

**COMPARATIVE EVALUATION OF THE EFFECT OF  
DIFFERENT SURFACE TREATMENTS ON THE SHEAR BOND  
STRENGTH BETWEEN SILICONE SOFT LINER AND  
DENTURE BASE RESIN – 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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**THE TAMILNADU Dr. M.G.R. MEDICAL UNIVERSITY  
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**DECLARATION BY THE CANDIDATE**

I hereby declare that this dissertation titled  
**“COMPARATIVE EVALUATION OF THE EFFECT  
OF DIFFERENT SURFACE TREATMENTS ON  
THE SHEAR BOND STRENGTH BETWEEN  
SILICONE SOFT LINER AND DENTURE BASE  
RESIN – AN *IN VITRO* STUDY”** is a bonafide and genuine  
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This dissertation is submitted to **THE TAMILNADU Dr. M.G.R. MEDICAL UNIVERSITY**, in partial fulfillment for the Degree of **MASTER OF DENTAL SURGERY – PROSTHODONTICS AND CROWN & BRIDGE, BRANCH I**. It has not been submitted (partial or full) for the award of any other degree or diploma.

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## INTRODUCTION

Prosthodontics is greatly concerned with the prevention and treatment of chronic soreness from denture and also with the preservation of supporting structures. The problem of managing patients with congenital or acquired anatomic abnormalities, detrimental psychological factors (bruxism), prosthesis with compromised retention or a combination of these problems with hard and rigid acrylic polymers is very difficult<sup>5,6,9,12,20,22,23,43</sup>.

With the advent of denture soft lining materials the management of these problems has been greatly enhanced by the softness and flexibility of these materials by virtue of their physical or chemical composition, and provide an opportunity to protect their supporting tissues from functional or parafunctional occlusal stresses.

Liners act as a cushion for the denture-bearing tissues by absorbing and redistributing forces transmitted to the stress-bearing areas of the edentulous ridges<sup>6,12,16,17,29,31</sup>. Liners also offer a valuable solution in the management of painful or fragile mucosa or ulcerated tissues associated with the wearing of dentures and provide comfort for patients who cannot tolerate occlusal pressures, such as in cases of alveolar ridge resorption, chronic soreness, and knife-edge ridges<sup>12,18</sup>. These materials have been found useful for treating patients with bony undercuts, bruxing tendencies, congenital or acquired oral defects requiring obturation, xerostomia, dentures opposing natural dentition in the opposing arch and for transitional prosthesis after

implant surgery<sup>6,7,9,17,22,23,32</sup>. The ideal properties for a soft liner include resilience, tear resistance, viscoelasticity, biocompatibility, adhesive bond strength, low solubility and low absorption in saliva, ease of adjustability, dimensional stability, colour stability, lack of adverse effect on denture base material, resistance to abrasion and ease of cleaning<sup>7,13,20</sup>.

Soft liners have been categorised based on durability, mode of processing and chemical composition. The ISO (International Organization for Standardization) categorizes a short term resilient liner as one used intraorally for a period of upto 30 days. They are also called as temporary soft liners or tissue conditioners. They are used for surgical procedures, diagnostic procedures, immediate placement of transitional removable partial dentures, immediate dentures, and other temporary situations to aid the healing of the tissues in contact with the denture. Liners intended to be used over a period of 1-6 months are categorized as intermediate liners. These are made of plasticized acrylic. They usually last for 1-2 months when placed in removable prosthesis, after which the liner loses the plasticizer and becomes stiff. They are mainly used when preprosthetic surgery is not indicated but the patient presents with bony undercuts or poor residual ridge anatomy, such as knife-edge ridge<sup>2,18</sup>.

Long term soft liners are those that are intended to function beyond and are indicated in situations when preprosthetic surgery is not indicated, but the patient has significant bony undercuts<sup>19</sup>.

Soft or resilient liners can also be classified as room temperature vulcanized (RTV) and heat temperature vulcanized (HTV). Soft liners can be further divided into 4 groups according to their chemical structure: a) plasticized acrylic resin either chemical or heat cured, b) vinyl resin, c) polymethane and poly phosphazine rubbers (d) silicone rubbers<sup>9,30</sup>.

Silicone based resilient lining material is similar in basic composition to silicone impression materials as both are dimethylsiloxane polymers<sup>33</sup>. Polydimethylsiloxane is a viscous liquid that can be cross linked to form a rubber with good elastic properties. Softness of these liners is controlled by the amount of cross-linking in the rubber and no plasticizer is necessary to produce a softening effect with this material<sup>43</sup>. Silicone liners have little or no chemical adhesion to PMMA resins and an adhesive is supplied to aid in bonding the liner to the resin denture base. Silicone liners have been reported to keep their softness for longer periods than acrylic resin liners<sup>12</sup>.

Common problems with the liners is interface colonization by *Candida albicans*, porosity, poor tear strength and loss of softness<sup>10,26,28,36</sup>. One of the most serious problems with these materials is bond failure between the resilient denture liner and denture base. According to glossary of prosthodontics terms, bond is the force that holds two or more units of matter together<sup>50</sup>. Bond failure creates a potential surface for bacterial growth, and plaque and calculus formation<sup>10,27</sup>. A variety of parameters affect the bond between resilient lining materials and the denture base, including water

absorption, surface primer use, nature of denture and temperature changes<sup>12,20,28,31</sup>. In vitro studies on the bond strength between soft liners and denture base resin have focussed on either tensile and/or shear bond strength testing. Since the forces that the lining material is clinically exposed are more closely related to shear and tear test, the shear test is considered an appropriate method for testing bond strength of soft liners to denture base resin<sup>25</sup>.

Several methods have been advocated to enhance the bonding of acrylic denture base to silicone soft liner. They can be broadly categorized into mechanical and chemical modifications or a combination of both<sup>6</sup>.

