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RESEARCH ARTICLE

COMPARATIVE EVALUATION OF SHEAR BOND STRENGTH OF TWO DIFFERENT RESIN CEMENT TO ZIRCONIA AFTER SURFACE TREATMENT USING ER:YAG LASER: AN IN VITRO STUDY

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Abstract

Zirconia (ZrO_2), a crystalline dioxide of zirconium, boasts metal-like mechanical properties and a tooth-like color. Creating a ZrO_2 core for prosthetic restorations necessitates a CAD/CAM system adept at handling zirconia. Resin-bonded luting is the top choice for zirconia ceramic restorations, with tribochemical treatment preferred for surface preparation before resin cement luting. Clinical trials indicate that resin cementation improves adhesion and mechanical properties of zirconia restorations. While studies on sandblasting's effect on shear bond strength to zirconia exist, more data is needed on the impact of sandblasting and laser treatment on yttria-stabilized tetragonal zirconia. This study examines the shear bond strength of two resin cements to zirconia after Er:YAG laser treatment.

Introduction:-

The use of high-strength zirconia in modern dentistry has significantly improved durability and longevity over traditional materials like porcelain. However, challenges remain in bonding zirconia restorations with conventional cementation methods. Since successful prostheses depend on effective cementation, enhancing zirconia bonding techniques is crucial for retention and long-term stability.[1]

Modifying zirconia surfaces improves bonding with substrates in dental restorations. Techniques like mechanical roughening, chemical modification, tribochemical silica coating, laser treatment, etching, and plasma treatment enhance adhesion by increasing surface roughness, energy, and wettability. These methods strengthen zirconia's bond with cement, ensuring long-term success in prosthetic applications.[2]

ZrO_2 crystals can adopt three distinct patterns: monoclinic (M), cubic (C), and tetragonal (T). Mixing ZrO_2 with other metallic oxides like MgO, CaO, or Y_2O_3 enhances molecular stability significantly. Among them Yttrium-stabilized zirconia (TZP) is popular for its strength, biocompatibility, stability, and wear resistance, ideal for prosthetics.[2,3]However, zirconia's surface stability presents challenges in forming durable bonds with cements, crucial for long-lasting dental restorations.[4]

Optimal resistance and retention in tooth preparation are critical, but dental cement plays a key role in preventing microbial leakage. It seals the interface between tooth and restoration, providing mechanical, chemical, or combined surface attachment. An ideal adhesive offers strong bonding, durability, tissue compatibility, and resistance to disintegration, ensuring proper restoration seating.[5,6]Surface treatment is essential for optimal adhesion between the

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luting agent and ceramic surfaces. While hydrofluoric acid etching and silane coupling agents improve bond strength in silica-based ceramics, they are ineffective on zirconium and alumina ceramics. Their high crystalline content makes them resistant to acid etching, hindering bond strength improvement.[7]

In recent advances Laser irradiation has been studied as a method to enhance zirconia's surface morphology and chemistry for better bonding. By creating microtextured surfaces and increasing surface energy, laser treatment, particularly Erlaser, has shown to improve zirconia's bond strength to resin cements, making it a promising option in fixed prosthodontics.[8]

Thus, the need for this study was to evaluate the shear bond strength of two different resin cements to zirconia after surface treatment using Er:YAG laser.

Aims And Objectives:-

The aim of this study was to evaluate the shear bond strength of two different resin cements to zirconia before after surface treatment using Er:YAG laser.

Methodology:-

Fabrication of zirconia blocks:

A wax pattern was prepared with dimensions of 10x10x4 mm using modelling wax (Figure 1). A putty index of the wax pattern was prepared using addition silicone elastomeric impression material. Die stone was poured into the putty index. The final block of die stone (Figure 2) thus obtained was sandpapered and scanned using an intra oral scanner. The scanned 3D image was used to fabricate the zirconia test specimens (Figure 3).

Fabrication of test specimens:

Forty standard test specimens of zirconia block of 10×10×4 mm were fabricated using CAD/CAM technique by dry milling followed by sintering. A metal mould of dimension 35x15x15mm was used to make auto polymerising resin blocks. Auto polymerising resin was mixed according to manufacturer’s instructions into the metal mould in which zirconia blocks were embedded before start of polymerization. During this process of embedding, one surface of zirconia is exposed which would be surface treated later (Figure 4).

Surface treatment of the Zirconia blocks:

All the forty zirconia specimens were surface treated with sand blasting using 50 µm aluminium oxide particles for two minutes under two bar pressure, at a distance of 10mm.

Fabrication of composite rods:

Forty composite rods (2.5 mm diameter × 3 mm length) were prepared using customised clear teflon mould and were light cured (Figure 5). These composite rods were fabricated by compacting the Tetric N Ceram composite material within the customized clear teflon mould and were light cured for 30 seconds.

Grouping of the zirconia specimens:

The forty sandblasted zirconia specimens were grouped as follows. The group I comprised of usage of glass powder treated with silane resin cement RelyX U200, group II comprised of BIS-GMA based resin cement Multilink N. Among the classified groups, the specimens of Group IA and Group IIA were not subjected to laser treatment. Whereas the specimens of Group IB and Group IIB were subjected to laser treatment using Erbium with yttrium aluminium garnet (Er:YAG) laser. The specimens of Group 1B and Group IIB were further treated with hard tissue Er:YAG laser with power output of 4W, pulse penetration rate of 10Hz, energy of 400mJ for 10 seconds.

Group	Number of specimens	Type of resin cement	Surface treatment of zirconia
Group IA	10	RelyX U200	Without laser
Group IB	10	RelyX U200	With laser
Group IIA	10	Multilink N	Without laser
Group IIB	10	Multilink N	With laser

Preparation of Group IA samples:
Ten zirconia specimens were subjected to sandblasting as mentioned above. Z-PRIMETM PLUS primer was applied onto the sandblasted surface and cured using light polymerizing unit for 30 seconds. RelyX U200 cement was dispensed from the clicker dispenser onto the mixing pad according to manufacturer's instructions. The base and catalyst were mixed for 30 seconds following which it was applied onto the zirconia block. Composite rod was placed on the cement, (Figure 6) excess resin cement was removed with an explorer before polymerization and light curing was performed for 20 seconds using intensity of 600 mW/cm2, with the light directed approximately 45 degrees from the intersection of the zirconia bonding site and composite rod (Figure 7).

