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# An *in vitro* study to analyze the effect of ultrasonic instrumentation and grit blasting of natural tooth on the bond strength of nickel-chromium alloy cemented with self-adhesive resin cement

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**Abstract**
**Aim:** To evaluate the effect of various surface treatments of natural tooth on the bond strength of nickel-chromium alloy when cemented with self-adhesive resin cement.
**Materials and Methods:** This *in vitro* study was conducted on 48 freshly extracted non-carious human premolars and class 1 inlay cavity was prepared on the occlusal surface. Wax pattern with a loop on the occlusal surface was prepared on each specimen using standard procedures and then inlays were casted with Ni-Cr alloy. Specimens were then divided into 4 groups of 12 each. Group 1: Control, Group 2: Grit blasting with 110µm $Al_2O_3$, Group3: Ultrasonic instrumentation and Group 4: Grit blasting followed by ultrasonic instrumentation. The specimens were then cleaned, cemented with self-adhesive resin cement and were loaded on universal testing machine, at a cross head speed of 1 mm/min. The loads at debonding were noted and tensile bond strength was calculated in MPa.
**Statistical Analysis Used:** One-way ANOVA and Post Hoc Test.
**Results:** The means for tensile bond strength for Group 1, 2, 3 and 4 were found to be (37.20±10.97), (43.09±13.47), (47.61±12.41) and (70.67±17.58) MPa respectively. Means of tensile bond strength among the groups were compared using one-way ANOVA and comparison between individual groups were made with Post Hoc Bonferroni test. Among all the groups, Group 4 (70.67±17.58 MPa) showed the highest bond strength values and p value is <0.001 which denotes that the difference in tensile bond strength between the resin cement bonded with base metal alloy in this study was statistically significant.
**Conclusion:** Among all types of surface treatments used in this study, sandblasting improves the bond strength and the best results can be achieved with a combination of gritblasting and ultrasonic instrumentation. Therefore surface treatment have an important role on the bond strength of ceramic-metal interface.

**Keywords:** Freshly extracted premolars, surface treatment, adhesion, dental alloy, self-adhesive

## Introduction
Base metal alloys were introduced in dentistry by Erdle and Prange in the 1930s. These alloys are based on more than 75% of the base metal elements and have immense value in dentistry because of their low cost and its influence on weight, strength, stiffness, improved ceramic bonding and corrosion resistance [1]. The bonding mechanism between the cast restoration and the tooth structure can be mechanical, chemical or combination of two. Mechanical retention is achieved through sealing irregular crevices along the tooth and the metal surface by an interweaving cement. The bond strength in such a situation depends on the strength of luting agent [2]. Introduction of newer cements with low solubility, high strength and fluoride releasing abilities overcomes the biologic failure of restoration [3]. The physical factors like intraoral forces, film thickness and flaws within the cement layer also influences the quality of the bond. The use of cements with high tensile strength, thin film thickness and/or a bond enhancing intermediate layer to maximize the effect of inherent strength on the restoration can enhance the bond strength of the cement. Recently published literature suggests the use of resin cements for obtaining optimum retention for indirect restorations [4]. The success of bonding of cast restoration to the human enamel with resin cements depends upon optimization of the following components [5].

1. Enamel to resin bond.
2. Cohesive bond of the composite resin.

3. Resin to framework bond.

Studies have suggested that the self-etching primer helps to improve the tensile bond strength of cement. Many such systems are currently being employed.[6] However, the luting procedure with those cements are carried out in two stages, initially by application of self-etching primer and followed by the application of the cement itself. The cohesive bond of the cement contributes to improved bonding of alloy to the tooth surface. Failure can also occur along the interfaces (cement-tooth interface and cement-prosthesis interface), if the bonding is mechanical.[7] The resin to framework bond is essential for successful restoration. It has been reported that the bonding between the metal and resin is purely mechanical. Attempts have been made to increase the bond strength by surface treatment of the metal with methods such as silicoating, acid etching, air abrasion, use of bonding agents as ultrasonic cleaning, acid soak and electrolytic etching. Among these methods, air abrasion has proved to be a simple yet adequate method of improving the resin-metal interface bonding.[8] The bond strength of the resin luting cements used to bond the base metal alloy to the tooth structure is an important feature that has an impact on any cement that has to be retained. Thus, the present study was conducted to evaluate the effect of ultrasonic instrumentation and grit blasting of natural tooth on the bond strength of nickel-chromium alloy cemented with self-adhesive resin cement.

