05$). Intergroup comparison also showed a significant difference ($P<0.05$). When pair wise comparison of mean bond strength of the three groups (I, II, III) by Turkey's multiple posthoc procedure was done, it was seen that there was a statistically significant difference between the pairs (Group I and Group II, Group II and Group III, Group III and Group I).

Discussion

A good adhesion to the dental tissues has been achieved successfully over a period of time. Current research efforts are also aimed at the optimization of the adhesion between composites to metals (alloys), composites to ceramics and composites to other composites. A number of surface conditioning methods have been developed over the last few decades to produce adequate adhesion to the adherent for restorations.

The present study examined the effect of different surface conditioning methods by determining bond strength of a resin cement to glass infiltrated zirconia ceramics. In this study, roughening the zirconia-reinforced ceramic surfaces with air particle abrasion and applying silane prior to cementation provided high bond strengths and silica coating followed by silanization evidently enhanced the bond between the luting cement and the ceramic surfaces. The silica layer left by silica coating on the ceramic surface provides a basis for silane to react. In the ceramic-resin bond, silane functions as a coupling agent, which adsorbs onto and alters the surface of the ceramic, thereby facilitating chemical interaction[12]. Oyagüe et al. noted a statistically significant decline in bond strength of MDP containing resin cement after 6 months of water storage when luting to either untreated or sandblasted zirconia surfaces; in contrast, no significant bond strength variability was seen for this type of resin cement after 6 months of water exposure when luting to silica-coated zirconia surfaces [13] . When the surface is air abraded, this would generate more hydroxyl groups on the surface and also enhance the micro-mechanical retention. Furthermore, the methoxy groups of silane would react with water to form silanol groups that in turn, will react with the surface hydroxyl groups to form siloxane network. Amphoteric alumina in the ceramic matrix could form chemical adhesion, covalent bridges, through its surface hydroxyl groups with hydrolyzed silanol groups of the silane:–Al–O–Si[6].

The In-Ceram ceramic system tested in this study, In-Ceram Zirconia (INC-ZR), is glass infiltrated. Most probably the glass infiltration facilitated better silane bonding and therefore increased bond strength values were obtained for this ceramic. These findings are in compliance with the study of Ozcan and Vallittu[6], even though a different experimental set up was used where a bis-GMA based resin cement and shear bond test were employed

Material selection and clinical recommendations on resin bonding are based on mechanical laboratory tests that show great variability in materials and methods. One of the most common testing method is the shear bond test. However the specific fracture pattern in shear testing may cause cohesive failure in the substrate that may lead to erroneous interpretation of the data while in micro tensile tests, stress distribution was reported to be more homogeneous[9]. Although, for this reason, micro tensile test was employed in this study, similar ceramic-cement performance was observed in dry conditions in the study of Ozcan and Vallittu[6].

The results of this study indicated that sandblasting with 110µm Al_2O_3 followed by silica coating with particle size of 110 µm SiO_2 produced statistically higher mean bond strength values than with chairside grit blasting using 110µm grain sized Al_2O_3 particles alone.

In another study by Valandro et al[14], ten blocks (5x6x8 mm^3) of In Ceram Zirconia and Procera ceramics were fabricated according to each manufacturer's instructions and duplicated in composites. The specimens were assigned to one of the two following treatment conditions: (1) airborne particle abrasion with 110-µm Al_2O_3 particles + silanization, (2) silica coating with 30µm SiO_2 particles (CoJet, 3M ESPE) + silanization. Each ceramic block was duplicated in composite resin. The composite blocks were bonded to the surface-conditioned ceramic blocks using a resin cement system (Panavia F, Kuraray, Okayama, Japan). One composite resin block was fabricated for each ceramic block. The bond strength tests were performed in a universal testing machine. Bond strength values were statistically analyzed using two-way ANOVA and Tukey's test (≤ 0.05). It was found that Silica coating with silanization increased the bond strength significantly for all three high-strength ceramics (18.5 to 31.2 MPa) compared to that of airborne particle abrasion with 110-µm Al_2O_3 (12.7-17.3 MPa) (ANOVA, $p < 0.05$).

In our study also, we found the highest shear bond strength in Group II (Laboratory Silica Coating) followed by Group I (Laboratory Gritblasting). The differences were found to be statistically significant ($P< 0.05$).

Ozcan M[9] in 2003 conducted a study where different conditioning methods were tested to promote the adhesion between some of the substrates and the resin composites. In this study, HF acid etching demonstrated satisfactory results for ceramics with glassy matrix.