Preparation of Group IB samples:
Ten zirconia specimens were subjected to sandblasting as mentioned above. The sandblasted surface was additionally subjected to laser (Figures 8 & 9) (Er:YAG laser, 2490nm with power output of 4W, pulse penetration rate of 10Hz, energy of 400mJ for 10 seconds). Z-PRIMETM PLUS primer was applied onto the sandblasted and laser treated surface and cured using light polymerizing unit for 30 seconds. RelyX U200 cement was dispensed from the clicker dispenser onto the mixing pad according to manufacturer's instructions. The base and catalyst were mixed for 30 seconds following which it was applied onto the zirconia block. Composite rod was placed on the cement. Excess resin cement was removed with an explorer. Light curing was performed for 20 seconds using intensity of 600 mW/cm2, with the light directed approximately 45 degrees from the intersection of the zirconia bonding site and composite rod.

Preparation of Group IIA samples:
Ten zirconia specimens were subjected to sandblasting as mentioned above. Z-PRIMETM PLUS primer was applied onto the sandblasted surface and cured using light polymerizing unit for 30 seconds. Multilink N cement was dispensed from the clicker dispenser onto the mixing pad. The base and catalyst were mixed for 30 seconds following which it was applied onto the zirconia block. Composite rod was placed on the cement. Excess resin cement was removed with an explorer. Light curing was performed for 20 seconds using intensity of 600 mW/cm2, with the light directed approximately 45 degrees from the intersection of the zirconia bonding site and composite rod.

Preparation of Group IIB samples:
10 zirconia specimens will be subjected to sandblasting as mentioned above. The sandblasted surface will additionally be subjected to laser (Er:YAG laser, 2490nm with power output of 4W, pulse penetration rate of 10Hz, energy of 400mJ for 10 seconds). Z-PRIMETM PLUS primer was applied onto the sandblasted and laser treated surface and cured using light polymerizing unit for 30 seconds. Multilink N cement was dispensed from the clicker dispenser onto the mixing pad. The base and catalyst were mixed for 30 seconds following which it was applied onto the zirconia block. Composite rod was placed on the cement. Excess resin cement was removed with an explorer. Light curing was performed for 20 seconds using intensity of 600 mW/cm2, with the light directed approximately 45 degrees from the intersection of the zirconia bonding site and composite rod.

All the zirconia specimens from all the groups were subjected to shear bond strength test using universal testing machine (UTM) and the values obtained were tabulated and assessed (Figure 10).

Testing of specimens:
Each specimen was mounted on the lower fixture of a universal testing machine. The bonded composite rods were then subjected to a shear force of 100 KN at a crosshead speed of 2 mm/min until fracture occurred and shear bond strengths were calculated using the formula:

$\sigma = P/A$

where σ is the tensile bond strength (MPa), P is the maximum force (N) and A is the interfacial area (mm2).
Statistical analysis: Two-way ANOVA was used to analyse the shear bond strength; followed by Tukey – Kramer post hoc test to compare between the mean values and identify the greater differences among the mean values. Statistical analysis was performed using the SPSS software at a significance level of 5%(p=0.05).

Evaluation of surface roughness: Surface roughness was assessed using scanning electron microscope (SEM). Surface roughness of sandblasted zirconia specimens were compared with sandblasted and laser treated zirconia specimens. The specimens were analysed under 2500 magnification.

Results:-

This in vitro study was carried out to evaluate the shear bond strength of two different resin cements to zirconia after surface treatment using Er:YAG laser. The study compromised of 40 zirconia specimens which were sandblasted and divided into two groups based on the different resin cements used. Further each cement group was divided based on the use of Er:YAG laser.

The following tests were observed:
Shear bond strength of the specimens:
Table 1 and Graph 1:
The mean shear bond strength for Group IA was 15.384 ± 1.051MPa, Group IB was 18.746 ± 0.666 MPa, Group IIA was 12.579 ± 0.736 MPa and for Group IIB was 17.122 ± 0.780MPa. This difference in the mean shear bond strength between 4 groups was statistically significant at $p<0.001$.

Table 2 and Graphs 2 – 8:
Depict multiple comparison of mean difference between groups revealed that the Group IB showed significantly highest mean shear bond strength as compared to Group IIB, Group IA & Group IIA and the mean differences were statistically significant at $p<0.001$ respectively. This was then later followed by Group IIB which showed significantly higher mean shear bond strength as compared to Group IA & Group IIA and the mean difference were statistically significant at $p<0.001$ respectively. This was then followed next with Group IA showing significantly higher mean shear bond strength as compared to Group IIA and the mean difference was statistically significant at $p<0.001$. This infers that the mean shear bond strength was significantly highest in Group IB followed by Group IIB, Group IA and least in Group IIA.

Sem Analysis:
The SEM analysis of zirconia specimen after sandblasting and Er:YAG laser surface treatment was compared with sandblasted zirconia specimen under 2500 magnification. Morphological analysis of the zirconia specimen without laser treatment showed densely sintered particles of zirconium oxide possessing some natural undercuts (Figure 11). However, the zirconia specimens with Er:YAG laser surface treatment showed significant surface irregularities. The laser irradiated zirconia surface showed altered surface with significant grooving and sharp edges (Figure 12). They also showed deep voids and sharp edges in a mesh like pattern showing surface ablation following the laser surface treatment.

Discussion:-

The clinical success of ceramic restorations is influenced by several factors, including the cementation procedure. The restoration can be secured using either conventional methods (zinc phosphate or glass ionomer cements) or more advanced adhesive systems and resin cements, each with its own advantages and challenges.[9]

The effectiveness of indirect ceramic restorations largely depends on the characteristics of the cement used and the preparation of the ceramic surface before applying the cement. The right combination of luting agent properties and proper surface preparation techniques is crucial for achieving a strong and durable bond, ensuring the long-term success of the restoration.[10, 11, 12]

Conventional luting cements have not reduced the fracture resistance of zirconia restorations. Tribochemical treatment is now favored before resin cementation, which enhances adhesion and mechanical properties. While studies have explored sandblasting’s impact on shear bond strength, this research evaluates the shear bond strength of two resin cements to zirconia after Erlaser treatment.