Mechanical modifications of denture base surface includes roughening with cutting or abrasive rotary instruments, placing diatorics and air abrading with aluminium oxide particles etc<sup>2,23,29</sup>. All these mechanical methods produces varying degrees of roughness and irregularities on the acrylic denture base surface which increases the surface area, thereby increasing the bond strength between silicone-based soft liner and acrylic denture base<sup>22</sup>. A significant increase in bond strength has been reported in the literature by employing one of these methods for enhancing retention<sup>2,25,27</sup>.

Sand blasting (Air abrasion) procedure involves spraying a stream of aluminium oxide particles against the material surface intended for bonding under high pressure. Air abrasion using aluminium oxide is one of the commonly followed micromechanical method of producing surface irregularities. Aluminium oxide of various particle sizes has been employed to

enhance the bond between the silicone based soft liner and denture base resin<sup>2,23,26</sup>.

Progress in laser technology has shown a quick adoption for being used by many in the field of dentistry due to the development of the first working laser by Maiman in 1960. Recently, lasers have been found to provide a relatively safe and easy means of altering the bonding surface of materials. Theoretically, it should benefit the bonding interface and result in stronger bond. Laser irradiation with various lasers like Er:YAG, Er,Cr:YSGG, Nd:YAG, KTP lasers have been reported to modify the intaglio surface of the denture before application of liner materials. It has been indicated in one study that Er,Cr:YSGG laser treatment may enhance the bond between silicone soft liner and denture base resin. However, literature using Er,Cr:YSGG laser as a method of surface modification of denture base before bonding with silicone soft liner is sparse<sup>27</sup>.

Soft denture liners are expected to function in the aqueous oral environment for long periods of time as well as under rapidly changing temperatures. However it must be noted that with cyclic temperature, as encountered in the oral cavity, the thermal behaviours of the structural components within a material can influence the latter's mechanical, physical properties especially the bond strength. In this connection, the thermocycling process conducted in invitro studies, can give useful data on the longevity of soft denture liners with respect to bond strength under conditions that simulate clinical usage. The effect of thermo cycling on the tensile bond strength of

denture liners has been widely reviewed by authors<sup>12,16,20,32,35,38</sup>. Adequate data on the effect of thermocycling on the shear bond strength of soft liners is lacking which is more critical than tensile loading, as shear bond strength can also impact the tensile bond strength<sup>6,16,40,49</sup>.

Surface modifications can bring about changes in surface topography which can affect the bonding between silicone resin and denture base resin<sup>36</sup>. Hence analysis of surface topography by 3D surface profilometry can provide valuable insights into the microsurface irregularities, surface roughness and their impact on the bond strength.

Scanning Electron Microscopy (SEM) analysis of debonded specimens have been studied to get useful information about mode of failure<sup>1,5,6,9,10,12,16,20,43,45</sup>. These qualitative interpretations can be used along with qualitative data obtained from bond strength testing for better interpretation and clarity of test results.

The paucity of data comparing the effect of laser surface treatment with other surface treatment methods on the shear bond strength between chair-side silicone-based soft liner and denture base resin prompted the present study, in view of its clinical impact and significance. Further, longevity of the bond between chair-side silicone reliners and denture base resins is still not clarified.

Hence, in light of the above, the aim of this present in-vitro study was to comparatively evaluate the effect of two different surface treatments on the

shear bond strength between silicone soft liner and heat polymerized denture base resin after thermocycling. The null hypothesis for the present study was that different surface treatments will not significantly affect the shear bond strength between silicone soft liner and heat polymerized denture base resin.

**The objectives of the present study were as follows:**

1. To assess the surface topography of one representative surface treated acrylic resin block from each test group by 3D surface profilometry.
2. To evaluate the shear bond strength between silicone-based soft liner and untreated surface of acrylic denture base resin using Universal Testing Machine.
3. To evaluate the shear bond strength between silicone-based soft liner and acrylic denture base resin surface treated by sandblasting using 110 $\mu$ m alumina using Universal Testing Machine.
4. To evaluate the shear bond strength between silicone-based soft liner and laser irradiated acrylic denture base resin surface treated by laser irradiation using Er,Cr:YSGG laser using Universal Testing Machine.
5. To compare between the mean shear bond strengths of silicone-based soft liner and untreated, sandblasted and laser irradiated acrylic denture base resin test samples respectively.

6. To compare between the mean shear bond strengths of silicone-based soft liner and untreated denture base resin test samples with that of silicone-based soft liner and sandblasted acrylic denture base resin test samples respectively.
7. To compare between the mean shear bond strengths of silicone-based soft liner and untreated denture base resin test samples with that of silicone-based soft liner and laser irradiated acrylic denture base resin test samples respectively.
8. To compare between the mean shear bond strengths of silicone-based soft liner and sandblasted denture base resin test samples with that of silicone-based soft liner and laser irradiated acrylic denture base resin test samples respectively.
9. To qualitatively analyse the mode of failure at the interface between silicone-based soft liner and untreated surface of acrylic denture base using scanning electron microscopy (SEM).
10. To qualitatively analyse the mode of failure at the interface between silicone-based soft liner and sandblasted acrylic denture base using scanning electron microscopy (SEM).
11. To qualitatively analyse the mode of failure at the interface between silicone-based soft liner and laser irradiated acrylic denture base using scanning electron microscopy (SEM).



## REVIEW OF LITERATURE

**Thomas J. Emmer et al (1995)<sup>17</sup>** evaluated the bond strength of five different soft lining materials (3 heat polymerized and 2 light polymerized) to heat processed PMMA resin using a new technique. The technique they developed represented an axial tensile mode of testing. The mode of failure was characterized using SEM analysis. Purely adhesive, purely cohesive, and mixed failures occurred depending on the type of relining material used.