The use of silica coating followed by silanization or acrylization (application of silanization in a special furnace) was not only very effective on reinforced ceramics but also gave very promising results when they were employed on titanium posts prior to adhering resin based core materials. One of the initial steps after all the surface treatment tested in this thesis, involves silane coupling agent application. Application of silane coupling agent to the preconditioned surfaces provides a covalent and hydrogen bond. The general outcome of this study suggested that in contrast to what was being regularly applied for adhesion principles creating macro-mechanical principles, relatively recent surface conditioning techniques based on a combination of micromechanical retention and chemical coating should be considered for durable restorations applied in adhesive dentistry[9].

Although HF acid gel worked fairly well in terms of receiving high bond strength on glass matrix ceramics, the results were poor when it was used for conditioning the reinforced ceramics[15-19.] Etching ceramics or composites with HF acid is a practical option since it can be easily applied at the chair side without any requirement of additional devices. However, it has certain drawbacks as it may lead to serious clinical manifestations. HF acid gel together with HCl (hydrochloric acid, a very strong acid) belongs to the group of hydrogen halogens.

These are corrosive, hazardous and cytotoxic acids. If HF vapors are inhaled, they can cause lung edema and after weeks, liver and kidney insufficiency can appear[20]. When compared to the above study, our study too showed similar findings.

Group III i.e. Hydrofluoric acid etching showed the least mean shear bond strength between the groups which was statistically significant ($P< 0.05$).

Qeblawi DM, Muñoz CA et al conducted a study [21] to evaluate the effect of mechanical surface treatment of yttria-partially stabilized zirconia on its flexural strength and the effect of mechanical and chemical surface treatments on its bond strength to a resin cement. For flexural strength evaluation, zirconia bars (4 x 5 x 40 mm) were prepared from zirconia blocks, finished using a diamond rotary cutting instrument, sintered, then assigned into 4 groups: (1) control (no treatment), (2) airborne-particle abrasion, (3) silicoating, and (4) wet hand grinding. Mechanical treatment included: (1) control (no treatment), (2) airborne-particle abrasion, (3) silicoating, or (4) wet hand grinding. Chemical treatment included: (1) control (no treatment), (2) acid etching followed by silanation, (3) silanation only, or (4) application of zirconia primer. Zirconia rods were bonded to dentin using a resin cement (Multilink Automix), then light polymerized. After storage, the specimens were loaded to failure with the notched shear bond test method in a universal loading apparatus. It was seen that airborne-particle abrasion and hand grinding significantly increased flexural strength. The highest shear bond strength values were achieved for the following groups:

silicoated + silanated > hand ground + zirconia primer > airborne-particle abraded + silanated >zirconia primer > airborne-particle abraded + zirconia primer.

It was concluded that mechanical modification of the surface increased the flexural strength of Y-TZP.In our study also we found the mean bond strengths in the following order: silicoated + silanated > airborne-particle abraded + silanated.

In another study by Yun JY [22] on effect of sandblasting and various metal primers on the shear bond strength of resin cement to Y-TZP ceramic found out that the bond strength of the specimens treated with sandblasting and metal primer (Alloy primer) was significantly higher than those of the other subgroups.

Chemical modification by acid etching followed by silane coating had the least bond strength. From the results of our study, clinical implications that could be derived are:

Relatively recent surface conditioning techniques based on the combination of micromechanical and chemical conditioning should be considered for improved adhesion of resin cements to glass-infiltrated zirconia ceramics.

More importantly, these methods seem to offset the importance of the varieties of the substrates and therefore could be applicable to a wide range of high-strength ceramics.

The equipment's used to apply these techniques were sophisticated and expensive during the last two decades but they are recently simplified and brought to the chair side.

The limitation of the study was that it is an in-vitro study. Although all possible efforts have been made to best simulate clinical situations, at best an in-vitro study can be used to rank performance and give an indication of likely clinical performance.

By employing chair side devices for airborne particle abrasion, contamination during delivery of the restoration from the laboratory to chair side could also be avoided.

As long as the available conditioning methods will not be optimized, the development in the high-strength ceramic field is expected to continue experiencing failures.

Conclusion

The effect of three surface conditioning methods on the micro tensile bond strength of resin cement to a glass infiltrated zirconia reinforced alumina based core ceramic was variable.

Roughening the ceramic surfaces with air particle abrasion with 110µm Al_2O_3 followed by silica coating with particle size of 110 µm SiO_2 and silanization prior to cementation provided higher bond strength when compared with air particle abrasion with 110µm Al_2O_3 and silanization.

Although Hydrofluoric acid gel worked fairly well in terms of receiving high bond strength on glass matrix ceramics, the results were poor when it was used for conditioning the reinforced ceramics as it showed the least mean shear bond strength.