The two self-adhesive resin cements used in this study were RelyX U200 and Multilink N resin cements. RelyX U200 resin cement is a dual-cure system featuring a base and catalyst, offering excellent bonding and mechanical properties. The base contains silane-treated glass powder, phosphoric acid, and TEGDMA, while the catalyst includes methacrylate monomers. It provides strong adhesion, reduced sensitivity, easy handling, moisture tolerance, and quick excess removal, ideal for various restorations.Multilink N is a dual-cure resin cement with a monomer matrix of BIS-GMA, dimethacrylate, and HEMA, and inorganic fillers like barium glass, ytterbium trifluoride, and spheroid mixed oxide. It provides high flexural and bond strength, low water solubility, easy excess clean-up, and high radiopacity, making it suitable for various ceramic and alloy restorations.[13]

Laser technology has become an advanced tool in dentistry, offering safe and effective ways to modify material surfaces. Its applications include reducing microleakage, etching metals for porcelain application, enhancing the surface roughness of Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP ceramics), and processing dental materials by fusing them onto or into tooth surfaces. The Erbium(Er) laser is widely used in clinical dentistry, where its effects on dental substrates are well-documented. This laser converts energy into heat upon substrate interaction, leading to particle removal through "ablation"—a process involving microexplosions and vaporization that reshapes the substrate.[8, 14]

Numerous studies have investigated the shear bond strength of zirconia bonded with different luting agents and several of these studies have consistently shown that self-adhesive resin cements outperform other luting agents, yielding significantly higher bond strength values when bonding to zirconia ceramics.[15, 16, 17, 18]

The results of the study showed that both resin cements tested in the study demonstrated significantly higher shear bond strength to the laser-treated zirconia surface as compared to the untreated group. However, there was no significant difference in bond strength between the two different resin cements. The results suggested that Er:YAG laser surface treatment effectively improved the bond strength of zirconia to resin cements.

However, the study encountered certain limitations. The in vitro testing cannot perfectly replicate clinical scenarios in every aspect. Moreover, when assessing adhesive systems in vitro, it's essential to consider various clinically relevant factors such as cavity or crown preparation configuration, dentin moisture, pulpal pressure, remaining dentin thickness, and the type of dentin (normal or sclerotic). Ultimately, the comprehensive evaluation of material performance should rely on long-term clinical studies that account for a myriad of parameters, including individual clinical variables. Furthermore, studies are to be conducted in-vivo using various methods of surface treatments, bonding protocols, along with evaluation of success of these restorations with long term follow-up.

Figures

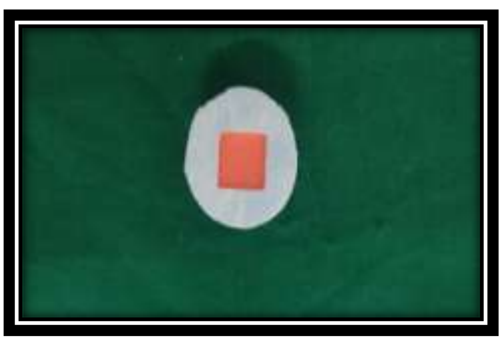


Figure 1:- Mock specimen made in wax embedded/mounted on scanner disc.

Figure 2:- Specimen made of die stone

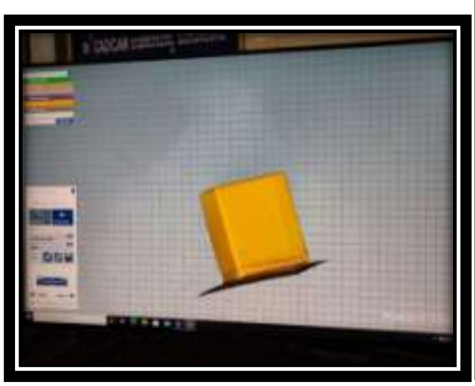


Figure 3:- The scanned image refined embedded in acrylic resin blocks

Figures 4:- Forty zirconia specimens

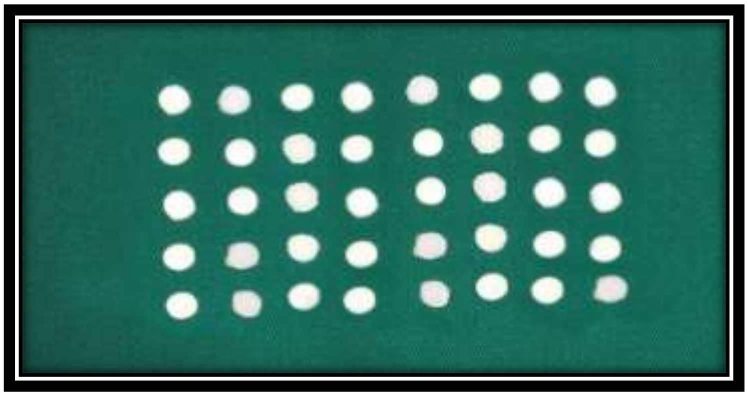


Figure 5:- Forty composite rods.

Figure 6:- Composite rod placed on zirconia specimen using resin cement

Figure 7:- Light curing of composite rod to zirconia specimen.

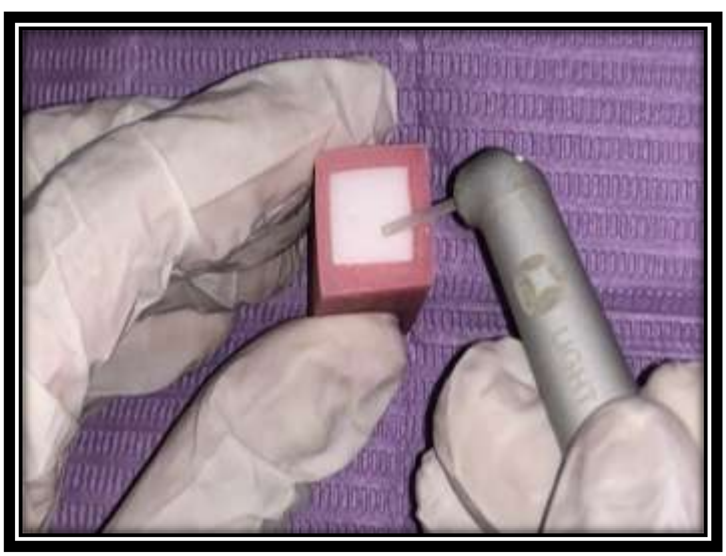


Figure 8:- Setting laser parameters.

Figure 9:- Zirconia surface treated using laser.