**Nancy L. Jacobsen (1997)<sup>23</sup>** evaluated the effects of a specific sandblasted or lased preparation on the interfacial bonding of PMMA and silicone and PEMA resilient liners by treating the fabricated PMMA with three surface treatments: untreated (control), sandblast (250microm aluminium oxide particles), lased (carbon dioxide). PEMA and silicone were added to I and tested with American society for its peel strengths. Altering PMMA with sandblasting significantly reduced peel strength for PMMA-PEMA, PMMA-silicone specimens. Altering PMM with laser produced low peel strengths and were statistically significant from controls for PMMA-PEMA but no so from PMMA-silicone specimens. Untreated PMMA-PEMA peel strengths were significantly higher than PMMA-silicone which implies that the mechanical surface preparation of denture bases before application of a resilient may not be warranted.

**Fumiaki Kawano et al (1997)<sup>26</sup>** evaluated the bond strength of six soft denture liners by a two phase tensile test .The soft liners investigated were VinaSoft ,Prolastic , Flexor, Molloplast–B, Novus and Supersoft. The samples were fabricated by processing them (1) against polymerized poly (methyl methacrylate) and (2) against unpolymerised poly(methyl methacrylate. The bond strength when processed against unpolymerised PMMA and polymerized PMMA was 0.4to 2.60MPa and 0.94 to 2.56 MPa respectively. The samples were tested using an Instron Universal Testing Machine .Four of the six liners investigated demonstrated increased bond strength when processed against polymerized poly(methyl methacrylate ).it was conclude that bonding can be influenced by the processing method

**A.K.Aydin et al (1999)<sup>9</sup>** did study to investigate the bonding properties of five lining materials to a denture base resin. Two hard liners (chemical cured resin “Kooliner” and light cured resin “Triad”) and three soft liners (chemical-cured resin “Express”, Heat-temperature vulcanized (HTV) silicone material, Molloplast-B and room-temperature vulcanized (RTV) Ufi Gel-P) were used. Bonding strength and adhesion properties of the liners to the conventional heat cured poly methylmethacrylate (PMMA) denture base resin were compared by tensile test and scanning electron microscope (SEM) analysis. After curing, an aging process was applied and the samples were immersed and stored in distilled water at  $37 \pm 1^\circ\text{C}$  and taken out at certain intervals at (0, 15, 30 and 90 days) for examination. A total of 168 specimens

were processed for tensile tests and 24 specimens were processed for fracture tests. The results showed Triad (a hard liner) has the closest tensile strength to the control, indicating the strongest bonding between the base and the liner. Also, during the aging process, formation of better adhesion was observed for Molloplast-B in SEM micrographs. Molloplast-B and Express as resilient liners were found to have adequate adhesive values for clinical use.

**Yutaka Takahashi et al (2001)<sup>49</sup>** had undertaken a study to characterize the shear bond strength established between four denture base polymers and four denture reline polymers. Specimens were immersed in water for four months and then thermocycled. The result showed significant difference in bond strength among the specimens because of the denture base polymer variable, the denture reline polymer variable and their interaction. A light activated denture base polymer (Triad) bonded adequately with a light activated reline polymer (Triad) but less with the other reline polymers tested. The bond strength established between some denture base polymers and a different light activated reline polymer (Rebaron LC) was relatively low. They concluded that the type of denture base polymer and denture reline polymer affected the shear bond strength between them.

**Yutaka Takahashi et al (2001)<sup>48</sup>** also did another study to assess the shear bond strength between three denture reline materials and a denture base acrylic resin. Cylindric columns of denture reline materials were bonded to columns of denture base resins that received one of the following surface

treatments: application of dichloromethane, the monomer of the denture base resin, the recommended bonding agent or the monomer of the denture reline material, polishing with 240grit silicon carbide paper and air abrasion. A control group without surface treatment was included for each material. Specimens were immersed in water for 1 day and then thermocycled. The result showed that the Triad bonding agent and denture base monomer should be used in conjunction with Triad and GC reline, respectively, when relining a denture base resin.

**M. Al- Athel et al (2002)<sup>3</sup>** did a study to know the effects of long term immersion in water at  $37\pm 1^{\circ}\text{C}$  and of accelerated ageing in water at  $50\pm 1^{\circ}\text{C}$  on the tensile and shear bond strength values of Molloplast-B bonded to a heat cured denture base material. Immersion in water for 1 week at  $37\pm 1^{\circ}\text{C}$  had no significant effect on the measured bond strength values. They concluded that reduction in Molloplast-B bond strength that occurs as a result of long term ageing of water at  $37\pm 1^{\circ}\text{C}$  can be achieved in a shorter period of time by ageing the specimens in water at a higher temperature.

**Robert G.Jagger et al (2002)<sup>24</sup>** studied the effect of roughening the denture base surface on the tensile and shear bond strengths of a poly (dimethylsiloxane) resilient material bonded to a heat cured acrylic resin denture base material. Three groups of 10 specimens each were constructed for both tensile and shear tests. In the first group, Molloplast-B was packed against cured PMMA denture base surface. In the second group Molloplast-B

was packed against PMMA denture base roughened with acrylic bur. In the third group, Molloplast-B was packed against PMMA denture base acrylic resin dough. In the result Molloplast-B exhibited significantly higher tensile and shear bond strengths when packed against acrylic resin dough. Roughening the denture base surface prior to the application of Molloplast-B had a statistically significant weakening effect on tensile bond strength compared with the smooth denture base and the acrylic resin dough. For the shear bond strength, roughening the surface produced a non-significant increase compared with the smooth surface, but the bond was weaker than when packed against acrylic resin dough.