**Objectives**

1. To study the effect of ultrasonic instrumentation of natural tooth on the bond strength of nickel-chromium alloy cemented with self-adhesive resin cement.
2. To study the effect of grit blasting of natural tooth on the bond strength of nickel-chromium alloy cemented with self-adhesive resin cement.
3. To study the effect of ultrasonic instrumentation and grit blasting of natural tooth on the bond strength of nickel-chromium alloy cemented with self-adhesive resin cement.
4. To comparatively evaluate the tensile bond strength as achieved by various surface treatment protocols (i.e., ultrasonic instrumentation, grit blasting and combination of both) of nickel-chromium alloy on natural tooth cemented using self-adhesive resin cement.

## Materials and methods

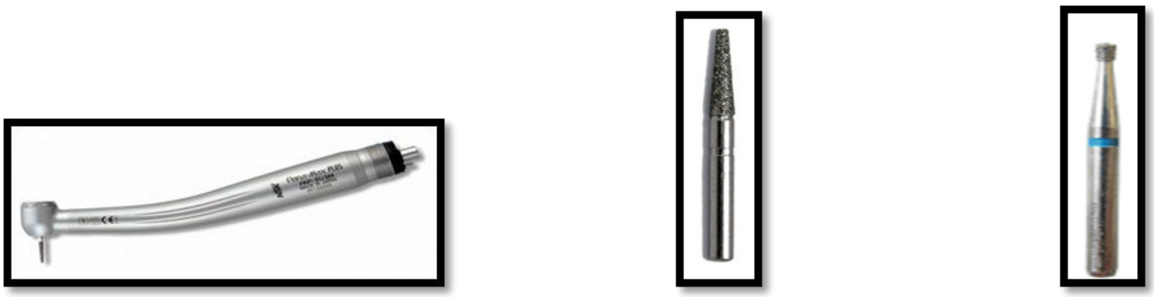
### I. Preparation of the Natural Tooth Specimens

48 freshly extracted non-carious permanent human premolars were used in the study and were stored in normal saline till the study commenced. For the purpose of the study the root portion of the teeth was embedded in Plaster of Paris with occlusal surface exposed. Each specimen was marked for future reference [Figure 1].

**Group 1** **Group 2** **Group 3** **Group 4**

**Fig 1:** 48 Extracted Pre-Molars Mounted in Plaster

A Class 1 inlay cavity was prepared on the occlusal surfaces of premolars with high speed airotor with Taper Flat End Bur (SSW-TF 21F) with minimum depth of 1.5 mm having divergent walls and pulpal floor flattened by Single Inverted Cone (SSW-SI 47) ensuring continuity of enamel, thereby ensuring that dentin is left unexposed [Figure 2].

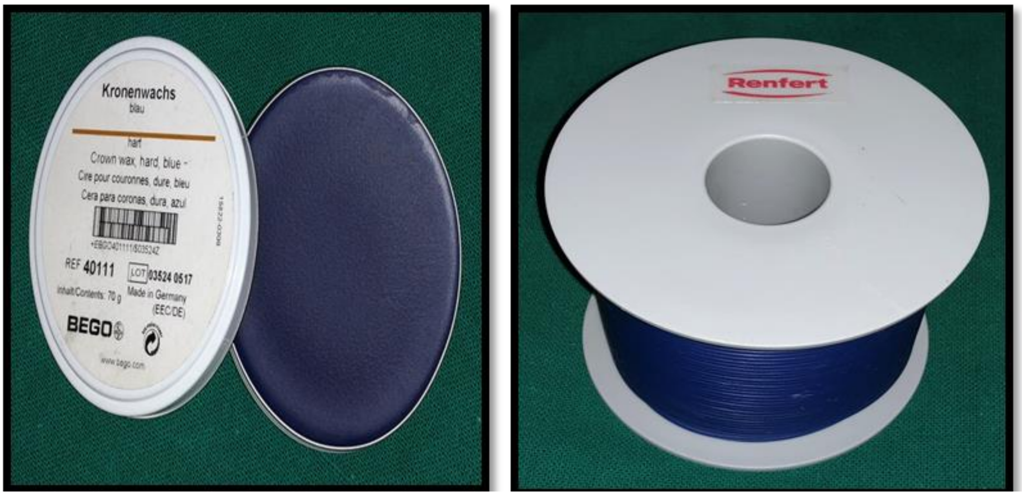


Pana-Max Plus (Nsk) Taper Flat End Bur (SSW-TF 21 F) Single Inverted Cone (SSW-SI 47)

**Fig 2:** Armamentarium for Inlay Cavity Preparation

### II. Preparation of Wax Pattern and Metal Inlays

After class 1 cavity preparation the occlusal surface was filled up with blue inlay wax for wax pattern fabrication. A U-shaped wax loop was placed on each tooth surface to serve as a sprue during investing and casting. Later, the same loop served as the attachment to be connected to the universal testing machine [Figure 3].