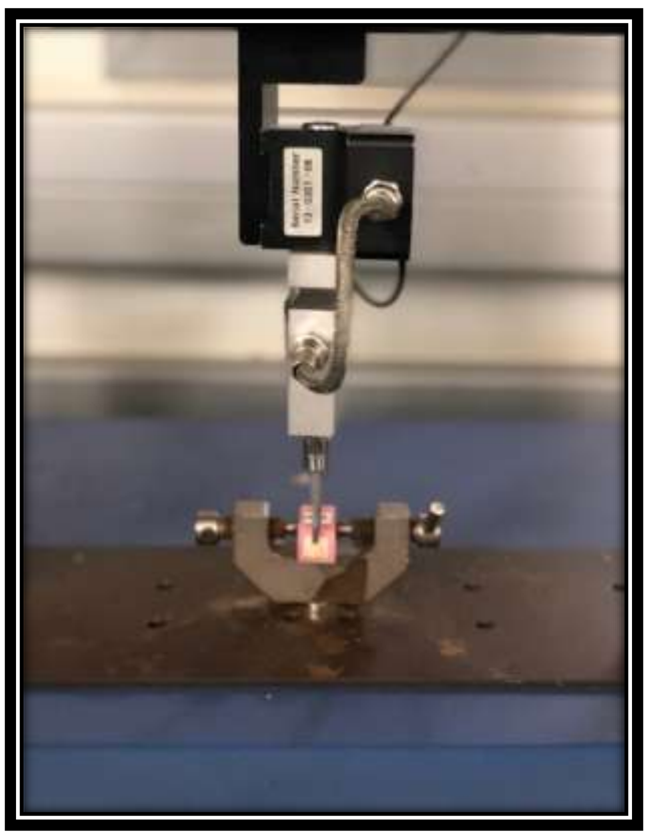


Figure 10:- Zirconia specimen subjected to shear test.

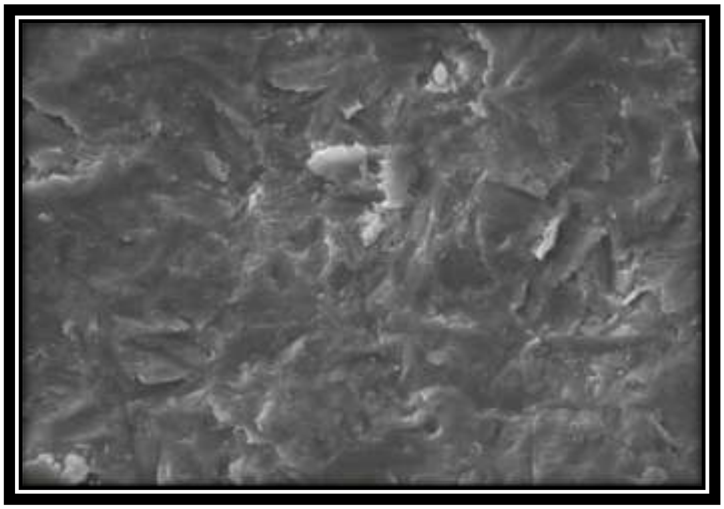


Figure 11:- Zirconia specimen surface after sand blasting with 50 micron aluminium oxide particles under SEM 2500x magnification.

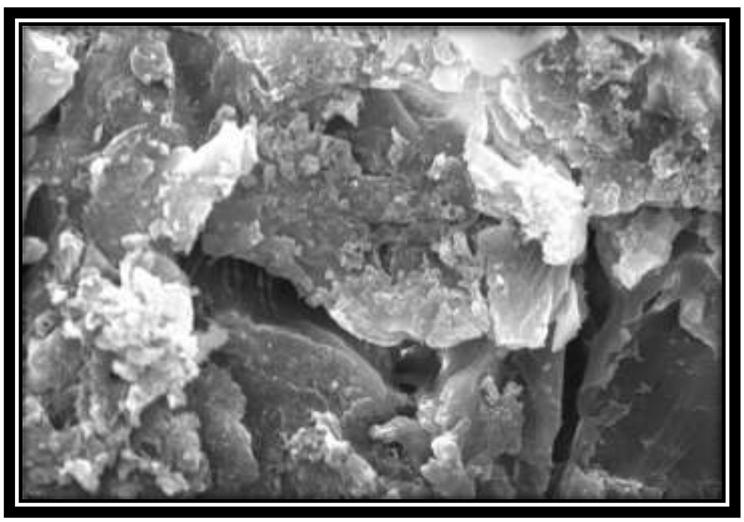


Figure 12:- Zirconia specimen surface after surface treatment with 50 micron aluminium oxide particles and Er:YAG laser under SEM 2500x magnification.

Tables and Graphs

Tables:-

Table 1. Comparison of mean shear bond strength (in MPa) between 4 groups using One-way ANOVA Test

Groups	N	Mean	SD	Min	Max	p-value
Group IA	10	15.384	1.051	14.22	17.07	<0.001*
Group IB	10	18.746	0.666	17.72	19.77	
Group IIA	10	12.579	0.736	11.47	13.51	
Group IIB	10	17.122	0.780	16.07	18.17	