**Yasemin Kulak Ozkan et al (2003)**<sup>28</sup> did a study on the effect of thermocycling on tensile bond strength of six silicone based resilient denture liners namely Ufigel C, Ufigel P, Molloplast-B, Mollosil, Permafix, and permafex. The bond strength was determined, in tension after processing to PMMA. Half of the specimens for each group were stored in water for 24 hours and the other half were thermocycled (5000 cycles) between baths of 5° C and 55°C. The maximum tensile stress before failure and mode of failure were recorded. The mode of failure was characterized as cohesive, adhesive, or mixed mode. Results of this study also indicated that the bond strengths of soft lining materials had significantly decreased after thermocycling except Ufigel C and Mollosil. The adequate adhesive value for soft lining materials is given as 4.5 kg/cm<sup>2</sup> and all of the materials were acceptable for clinical use.

**Duygu Sarac et al (2006)**<sup>43</sup> did a study on the micro leakage and bond strength of a silicone based resilient liner following denture base surface pretreatment. Forty two PMMA denture base resin specimens consisting of two plates measuring 30 x 30 x 2 mm were prepared and divided into seven groups. Specimens were surface treated by immersing in acetone or methyl methacrylate and methylene chloride. One group with no surface treatment was served as the control group. The results showed that treating a denture based acrylic resin surface with chemical etchants prior to adhesive application reduced the micro leakage and increased the bond strength when using silicone based resilient liners. However, these chemical treatments decreased the flexural strength of the acrylic resin when compared to the untreated group.

**Karin Hermana Neppelenbroek et al (2006)**<sup>39</sup> assessed the shear bond strength of four hard chair side reline resins to a rapid polymerizing denture base resin (QC-20) processed using two polymerization cycles (A or B) before and after thermocycling. Cylinders (3.5mm x 5.0 mm) of the reline resins were bonded to cylinders of QC-20 polymerized using cycle A (boiling water 20 minutes) or B (boiling water, remove heat 20 minute; boiling water 20 minutes). For each reline resin/polymerization cycle combination, ten specimens were thermally cycled and the other ten were tested without thermal cycling. The result showed QC-20 displayed the lowest bond strength values in all groups. In general, the bond strengths of the hard

chair side resins were comparable and not affected by polymerization cycle of QC-20 resin and thermal cycling.

**Andrea Azevedo et al (2007)<sup>18</sup>** did a study to evaluate the effect of water immersion on the shear bond strength between chair side relines and denture base acrylic resins. The effect of water immersion on the shear bond strength between one heat polymerizing acrylic resin (Lucitone 550-L) and four autopolymerizing relines resins (Kooliner-K, New Truliner-N, Tokuso rebase fast-T, Ufi gel Hard-U) was investigated. Shear tests were performed on the specimens after polymerization and after immersion in water at 37°C for 7, 90 and 180 days. All fractured surfaces were examined by scanning electron microscope (SEM) to calculate the percentage of cohesive fracture (PCF). They concluded their study saying that the long term water immersion did not adversely affect the bond of materials Kooliner, New Truliner, Tokuso rebase and Ufi gel hard and decreased the values of resin Lucitone. Materials Lucitone 550-L and Ufi gel hard failed cohesively and Kooliner, New Truliner and Tokuso rebase failed adhesively.

**Ayese Mese et al (2008)<sup>34</sup>** did a study to evaluate the effect of storage duration on the tensile bond strength and hardness of acrylic-resin and silicone based resilient liners that were either heat or auto polymerized onto denture base acrylic resin. The denture liners investigated were a definitive heat polymerized acrylic resin based (Vertex Soft), interim auto polymerized acrylic resin based (Coe-Soft), definitive heat polymerized silicone based

(Molloplast-B), and definitive auto polymerized silicone based (Mollusil Plus) resilient liner. The resilient liners were processed according to manufacturer's instructions. The definitive heat polymerized silicone based Molloplast-B resilient liner had significantly higher bond strength and lower hardness values than the others. Prolonged exposure to water produced significantly higher hardness values and lower bond strength values, which suggested that the use of this resilient liner may not provide long term clinical success.

**Daniela Maffei Botega et al (2008)<sup>12</sup>** evaluated the effects of thermocycling on the tensile bond strength of three permanent soft denture liners (PermaSoft, Dentuflex and Ufi-gel). Ten specimens were prepared for control and test groups of each material for a total of 60 specimens. All controls were stored in water (37°C) for 24 hours before testing. All test groups received 3000 thermal cycles consisting of 1 minute at 5°C and 1 minute at 65°C. All specimens were submitted to a tensile test using a universal testing machine at a crosshead speed of 5mm/min. Despite presenting greater bond strength, thermocycling had a deleterious effect in Dentuflex; Ufi-gel may be adequate for short term use.

**Hiroyuki Minami et al (2008)<sup>35</sup>** evaluated the invitro effect of thermal and mechanical fatigues on the bonding of an autopolymerising Soft Denture Liner to denture Base Materials using different Primers. Resin denture base specimens were pretreated with Sofreliner Primer or Reline Primer for resin . Metal specimens treated with Reline Primer for metal followed by application



of Sofreliner Primer .Repetitive mechanical stressing was performed by using a university of Alabama-type wear –testing apparatus as a stress generator. Vertical 75N load with 15 degree rotation as applied, then residual tensile resistance to failure was measured. The study conclude that the application of Sofreliner Primer for a resin denture base provided better bonding after thermocycle and cyclic load testing than did Reline Primer.

**Fauziah Ahmad et al (2009)<sup>1</sup>** did a study to evaluate the shear bond strength of light polymerized urethane dimethacrylate (Eclipse) and heat polymerized polymethylmethacrylate (Meliodent) denture base polymers to intra oral and laboratory processed reline materials. Thirty disks measuring 15mm diameter and 2mm thick were prepared for each denture base material following the manufacturer’s recommendations. They were relined with Meliodent RR, Kooliner, and Secure reline materials after one month of water immersion. Ten additional Eclipse specimens were relined using the same Eclipse resin. Meliodent denture base showed adhesive, cohesive and mixed failure, while all Eclipse showed adhesive failure with various reline materials. The two chemically different denture base polymers showed different shear bond strength values to corresponding reline materials.