Blue Inlay Wax (Kronenwachs, Bego) Sprue Wax (Renfert)

**Fig 3:** Materials for Wax Pattern Fabrication

The sprue former with reservoir was attached to the U-shaped loop. The complete pattern with sprue former was attached to the crucible former [Figure 4]. Investing procedure was carried out using phosphate bonded investment material [Figure 5].


**Fig 4:** Crucible Former (Bego)


**Fig 5:** Phosphate Bonded Investment (Deguvest Impact, Degudent)

After wax elimination procedure, casting was done in the induction casting machine [Figure 6]. The Nickel-Chromium alloy [Figure 7] was heated sufficiently till the alloy turned to molten state and investment was allowed to cool down to room temperature and the same procedure was followed for all 48 test samples.

**Fig 6:** Induction Casting Machine
(Formax, Bego)

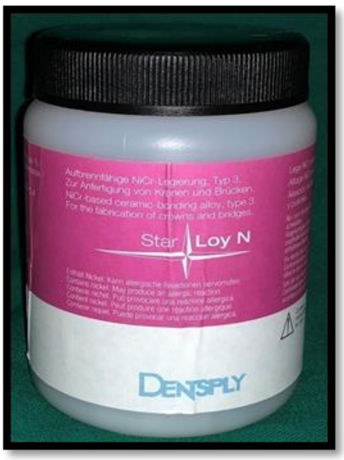
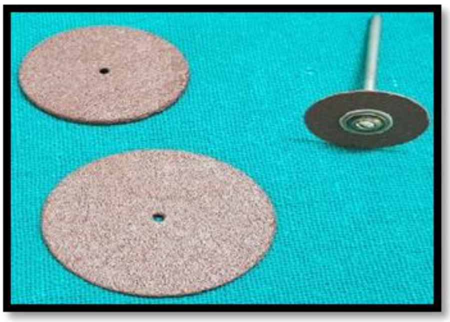



**Fig 7:** Nickel-Chromium Alloy
(Star Loy N, Dentsply)

The investment cylinder was cleaned along its long axis, and the casting was lifted free. Sandblasting using micro abrasive $Al_2O_3$ was used to divest the casting. The sprue was removed with an abrasive disc. The cast inlays were steam cleansed and checked visually. The surface of the casting was inspected and finishing procedures were done to make sure all castings adapted to corresponding tooth surface [Figure 8]. Each inlay was also labelled and stored in individual boxes with the tooth samples for future identification.

Labrotoary Micromotor (Marathon-4)



Abrasive Disks (L.M.)

Polishing Kit (Shofu)

**Fig 8:** Armamentarium for Finishing and Polishing Inlay

The casted inlays were randomly cemented to prepared teeth divided into 4 groups viz; Group 1(control group) with no surface treatment, Group 2 surface treated with ultrasonic instrumentation for 5 minutes [Figure 9], Group 3 surface treated by grit blasting with 110µm $Al_2O_3$ [Figure 10] and Group 4 surface treated by both ultrasonic instrumentation and grit blasting. Following the above mentioned surface treatment protocols the metal inlays were cemented to the tooth structure by self-adhesive resin cement (Relyx U200) and light-cured according to manufacturer's instructions and the samples thus mentioned were ready for testing at universal testing machine [Figure 11].


Ultrasonic Scaler (Wood Pecker, Uds-J)

**Fig 9:** Armamentarium for Ultrasonic Instrumentation

Aluminium Oxide (110 Microns, Bioart)

Intra-Oral Micro Sandblaster (Bioart)

**Fig 10:** Armamentarium for Grit-Blasting

3M ESPE RelyX (U200) Visible Light Cure Unit (QHL75, DENTSPLY)

**Fig 11:** Armamentarium for Cementation and Curing

**III. Equipment and Testing of Retentive Force**

The universal testing machine with a computer interface was used to test the study groups. The horizontal load frame, which was driven by a motor, induced the separation of the samples tested. This was set at a constant cross head speed of 1 mm/min for the material to pull out. The test model was stabilized with a clamp to a stainless steel plate centered on the testing machine. The models were positioned so that the dislodging forces of the three-point pull were directed upwards against the path of insertion of the cast metal framework. A custom made hook was used for engaging into the loops incorporated in the cast metal framework by using universal testing machine [Figure 12].