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Legends Table and Figures

Sample No.	Result		
	Group -I	Group -II	Group -III
1	17.3	25.5	9
2	16.7	27	8.8
3	20.7	27.3	10.6
4	23	26.9	11.1
5	20.9	28.6	12.2
6	23.5	29.5	12.7
7	19.1	28.9	8.3
8	21.4	29.1	10.2
9	18.3	28.8	9.9
10	21.8	23.7	11.3

Table 1: Shear bond strength in MPa of the test samples in the three groups

N	Mean	SD	SE	CV
GROUP I				
10	20.27	2.33	0.74	11.50
GROUP II				
10	28.23	1.53	0.48	5.41
GROUP III				
10	10.41	1.46	0.46	13.99

Table 2: Mean, SD, SE and coefficient of variation of the bond strength in three groups (I, II, III)

Sum of squares	Degrees of freedom	Mean sum of squares	F-value	p-value
BETWEEN GROUPS				
1593.78	2	796.89	241.8317	0.00001*
WITHIN GROUPS				
88.97	27	3.30		
TOTAL				
1682.75	29			

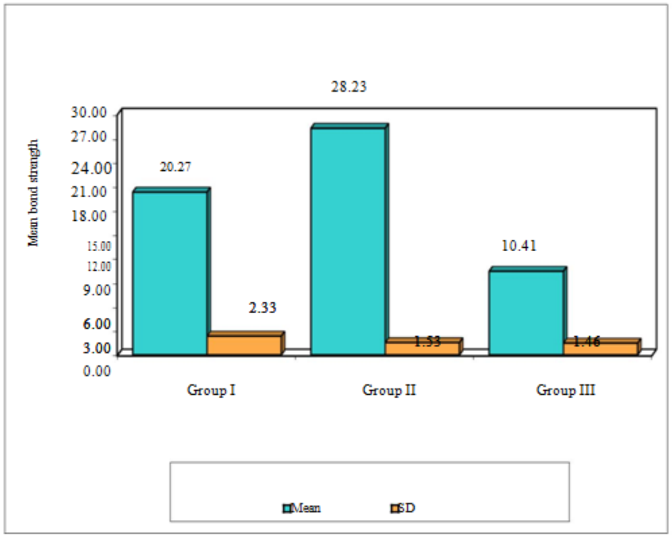
*p<0.05

Table 3: Comparison of the bond strength of three groups (I, II, III) by one way ANOVA

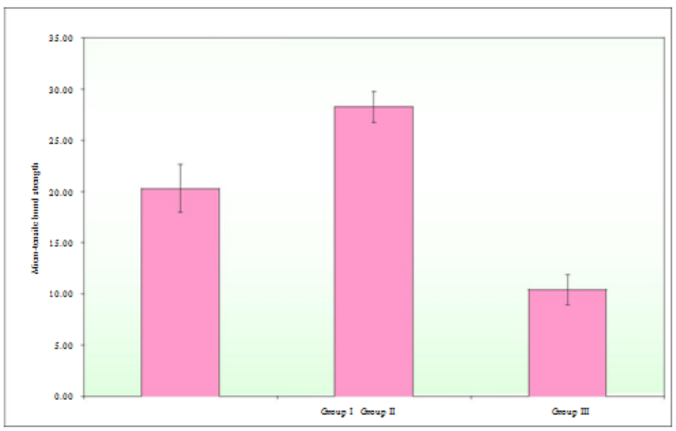
GROUPS	GROUP I	GROUP II	GROUP III
Mean	20.2700	28.2300	10.4100
SD	2.3310	1.5268	1.4564
GROUP I	-		
GROUP II	P=0.0001*	-	
GROUP III	P=0.0001*	P=0.0001*	-

*p<0.05

Table 4: Pair wise comparison of the bond strength of three groups (I, II, III) by Tukeys multiple posthoc procedure



Graph 1: Comparison of mean bond strength of the three groups (I, II, III)



Graph 2: Comparison of mean bond strength of the three groups (I, II, III)

Master Chart

SL NO	GROUP I (Alumina + Silane)			GROUP II (Alumina + Silica + Silane)			GROUP III (Hydrofluric Acid + Silane)		
	SBS MPa	MEAN	SD	SBS MPa	MEAN	SD	SBS MPa	MEAN	SD
1.	17.3	20.27	2.33	25.5	28.23	1.53	9	10.41	1.46
2.	16.7			27			8.8		
3.	20.7			27.3			10.6		
4.	23			26.9			11.1		
5.	20.9			28.6			12.2		
6.	23.5			29.5			12.7		
7.	19.1			28.9			8.3		
8.	21.4			29.1			10.2		
9.	18.3			28.8			9.9		
10	21.8			30.7			11.3		