**Saloni Gupta (2010)<sup>44</sup>** evaluated the effect on the tensile bond strength of silicone based liner and flexural strength of denture base resin when the latter is treated with different chemical etchants prior to application of the resilient liner. The specimens were divided into 4 subgroups of which one

acted as a control and the rest were subjected to surface treatment with acetone for 30s, MMA monomer for 180s, methylene chloride for 15s, respectively. Of the 4 subgroups 180s of MMA monomer treatment was found to be the most effective in improving the bonding between the liner and denture base resin .

**Neeraja Mahajan et al (2010)<sup>33</sup>** did an in vitro study on the comparison of bond strength of Auto polymerizing and Heat cure Soft denture liners with denture base resin. The tensile bond strength of two commercially available silicone based heat cured (Molloplast-B) and auto polymerizing (Mollosil) soft denture liners to denture base material (Trevalon) was compared. Lloyds Universal testing machine was used to test 60 samples. Results showed Molloplast-B having greater bond strength than Mollosil soft denture liner. It was even greater when packed against trevalon in an n-polymerized form than an already polymerized trevalon using primo adhesive. Both the soft lining materials used are acceptable for clinical usage.

**Hankan Akin et al (2011)<sup>2</sup>** evaluated the effect of sandblasting with different size of Aluminium oxide particles on tensile bond strength of resilient liner to denture base. PMMA test specimens were fabricated and assigned into 5 groups ,based on the treatment applied ,untreated ,sandblasted with 50 micrometer particles,60 micrometer particles,120 micrometer particles ,and 250 micrometer particles. The resilient liner specimens were processed between 2 PMMA blocks. the bonding strength was measured using a universal testing machine at a crosshead speed of 5mm/min.The study

concluded different particle size affect the bond strength and the 120 micrometer Aluminium particles improve the strength better.

**Rahul Shyamrao Kulkarni et al (2011)<sup>29</sup>** did this study to evaluate the effect of two surface treatments, sandblasting and monomer treatments, on tensile bond strength between two long term resilient liners and poly methyl methacrylate denture base resin. Two resilient liners Super-Soft and Molloplast-B were selected. Each group was surface treated by sandblasting, monomer treatment (for 180 sec) and control (no surface treatment). The result showed monomer pretreatment of acrylic resin produced significantly higher bond strength for both the liners when compared to monomer pretreatment and control. They concluded that surface pretreatment of the acrylic resin with monomer prior to resilient liner application is an effective method to increase bond strength between the base and soft liner. Sandblasting on the contrary, is not recommended as it weakens the bond between the two.

**Mohammad Q. Al Rifaiy et al (2011)<sup>4</sup>** to assess the bonding characteristics of Triad VLP direct hard reline resin to heat polymerized denture base resin subjected to long term water immersion. Ninety circular disks, 15mm in diameter and 3mm thick of denture base resin were polymerized from a gypsum mold. Thirty water immersed specimens were dried with gauze (group 1), 30 water immersed specimens were dried with a hair dryer (group 2) the remaining dry specimens represented the control group (group 3). All specimens were air abraded and painted with bonding

agent before packing Triad VLP direct hard reline resin. Specimens in each group were subjected to thermal cycling for 50,000 cycles between 4°C and 60°C water baths with one minute dwell time at each temperature. The results showed significant difference in mean shear bond strength among the specimens existed because of variable water content in the denture base resin. The mean shear bond strength for Group 3 (dry) was higher than group 2 (desiccated) and the lowest was group 1 (saturated).

**Salah A. Mohammed et al (2011)<sup>36</sup>** did their in vitro study to compare four silicone based soft liner materials (Permaflex and Molloplast, Ufi-gel SC and permafix) in shear bond strength, water sorption and solubility and surface roughness test. Seventy two specimens of four silicon based soft lining material was used, the specimens of shear bond strength test were subjected to tension in Instron machine with speed rate was 0.5mm/min to measure shear bond strength by N/mm. The result indicated that Permaflex shows better properties when compared with other soft liner materials and that hot cure polymerizing soft liner material showed proper properties when compared with auto polymerizing soft liner material.

**Jessica Mie Ferriera Koyama Takahashi et al (2011)<sup>46</sup>** did their study to evaluate the effect of different accelerated aging times on permanent deformation and tensile bond strength of two soft chair side liners, acrylic resin (T) and silicone (MS) based. Different specimens were made for each test of each reliner. The specimens were submitted to accelerated aging for 2,

4, 8, 16, 32, and 64 cycles. Mann-Whitney test was done to compare the materials at different times and Kruskal-Wallis and Dunn tests were used for comparing aging intervals within a given reliner. The result showed MS with lower permanent deformation and higher tensile bond strength than T. Although T presented changes in those properties after accelerated aging, both materials might be suited for long term use.

**Nishitha Madan et al (2012)**<sup>32</sup> made a study to assess the effect of simulated mouth conditions reproduced with thermocycling on the tensile bond strength of two silicone based resilient denture liners with acrylic resin bases. Specimens were divided into a control group that was stored for 24 hours in water at 37°C and a test group that was thermocycled (2500 cycles) between baths of 5°C and 55°C. Heat polymerized resilient denture liner Molloplast-B had higher tensile bond strength than auto polymerizing liner Mollosil regardless of thermocycling. The bond strength of Mollosil increased after thermocycling while that of Molloplast-B decreased after thermocycling.