**Fig 12:** Universal Testing Machine (ASIAN)

**IV. Evaluation of Tensile Bond Strength**

The breaking load values were recorded through a computer connected to universal testing machine. The values obtained were in Kg and bond strength was calculated in MPa using the formula mentioned below:

Newton (N) = Kg x 9.81

$$\text{Bond Strength (MPa)} = \frac{\text{Load}}{\text{Surface Area (mm2)}}$$

The data collected was subjected statistical analysis using SPSS Version 20.

**V. Statistical Analysis**

The SPSS software was used for statistical analysis (SPSS for Windows 8.0). Mean and standard deviation were estimated from the sample for each study group. Mean values were compared by F-Test. Equality of variances was studied by L-Test. In the present study, $P<0.05$ was considered as the level of significance.

**Results**

**Table 1:** Observed and Summary values of tensile bond strength of samples in Group 1 (Control Group) Observed values are 20.5, 26.4, 29.4, 23.5, 32.3, 33.3, 41.1, 44.1, 46.0, 48.0, 49.0, 52.9 MPa

| Group 1 (Control Group) | Sample Size (n) | Mean | Standard Deviation | Median | Standard Error | IQR | Minimum | Maximum |
|---|---|---|---|---|---|---|---|---|
| 1 | 12 | 37.20 | 10.97 | 37.20 | 3.16 | 27.15-47.50 | 20.50 | 52.90 |

Table 1 represents the Mean, Median, Standard deviation and IQR of the control group where a minimum value of 20.50 MPa and a maximum value of 52.90 MPa for tensile bond strength was recorded with a mean of 37.20 MPa and standard deviation of 10.97 MPa.

**Table 2:** Observed and Summary values of tensile bond strength of samples in Group 2 (Ultrasonic Instrumentation) Observed values are 48.0, 64.7, 38.2, 68.6, 30.4, 53.9, 39.2, 23.3, 39.2, 41.1, 44.1, 26.4 MPa

| Group 2 (Ultrasonic Instrumentation) | Sample Size (n) | Mean | Standard Deviation | Median | Standard Error | IQR | Minimum | Maximum |
|---|---|---|---|---|---|---|---|---|
| 2 | 12 | 43.09 | 13.97 | 40.15 | 4.03 | 32.35-52.42 | 23.30 | 68.60 |

Table 2 represents the Mean, Median, Standard deviation and IQR of the ultrasonic group where a minimum value of 23.30 MPa and a maximum value of 68.60 MPa for tensile bond strength was recorded with a mean of 43.09 MPa and standard deviation of 13.97 MPa.

**Table 3:** Observed and Summary values of tensile bond strength of samples in Group 3 (Grit Blasting) Observed values are 48.0, 54.9, 38.2, 44.1, 69.6, 68.6, 56.8, 45.1, 41.1, 37.2, 34.3, 33.5 MPa

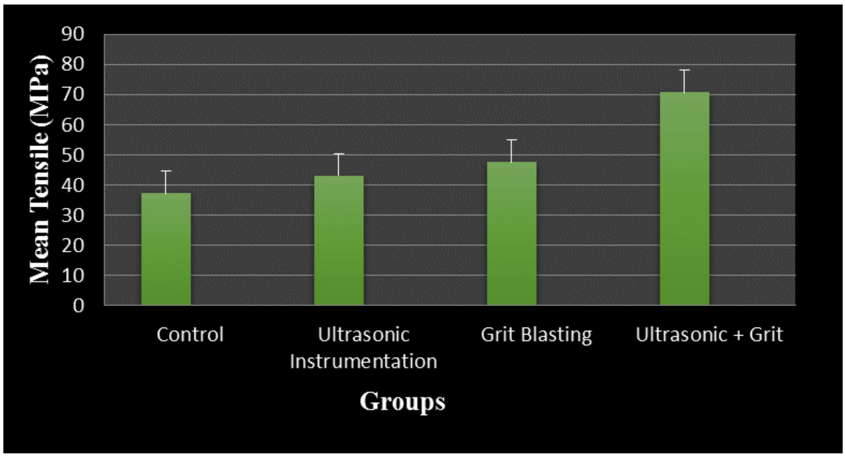
| Group 3 (Grit Blasting) | Sample Size (n) | Mean | Standard Deviation | Median | Standard Error | IQR | Minimum | Maximum |
|---|---|---|---|---|---|---|---|---|
| 3 | 12 | 47.61 | 12.41 | 44.60 | 3.58 | 37.45-56.32 | 33.50 | 69.60 |

