

**EVALUATION OF BOND STRENGTH OF METAL-RESIN USING DIFFERENT  
MODES OF MECHANICAL RETENTION- AN IN VITRO STUDY**

*Dissertation submitted to*  
**THE TAMILNADU Dr. M.G.R. MEDICAL UNIVERSITY**  
*In partial fulfillment for the Degree of*  
**MASTER OF DENTAL SURGERY**



**BRANCH I**  
**PROSTHODONTICS AND CROWN & BRIDGE**  
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## **CERTIFICATE**

This is to certify that the dissertation titled “**EVALUATION OF BOND STRENGTH OF METAL-RESIN USING DIFFERENT MODES OF MECHANICAL RETENTION-AN IN VITRO STUDY**” is a bonafide record of work done by **Dr. S.SOWMIYA LAKSHIMI** under my guidance during her postgraduate study period 2010 – 2013.

This dissertation is submitted to **THE TAMILNADU Dr. M.G.R. MEDICAL UNIVERSITY**, in partial fulfillment for the degree of **MASTER OF DENTAL SURGERY** in Branch I - **Prosthodontics and Crown and Bridge**.

It has not been submitted (partially or fully) for the award of any other degree or diploma.

### **H. O. D AND GUIDE**

**Prof (Dr). V.R.Thirumurthy. M.D.S.,**  
Vice Principal,  
Department of Prosthodontics including Crown  
and Bridge and Implantology  
Sri Ramakrishna Dental College and Hospital,  
Coimbatore.

### **PRINCIPAL**

**Prof (Dr). V.PRABHAKAR, M.D.S.,**  
Sri Ramakrishna Dental College and Hospital,  
Coimbatore.

**Date :**

**Place :** Coimbatore.

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## **ABSTRACT**

**AIM AND OBJECTIVE:** To evaluate and compare the bond strength of Ni-Cr metal and ADORO resin processed by “Lost salt crystal technique” using salts of different crystal shapes. [Sodium chloride (Cube shape), Potassium permanganate (Irregular shape) and Ammonium chloride (Spherical shape)].

**MATERIALS AND METHODS:** A total of 90 samples (30 for each group) were prepared in wax with a handle. The salt crystals were embedded into the 8mm disc of the wax pattern (Group A with Cube shape, Group B with Irregular shape, Group C with Spherical shape) and washed in tap water. The patterns were sprued, invested and casted. A transparent matrix was used to standardize the 2mm thickness of SR ADORO resin over the casted pattern. The SR ADORO resin was added to the casted patterns as per manufacturer’s instructions. The finished samples were mounted in a jig and was tested for shear bond strength in Instron Universal testing machine.

**RESULT:** One way ANOVA test showed a significant difference in mean shear bond strength ( $p < 0.05$ ) among the three groups. t-test showed a significant difference in mean shear bond strength of Group A, when compared to Group B & C. Chi-square test showed significant difference ( $p < 0.05$ ) in modes of failure among the three groups.

**CONCLUSION:** It was concluded that the shear bond strength of Group A (Cube shape) was better followed by Group B (Irregular shape) and Group C (Spherical shape).

**KEY WORDS:** SR ADORA resin, salt crystals, shear bond strength, Ni-Cr alloy.

# *Introduction*

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# INTRODUCTION

“A smile is an inexpensive way to change your looks” may not be exactly true in today’s age of acute need for the ideal face or to be more specific the ideal smile. The focus on esthetics has encouraged the general public to enquire about options for improving the appearance of their teeth and smile. This esthetic demand has risen through the years and is achieved in many instances by veneering the metal base of a restoration with resin or porcelain. Resin has been used as a veneering material since the early 1940s. Clinicians have encountered problems with these early generation methyl methacrylate resins, such as marginal percolation, discoloration, low abrasion resistance, high thermal expansion coefficient, high elastic deformation, low yield strength and poor dimensional stability. The problems continued even after the introduction of a number of techniques to improve metal to resin bonding.<sup>6,10,17</sup>

Porcelain facings do not wear, they have good colour stability and are devoid of marginal leakage, yet they have their limitations such as brittleness, abrasion of opposing teeth, subject to crack propagation and difference in coefficient of thermal expansion between metal and porcelain<sup>6,7,10</sup>. In addition, patients exhibiting signs of parafunctional bruxism or clenching can subject ceramic-veneered restorations to abnormally high stress resulting in their failure<sup>42</sup>.

To overcome the drawbacks of these materials, laboratory cured composite veneers have been introduced as an alternative veneering material to porcelain and acrylic resin. They are microfilled composite resins based on the Bis GMA and UDMA resin systems. These materials vary in their composition and physical properties. Principle variations in chemical composition are monomer composition and concentration of filler particles<sup>6,7,10</sup>. Composites are not as popular as porcelain, but they are still widely used to cover metal because of low

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cost and because the laboratorial procedures are faster and simpler. In addition, it is possible to repair composites intraorally.

SR ADORO (1996) is a new generation light and heat cured, highly filled laboratory composites containing approximately 66% inorganic fillers, and 33% resin matrix by volume, whereas former generation composite resins contained 33% inorganic fillers. In addition to a change in the size and distribution of filler particles, significantly improved physical and mechanical properties have improved these new composites<sup>10,45</sup>.

The advantages of these resins to name a few are, excellent esthetics, colour stability, abrasion resistance similar to tooth structure, biocompatibility, repairability and compatibility with dental casting alloys. These laboratory composites have been used for various applications such as fixed partial dentures subject to flexing forces, veneered crowns, custom denture teeth, veneers on complete cast overdentures, pontics for resin bonded fixed prosthesis, custom labial veneers, provisional restorations of implant prosthesis and repairs for fixed partial dentures<sup>17</sup>.

Bonding of laboratory polymerised composite resins to metal has traditionally been provided by mechanical retention such as beads<sup>3,42,50</sup>, mesh<sup>49</sup>, loops. Rochette introduced perforated cast metal splint in 1973<sup>35</sup>, followed by introduction of mechanical retentive beads and cast mesh patterns by Shen in 1984<sup>35</sup>. From available information mechanical retention has the following disadvantages: unreliable bonding values, gap formation and microleakage resulting from difference in thermal coefficients of expansion. Furthermore, mechanical retention requires thickness of material that might result in over contouring of the restoration. Micromechanical bonding systems involved sandblasting by cleaning oxides or greasy materials from metal surfaces and it was found that bond strengths obtained from sandblasting were insufficient<sup>35</sup>.

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Moon and Hudging et al introduced “Lost salt technique” using salt crystals to create retentive surface. This method was introduced for resin bonded prosthesis to provide retention between the prosthesis and the luting cement. Salt crystals were incorporated into wax and removed in solution leaving retentive pits<sup>34,15</sup>.

Electrochemical etching was developed in 1982 by Livaditis and Thompson<sup>27,28</sup>. Fine irregularities in the surface of base metal alloys were formed to increase the bond strength. Furthermore metal could be made thinner to avoid over contouring and massive tooth reduction. Electrolytic etching has the disadvantage of difficulty in creating a properly etched surface and does not work well with precious alloys<sup>27,28,35</sup>. Electrolytic tin plating was introduced later which was applicable for precious metal alloys. Studies noted that tin oxides form crystals on the surface of the alloy providing micromechanical and chemical retention<sup>35</sup>.

Chemical etching was introduced by Livaditis in 1986 as an alternative to electrochemical etching<sup>28,35</sup>. It provided better retention and has the following advantages of being very simple to use on chair side, restoration can be re-etched in case of failure without need for sophisticated laboratory procedures<sup>35</sup>. Over the last decade a major breakthrough was made in the development of new metal – resin bonding techniques that resulted in a chemical bonding between resin and the metal. These techniques usually involve some pretreatment of the alloy surface with chemical agents<sup>10</sup>. SR Spectra Link (Ivoclar) creates a physicochemical bond between metal and resin without silane coating<sup>35</sup>. Silicoater Classical (Heraeus-Kulzer) was developed by Tiller et al in 1984. This created an intermediate layer containing silicon dioxide. The technique has the disadvantage of being expensive, and time consuming<sup>35</sup>. Silicoater MD (metal-dotted) is the new version of 14-yr old Silicoater Classical technique. The difference between the techniques is that in the new method the metal surface is coated with Siox as a liquid and is fired at highly controlled and carefully regulated temperatures<sup>35</sup>. Rocatec system presented in 1989 provides tribochemical

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application of a silica layer by means of sandblasting. The advantages are speed and accuracy of coating, adhesive layer can be checked visually and thermal stressing of framework is avoided. Kevloc AC (Heraeus Kulzer) is a relatively new system, introduced in 1995 that offers a combination of chemical and mechanical bonding. Siloc system is another chemical bonding technique introduced later<sup>35</sup>.

A strong and stable bond between veneering material and metal framework considerably assists in the esthetic appearance and clinical longevity of a restoration. There can be various types of bond failures between veneer and base metal alloy due to mechanical, chemical and thermal stresses<sup>43</sup>. It has been shown that temperature increase during manufacturing process can cause stresses between framework and veneer, which may induce cracking and chipping of the composite, especially in posterior region<sup>41</sup>.

It has been claimed that a newly developed heat protection paste increases the fracture resistance of composite veneering. This paste is meant to be applied to the unveneered metal surfaces to achieve a thermal absorption effect (cooling effect), which is expected to minimise the tension at the interface between the metal and the UDMA based veneering composite<sup>41,44,45</sup>.

Though numerous techniques has been advocated by several authors for metal- resin bonding, the chemico - mechanical system was widely advocated for bonding metal alloys to composites<sup>17,29</sup>.

Conventional mechanical retention techniques using beads, loops, mesh, wires were used earlier. The use of these mechanical retentive features resulted in restorations that were bulkier and were not esthetically pleasing, which was considered as a major disadvantage. To overcome the drawbacks of the previous mechanical retention features, the use of “Lost salt technique”, which was introduced for resin bonded prosthesis was advocated in this study.

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This method reduces the bulk of the veneering material by creating depressions on the surface rather than elevations as in case of retentive beads and loops at the same time providing the needed mechanical retentive features.

This in vitro study was conducted to evaluate the chemico-mechanical bond strength of nickel – chromium alloy to SR ADORO resin using commonly available salts with different shapes.

The aim of the study is

- To evaluate and compare the bond strength of metal and ADORO resin processed by “Lost salt crystal technique” using salts of different crystal shapes. [ sodium chloride (cubic shape), potassium permanganate (irregular shape) and ammonium chloride (spherical shape)]
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*Review of*  
*Literature*

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## REVIEW OF LITERATURE

### **Peter C. Moon in 1987<sup>34</sup>**

Shear bond tests were described for a new lost salt procedure for developing retention to the metal framework for a resin-bonded fixed partial denture. It was found that different salt crystal sizes influence the bond strength. The salt crystal size ranging from 150  $\mu\text{m}$  to 250  $\mu\text{m}$  was significantly stronger than the smaller and larger size ranges tested. This size range produced a surface more easily evaluated visually than an etched metal surface for retention. The bond failure observed was a fracture of the bonding resin and not an adhesive pulling out of the resin from the cubic surface voids.

### **Barzilay et al in 1988<sup>17</sup>**

This study was divided into two parts-1) Independent chemical and mechanical retention techniques, 2) Combined chemical and mechanical retention techniques.

#### **Part 1:**

The four chemical retention techniques were as follows:

1. A pyrolytically applied silica surface to which composite was bonded with a silane coupling agent (Silicoater, Kulzer Inc.)
  2. 4-META acrylic resin (Super Bond C&B, Sun Medical Co., Ltd., Kyoto, Japan)
  3. 4-META resin applied to an oxidized metal surface
  4. Gold Link bonding agent (Den-Mat Corp., Santa Maria, Calif.)
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The mechanical retention techniques included were as follows:

5. An electrolytically etched metal surface
6. Small bead retention-350  $\mu\text{m}$  diameter resin beads (Lang Dental Mfg. Co., Chicago, III.)
7. Large bead retention-430  $\mu\text{m}$  diameter resin beads (Lang Dental Mfg. Co., Chicago, III.)

The seven techniques were used in combination with (1) Litecast B(Williams Gold Co., Ltd., Buffalo, NY.)(2) Ticonium (Ticonium, Inc., Albany, N. Y.) ; and (3) Vitallium (Howmedica, Chicago, III.) alloys.

**Part 2:**

Three new groups based on a combination of chemical and mechanical retention were formed as follows:

1. Ticonium alloy was etched and 4-META monomer with polymer, followed by the composite, was applied to the etched metal surface.
2. The surface of the Ticonium small-beaded samples were treated with 4-META followed by composite.
3. Litecast B small-beaded samples were processed with the Silicoater unit and composite was then placed.