**Fatih Mehme (2013)**<sup>27</sup> investigated the effect of laser parameters and air abrasion on the peel strength of silicon based soft denture liner to different denture resins. Silicone based soft denture liner was applied to the specimens (N=180) which were prepared out of three different denture base resins: Rodex, crosslinked denture base acrylic resin; Paladent heat cure acrylic resin; Deflex, Polyamide resin (75mm\*25mm\*3mm); after the following methods: air-

abrasion (50Hz, d) ErCr:YSGG laser (waterlase MD turbo, biolase technology) at 2 W-20 Hz, Er:Cr; YSGG laser at 2 W-30Hz; Er,Cr:YSGG laser at 3W-20 Hz; Er,Cr:YSGG laser at 3 W-30 Hz; non conditional group-control. Peel test was done in universal testing machine and failure modes were evaluated visually. Data were analysed using 2 way ANOVA and Turkey test. Denture liner tested showed increased peel strength after laser treatment compared to the control and air abraded groups, but the results were not statistically significant except for Paladent with the pretreatment of Er,Cr:YSGG laser at 3 W-20 Hz. Polyamide resin after air-abrasion showed significant peel strength than those of other groups thus concluding that Heat cured acrylic resin PMMA may benefit from Er,Cr:YSGG laser treatment at 3 W-20 Hz. Air-abrasion should be avoided.

**Farideh Geramipناه et al (2013)<sup>20</sup>** evaluated the effect of thermocycling on Tensile Bond strength of two soft liners (Acropars, Molloplast –B) to denture base resin .ten specimens were maintained in 37degree C water for 24 hours and 10 were thermocycled among bath of 5 degree and 55 degree C .The tensile bond strength was measured using a universal testing machine at a crosshead speed of 5mm/min.There was no significant difference in the mean and standard deviation before and after thermocycling. Mode of failure in Acropars and Molloplast –B were adhesive and cohesive. The bond strength of Acropars was significantly higher than Molloplast –B (P<0.05).

**Hemchand Surapaneni et al (2013)<sup>22</sup>** studied the comparative evaluation of tensile bond strength of silicone-based soft lining materials (Ufi Gel P and GC Reline soft) with different surface treatments of heat cure PMMA denture base acrylic resin. Two types of addition silicone –based soft lining materials : Ufi Gel P and GC Reline soft) were selected ,these samples were further divided into 4 subgroups based on the pre-treatment .sub group 1 –without any surface treatment ,Sub group 2 –sand blasted, subgroup 3 – treated with methyl methacrylate monomer ,sub group 4 with chemical etchant Acetone. The specimens of the GC Reline soft treated with MMA monomer showed superior bond strength than other surface treatment.

**Mayank Lau et al (2015)<sup>30</sup>** evaluated the tensile and shear bond strength of hard and soft denture relining materials to the conventional heat cured acrylic denture base resin. 4mm sections in the middle of 160 acrylic cylindrical specimens(20mmx8mm) were removed ,packed with Mollosil ,GC Reline Soft, GC Reline Hard and Ufi Gel Hard and polymerized . The samples were tested using an Instron Universal Testing Machine by the equation  $F/A$  (F-maximum force exerted and A-bonding area =50.24 square mm).the Tensile and Shear Bond strength values of denture soft reliners were significantly lower than denture hard reliners.

**Anshul Khanna (2015)<sup>6</sup>** did a comparative evaluation of shear bond strength between 2 commercially available heat cure resilient liners and denture base rein with different surface treatments. Soft denture liner Luci-soft

and super-soft and PMMA were used. 80 samples were made, 40 each for each of the 2 materials under investigation which were further divided into 4 groups containing 10 samples each. Group 1: untreated surface of PMMA-control; Group 2: PMMA surface sandblasted; Group 3: PMMA surface treated with monomer; Group 4: liner materials processed with acrylic dough. Samples after thermocycling for 500 cycles at  $5^{\circ}\pm 1^{\circ}$  -  $55^{\circ}\pm 1^{\circ}$  at 60sec dwell time were subjected to shear loading on universal testing machine at cross head speed of 20mm/sec. Scanning electron microscope and stereomicroscope analysis of the bond interface between the liner and denture was conducted for all groups. Data was analyzed using independent samples t-test analysis of variance and post-hoc analysis. significant levels of  $\alpha=.05$  was used. The bond strength was significantly different between super soft and luci soft ( $p<0.05$ ) for all surface treatment. Electron microscope observations showed that application of surface treatments modified the surface of denture base, thus concluding that super-soft has significantly higher bond strength than luci -soft.



## **MATERIALS AND METHODS**

The present in-vitro study was conducted to comparatively evaluate the effect of two different surface treatments on the shear bond strength between silicone soft liner and heat polymerized denture base resin after thermocycling.

**The following materials, instruments and equipments were used for the study:**

### **MATERIALS EMPLOYED:**

- Addition polymerizing polyvinylsiloxane putty impression material, (Densply, USA) (Fig.1a)
- Addition polymerizing polyvinylsiloxane light body, regular set (Densply, USA) (Fig.1b)
- Dispensing gun (Densply, USA) (Fig.1c)
- Auto mixing spiral tips (Densply, USA) (Fig.1d)
- Tray adhesive (Densply, USA) (Fig.2)
- Acetate sheet (Fig.3)
- Modelling Wax (The Hindustan Dental Products, Hyderabad) (Fig.4)
- Type-II Dental Plaster (Ramaraju Mills Ltd., India) (Fig.5)
- Separating medium (DPI-Mumbai) (Fig.6)
- Heat cure acrylic resin (DPI-heat cure polymer and monomer) (Fig.7)

- Sand paper 100 and 120 grit sizes (continental abrasives, Chennai)  
(Fig.8)
- Silicone-based soft resilient liner (GC reline soft) (Fig.9)
- Petroleum Jelly (Tejpal Industries Ltd) (Fig.10)
- Distilled Water (Diet. Pondicherry) (Fig.11)

### **INSTRUMENTS EMPLOYED:**

- Clamp and denture flask (Jabbar, India) (Fig.12a,b)
- Rubber bowl & Spatula (classic, India) (Fig.12c,d)
- Wax Knife, Wax carver (Fig.12e,f)
- Acrylic Trimmers (Shofu, Japan) (Fig.13a)
- Sand paper mandrel (Fig.13b)