Table 3 represents the Mean, Median, Standard deviation and IQR of the grit blasting group where a minimum value of 33.50 MPa and a maximum value of 69.60 MPa for tensile bond strength was recorded with a mean of 47.61 MPa and standard deviation of 12.41 MPa.

**Table 4:** Observed and Summary values of tensile bond strength of samples in Group 4 (Ultrasonic Instrumentation and Grit Blasting) Observed values are 71.6, 64.0, 70.6, 78.4, 62.7, 70.6, 72.5, 86.2, 91.2, 50.9, 66.6, 62.7 MPa

| Group 4 (Ultrasonic Instrumenation + Grit Blasting) | Sample Size (n) | Mean | Standard Deviation | Median | Standard Error | IQR | Minimum | Maximum |
|---|---|---|---|---|---|---|---|---|
| 4 | 12 | 70.67 | 17.58 | 70.60 | 3.73 | 62.70-76.92 | 50.90 | 91.20 |

Table 4 represents the Mean, Median, Standard deviation and IQR of the combination of ultrasonic instrumentation & grit blasting group where a minimum value of 50.90 MPa and a maximum value of 91.20 MPa for tensile bond strength was recorded with a mean of 70.60 MPa and standard deviation of 17.58 MPa.




**Graph 1:** Observed Mean ± Standard Deviation of the tensile bond strength values in all the 4 groups

Graph 1 showing the observed Mean ± Standard Deviation of tensile bond strength of all the four groups in which group 4 (ultrasonic + grit blasting) showed the highest mean and standard deviation of 70.67 ± 17.58.

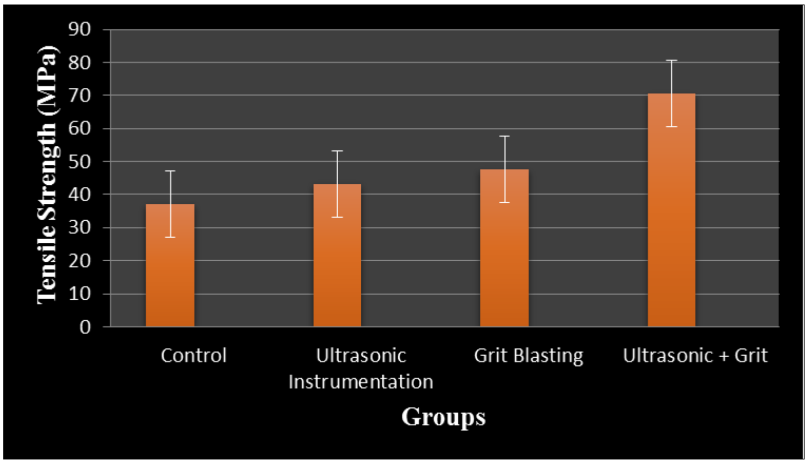
**Table 5:** Comparison of tensile bond strength in all the 4 groups (One Way ANNOVA)

| Groups | Tensile Strength (Mean ± Standard Deviation) |
|---|---|
| 1 (Control) | 37.20 ± 10.97 |
| 2 (Ultrasonic Instrumentation) | 43.09 ± 13.97 |
| 3 (Grit Blasting) | 47.61 ± 12.41 |
| 4 (Ultrasonic Instrumentation + Grit Blasting) | 70.67 ± 17.58 |

Tensile Bond Strength

| Groups | Sum of Squares | Df | Mean Square | F | P-Value |
|---|---|---|---|---|---|
| Between Groups | 7723.697 | 3 | 2574.566 | 17.491 | <0.001 |
| Within Groups | 6476.662 | 44 | 147.197 | | |
| Total | 14200.369 | 47 | | | |

Table 5 on analyzing the data using one way ANOVA it was observed that there was a statistically significant with $p<0.001$ in the tensile bond strength values in all the 4 study groups with Group 1 (control) showing minimum of 20.50 MPa and Group 4 (ultrasonic instrumentation + grit blasting) showing maximum of 91.20 MPa tensile bond strength.