The following conclusions were drawn:

1. Chemical retention techniques with 4-META and Silicoat materials were superior to conventional mechanical techniques.
  2. Mechanochemical techniques with etched 4-META and small-bead Silicoat material recorded elevated bond strengths but were not significantly different from 4-META material.
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3. Chemical bonding minimized gap formation at the composite-metal interface.

### **Naegeli et al in 1988<sup>6</sup>**

This study evaluated the shear bond strength of two commercial fixed prosthodontic composites to a cast alloy using three recently introduced adhesive bonding systems. The three resin-metal bonding systems evaluated were (1) Silicoater system (Kulzer Inc., Laguna Hills, Calif); (2) Lee metal primer (Lee Pharmaceuticals, South Elmonte, Calif); (3) Gold link (Den-Mat Corp., Santa Maria, Calif). The veneering materials used were, Dentacolor (Kulzer Inc.) and Visio-Gem (ESPE-Premier, Norristown, Pa.). Shear bond strengths were determined by using an Instron universal testing machine with a strain rate of 1 mm/min.

The results obtained were

1. Shear bond strengths showed that the silicoater system with Dentacolor composite had significantly higher bond strength than any other combinations.
2. The Silicoater/Visio-Gem combination and the Lee primer/Dentacolor combination were not significantly different.
3. The Lee primer/Visio-Gem, Gold Link/Dentacolor and the Gold Link/Visio-Gem combinations showed significantly lower shear bond strengths than other combinations
4. After use of Silicoater system, cohesive failure was normally observed during shear bond testing.



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**Re et al in 1988<sup>40</sup>**

This study compared three methods of etching metal for resin-bonded retainers namely: Met-Etch gel (Gresco Products, Inc., Stafford, Tex.), Electrolytic etching with 10% sulphuric acid for 3 min at 300mA, Silicone coating (Kulzer & Co., GmbH, Friedrichsdorf, Germany). All the specimens were treated identically to bond Comspan (L . D .Caulk Co., Milford, Del) composite luting material. Shear bond strength was tested in an Instron Universal testing machine. Superior resin-alloy shear strengths were obtained by silicone coating and electrolytic etching. Coating with silicone produced the greatest resin-alloy bond whereas Met-Etch gel recorded the lowest mean resin-alloy shear strengths. All three methods are considered clinically suitable.

**Xuan-Xiang Ma, Jun-wu Xu, He-ming Xie in 1991<sup>59</sup>**

The purpose of this study was to determine the relationship between metal-resin bond strength and the shape and size of the salt crystals used to roughen the metal surface and to evaluate the influence of roughening treatment on the bending strength of metal. It was found that roughening of metal surfaces with lost salt technique considerably improved metal-resin bond strength. The bond strength found between the Ni-Cr alloy and EM bonding agent was as high as 377.8 kg/cm<sup>2</sup>.

There was no relationship between metal-resin bond strength and size of the salt crystal. Among the four shapes (spherical, cubical, cuboidal, irregular) of salt crystal tested, cuboid crystals provided greatest bond strength. The bending strength was reduced by roughening treatment, but after coating with acrylic resin, the bending strength was much higher than that of sandblasted and control groups.

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**Lee, Pierpont and Strickler in 1991<sup>25</sup>**

This study compared the difference in tensile bond strength between the composite resin veneer and the cast Ni-Cr disk when different bead adhesives were used to make the laboratory patterns. Visio-Gem, cyanoacrylate, and shellac were the adhesives tested. The results showed that the mean tensile bond strength of the cyanoacrylate group was significantly higher than the other two groups. No significant difference was observed between Visio-Gem and shellac groups.

The high tensile bond strength in the cyanoacrylate group is thought to be attributed to the low rheological property of the adhesive that allowed greater exposure of the bead for retention. Using different adhesives in the fabrication of composite resin veneered castings may affect the bond strength in the composite resin-metal interface.

**D. Vojvodic in 1995<sup>57</sup>**

The silicoater technique, or silicoating, is a chemical bond system (makes mechanical retentions unnecessary) which prevents the rising of a gap in the margin area between resin and metal. This procedure solves the problems of fracture and detachment of the veneer or its discoloration. The purpose of this investigation was to find out the values of the bond strength achieved by using the silicoater technique on Ag-Pd alloy with smooth surface and with mechanical retentions on the surface. The silicoater technique gave the best results in combination with Dentacolor XS veneer material and mechanical retentions on the surface of the dental alloy.

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**Masami Mukai in 1995<sup>31</sup>**

This study investigated the effect of sandblasting on composite resin-alloy bond strengths with the Silicoating technique. Ag-Pd and Ni-Cr alloys were prepared for casting. The specimens were sandblasted with 37  $\mu\text{m}$  or 250  $\mu\text{m}$  A1203 particles under the following conditions: 3  $\text{kg}/\text{cm}^2$  for 30 seconds; 5  $\text{kg}/\text{cm}^2$  for 10 seconds, 30 seconds, and 45 seconds; and 7  $\text{kg}/\text{cm}^2$  for 30 seconds. After sandblasting, the alloy surface was silica-coated, and light cured composite resin was bonded to the alloy. Specimens were divided, stored in dry air for 1 week, and thermocycled from 4° to 60° C for 10<sup>2</sup> cycles. Bond strength was measured by applying compressive shear stress. It was found that sandblasting made the alloy-water contact angle smaller and wettability greater. The adhesive strength of composite resins with alloys was influenced by sandblasting.

**Stefanos G Kourtis in 1997<sup>46</sup>**

Bond strengths of different resin-to-metal chemical bonding systems were evaluated in this study. Six different systems ( Silicoater, Silicoater MD, Rocatec, OVS, Sebond, and Spectra-Link.) were tested with bending and tensional tests after dry storage, water storage, and thermocycling. It was concluded that mechanical retention for resinous material on resin-veneered crowns was not needed for the Silicoater, Silicoater MD, Sebond, and Spectra-Link bonding systems. For the other current bonding systems, OVS and Rocatec, additional invitro research is required to investigate the mechanism of bond weakening after water storage and thermocycling.

**M. Zalkind in 1998<sup>63</sup>**

The tensile strengths of laboratory versus clinical photocured composite resins have been investigated. Metal surfaces were bonded to photocured composite resin by either retentive beads, Sebond or Silicoating. The metal/facing bond strength was the highest in samples fabricated by the Silicoating technique, the bond strength exceeding the cohesive forces in the composite resin facing. The tensile strengths of metal/facing bonded by Sebond and retentive beads were similar.

**Rothfuss et al in 1998<sup>24</sup>**

This study compared the shear bond strength of composite to metal with two commercially available chemical bonding systems: Silicoater and Kevloc system. Mean bond strength for the Silicoated sample was 10.93 MPa and for the Kevloc system was 11.44 MPa. Visual examination of the fracture surfaces revealed that the failure was adhesive at the resin-metal interface in almost all the specimens.

**K. Yoshida in 1999<sup>61</sup>**

This study evaluated the effect of five adhesive primers on the shear bond strength of a self-curing resin to cobalt-chromium (Co-Cr) alloy. The adhesive primers Acryl Bond® (AB, Shofu), Cesead Opaque Primer\* (COP, Kuraray), Metacolor Opaque Bonding Liner® (MOBL, Sun-Medical), Metal PrimerII\* (MPII, GC) and MR. Bond\* (MRB, Tokuyama) were used. All of the primers examined, except MOBL, improved the shear bond strength between the resin and Co-Cr alloy compared with non-primed specimens prior to thermal

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cycling. Regardless of which primer was used, the shear bond strength significantly differed between thermal cycles 0 and 50000. However, after 50000 thermal cycles, the bond strengths of resin to Co-Cr alloy primed with COP or MPII were significantly greater than those of specimens primed with AB, MOBL or MRB and non-primed controls. This study indicated that COP and MPII are effective primers to obtain higher bond strength between resin and Co-Cr alloy.

#### **Petridis et al in 1999<sup>10</sup>**

The purpose of this study was to compare the shear bond strength between a laboratory-cured composite and a high-noble Au-Pd-Ag alloy that used a commercial metal-resin bonding system with the bond strength of conventional porcelain fused to metal. Effects of water storage and thermocycling were also evaluated. Mean shear bond strength values before and after wet storage and thermocycling were 29.66 and 22.91 MPa for the PFM group; 21.43 and 17.92 MPa for the Siloc group; and 19.34 and 15.64 MPa for the etched group, respectively. Wet storage and thermocycling caused a significant decrease in shear bond strength of all specimens. Shear bond strength of conventional feldspathic PFM was significantly higher than that of the 2 metal-resin bonding techniques tested.

#### **Hideo Matsumura in 2001<sup>14</sup>**

The purpose of this study was to evaluate the surface preparation effects of 4 metal conditioners and 1 adhesive system on bonding between a prosthetic resin composite veneering material and a gold casting alloy. Four primers containing sulfur derivative monomer and designed for conditioning noble metal alloys (Alloy Primer,

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Infis Opaque Primer, Metal Primer II, and Metaltite) and a surface modification technique (Siloc) were assessed. It was concluded that all of the primed and Siloc-treated groups showed improved 24-hour shear bond strengths compared with the control group. After thermocycling, the groups either primed with the Metaltite conditioner or treated with the Siloc system exhibited the highest mean shear bond strengths. The Metaltite conditioner and Siloc system each represent a useful method for improving the bond between the gold alloy and resin composite material tested.

#### **Eunice Teresinha Giampaolo in 2001<sup>7</sup>**

This investigation evaluated the bond strength of two composite resin veneer systems to Nickel-Chromium (Ni-Cr) castings using three methods of promoting bonding of the resin to metal. The three resin-to-metal bonding systems used were mechanical bonding (S<sub>1</sub>), Silicoater system (S<sub>2</sub>) and mechanical bonding with the Silicoater system (S<sub>3</sub>). Two veneering resin materials were evaluated Chromasit - (M<sub>1</sub>); Dentacolor - (M<sub>2</sub>). The M<sub>2</sub>/S<sub>3</sub> combination had significantly higher bond strength than any of the other combinations. Mechanical bonding system (S<sub>1</sub>) provided better retention than the other bonding systems for material M<sub>1</sub>. The bonding system S<sub>2</sub> gave the lowest bonding strength values for both resins.

#### **Mihai Rominu in 2002<sup>33</sup>**

This study investigated cervical microleakage between a Co-Cr alloy and 4 indirect polymeric veneering materials used with different conditioning systems. Sixteen metallic frameworks (copings) were obtained by fabricating 0.6-mm calibrated wax patterns on a master cast abutment. The wax patterns were provided with 0.4-mm beaded retention on the veneering surfaces and cast in a Co-Cr based alloy (Biosil F) used for fixed partial dentures.

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The Co-Cr copings were divided equally into 4 groups and veneered with 4 polymeric materials (Signum, Solidex, Superpont C+B, and Targis). Three chemical systems (Siloc, Targis-Link, and Silicoater MD) were used with the respective veneering system recommended by the manufacturer; Conolar opaque was used for Superpont C+B. It was concluded that the extent of cervical microleakage between the coping and veneer depended on the particular polymeric material used for veneering. The extremes of the study were Targis/Targis-Link (lowest leakage scores) and Superpont C+B (highest leakage scores). Differences among the chemical retention systems tested were not significant.

#### **Takahashi et al in 2002<sup>48</sup>**

This study compared the probability of failure of three highly filled resin-veneered restorations (Artglass, Targis, Estenia) to that of conventional metal-ceramic restorations when used as implant-supported prosthesis. The effect of the location of load application on the fracture resistance of the restorations was also studied. Samples were prepared by application of three resins on Type IV gold. Compressive load was applied vertically at 1 mm and 2 mm from the periphery of the occlusal table until the restorations failed.

The probability of failure of resin-veneered restorations tested was not significantly different from that of the metal-ceramic restoration. Loading the resin-veneered restorations at the 1 mm location significantly increased their probability of failure when compared to the 2 mm loading location. Thus, eccentric loading of resin-veneered restorations should be minimized as higher probability of failure was associated with such a loading condition.

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**P. K. Vallittu in 2003<sup>56</sup>**

The aim of this study was to measure push-out bond strength of gold alloy and pure titanium bars to dimethacrylate polymer-glass fibre composite. Metal bars were either left untreated, or sandblasted with 110 µm grain size aluminium oxide, pyrolytically silica-coated (Silicoater, HeraeusKulzer) and silanized, tribochemically silica-coated, silanized (Rocatec, 3M-ESPE) and heat treated (100 °C / 10 min), or tribochemically silica-coated and left unsilanized. Light-polymerizable opaquer was applied on the metal surface and unidirectional glass fibres with Bis-GMA-PMMA monomer-polymer gel matrix was placed in contact to the bars and polymerized.