***Statistically significant**

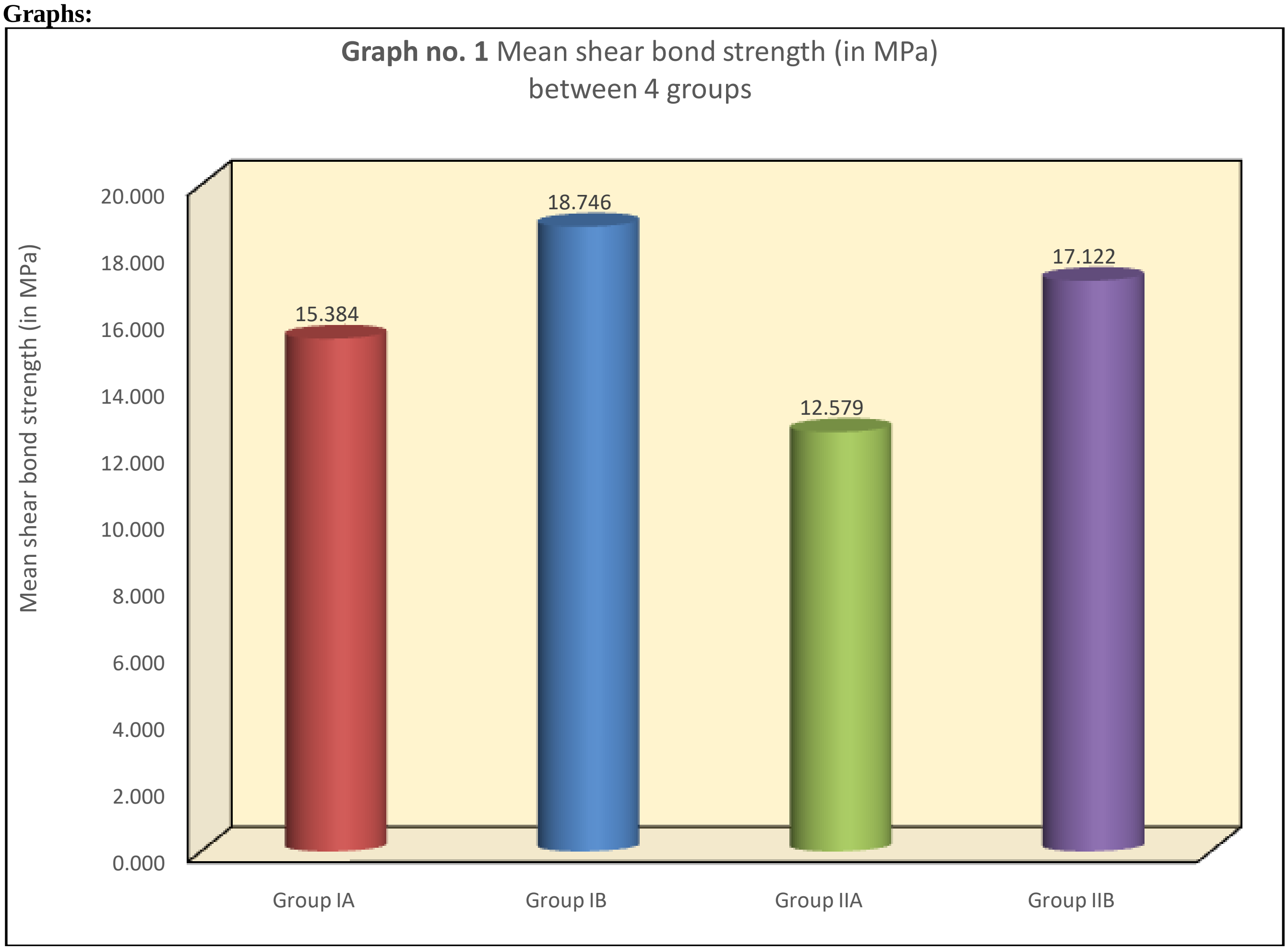
Table 2. Multiple comparison of mean difference in mean shear bond strength between 4 groups using Tukey's Post hoc Test

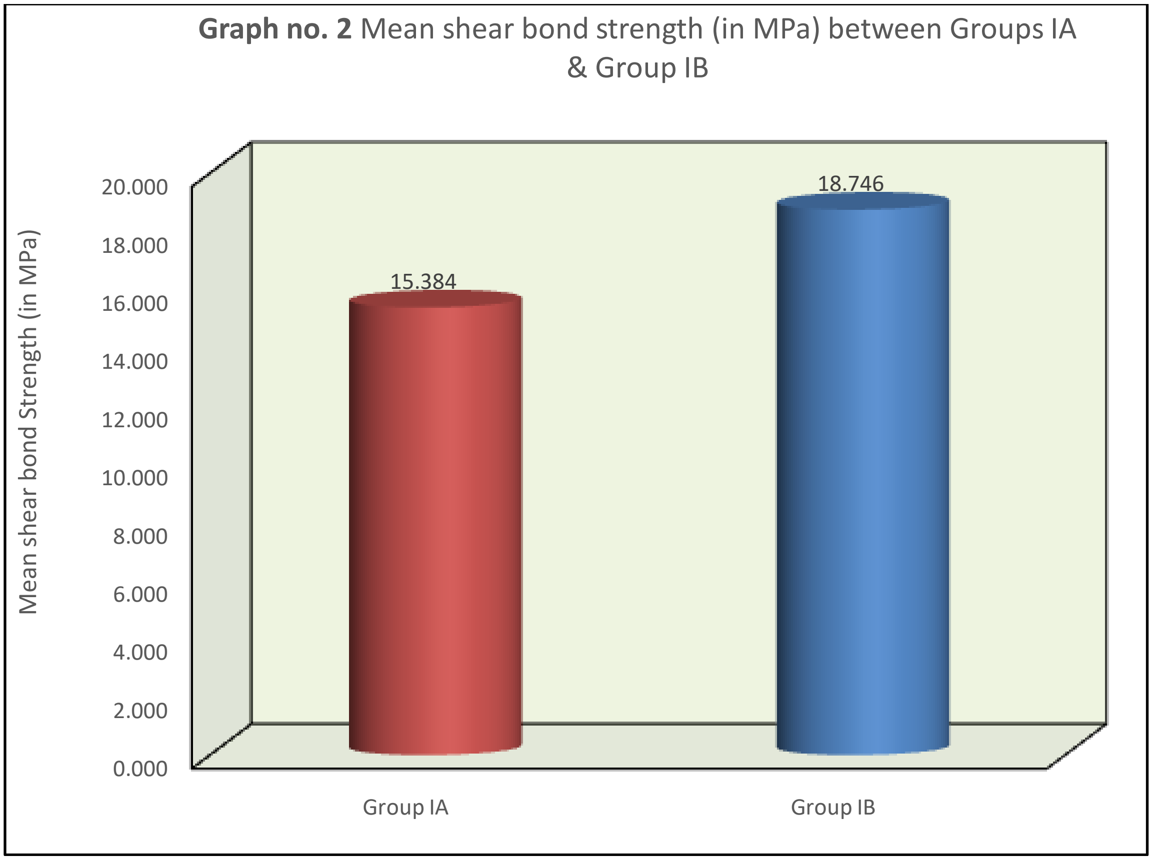
(I) Groups	(J) Groups	Mean Diff. (I-J)	95% CI for the Diff		p-value
			Lower	Upper	
Group IA	Group IB	-3.362	-4.351	-2.373	<0.001*
	Group IIA	2.806	1.816	3.795	<0.001*
	Group IIB	-1.737	-2.726	-0.748	<0.001*
Group IB	Group IIA	6.167	5.178	7.157	<0.001*
	Group IIB	1.625	0.635	2.614	<0.001*
Group IIA	Group IIB	-4.543	-5.532	-3.554	<0.001*