### **EQUIPMENTS USED:**

- Acrylizer (confident Dental Equipments Limited, Bangalore, India)  
(Fig.14)
- Dental Lathe (Suguna Industries Ltd) (Fig.15)
- Sand blaster (Basic Professional, Renfert GmbH, Germany) (Fig.16)
- Er,Cr:YSGG laser unit (Waterlase iPlus laser unit, Biolase  
Technology, CA, USA) (Fig.17)
- 3-D Surface Profilometer (Talysur fCCI, Ametek, UK) (Fig.18)
- Automated Thermocycling Unit (Haake Willytec, Germany) (Fig.19)
- Universal Testing Machine (Instron, Lloyd Instruments, UK) (Fig.20)
- Scanning Electron Microscope- Sputtering Machine (Fig.21)

- Scanning Electron Microscope (SA400N, Canada) (Fig.22)

### **Description of Thermocycler:**

In this study, thermocycler (Haake, W15, Germany) was used for thermocycling the test samples to simulate the temperature changes in the oral cavity. It consists of two water baths, each maintained at different temperatures. Bath one has temperature variation from 25°C to 100°C and bath two has temperature variation from -5°C to 100°C. The required cycles can be easily adjusted via display from 0-9999 cycles. It has automatic refills for the baths to compensate evaporation during the long duration test. It has an auto start capability. Bath two is connected to a cooling device. The two baths are connected by a rolling unit with an open sample container in the centre for holding the test samples. The open sample container with the test samples is immersed cyclically in baths of warm and cold water. Simulation of exposure of samples to various temperature fluctuations can reveal bond durability of the samples.

### **Description of the Universal Testing Machine:**

Universal mechanical testing machine (Instron, Lloyds Universal Testing Machine, U.K.) was employed in the present study for obtaining shear bond value for the test samples. This machine rests on a table top. It consists of a lower chamber, upper chamber a display board to display the amount of force needed and is connected to a computer. The upper member is attached to the lower with help of two horizontal bars, which also houses the hydraulic

pressure machine attached to upper member. The lower portion has a bench vice test specimen fixture to hold the test specimens. The upper portion has a clevis grip on which a mono-beveled chisel blade can be attached. The whole unit is attached to a computer for recording and converting the data.

### **Description of the Scanning Electron Microscope:**

In this present study, the surface of the test samples was analyzed using Scanning Electron Microscope (SA400N, Canada). Scanning Electron Microscope uses a beam of highly energetic electrons to examine objects on a very fine scale. The specimens to be magnified are coated with a platinum layer to prevent the charging up and in order to increase the secondary emissions. Additional sputter coating with gold produces high contrast and resolution. The incident electron probe scans the sample surface and the signals produced are used to modulate the intensity of a synchronously scanned beam on a CRT screen. The electrons which are back scattered from the specimen are collected to provide (i) topographical information if low energy secondary electrons are collected (ii) atomic number and reorientation information if the higher energy, back scattered electrons are used, or if the leakage current to the earth is used. The magnification is given immediately by ratio of the CRT scan size to the specimen scan size.

### **Description of the laser unit:**

In this present study Er,Cr:YSGG laser unit (Waterlase iplus laser unit, Biolase Technology, CA, USA) was used to etch the test surface of the acrylic denture base. The laser system is categorized under classification IV (Medical

laser) of ANSI laser safety standard. Er,Cr:YSGG laser is basically a hard and soft tissue laser with a wavelength of 2780nm. The maximum pulse energy of the laser is 600mj. Pulse repetition rates of the laser energy can be adjusted to 5-100 Hz and the pulse duration can be regulated to 60 and 700 $\mu$ sec. The laser unit has maximum power output of 10watts. The graphical display present in front portion of the laser unit can be programmed to the required setting. The atomized spray of water and air from the BIOLASE proprietary hand piece continually re-hydrates the acrylic denture base, preventing thermal injury. The waterlase Iplus works in non-contacting mode at the distance of 10mm away from acrylic denture base surface. It has précised radial firing tip which is uniquely designed that tapers to the end of the trip, allowing for a more effective irradiation. Laser source comes out from tip as a beam and breaks off the water molecules present on the substrate, thereby etching the surface.

### **Description of 3-D surface profilometer:**

In this present study the surface texture of one representative acrylic blocks was analysed using a 3-D surface profilometer (talysurfCCI, ametek,UK). In the present study a non-contact optical 3-D surface profilometer was used to measure the 3-D surface texture. It is an advanced type of measurement interferometer. The 3-D Non-contact profilometer is designed with leading edge optical lens using superior green light axial chromatism. 3-D Non-contact profilometer optical lens have zero influence from sample reflectivity, variation require no sample preparation and have

advance ability to measure high surface angles. It can easily measure any material which is transparent, opaque, specular, diffusive, polished, rough etc. unlike other optical measurement techniques, large surface area can be precisely measured without any image stitching. From this, 3-D and advanced 3-D images can be viewed as well as average mean surface roughness (Ra) value can be calculated.