For titanium and gold alloy silicacoatings with the silane treatments resulted in the highest bond strengths. Values for titanium were somewhat higher than those for gold alloy. Silica-coating without silane treatment gave lower bond strengths but higher than that obtained with aluminium oxide sandblasting. The results suggest that pyrolytical silica-coating with silane application resulted in highest bond strengths of dimethacrylate based fibre-reinforced composite to pure titanium and gold alloy.

**Joon-Young Kim in 2003<sup>20</sup>**

The purpose of this in vitro study was to evaluate the effect of steam cleaning, boiling, ultrasonic cleaning (laboratory procedures), and thermocycling on shear bond strength of resin bonds to metal and denture teeth. Resin-metal bonding systems and their specific veneer resins (Rocatec, Sinfony; Rocatec, Visio-Gem, HLC-BOND, Zeta LC and Ducera experimental veneer resin) were tested on a Co-Cr alloy (Wirobond C). Shear strength tests (MPa) were performed with a universal testing machine until fracture.



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Simulation of laboratory procedures and thermocycling caused a significant drop in shear bond strength of metal-resin and denture tooth-resin bonds for most of the tested veneer resins. Thermocycling before shear testing had the same effect on veneer resin bond strength as the simulated laboratory procedures.

### **Luciana Silva Andrade Tarozzo in 2003<sup>29</sup>**

The aim of this invitro study was to analyze and compare the tensile strength between the base metal Ni-Cr alloys: (Wirocer and Wiron 99) and 2 composites (Artglass and Solidex) with 2 types of mechanical retention (0.4-mm and 0.6-mm beads); 2 types of chemical treatment (Siloc system and Metal Photo Primer system) and the combination of both treatments on a metal framework; to verify whether these composite systems were strong enough to be used as alternatives to porcelain.

It was concluded that mechanical retention with 0.6-mm beads and chemical/mechanical retention with 0.6-mm beads increased the metal-composite tensile bond strength. No significant difference was found between these 2 retention systems. Chemical and airborne particle abrasion retentions were statistically weaker than the other types of retention tested. All values observed were higher than that recommended for metal-ceramic dental restorative systems (25 MPa-ISO 9693)

### **Petridis et al in 2003<sup>11</sup>**

The purpose of this study was to compare the shear bond strength between a medium-gold, high-noble alloy and three resin composite veneer systems (Artglass, Sculpture, Targis) with that between the same alloy and a feldspathic porcelain. The

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mean shear bond strength values (in MPa), before and after wet storage and thermocycling, were 30 and 23 for the metal-ceramic group, 29 and 23 for the Artglass group, 20 and 19 for the Sculpture group, and 17 and 14 for the Targis group, respectively. No group exceeded the shear bond strength of the metal-ceramic group, but the Artglass group with its respective metal-resin bonding system exhibited similar bond strengths. The Sculpture group showed a stable bond after water storage and thermocycling.

### **M. Rosentritt in 2005<sup>41</sup>**

The aim of this invitro study was to determine the thermal mechanical properties of veneering composites after polymerization with the appropriate polymerization device. Fracture tests were performed to investigate the effect on fixed partial dentures (FPDs). Dynamic mechanical thermal analysis was used to determine the temperature-dependent mechanical properties. To approximate the clinical situation, the fracture resistance of three-unit metal-based FPDs with different composite veneering was investigated after a simulated 5-year oral wearing period. The restorations were made of a high gold alloy and veneered with three different composites. To determine the influence of fabrication, one composite was used in a light-polymerizing and a heat/pressure- curing version and, in addition, a newly developed heat protection paste was used. After a 5-year simulation period, the fracture resistance was determined. The Adoro-veneering with the heat protection paste showed the highest median fracture strength (1700 N), followed by Adoro LC (1555 N), Belleglass (1051 N), Adoro HP (1150 N) and Sinfony (909 N). The most common failure type occurring in all FPDs was a cracking of the composite, exposing the metal framework. All FPDs showed stress cracking of the composite. The heat protection paste seemed to reduce the crack formation after fabrication and increased the fracture resistance of the composite veneering.

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Stress cracking after thermal cycling and mechanical loading affected all composites, but all veneered three unit alloy FPDs showed a fracture resistance sufficient for posterior application

**Seimenis et al in 2006<sup>43</sup>**

This study compared the shear bond strength of three commercial veneering resins (Artglass, Solidex, Signum+) to a Ni-Cr alloy, using two different bonding procedures. The effect of different numbers of thermal cycles on the durability of the bond was also investigated. The cast specimens to which the veneering resins were applied contained 150 µm diameter beads. The shear bond strength values of all veneer resin surface treatment combinations tested exceeded the requirements of ISO 10477.

Solidex resin/Silco combination at 1000 cycles exhibited the highest bond strength. There was no significant effect of the different number of thermal cycles and the two surface treatments on the metal-resin bond strength, when Solidex and Signum+ were used. Solidex exhibited an adhesive failure mode, while combined failures were observed with Artglass and Signum+ for all surface treatments and thermal cycles used.

**Ciftci et al in 2007<sup>60</sup>**

The aim of this study was to evaluate the shear bond strength of four esthetic veneering materials on nickel-chromium (Ni-Cr) alloy. Four treatments for veneering: conventional porcelain-fused to-metal (PFM), Artglass, Targis/Vectris, and Biodent light-cured prosthodontic composite resins were used. After sandblasting of the cast metal surfaces with 50µm alumina, the composites were applied to the surfaces

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according to manufacturer's recommendations. Shear bond strength was determined at a crosshead speed of 0.5 mm/min. Mean shear bond strength values were 34.96 MPa for PFM, 14.17 MPa for Targis/Vectris, 13.64 MPa for Artglass, and 10.56 MPa for Biodent. It was concluded that the PFM group exhibited significantly higher bond strength values compared with the other three groups.

#### **A C L Faria et al in 2008<sup>8</sup>**

This study evaluated the effect of chemical and mechanical surface treatments for cast metal alloys on the bond strength of an indirect composite resin (Artglass) to commercially pure titanium (cpTi). The metal rods in grade-1 cpTi received the following surface treatments: sandblasting; chemical treatment; mechanical treatment-0.4 mm beads; mechanical treatment-0.6 mm beads; chemical/mechanical treatment-0.4 mm beads ; chemical/mechanical treatment-0.6 mm beads. Artglass rings were light cured around the cpTi rods.

It was found that chemical retention and sandblasting showed statistically similar results and both had significantly lower bond strengths than the other treatments. In conclusion , mechanical retention, either associated or not to chemical treatment, provided higher bond strength of the indirect composite resin to cpTi.

#### **Manish Katyayan in 2011<sup>30</sup>**

The objective of this investigation was to evaluate and compare the shear bond strength of UDMA based composite, restorative composite, and heat cure acrylic when veneered to Ni-Cr alloy and to evaluate the type of bond failure. The greatest mean shear bond strength was recorded in relation to the UDMA based composite material when thermal

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conducting paste was used during the curing (10.51 MPa). The mean bond strength values of UDMA based composite without thermal conducting paste (8.92 MPa), heat cured acrylic veneering material (4.24 MPa) and restorative composite material (5.03 MPa) were significantly different from each other ( $p > 0.05$ ). UDMA based composite veneering material when used with heat protection paste exceeds the shear bond strength requirement as suggested by Matsumura et al. ( $> 10$  MPa). A statistically significant association between the test groups and the type of failure was observed.

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*Materials &*

*Methods*

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## MATERIALS AND METHODS

The present in vitro study was conducted to evaluate and to compare the shear bond strength between Nickel-Chromium metal and SR ADORO resin processed by “Lost salt crystal technique” using salts of different crystal shapes such as sodium chloride (cubic shape), potassium permanganate (irregular shape), ammonium chloride (spherical shape). The materials used in the study are,

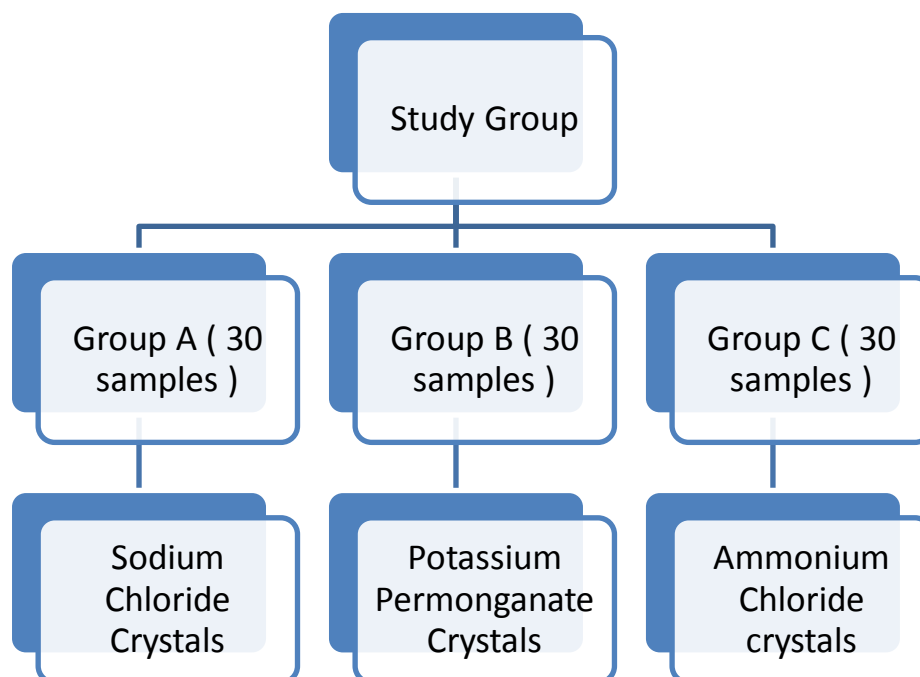
| S.NO | MATERIALS                                                                         | MANUFACTURER                                                                      |
|------|-----------------------------------------------------------------------------------|-----------------------------------------------------------------------------------|
| 1.   | Addition polymerization polyvinyl siloxane impression material- Putty consistency | Reprosil Vinyl Polysiloxane<br>Impression Material<br>Dentsply USA                |
| 2.   | Universal wax                                                                     | Dentic All wax No. 702-0500(Blue, Transparent),<br>Renfert, Germany               |
| 3.   | Sprue wax                                                                         | (GEO Wax Wire, Turquoise,<br>Hard, No. 676-2050, 4.0<br>mm/6 ga) Renfert, Germany |
| 4.   | Salt crystals-Sodium chloride, Potassium permanganate, Ammonium chloride          | SDFCL, Mumbai                                                                     |
| 5.   | Debubblizer (Waxit)                                                               | Degu Dent GmbH, Germany                                                           |
| 6.   | Ring liner                                                                        | Fiber ring asbestos-free ring<br>liner, No. 1752-1005,                            |

|     |                                                                                                              |                                                                        |
|-----|--------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------|
|     |                                                                                                              | Renfert, Germany                                                       |
| 7.  | Phosphate bonded investment                                                                                  | Bellavest SH , Bego,<br>Germany                                        |
| 8.  | Nickel-Chromium alloy                                                                                        | MEAlloy, Dentsply, UK                                                  |
| 9.  | Dental stone (Type IV-Gypsum die stone)                                                                      | GC Asia Dental PTE Ltd,<br>India                                       |
| 10. | Vaccum forming sheet                                                                                         | Easy-Vac Gasket-GB 040 1.0<br>mm,size-12.7×12.7 cm, 3A<br>MEDES, Korea |
| 11. | SR ADORO resin kit<br>-SR Link<br>-SR ADORO Opaquer<br>-SR ADORO Dentin<br>- SR ADORO Thermoguard<br>-SR Gel | Ivoclar Vivadent, AG<br>Schaan/Liechtenstein                           |
| 12. | Autopolymerizing resin (Pink colour)                                                                         | DPI-RR Cold Cure, Dental<br>Products of India, Mumbai                  |



## GROUPING OF THE SAMPLES:

The samples were grouped as follows.



A total of 90 samples were prepared and grouped into the following 3 groups (30 samples per group)

**Group A :** Samples impregnated with sodium chloride crystals

**Group B :** Samples impregnated with potassium permanganate crystals

**Group C :** Samples impregnated with ammonium chloride crystals

## **STUDY METHODOLOGY:**

For evaluation of bond strength the methodology followed was,

- 1. Fabrication of wax pattern**
- 2. Impregnation of salt crystals in the wax pattern**
- 3. Elimination of salt crystals**
- 4. Spruing of the pattern**
- 5. Investing, Casting, Finishing and Polishing of the samples**
- 6. Fabrication of transparent matrix**
- 7. Veneering of ADORO resin**
- 8. Mounting of test samples in the shear testing jig**
- 9. Evaluation of shear bond strength**

## **FABRICATION OF WAX PATTERN**

A Split metal die was made to produce the wax pattern for the test samples. The die was constructed in 2 parts enclosing the mold space to produce the wax pattern.