**Graph 2:** Comparison of all the 4 study groups

Graph 2 showing the comparison of all the 4 study groups which reveals the highest values was achieved by Group 4 (ultrasonic + grit) of 70.67 MPa, while the lowest tensile value was achieved by Group 1 (Control) of 37.20 MPa.

**Table 6:** Comparison of tensile bond strength between the study groups

| Groups | N | Mean Retentive Force (MPa) | Standard Deviation | Minimum | Maximum | P-Value |
|---|---|---|---|---|---|---|
| Group1 vs Group2 | 12 | 58.74 | 17.95 | 32.15 | 87.20 | 1.00 |
| Group1 vs Group3 | 12 | 61.05 | 17.89 | 43.75 | 87.70 | 0.248 |
| Group1 vs Group4 | 12 | 12.00 | 66.89 | 20.17 | -18.20 | 9.15 |
| Group2 vs Group3 | 12 | 78.42 | 22.76 | -41.25 | -13.89 | <0.001 |
| Group2 vs Group4 | 12 | 82.94 | 30.00 | -36.73 | -9.36 | <0.001 |
| Group3 vs Group4 | 12 | 89.27 | 19.76 | 45.95 | 117.65 | <0.001 |

Table 6 showing the comparison of tensile strength between the study groups which on observation reveals that while comparing the group 3 vs 4 revealed the highest maximum value and p-value was statistically significant, thus has greater tensile bond strength.