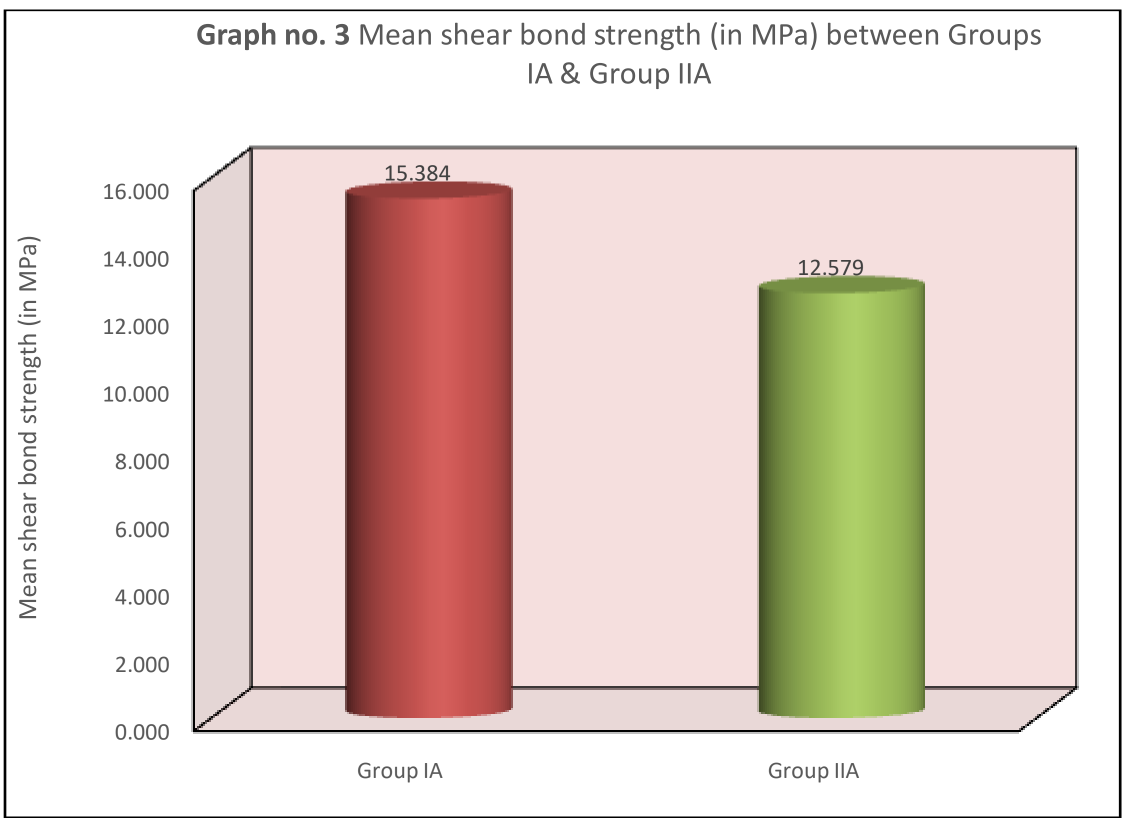
***Statistically significant**

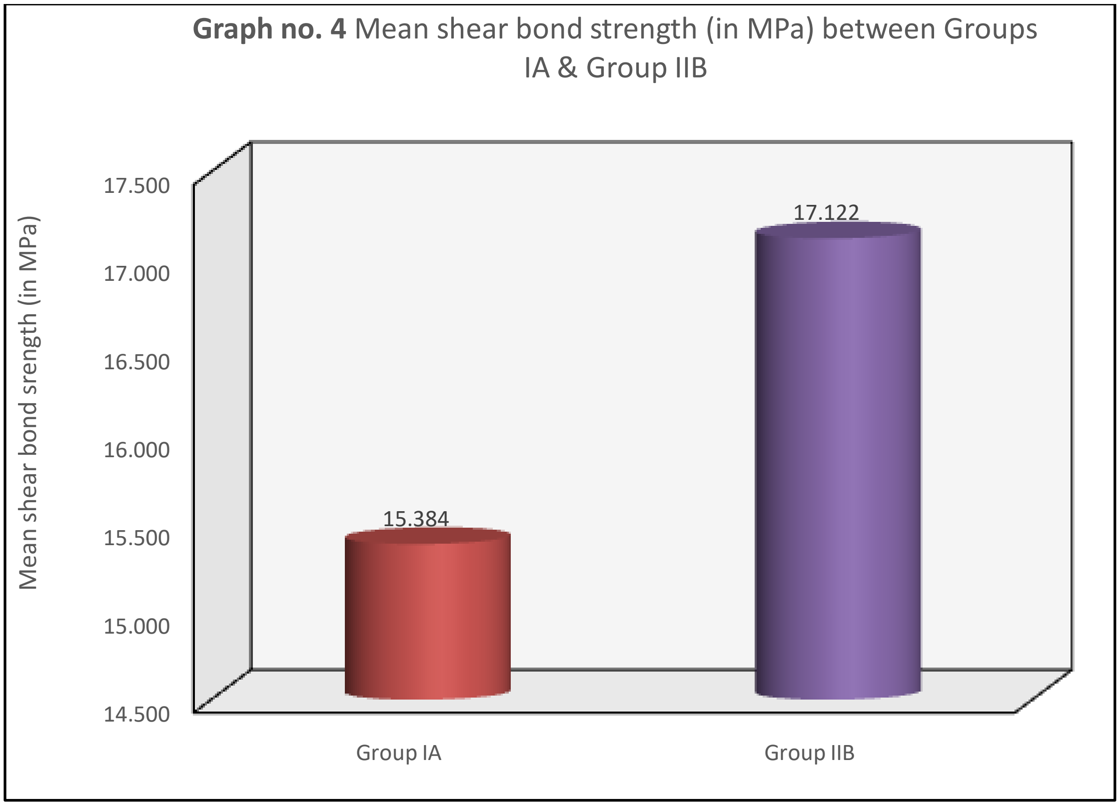
Shear bond strength: Group IB > Group IIB > Group IA> Group IIA at p<0.001.