### **Parts of Metal Die:**

The metal die consists of 2 parts: A Lower part and an Upper part

### **Lower part of the die:**

It has the indentations for the larger size disc measuring about 12 mm in diameter and 4 mm in thickness.

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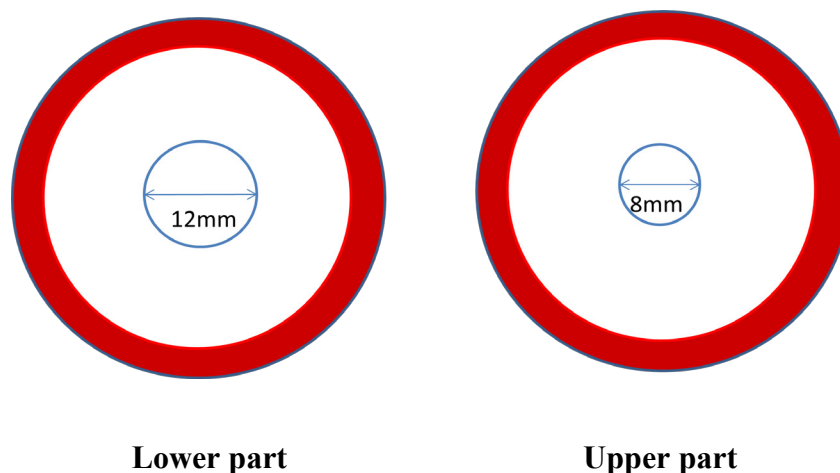
**Upper part of the die:**

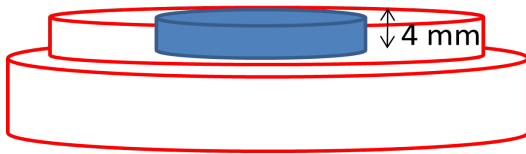
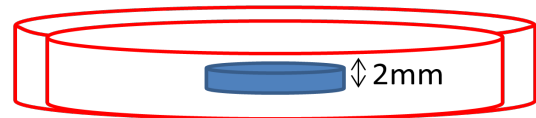
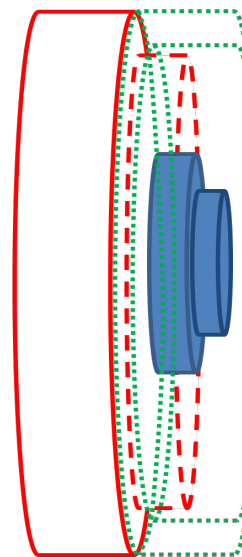
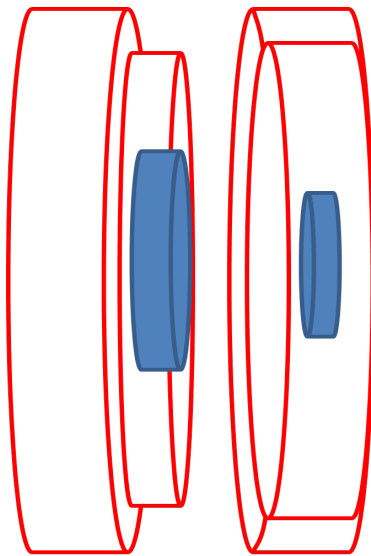
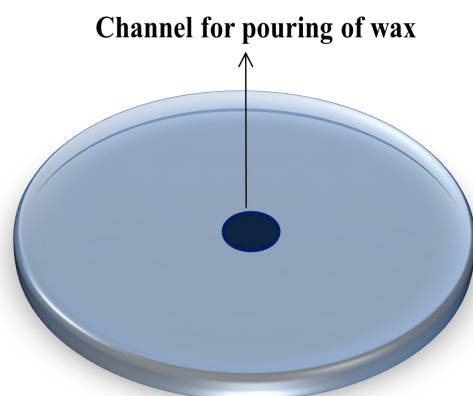
It has the indentations for the smaller size disc measuring about 8 mm in diameter and 2 mm in thickness. There is a through and through hole of 4mm diameter in the centre of the 8 mm diameter indentation through which wax was poured into the mould space when approximated.

**Orientation platform:**

The two parts of the metal die can be oriented with the help of the orientation platform. The lower part of the die has an elevated platform all around the 12 mm diameter indentation, a few millimetres from the outer rim of the die. Similarly the upper part of the die has depression all around to engage the elevated platform on the opposite side. This helped in orientation of the two parts of the die. The schematic diagram of the metal die used to prepare the wax pattern is shown in **Fig 1. 1**.

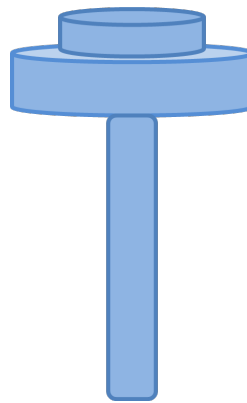
**FIG 1.1.SCHEMATIC REPRESENTATION OF THE METAL DIE**

**A) TOP VIEW**

**B) LATERAL VIEW****Lower part****Upper part****C) ORIENTATION OF TWO PARTS OF THE DIE****D) APPROXIMATED METAL DIE****E) WAX PATTERN**

Using the metal die, 90 patterns (Thirty patterns for each group) were prepared with Universal wax (**Fig 1.7** Dentic All wax No. 702-0500 (Blue, Transparent), Renfert, Germany) (**Fig 1.8**). A straight sprue (**Fig 1.7** GEO Wax Wire, Turquoise, Hard, No. 676-2050, 4.0 mm/6 ga) Renfert, Germany) of dimension 6 gauge was attached to the rear end of the 12 mm disc (**Fig 1.9**). It helped in handling of the pattern during embedment of salt crystals (SDFCL, Mumbai) and also in future mounting of disc in the shear testing jig. The schematic diagram of the wax pattern with the handle is shown in **Fig 1. 2**

**Fig 1. 2 WAX PATTERN WITH HANDLE**



### **IMPREGNATION OF SALT CRYSTALS IN THE WAX PATTERN**

The salt crystals (**Fig 1.10** SDFCL, Mumbai ) used in the study were sodium chloride (cubic shape), potassium permanganate(irregular shape), ammonium chloride(spherical shape).

The salt crystals (SDFCL, Mumbai) were spread out in a sheet of paper and was kept over the vibrator to allow even distribution of salt crystals (SDFCL, Mumbai) (**Fig 1.11**). The working end of the wax pattern was blown in flame and was pressed over the salt crystals (SDFCL, Mumbai). The salt crystals (SDFCL, Mumbai) were embedded into the wax pattern (**Fig 1.12**).

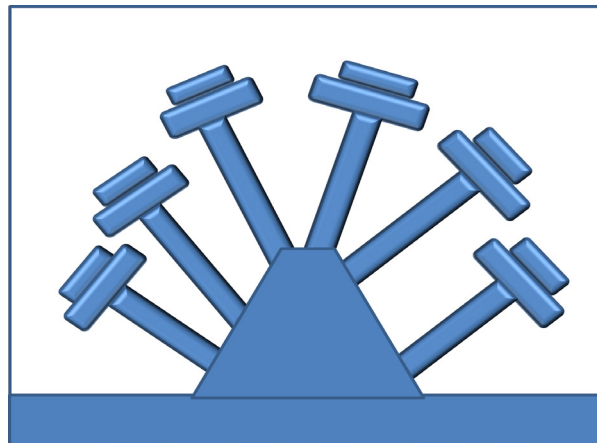
## ELIMINATION OF SALT CRYSTALS

The embedded salt crystals were then washed under running tap water (**Fig 1.13**). The surface tension of the wax was dispelled with alcohol.

## SPRUNG OF WAX PATTERN

The wax patterns were sprued in such a way that the pattern itself served as the sprue. A total of ten patterns were attached to the base of the crucible former and debubbler (Waxit, Degu Dent GmbH, Germany) was applied (**Fig 1.14**). All the patterns were sprued in groups and casted as described below. The patterns were attached to the crucible former as shown in **Fig 1.3**.

**Fig 1.3 SPRUING OF SAMPLES**



## INVESTING, CASTING, FINISHING AND POLISHING OF THE SAMPLES

Appropriate metal ring (no. 9) was selected such that the patterns were approximately 6mm inside from the top surface of the metal ring and had approximately 3mm clearance from the side wall of the ring. The metal ring was lined with a single layer of ring liner (Fiber ring asbestos-free ring liner, No. 1752-1005, Renfert, Germany) approximately 3mm short from both the ends of the ring. It was wet with water to allow hygroscopic expansion.

Phosphate Bonded Investment (Bellavest SH, Bego, Germany) (**Fig 1.15**) was mixed according to manufacturer's instructions (160 gm : 35 ml). The metal ring was placed on the vibrator and a small amount of investment (Bellavest SH, Bego, Germany) was painted on the pattern with brush and then the rest of the ring was invested (**Fig 1.16**). The ring was allowed to set at room temperature without disturbance and kept for burnout after one hour.

The casting ring was placed in the furnace at room temperature and the temperature of the furnace was increased to 950° C over a period of 1 1/2 hours and then maintained at that temperature for another 30 min as specified by the manufacturer.

The crucible was preheated. The nickel-chromium alloy (MEAlloy, Dentsply, UK) required for each group was weighed using an electronic weighing machine. The amount of nickel-chromium alloy (MEAlloy, Dentsply, UK) required for each casting was determined by the following formula.

$$\text{GRAM OF ALLOY REQUIRED} = \text{WEIGHT OF PATTERN} \times \text{DENSITY OF ALLOY (8.4 gm)}$$

The nickel-chromium alloy (MEAlloy, Dentsply, UK) was melted in the induction casting machine and when the margins of the alloy started rounding the spring was released.

The casting rings were allowed to cool to room temperature and then divested. The castings were then retrieved and were immersed in 36% hydrofluoric acid solution for 30 sec to facilitate removal of the investment material, followed by cleaning in an ultrasonic cleaner for 15 min and then dried. Since sandblasting the salt crystals treated surface will alter the surface morphology, the rest of the areas except for the salt crystals treated surface were sandblasted using 50 $\mu$  grit aluminium oxide.

The sprues were cut using carborundum disc and the surface of the samples except the bonding surface were smoothed using tungsten carbide trimmer to remove surface irregularities (**Fig 1.17, 1.18, 1.19**).

## **FABRICATION OF TRANSPARENT MATRIX**

Universal wax (**Fig 1.7** Dentic All wax No. 702-0500 (Blue, Transparent), Renfert, Germany) was added over the treated surface of the casted pattern to a thickness of 2mm (**Fig 1.20**). The metal sample together with the wax-up was embedded in addition polymerization polyvinyl siloxane impression material- putty consistency (Reprosil Vinyl Polysiloxane Impression Material Dentsply USA) to the level of the metal disc leaving the handle to create a mould space (**Fig 1.21**).

Type IV dental stone (GC Asia Dental PTE Ltd, India) was poured into the mould space and the stone dies were obtained (**Fig 1.22**). Vacuum forming sheet (**Fig 1.23** Easy-Vac Gasket-GB 040 1.0 mm, size-12.7×12.7 cm, 3A MEDES, Korea) of thickness 1mm was vacuum pressed over the stone dies obtained. The matrix thus obtained had room for the SR ADORO resin (Ivoclar Vivadent, AG Schaan/Liechtenstein) which was later added over the metal casting (**Fig 1.24**).

## **VENEERING OF ADORO RESIN**

SR Link (Ivoclar Vivadent, AG Schaan/Liechtenstein), a metal-composite bonding agent that provides a covalent bond between the metal and SR ADORO (Ivoclar Vivadent, AG Schaan/Liechtenstein) composite resin, was applied. A clean disposable brush was used to apply SR Link (Ivoclar Vivadent, AG Schaan/Liechtenstein) and allowed to react for 3 min (**Fig 1.25**). Precaution was taken not to soak the alloy surfaces in SR Link (Ivoclar Vivadent, AG Schaan/Liechtenstein). The desired amount of opaque paste (SR ADORO Opaquer, Ivoclar Vivadent, AG Schaan/Liechtenstein) was removed from the syringe and



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applied after slightly spreading out on a mixing pad using a disposable brush and precured (initial curing) for 20 sec, using the Quick curing light (Quick, Ivoclar Vivadent, AG Schaan/Liechtenstein). Second opaquer layer was applied in such a way to completely cover the first layer and precured for 20 sec with the quick light curing unit (Quick, Ivoclar Vivadent, AG Schaan/Liechtenstein). After precuring, the samples were polymerised directly in the Lumamat (program 2/11 min) (Ivoclar Vivadent, AG Schaan/Liechtenstein). A disposable sponge was used to thoroughly remove the inhibition layer formed during the curing. It was made sure that the surface of the opaque had a silky mat finish (**Fig 1.26**).