## Discussion

*In vitro* techniques have demonstrated that high stresses are imposed on luting cements, particularly in the biologically important marginal areas and such localized stress concentrations are probably the initial sites of cement failure [9]. In practice, ideal axial wall convergence is rarely achieved, and lack of retention is a common cause of fixed prosthesis failure. Retention depends on magnitude of dislodging force, geometry of tooth preparation, roughness of fitting surface, materials being cemented and film thickness of the luting cement.[10] Studies have also shown the effect of conditioning of the metal and enamel surface before luting on the bond strength of the luting agents. Surface treatment such as air abrasion, acid etching, tin plating were done on the alloy surface, whereas etching and intra-oral air abrasion done on the enamel surface. The summarized results of some of these comparisons revealed considerable variation in the bond strength. The enamel to resin bond is micromechanical in nature for resin cements. It depends upon the proper etching and bonding procedures [11]. Though resin cements have been found to provide optimum retention, they may cause postoperative sensitivity in some cases. In order to reduce the postoperative sensitivity, Christensen G.J [12] has recommended the use of resin cements with an acceptable bonding agent and reported that the most popular brand of resin cement for routine use, was a resin system in which a self-etched primer was used for conditioning the tooth surface followed by application of a dual cure cement (Panavia F.2) and a newly introduced self-adhesive universal resin cement (RelyX Unicem) in which self-etching primer was incorporated in the cement itself and thus separate conditioning of tooth surface was not necessary. Piwowarczyk *et al.* [13] studied the shear bond strength of cementing materials and concluded that the self-adhesive universal cement (RelyX Unicem) had the highest bond strength values. However in the literature, studies comparing the tensile bond strength between the resin cement system in which the self-etched primer is not incorporated into the dual cure system (Panavia F.2) and self-etched primer incorporated in the dual cure cement system (RelyX Unicem) are scarce. Various surface treatments including surface roughening, chemical and electrolytic etching have been recommended to improve the bond strengths of casting alloys. The mechanical removal of the debris can also improve wetting before the application of adhesives. This method is inexpensive and eliminates numerous problems associated with etching [14]. Musil and Tiller had suggested that by sandblasting the base metal alloy surface could increase the adhesion. Sen *et al.* [15] did surface treatment with $Al_2O_3$ and obtained good bond strength for base metal alloys compared to noble and high noble alloys bonded with resin cements proper bonding. Though literature has evidenced studies that the effects of layer thickness, adherence energy, polymerization shrinkage, storage conditions of cast restoration on the bond strength of resin cements, the bond strength between the resin luting agent and the dental structure still warrants investigation along with any technique which enhances this bond must also be explored. Generally, adhesive capacity has been evaluated with *in vitro* testing, with shear and tensile tests. Finite element analysis have revealed that shear test were the most efficient to disclose the cohesive resistance of a material, whereas tensile tests were better to investigate the adhesion at the interface. Since the purpose of the present study was to evaluate the adhesive capacity of the resin cement rather than the stress produced during clinical function a tensile test was used and the study was to be conducted with an aim to compare tensile bond strength of resin cements used to bond the base metal alloy to human tooth after subjecting it to surface treatments i.e., ultrasonic instrumentation and grit blasting and a combination of the above. For the purpose of the study, freshly extracted non-carious human permanent premolars were used and teeth were mounted in a plaster of paris block such that it would facilitate the placement of specimen at one end of universal testing machine without producing any deleterious effect on the tooth specimens. The sprues were attached, invested and were casted. In this way, 48 test specimens corresponding to natural tooth made of metal inlays were obtained. These inlays were randomly cemented on to tooth into 4 groups of 12 samples in each group depending on the surface treatments. Group 1 (control group), Group 2 treated with ultrasonic instrumentation for 5 minutes. Group 3 grit blasted by 110µm $Al_2O_3$ and Group 4 surface treated with both ultrasonic instrumentation and grit blasting and then bonded to treated natural tooth test specimen with self-adhesive resin cement. (RelyX U200). The results of this study after tensile testing have been tabulated and interpretation of this data was done by statistical analysis. The basic data (Table No.1) shows a mean value of 37.20 MPa of tensile strength for control group with no surface treatment (Group1). The (Table No.2) shows a mean value of 43.09 MPa for ultrasonic instrumentation for 5 minutes on the tooth surface (Group2). The (Table No.3) shows a mean value of 47.61 MPa for 110µm air abrasion on the tooth surface (Group3). The (Table No.4) shows a mean value of 70.67 MPa for combination of both the surface treatments of (Group 2 and 3) on the tooth surface. The statistical analysis by Levene's test for equality of variance and Student t-test, indicated that the difference in the tensile bond strength for the resin cement bond with base metal alloy showed the p value of <0.001 (Table 5). This denotes that the difference in tensile bond strengths between the test resin cement bonded with base metal alloy was statistically significant. A wide range of enamel-resin composite-metal bond strengths has been reported in the literature and the results of this study are within the ranges, as documented earlier [16]. Previous studies by Cotert HS *et al.* [17] and Tjan AH *et al.* [18] indicated that the differences among the bond strengths of various resin composites were statistically significant. However, they observed no statistically significant difference between the tensile bond strength of dual cure resin cement system (Panavia F.2) in which self-etching primer was supplied separately and a dual cure resin cement system (RelyX Unicem) in which the self-etching primer is incorporated within the cement itself was observed. Atta *et al.* [19] in their study, while investigating the effect of air abrasion of $Al_2O_3$ stated that they produced statistically significant results i.e., surface treatment with 110µm of air abrasive on the alloy and tooth surface produced higher mean tensile bond strengths for both the test cements as compared to surface treatment with 250µm. These findings were in accordance with those obtained by Sen *et al.* who in their study concluded that surface treatment with $Al_2O_3$ yielded good results of bond strength for base metal alloy samples compared to noble and high noble alloys. The results obtained in the present study also correlates with the above mentioned findings wherein surface treatment with 110µm $Al_2O_3$ yielded better bond strength and retention value of 70.67 MPa. Chen LY *et al.* [20] through a SEM study revealed that tooth surfaces after ultrasonic instrumentation are rougher and there is no significant difference of resin restoration and the prepared tooth surface. On the other hand, Conde *et al.* [21] in their study stated that the smear layer removal increases the adhesive penetration in dentin thus promoting better interlocking micromechanically and it is believed that removal of the smear layer treated with ultrasonic tips increases the bond strength. Researchers have also shown that the ultrasonic treatment alone does not promote the total removal of smear layer and also it is believed that both the dentinal cleanliness and the roughness influence the micro tensile bond strength [22]. Thus by correlating the evidence in literature it can be concluded with the present study that a clean surface as obtained after ultrasonic instrumentation and a surface treatment like sandblasting yields a better bond strength when using resin cement as luting agent for Ni-Cr alloy.

## Conclusion

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

1. The comparative mean tensile bond strengths of resin cement bonded to base metal alloy cemented on to tooth surface via various surface treatments was statistically significant.
2. Surface treatment of teeth with procedures like ultrasonic instrumentation and grit blasting can be recommended as a routine protocol to enhance retention when cementing Ni-Cr alloys to human tooth.

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