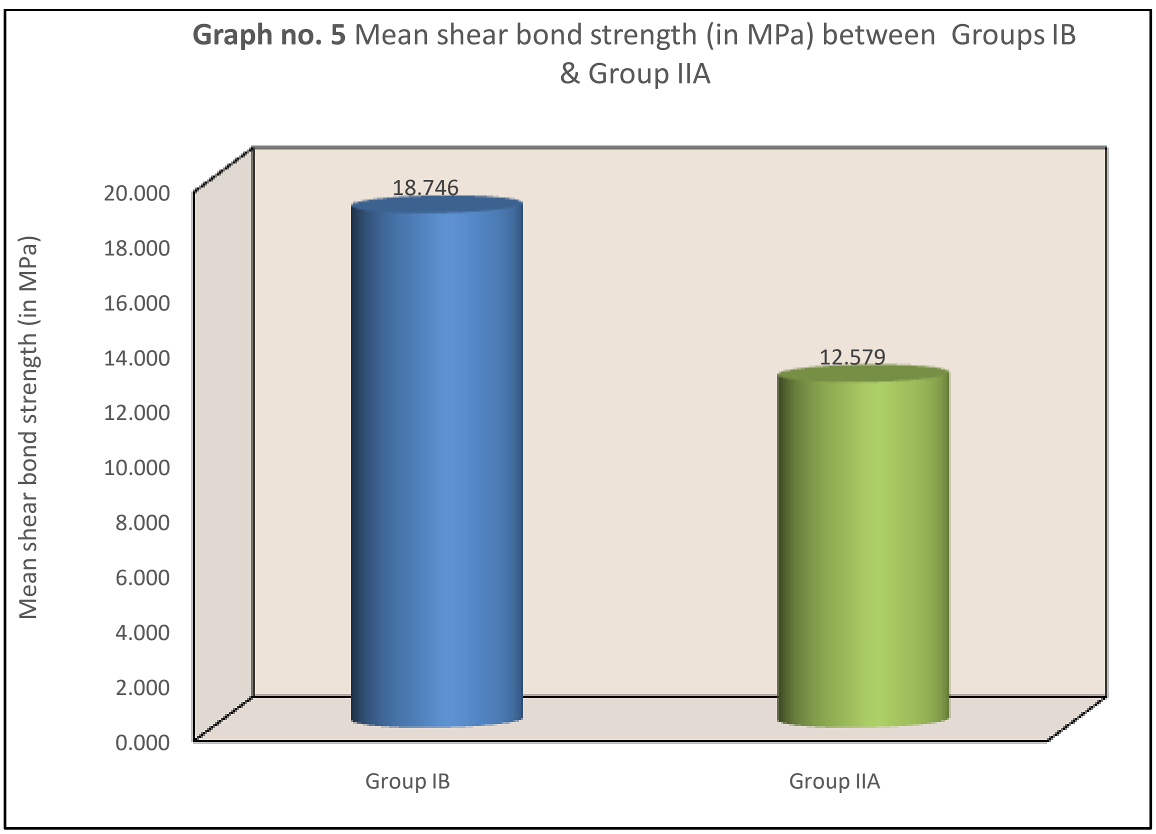
Graphs:



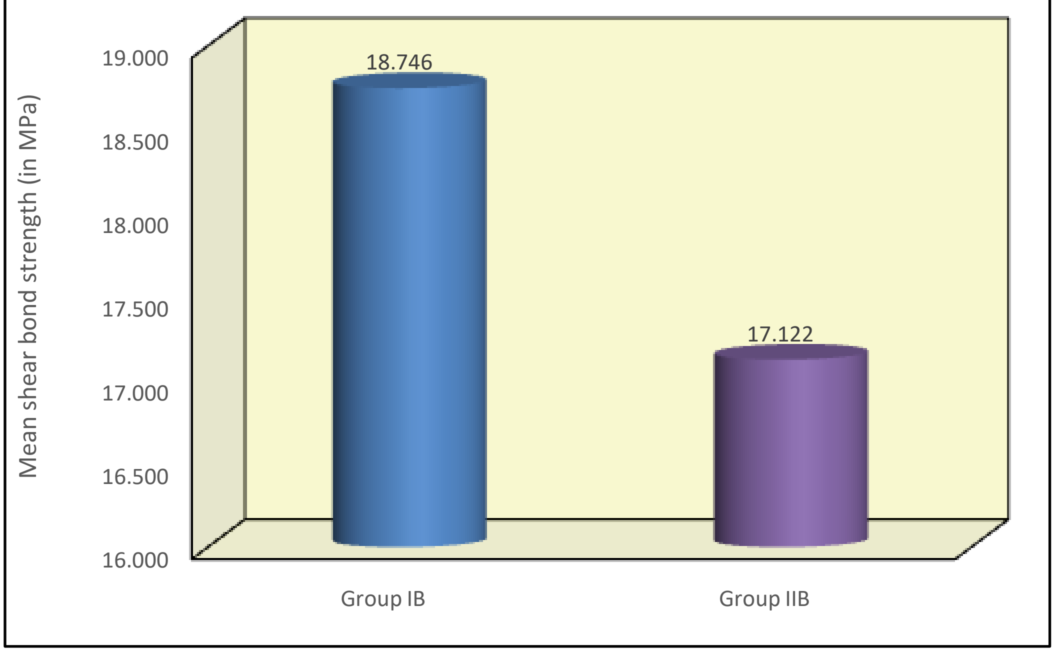

Graph no. 2 Mean shear bond strength (in MPa) between Groups IA & Group IB
Mean shear bond Strength (in MPa)
20.000
18.000
16.000
14.000
12.000
10.000
8.000
6.000
4.000
2.000
0.000
15.384
18.746
Group IA
Group IB


Graph no. 3 Mean shear bond strength (in MPa) between Groups IA & Group IIA
Mean shear bond strength (in MPa)
16.000
14.000
12.000
10.000
8.000
6.000
4.000
2.000
0.000
15.384
12.579
Group IA
Group IIA