The dentin veneering resin (SR ADORO Dentin, Ivoclar Vivadent, AG Schaan/Liechtenstein) was then applied over the opaque layer and the transparent matrix fabricated was used as an index to veneer the resin (SR ADORO Dentin, Ivoclar Vivadent, AG Schaan/Liechtenstein) to a thickness of 2mm (**Fig 1.27**). The veneered resin (SR ADORO Dentin, Ivoclar Vivadent, AG Schaan/Liechtenstein) along with the transparent matrix was precured for 20 seconds (**Fig 1.28**). After precuring, the transparent matrix was removed (**Fig 1.29**).

SR Gel (Ivoclar Vivadent, AG Schaan/Liechtenstein), a glycerine-based masking gel that is impervious to oxygen was applied to the restorations before final polymerization to minimize the formation of inhibition layer on the surface of the veneering composite. Consequently the gel ensures complete curing of restoration surfaces.

SR Gel (Ivoclar Vivadent, AG Schaan/Liechtenstein) was applied on the entire veneering surface in a fully covering manner. After applying SR Gel (Ivoclar Vivadent, AG Schaan/Liechtenstein), all exposed alloy parts of the specimen were covered homogeneously with thermal conducting paste (SR ADORO Thermo Guard, Ivoclar Vivadent, AG

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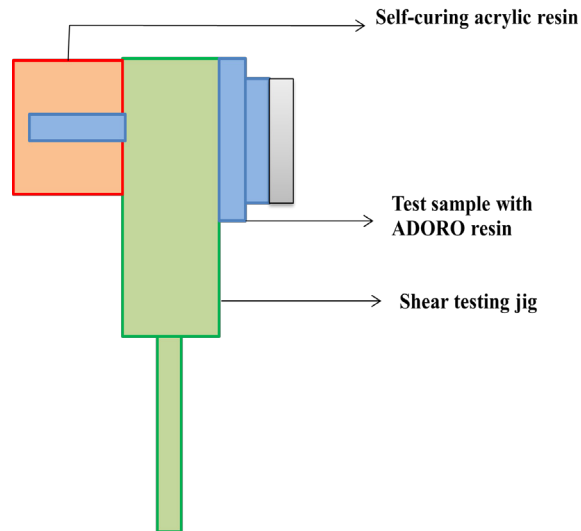
Schaan/Liechtenstein) (**Fig 1.30**). This paste is applied to provide a thermal absorbing effect, thereby minimizing the internal tension at the interface between metal and veneering resin.

The final curing was carried out as per the manufacturer's instructions, which included polymerization for 25 min (program 3) in Lumamat 100 (Ivoclar Vivadent, AG Schaan/Liechtenstein). After completion of polymerization procedure, SR Gel (Ivoclar Vivadent, AG Schaan/Liechtenstein) and the thermal conducting paste (SR ADORO Thermo Guard, Ivoclar Vivadent, AG Schaan/Liechtenstein) was completely removed from the specimen using a steam blaster.

The veneered resin was finished with cross-cut tungsten carbide burs and fine diamonds under low rpm and light pressure. The finished surfaces were polished with rubber polishing wheels (**Fig 1.31, 1.32,1.33**). The samples were tested after 24 hours of dry storage at room temperature.

#### **MOUNTING OF TEST SAMPLES IN THE SHEAR TESTING JIG**

A total of 90 machined rectangular jigs measuring 25 mm × 12mm with a handle on one end and a through and through hole on the other end were fabricated (**Fig 1.34**). The hole on one end enabled insertion of the metal-resin test sample from one side (**Fig 1.35**) and secured on the other side with the help of self – curing acrylic resin (DPI-RR Cold Cure, Dental Products of India, Mumbai) (**Fig 1.36**). The handle on the other end was used to mount the samples in the universal testing machine (**Fig 1.37** Instron Universal Testing Machine, Model no 5500R,Atwood,USA). The test samples are mounted in the shear testing jig as shown in **Fig1.4**.

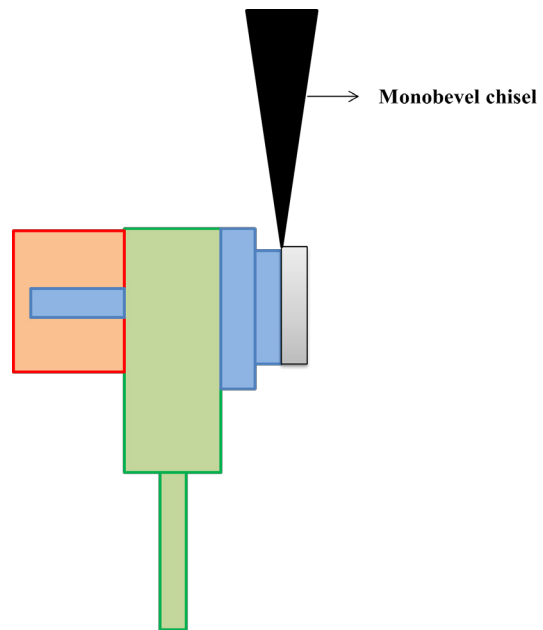
**Fig 1. 4 MOUNTING OF SAMPLE IN SHEAR TESTING JIG**

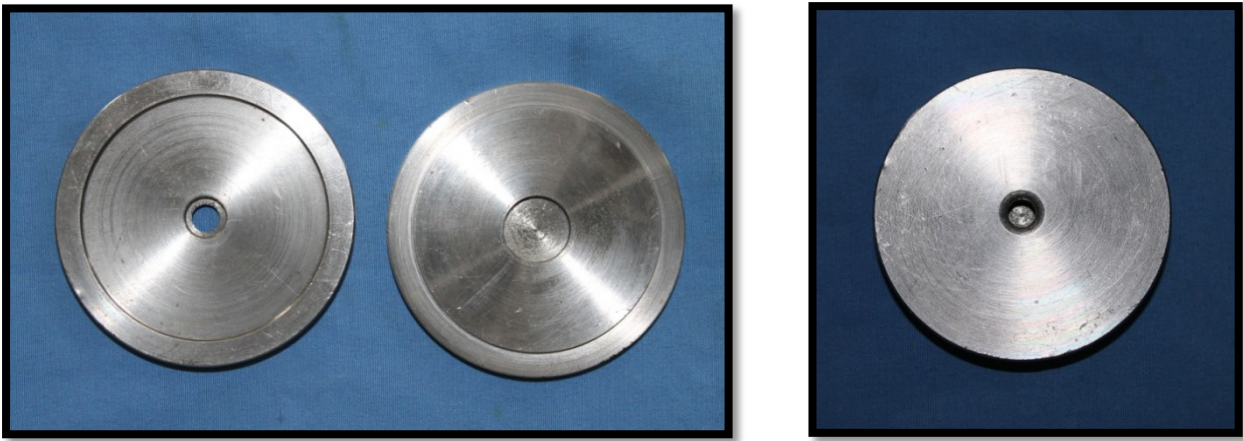
### EVALUATION OF SHEAR BOND STRENGTH

The mounted samples were tested for shear bond strength on an Universal testing machine ( Instron Universal Testing Machine, Model no 5500R, Atwood,USA). A specially made mono bevel chisel (**Fig 1.38**) was used to apply a shearing force on the test sample at the metal-resin interface as shown in **Fig 1.5**. The specimens were loaded to failure and shear bond strength was measured with the Universal testing machine (Instron Universal Testing Machine, Model no 5500R, Atwood,USA) at a crosshead speed of 1mm/min. A computer attached to the testing machine recorded the load at which the bond failure occurred. The values were obtained in Megapascals.

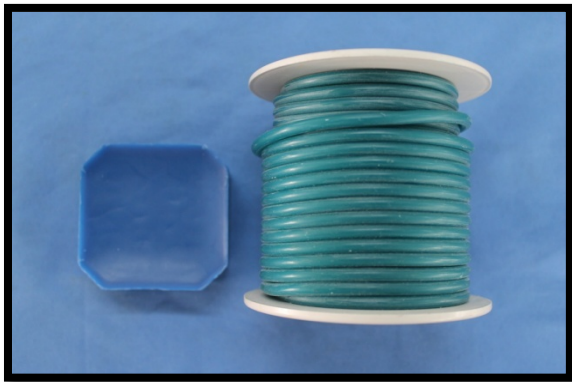
The mode of failures were evaluated visually. On visual examination , when there was no traces of resin material (SR ADORO, Ivoclar Vivadent, AG Schaan/Liechtenstein) adhering to the metal surface, it was termed adhesive failure (**Fig 1.42**) and if part of or traces of resin (SR ADORO, Ivoclar Vivadent, AG Schaan/Liechtenstein) was seen adhering to the metal surface , it was termed cohesive failure (**Fig 1.41**).

**Fig 1. 5 TESTING OF SHEAR BOND STRENGTH**

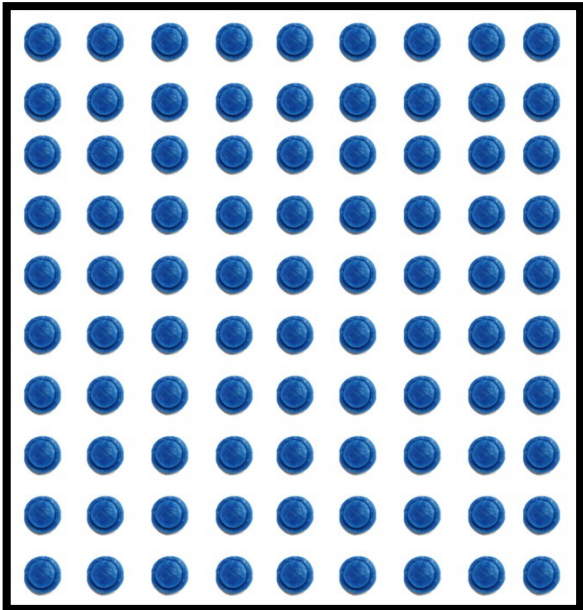




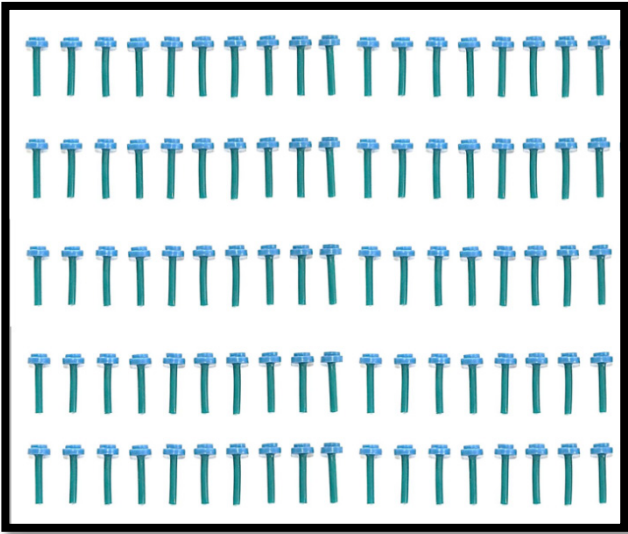
**Fig 1.6 Metal die**



**Fig 1.7 Universal wax (Dentic All wax No. 702-0500(Blue, Transparent), Spue wax (GEO Wax Wire, Turquoise, Hard, No. 676-2050, 4.0 mm/6 ga) Renfert, Germany)**



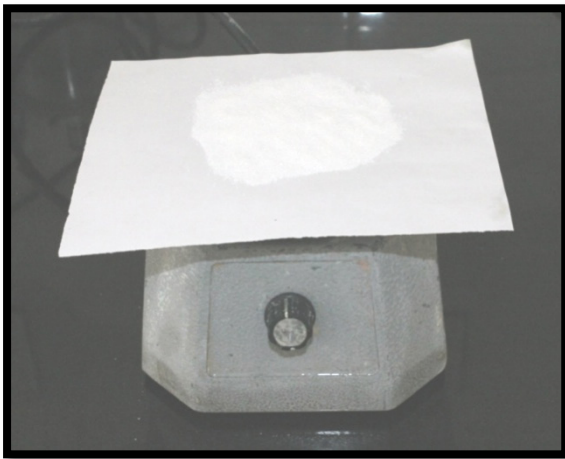
**Fig 1.8 Wax pattern**



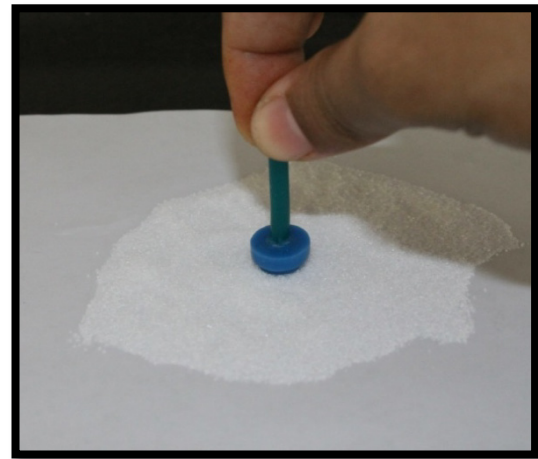
**Fig 1.9 Wax pattern with handle**



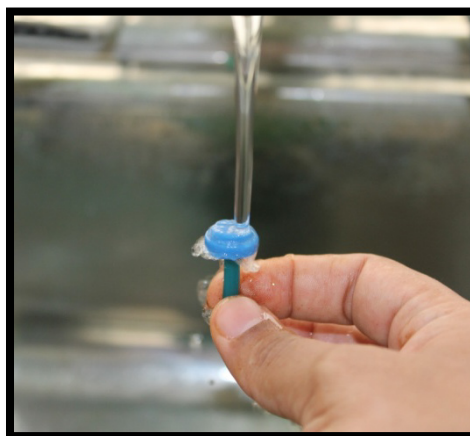
**Fig 1.10 Salt crystals (SDFCL, Mumbai)**



**Fig 1.11 Placement of salt crystals over vibrator**



**Fig 1.12 Embedment of salt crystals**



**Fig 1.13 Washing in running tap water**



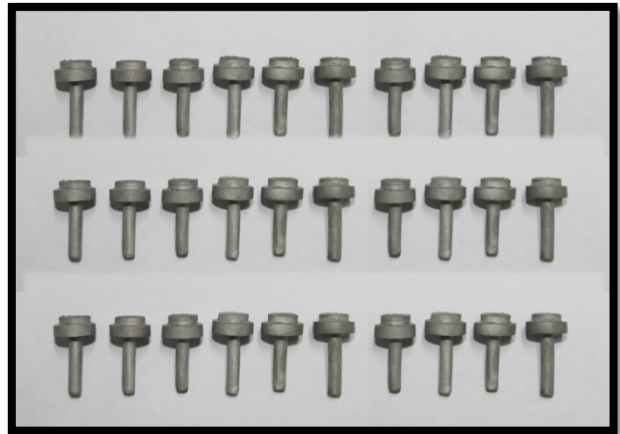
**Fig 1.14 Spruing of wax pattern**



**Fig 1.15 Armamentarium for investing**



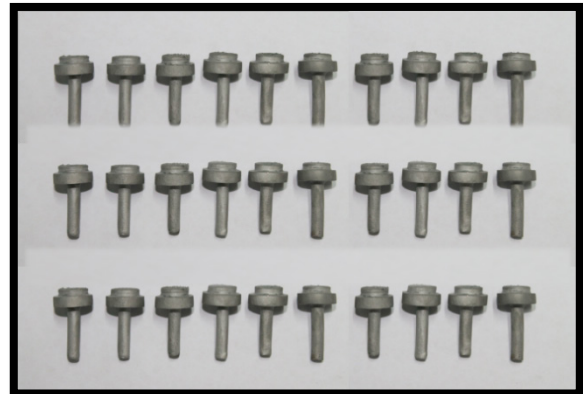
**Fig 1.16 Investing of the wax pattern**



**Fig 1.17 Casted samples-Group A**



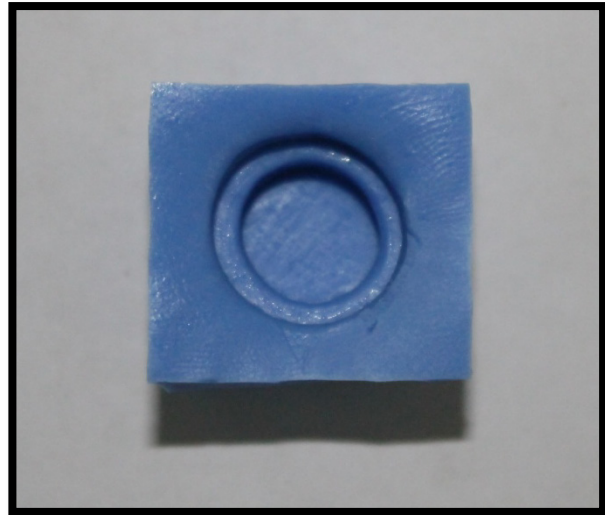
**Fig 1.18 Casted samples-Group B**



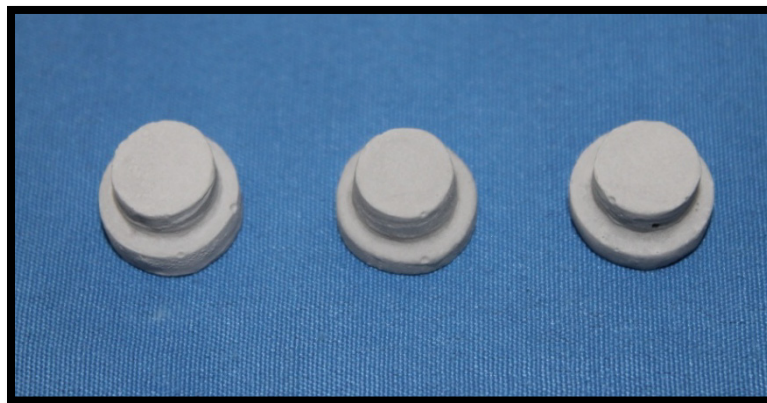
**Fig 1.19 Casted samples-Group C**



**Fig 1.20 Wax up for resin over casted pattern**



**Fig 1.21 Putty index of the waxed up pattern**



**Fig 1.22 Stone model**



**Fig 1.23 Vaccum forming sheet (Easy-Vac Gasket-GB 040 1.0 mm,size-12.7×12.7 cm, 3A MEDES, Korea)**



**Fig 1.24 Transparent matrix**

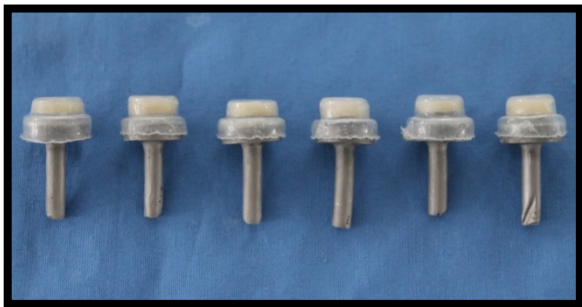




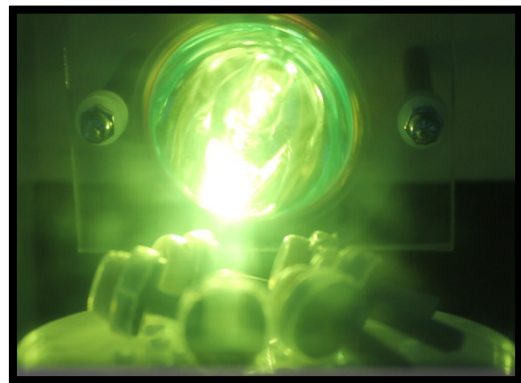
**Fig1.25 Application of SR Link (Ivoclar Vivadent, AG Schaan/Liechtenstein)**



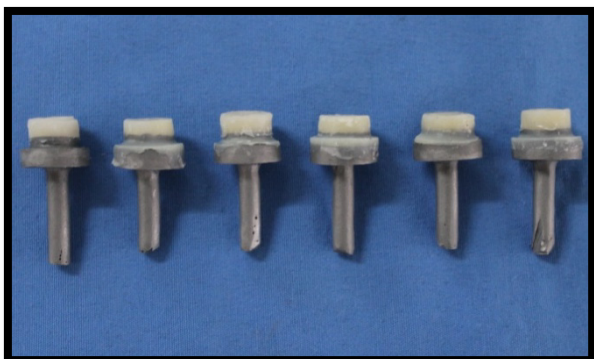
**Fig 1.26 Application of opaquer (Ivoclar Vivadent, AG Schaan/Liechtenstein)**



**Fig 1.27 SR Adoro resin (Ivoclar Vivadent, AG Schaan/Liechtenstein) application using the transparent matrix**



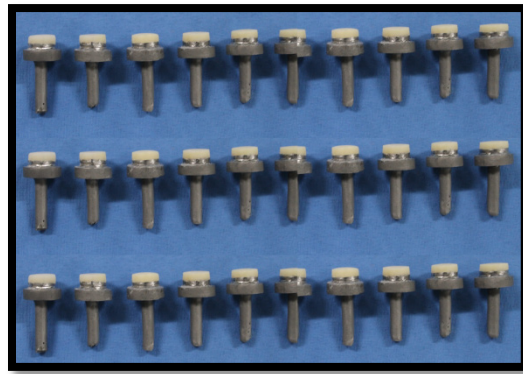
**Fig 1.28 Curing of samples**



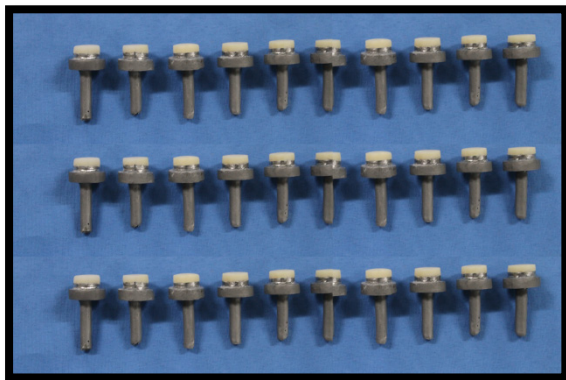
**Fig 1.29 Samples after initial curing**



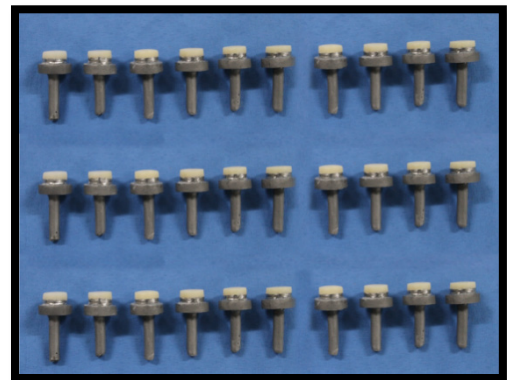
**Fig 1.30 Application of SR Gel and SR Thermoguard (Ivoclar Vivadent, AG Schaan/Liechtenstein)**



**Fig 1.31 Finished samples-Group A**



**Fig 1.32 Finished samples-Group B**



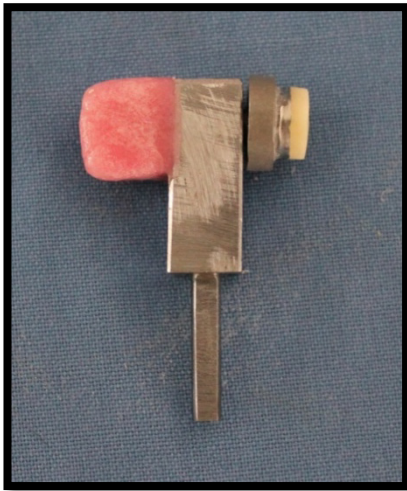
**Fig 1.33 Finished samples-Group C**



**Fig 1.34 Shear testing jig**



**Fig 1.35 Placement of sample in the jig**



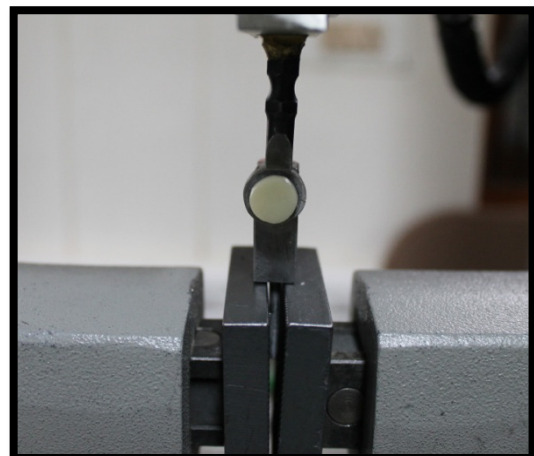
**Fig 1.36 Test sample mounted in the jig using autopolymerising resin (DPI-RR Cold Cure, Dental Products of India, Mumbai)**



**Fig 1. 37 Instron Universal testing machine Model.5500,Atwood, USA**



**Fig 1.38 Force application tip**



**Fig 1.39 Mounting of sample in the testing machine**



**Fig 1.40 Application of shear force in the testing machine**



**Fig 1.41 Test sample showing cohesive failure**



**Fig 1.42 Test sample showing adhesive failure**

# *Results*

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## RESULTS

The present in vitro study was conducted to evaluate and to compare the bond strength of metal and SR ADORO resin processed by “Lost salt crystal technique” using salts of different crystal shapes.