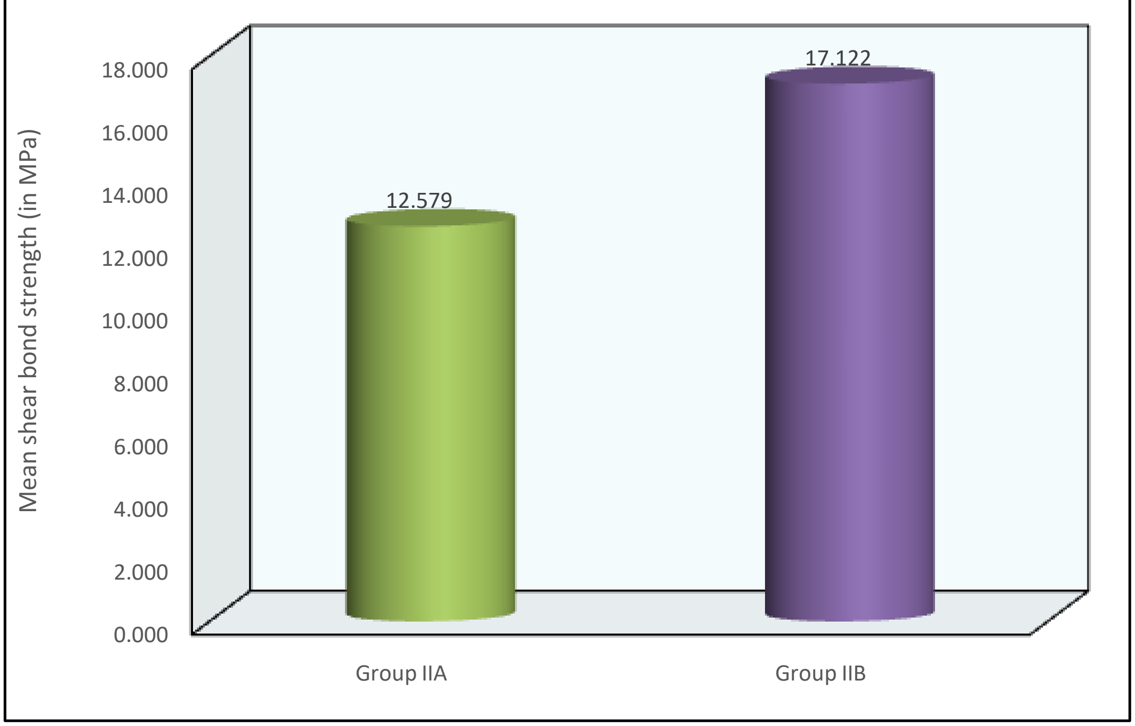

Graph no. 4 Mean shear bond strength (in MPa) between Groups IA & Group IIB
Mean shear bond srength (in MPa)
17.500
17.000
16.500
16.000
15.500
15.000
14.500
15.384
17.122
Group IA
Group IIB

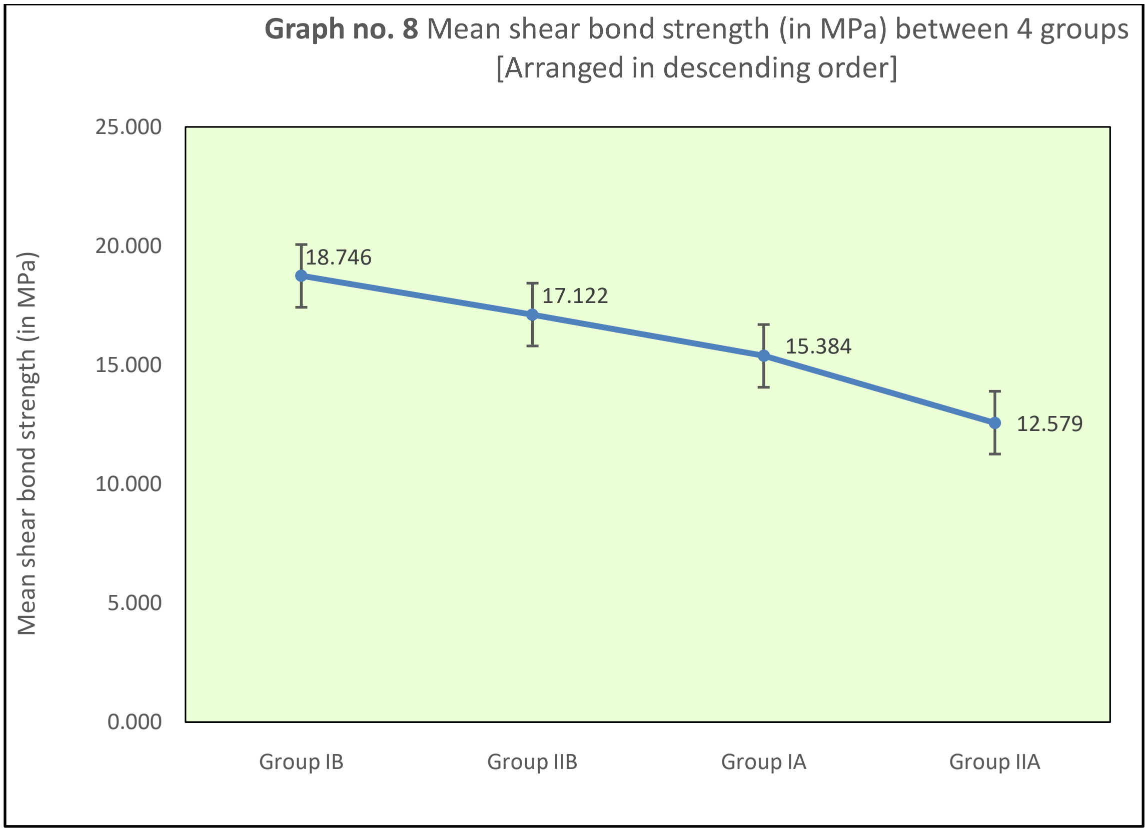

Graph no. 5 Mean shear bond strength (in MPa) between Groups IB & Group IIA
Mean shear bond strength (in MPa)
20.000
18.000
16.000
14.000
12.000
10.000
8.000
6.000
4.000
2.000
0.000
18.746
12.579
Group IB
Group IIA

Graph no. 6 Mean shear bond strength (in MPa) between Groups IB & Group IIB



Graph no. 7 Mean shear bond strength (in MPa) between Groups IIA & Group IIB





Conclusion:-

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

- The shear bond strength of silane-treated glass powder cement (RelyX U200) to commercially available zirconia, without laser surface treatment. After surface treatment with laser, the shear bond strength of silane-treated glass powder cement (RelyX U200) to commercially available zirconia showed a highly statistically significant increase.
- Among the shear bond strength values of BIS-GMA based resin cement (Multilink N) to commercially available zirconia without laser surface treatment, the lowest value was observed.After surface treatment with laser, the shear bond strength values of BIS-GMA based resin cement (Multilink N) to commercially available zirconia were found to fall between the values observed for Group IB and Group IA.
- In intergroup comparisons, non-laser treated zirconia surface Group IA exhibited significantly higher shear bond strength compared to non-laser treated zirconia surface Group IIA, and significantly lower shear bond strength compared to the laser surface treated groups, Group IB and Group IIB.
- The comparative analysis of shear bond strength revealed the following order: Group IB > Group IIB > Group IA > Group IIA.

SEM analysis revealed that Er:YAG laser irradiation resulted in smooth areas surrounded by cracks on the surfaces, whereas sandblasting altered the surface

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