The bond strengths of Group A (samples impregnated with sodium chloride crystals), Group B (samples impregnated with potassium permanganate crystals), and Group C (samples impregnated with ammonium chloride crystals) were compared. The bond strengths of the three groups and the mean values are given in the **Table1**.

**Table 1 SHEAR BOND STRENGTH VALUES OF THREE GROUPS AND THEIR MEAN VALUES**

| S. No | Group A<br>(30 Samples)<br>Sodium Chloride<br>(MPa) | Group B<br>(30 Samples)<br>Potassium<br>Permanganate<br>(MPa) | Group C<br>(30 Samples)<br>Ammonium<br>Chloride<br>(MPa) |
|-------|-----------------------------------------------------|---------------------------------------------------------------|----------------------------------------------------------|
| 1.    | 19.4535                                             | 14.9625                                                       | 13.812                                                   |
| 2.    | 18.108                                              | 14.841                                                        | 14.718                                                   |
| 3.    | 17.568                                              | 12.0375                                                       | 12.048                                                   |
| 4.    | 18.54                                               | 15.2955                                                       | 13.284                                                   |
| 5.    | 17.7795                                             | 15.6555                                                       | 14.628                                                   |
| 6.    | 18.873                                              | 11.007                                                        | 15.012                                                   |
| 7.    | 18.6345                                             | 13.3875                                                       | 12.81                                                    |
| 8.    | 17.9235                                             | 13.941                                                        | 13.47                                                    |
| 9.    | 20.034                                              | 15.534                                                        | 12.054                                                   |

|             |               |                |                |
|-------------|---------------|----------------|----------------|
| 10.         | 18.099        | 15.237         | 12.612         |
| 11.         | 16.9065       | 15.129         | 15.012         |
| 12.         | 20.853        | 14.058         | 15.034         |
| 13.         | 20.5515       | 13.3515        | 15.204         |
| 14.         | 16.0515       | 14.283         | 12.81          |
| 15.         | 20.07         | 15.6555        | 13.47          |
| 16.         | 19.4535       | 14.157         | 12.054         |
| 17.         | 18.108        | 12.0375        | 12.612         |
| 18.         | 17.568        | 15.1425        | 15.012         |
| 19.         | 18.54         | 12.0375        | 12.774         |
| 20.         | 17.7795       | 13.3515        | 12.03          |
| 21.         | 19.323        | 13.0725        | 13.452         |
| 22.         | 18.6345       | 11.0385        | 12.81          |
| 23.         | 17.9235       | 14.3415        | 13.47          |
| 24.         | 20.034        | 15.4575        | 12.054         |
| 25.         | 18.099        | 15.552         | 12.612         |
| 26.         | 16.9065       | 12.4605        | 15.012         |
| 27.         | 20.853        | 14.3055        | 12.738         |
| 28.         | 20.0565       | 14.157         | 14.898         |
| 29.         | 21.9555       | 14.2875        | 13.852         |
| 30.         | 20.07         | 17.8515        | 15.198         |
| <b>Mean</b> | <b>18.825</b> | <b>14.9625</b> | <b>13.5518</b> |

**Table 1** shows the shear bond strengths of three groups. The mean values of bond strength of groups A, B, and C were 18.825 MPa, 14.9625 MPa and 13.5518 MPa respectively.

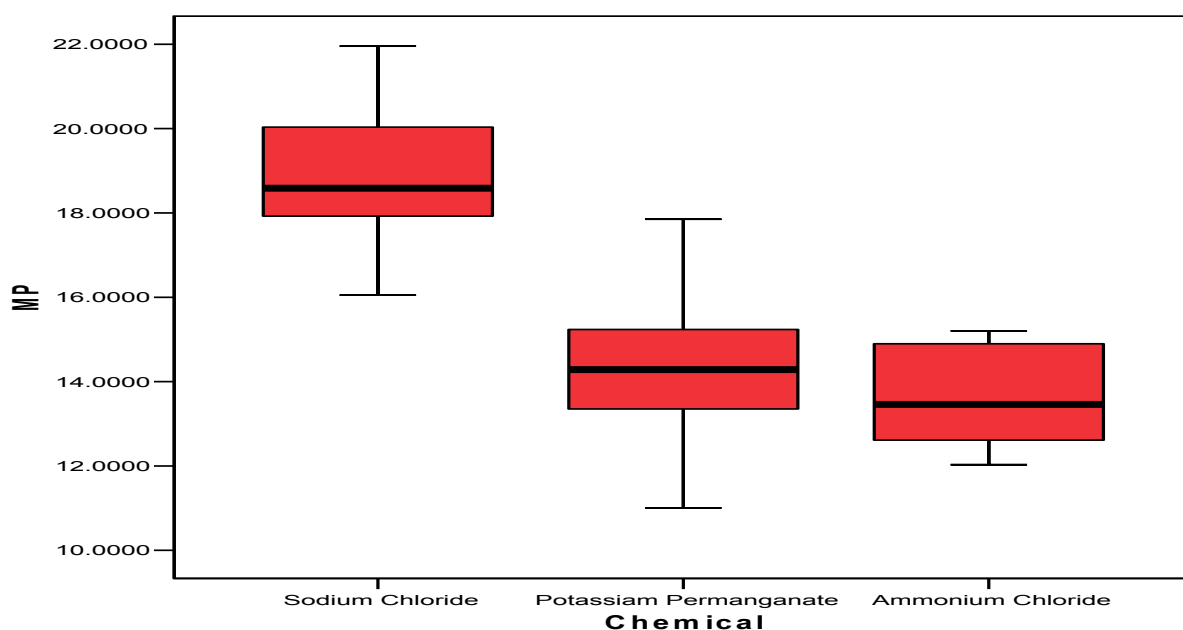
**TABLE 2 NATURE OF FAILURE AND THE PERCENTAGE OF FAILURE IN EACH GROUP**

|       |   | Failures      |                | Total |
|-------|---|---------------|----------------|-------|
|       |   | Adhesive      | Cohesive       |       |
| Group | A | 3<br>(10%)    | 27<br>(90%)    | 30    |
|       | B | 7<br>(23.33%) | 23<br>(76.66%) | 30    |
|       | C | 12<br>(40%)   | 18<br>(60%)    | 30    |
| Total |   | 22            | 68             | 90    |

**STATISTICAL ANALYSIS**

**BOX AND WHISKER PLOT**

**Graph 2.1 COMPARISION OF DISTRIBUTION OF THREE GROUPS**





**TABLE 3 DESCRIPTIVE MEASURES SHOWN IN BOX AND WHISKER PLOT**

| Chemical                                      | N  | Mean   | Std. Deviation | 95% Confidence Interval for Mean |             | Minimum | Maximum |
|-----------------------------------------------|----|--------|----------------|----------------------------------|-------------|---------|---------|
|                                               |    |        |                | Lower Bound                      | Upper Bound |         |         |
| Group A(30 samples)<br>Sodium Chloride        | 30 | 18.825 | 1.363          | 18.315                           | 19.334      | 16.051  | 21.955  |
| Group B(30 samples)<br>Potassium Permanganate | 30 | 14.120 | 1.535          | 13.547                           | 14.694      | 11.007  | 17.851  |
| Group C(30 samples)<br>Ammonium Chloride      | 30 | 13.551 | 1.139          | 13.126                           | 13.977      | 12.030  | 15.204  |
| Total                                         | 90 | 15.499 | 2.728          | 14.927                           | 16.070      | 11.007  | 21.955  |

Since the standard deviation is lower for Group C (1.39) and more for Group A (1.363) and Group B (1.535), the observations may vary in broader interval for Group A and Group B, but for Group C it varies moderately.

#### **STATISTICAL ANALYSIS:**

The values were statistically analysed using One-way ANOVA test.

#### **ONE-WAY ANOVA:**

One-way ANOVA is essentially a procedure for testing the difference among different groups of values for homogeneity of the groups.

**Hypothesis:**

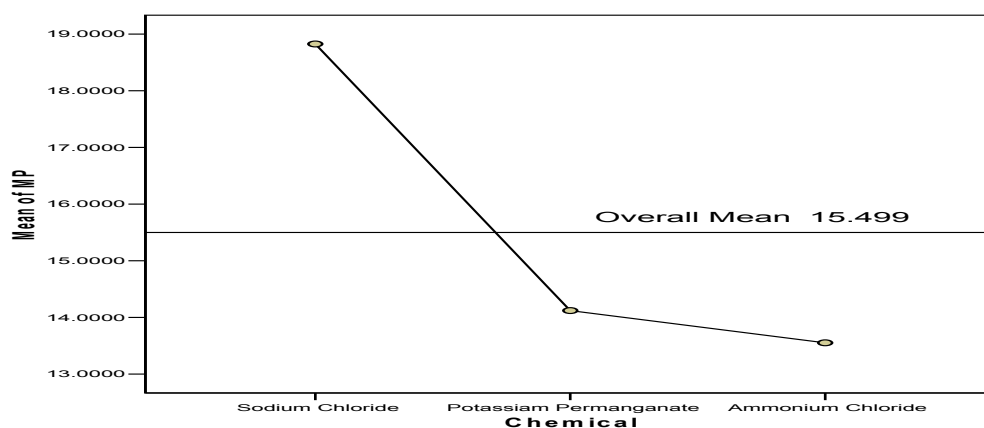
**Null hypothesis  $H_0$** - There is no significant difference between the shear bond strength values of three groups.

**Alternative hypothesis  $H_1$** -There is significant difference between the shear bond strength values among three groups.

**TABLE 4 ONE WAY ANOVA**

| Source of Variation                | Sum of Squares | D.F. | Mean Square | F       | Sig. Value (p) | Significant or Not |
|------------------------------------|----------------|------|-------------|---------|----------------|--------------------|
| <b>Between Groups A, B &amp; C</b> | 502.587        | 2    | 251.294     | 136.723 | 0.0            | Significant        |
| <b>Within Groups A, B &amp; C</b>  | 159.904        | 87   | 1.839       |         |                |                    |
| <b>Total</b>                       | 662.491        | 89   |             |         |                |                    |

From the **Table 4**, the one way ANOVA test was performed to test the Null Hypothesis. Since the p value is less than 0.05, the Null hypothesis was rejected at 5% level of significance. The alternative hypothesis was accepted and it was concluded that there is significant difference between the shear bond strength value among three groups

**Graph 2.2 MEAN SHEAR BOND STRENGTH VALUES OF THREE GROUPS**

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In one way ANOVA when the null hypothesis is rejected, it is concluded that all group means are not equal. But the test does not indicate the comparative equality of pairs of means. Therefore t-test is applied.

t- test is a method to do multiple pair wise comparisons. If the difference between two group's means is considerably bigger than the general variation, then it is inferred that there is a significant difference. Since there is a significant difference among groups, t- test is employed to compare the groups pair wise based on mean values. The t statistic values and p values are given in **Table 6**.

### **t-TEST FOR INDEPENDENT SAMPLES**

#### **NULL HYPOTHESES FOR t-TESTS:**

##### **For t-test 1:**

$H_0$  – There is no significant difference between the mean bond strength values of Sodium chloride and Potassium permanganate impregnated samples.

##### **For t-test 2:**

$H_0$  – There is no significant difference between the mean bond strength values of Sodium chloride and Ammonium chloride impregnated samples.

##### **For t-test 3:**

$H_0$  – There is no significant difference between the mean bond strength values of Potassium permanganate and Ammonium chloride impregnated samples.

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**TABLE 5 GROUP STATISTICS**

| Chemical               | n  | Mean   | Std. Deviation |
|------------------------|----|--------|----------------|
| Sodium Chloride        | 30 | 18.825 | 1.363          |
| Potassium Permanganate | 30 | 14.120 | 1.535          |
| Ammonium Chloride      | 30 | 13.551 | 1.1391         |

**TABLE 6 t-TEST FOR EQUALITY OF MEANS**

| t-test no. | t-test for Comparison b/w                  | t-test for Equality of Means |           |        |     |              |                    |
|------------|--------------------------------------------|------------------------------|-----------|--------|-----|--------------|--------------------|
|            |                                            | Mean Difference              | Std Error | T      | d f | Sig. p-value | Significant or not |
| 1          | Sodium Chloride & Potassium Permanganate   | 4.704                        | 0.000     | 12.548 | 58  | 0.000        | Significant        |
| 2          | Sodium Chloride & Ammonium Chloride        | 5.273                        | 0.108     | 16.255 | 58  | 0.000        | Significant        |
| 3          | Potassium Permanganate & Ammonium Chloride | 0.569                        | 0.3490    | 1.630  | 58  | 0.108        | Not significant    |

From **Table 6**, t-test was used to test the Null Hypothesis. Since the p value is less than 0.05, the Null hypothesis was rejected at 5% level of significance for tests 1 and 2, whereas the null hypothesis was accepted for test 3. The alternative hypothesis was accepted for tests 1 and 2 and it was concluded that :

- There is a significant difference between the mean bond strength values of Sodium chloride and Potassium permanganate impregnated samples.
- There is a significant difference between the mean bond strength values of Sodium chloride and Ammonium chloride impregnated samples.

- There is no significant difference between the mean bond strength values of Potassium permanganate and Ammonium chloride impregnated samples.

**TABLE 7 EVALUATION OF MODE OF FAILURE**

|       |   | Failures |          | Total |
|-------|---|----------|----------|-------|
|       |   | Adhesive | Cohesive |       |
| Group | A | 3        | 27       | 30    |
|       | B | 7        | 23       | 30    |
|       | C | 12       | 18       | 30    |
| Total |   | 22       | 68       | 90    |

**Null hypothesis  $H_0$** -There is no significant difference in mode of bond failure among the three groups

**Alternate hypothesis  $H_1$** -There is a significant difference in mode of bond failure among three groups

**TABLE 8 CHI-SQUARE TEST**

| Chi-square value ( $\chi^2$ ) | Significant p-value | Significant / Not |
|-------------------------------|---------------------|-------------------|
| 7.34                          | 0.025               | Significant*      |

Chi square test was used to test the null hypothesis. Since the p value was less than 0.05, the null hypothesis was rejected at 5% level of significance. The alternative hypothesis was accepted and it was concluded that there is a significant difference in failure modes among three groups.

# *Discussion*

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## DISCUSSION

The growing demand for esthetics has made people to seek restorations that closely resemble natural teeth. This esthetic demand has risen through the years and is achieved in many instances by veneering the metal base of a restoration with resin or porcelain.

Although resin veneers have been a part of dental practice for over 40 years, their use has been generally contraindicated due to lack of certain important physical properties. These include low strength, abrasion resistance, lack of colour stability, high coefficient of thermal expansion and the lack of an adequate bonding system to the metallic substrate. The advantages often cited with resin veneering materials include ease of fabrication, low cost, lack of wear to opposing dentition and the ability to repair veneers clinically<sup>9</sup>.

Though porcelain is being considered superior to resin in certain properties<sup>6,7,10</sup>, the cost factor, high abrasion of opposing teeth and need for resin-veneered metal crown in case of screw-retained implant-supported prosthesis have raised the demand for resin veneered restorations<sup>48</sup>.

The new generation highly filled laboratory composites meet up to the expectations by being cost effective, having abrasion resistance similar to tooth structure, low coefficient of thermal expansion and transfer of minimal load underneath when used with implants<sup>6,17,10,48</sup>.

Several attempts have been made in the past to create and develop techniques, for bonding composite materials and metals. This led to the development of various surface-conditioning techniques.

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With the advent of adhesive dentistry, competition in developing techniques and adhesive materials with increased bond strength has emerged between dental scientists and manufacturers. Though a number of chemical methods of retention have been introduced, mechanical retention method is still considered valuable in bonding resin to metal<sup>7</sup>.

**Jones et al** stated that when resins were attached to the alloy with retentive beads, higher shear bond strengths were evident than with chemical bonding techniques<sup>18,19</sup>.

**Tanaka et al** stated that mechanical retention with the aid of spherical powders of acrylic resin provided better retention when compared to commercially available mechanical retention methods<sup>50</sup>.

Tin plating is a surface treatment recommended for improving the strength and durability of the bond between adhesive resins and the metal<sup>35</sup>. With electrochemical etching, on the other hand, it is difficult to create a properly etched surface, and the technique does not work well with precious alloys. The method is also technique sensitive, requires expensive equipment and is time consuming<sup>27,28,35</sup>.

A frequently mentioned criticism of etching non-precious alloys is the complexity and technique sensitivity of the treatment. In this regard, Silicoater is also complex and technique sensitive<sup>35</sup>.

Rocatec system is simpler than tin plating. It was developed to permit chemical bonding between resin veneers and the metal framework, but the simplicity of the technique makes it a potentially useful surface treatment in fixed prosthodontics. Rocatec, Silicoater and Tin plating surely improve bond strength compared to sandblasting.

Mechanical retention systems using beads, wires, loops, mesh have been used traditionally. These methods require more space for the metal structure and more tooth

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reduction in order to provide enough thickness of esthetic material. Another disadvantage of the mechanical retention was the development of gap between the metal and the resin<sup>3,42,49,50</sup>.

Though every method had its own advantages and disadvantages, the mechanochemical retention system was recommended for bonding metallic alloys to composites<sup>2,4,17,18,19,25,29,32,37,57,58</sup>.

**Barzilay et al** evaluated the mechanical and chemical retention of laboratory light-cured composites to base metal and found mechanochemical retention techniques to be better when compared to individual retention systems<sup>17</sup>.

The present invitro study was taken up to evaluate the mechanochemical bond strength of nickel-chromium alloy to SR ADORO resin. The commonly followed mechanical retention method for SR ADORO resin involves fabrication of the metal framework with retention beads. Acrylic retention beads are incorporated in the wax pattern and casted. Acrylic retention beads for SR ADORO resin as recommended by the manufacturer are available in two sizes: 200-300  $\mu\text{m}$  (micro retention) and 400-600  $\mu\text{m}$  (macro retention).

The casted metal framework with the retention beads are usually over contoured. To provide room for the veneering resin and to make the restoration esthetically pleasing, the manufacturers of SR ADORO resin recommend flattening of the retention beads by trimming the surface of the retention beads till the height of contour. This provides the needed undercut for the resin material to get locked and at the same time prevents over contouring of the restoration<sup>44,45</sup>.

Though this method offers a good mechanical retentive feature, the entire procedure is time consuming and careful monitoring of the thickness of the metal framework is mandatory to avoid over contouring.

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This in vitro study was conducted in an attempt to overcome the difficulties of the retention beads by use of salt crystals to provide the needed mechanical retentive feature.

The “Lost salt technique” introduced by **Moon and Hudging et al** was introduced for resin bonded prosthesis to provide retention between the prosthesis and the luting cement<sup>15,34</sup>. Conventional mechanical retention techniques using beads, loops, mesh, wires were used earlier<sup>3,42,49,50</sup>. Their use resulted in restorations that were bulkier and was not esthetically pleasing, which was considered a major disadvantage. To overcome the drawbacks of the previous mechanical retention features, the use of “Lost salt technique” was advocated in this study. This method reduces the bulk of the veneering material by creating depressions on the surface rather than elevations as in case of commercially available mechanical retentive systems like beads, loops, wires and mesh, at the same time providing the needed mechanical retentive feature. SR link, a component of SR ADORO resin kit provides the chemical bond by forming a covalent bond between the metal and SR ADORO resin. The SR ADORO opaquer also promotes a reliable bond to SR Link and the veneering composite.

A total of 90 samples were prepared and were divided into 3 groups (30 samples per group). Group A included samples impregnated with sodium chloride crystals(cubic shape), Group B included samples impregnated with potassium permanganate crystals(irregular shape) and Group C included samples impregnated with ammonium chloride crystals(spherical shape).

The salt crystals before impregnation were spread out over a sheet of paper and kept over a vibrator to ensure uniform thickness of salt crystals. The casting variables like alloy-investment combinations, liquid-powder ratio of investment, burn-out temperature, heat source and casting method were optimized to achieve identical groups of samples.

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In clinical situations, the thickness of a resin facing measures roughly about 2mm. To mimic the exact thickness in the study, a transparent matrix was prepared to ensure uniform thickness of veneering resin in all the samples.

The samples were subjected to shear bond strength analysis in the Universal testing machine to mimic the shear force in the oral environment to which the facings are subjected to. The mean values of shear bond strength of groups A, B and C were 18.825 MPa, 14.9625 MPa and 13.5518 MPa respectively (Table1).

According to **ISO 10477:2004 Dentistry-Polymer Based Crown and Bridge Materials<sup>16</sup>**, the bond strength requirement of metal-resin was stated to be a minimum of 5 MPa. The bond strength values obtained in this study were well above the ISO limits.

The failures were predominantly cohesive in nature ,suggestive of better bond strength with the present study. In Group A, there were 3 adhesive failures & 27 cohesive failures; in Group B, there were 7 adhesive failures & 23 cohesive failures; in Group C, there were 12 adhesive failures & 18 cohesive failures (Table 2).

One way ANOVA test was used to test the variation in bond strength among the three groups. The one way ANOVA test was performed to test the null hypothesis. Since the p value is less than 0.05, it was concluded that there is significant difference between the shear bond strength values among the three groups (Table 4). But the test did not indicate the comparative equality of pairs of means. So t-test was done to do multiple comparisons. From the test result it was inferred that there is significant difference between the mean shear bond strength of Group A & Group B(4.704) and Group A & Group C(5.273), but there is no significant difference between Group B & Group C (Table 6). From the result, it could be inferred that the shear bond strength of Group A was superior to Groups B and C.

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Chi-square test was used to test the significance in mode of failure between the three groups. Since the p value was less than 0.05, it was concluded that, there is a significant difference in the failure modes among the three groups (Table 8).

Mechanical or chemical retention of metal to resin is always related to bonding area. The larger the bonding area, the stronger the retention between metal and resin. A larger bonding area can be obtained by roughening with lost salt crystals<sup>59</sup>. As per mathematical formula, the surface area of cube is larger than the surface area of sphere. The cavities made by the cube shaped salt crystals have five walls, thus providing an increased surface area compared with the area available on a smooth surface. In fact, sphere is the shape with minimal surface area among all bodies of the same volume, by the isoperimetric equality. Since the surface area of the irregular shaped crystals could not be calculated and the samples impregnated with that shape of crystals exhibited a lower bond strength compared to those impregnated with sodium chloride crystals, sodium chloride was considered superior in providing the needed mechanical retention feature.

In addition the SR Link, a component of SR ADORO kit provided the chemical bond by forming a covalent bond between metal and the SR ADORO resin.

It has been claimed that the newly developed heat protection paste increases the fracture resistance of composite veneering. This paste is meant to be applied to the unveneered metal surfaces to achieve a thermally absorbing (cooling effect), which is expected to minimize the tension at the interface between the metal and SR ADORO resin.

In this study, the samples were tested after 24 hours of dry storage at room temperature. The study was done to compare the shear bond strength of metal to SR ADORO resin by using salt crystals of three different shapes as a means of mechanical retention

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feature. The condition of oral environment was not mimicked by wet storage and thermocycling which are other criterias to be considered for the success of the restoration.

Further in vivo studies mimicking the oral environment and in vitro studies are needed to validate the use of salt crystals as a mode of mechanical retentive feature.

Hence from this in vitro study it can be inferred that combination of mechanical retention provided by “Lost salt technique” and chemical retention by SR Link provided a better bond strength needed for the success of a resin-bonded prosthesis.

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*Summary &  
Conclusion*

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## SUMMARY AND CONCLUSION

This study compared the bond strength of Nickel-Chromium alloy and SR ADORO resin processed by “Lost salt crystal technique” using salts of different crystal shapes and evaluated the type of bond failure.

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

1. The shear bond strength values between SR ADORO resin and metal using the three different salt crystals exceeded the requirements of ISO 10477.
  2. Statistically significant differences were observed between the shear bond strengths of SR ADORO resin and metal treated with three different salt crystals.
  3. The greatest mean shear bond strength was between the metal and SR ADORO resin treated with cube shaped salt crystals (18.825 MPa).
  4. The least mean shear bond strength was between the metal and SR ADORO resin treated with ammonium chloride salt crystals (13.5518 MPa).
  5. Among the three shapes of salt crystal tested, cube shaped crystals provided the greatest increase in metal-resin bond strength. The greatest bond strength was attributed to the increase in the surface area compared to the area available on a smooth surface.
  6. Roughening of metal surfaces with “Lost salt crystal technique” will considerably improve metal-resin bond strength.
  7. The majority of the failure modes were cohesive in nature. But the percentage of cohesive failure varied among the three groups.
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8. A Combination of mechanical and chemical retention should be provided for successful clinical outcome.
  9. Use of “Lost salt crystal technique” to attain mechanical retention reduces the bulk of the veneering material .This together with the use of SR Link provided the chemical retention thereby providing a better bond between the metal and resin.
  10. Further studies should be performed to assess these materials, with the ultimate goal of long-term prospective clinical trials.
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