## **METHODOLOGY**

- I. Fabrication of custom made stainless steel mold**
- II. Preparation of duplicating index**
- III. Fabrication of heat polymerized acrylic denture base resin blocks**
  - a. Preparation of wax blocks
  - b. Flasking procedure
  - c. Dewaxing procedure
  - d. Packing of acrylic resin
  - e. Curing procedure
  - f. Deflasking procedure
  - g. Finishing
  - h. Storage of acrylic blocks
- IV. Grouping of acrylic resin blocks**
- V. Preparation of test surfaces (Bonding surfaces) of acrylic resin blocks**
  - a. Preparation of Group A (Control Group) acrylic resin blocks
  - b. Preparation of Group B (sandblasted Group) acrylic resin blocks

c. Preparation of Group C (Laser irradiation Group) acrylic resin blocks

**VI. 3-D Non-contact surface profilometer analysis of Group A, Group B, Group C**

**VII. Bonding of resilient liner material**

a. Fabrication of custom made Teflon jig

b. Application of primer to the test surfaces

c. Assembling of acrylic resin blocks and Teflon jig

d. Bonding of silicone based soft liner to heat polymerized acrylic resin blocks

**VIII. Thermocycling of Test samples**

**IX. Shear bond strength testing of the Test samples**

**X. Qualitative analysis of bond strength and mode of failure by Scanning Electron Microscopy (SEM)**

**XI. Data tabulation and Statistical analysis**

### **I. Fabrication of custom made stainless steel block: (Fig.23, 24)**

A stainless steel block (MRS industries, Chennai) was custom milled in a milling machine with the standardized dimension of 14mmx14mmx25mm by subtractive method. This block was to serve as a template for duplication of wax blocks of similar dimensions and then to be converted to heat cure acrylic resin blocks of uniform dimension to be used in the present study.

### **II. Preparation of duplicating index: (Fig.25-34)**

The duplicate index in the present study was obtained using addition polymerizing putty and light body impression material using the two-stage technique. Tray adhesive was applied on a rigid plastic container that served as a counter. Addition polymerizing polyvinylsiloxane putty impression material was hand mixed by taking equal quantities of base and catalyst. It was kneaded to obtain homogeneously mixed dough. The rigid, plastic container was filled with the mixed dough and an acetate spacer sheet was placed over the dough and the custom made stainless steel block was impressed into it while the putty was still in a soft pliable state. It was left undisturbed until the setting of the putty material. After the material had set, the block and spacer sheet were removed. Addition polymerizing siloxane light body material in a cartridge was loaded in an automixing gun and a spiral mixing tip was syringed over the putty index to fill it and the stainless steel block was resealed into the putty index. Excess material was wiped away. It was held with firm finger pressure until set. After setting, the block was removed and



the index was inspected for acceptability. This index of 14mmx14mmx25mm dimensions thus obtained was used to obtain wax blocks of standardized dimensions for the study.

### **III. Fabrication of heat polymerized acrylic denture base resin blocks**

**(Fig. 35-42)**

#### **a. Preparation of wax blocks: (Fig.35, 36)**

Modeling wax (Hindustan manufacturer, Hyderabad) was melted and poured into the mold space and allowed to cool. After the wax had completely hardened, the wax blocks were retrieved carefully and placed in a container of distilled water at room temperature. Thirty three such wax blocks of 14mmx14mmx25mm dimensions were fabricated.

#### **b. Flasking procedure: (Fig.37)**

The fabricated wax blocks were invested in a denture flask using Type II dental plaster. A two pour technique was followed for flasking the wax specimens. Type II dental plaster was mixed with water using a stainless steel straight spatula in rubber bowl and poured into the lubricated base portion of the denture flask. The wax blocks were placed into the in the denture flask. The number of samples per denture flask was restricted to a maximum of five to ensure adequate space between the samples. After the plaster had set, separating medium was painted over the plaster surfaces, and the lubricated body of the flask was placed over the base. It was filled with a fresh mix of

Type II dental plaster and the lid was closed. The denture flask was tightened with a flask carrier and the excess plaster removed.

**c. Dewaxing procedure: (Fig.38)**

The plaster was allowed to harden for 1 hour before the denture flask was placed in a boiling water bath. The clamps were loosened and flasks were placed in boiling water for 15 minutes. The flasks were removed from the water and the appropriate segments of the flask were carefully separated in a vertical direction to avoid fracture of the invested plaster. The softened wax was flushed out from the surface of the mold with hot water. Wax solvent and warm detergent solution were used to remove wax residues and oily films respectively. Finally the molds were flushed well with clean hot water. Both the halves of the flasks were placed slanting on the laboratory bench for several minutes to allow the water to drain completely. The flasks were allowed to cool completely prior to packing. After dewaxing, the rectangular mold spaces in the base of the denture flask were ready for packing of heat cure denture base acrylic resin.

**d. Packing of acrylic resin: (Fig.39)**

A thin coating of separating medium was painted on the plaster surfaces. Heat cure acrylic resin was mixed in the porcelain cup with a powder/liquid ratio as per the manufacturer's instructions. The porcelain cup was closed with a lid until the mix reached the dough stage. Required quantity

of acrylic resin was packed individually into each rectangular mold space. A thin acetate separator sheet was placed over the lower halves and then the upper halves were seated over them. Pressure was applied to allow excess resin to displace out of the denture flask.

Once the flask was fully closed, it was opened and the polyethylene sheet was removed and excess was removed by using a sharp wax carver/wax knife. Trail closure with fresh polyethylene sheet is repeated till there is no longer apparent flash. The two halves of the flask were closed and the flask was placed under the bench press and tightened. The excess resin extruding from the flask was removed.

**e. Curing procedure:**

The packed denture flasks were bench cured for 60 minutes as per the manufacturer's instructions and the flasks were removed from the bench press. The flasks were tightened under their respective flask carriers and placed in the acrylizer for resin polymerization. A curing cycle of 74°C for approximately 2 hours was carried out followed by increasing the temperature of the water bath to 100°C and processing for 1 hour as per standard recommendations. This was followed for all the packed acrylic blocks.

**f. Deflasking procedure: (Fig.40)**

After completion of the polymerization cycle, the flasks were removed from the water bath and bench cooled for 30 minutes and then kept under

running tap water for 15 minutes. Following this, the deflasking of the specimens was done. Excess plaster was removed from the blocks with a sharp knife.

**g. Finishing: (Fig.41,42)**

Acrylic burs were used to trim excess resin flash. Sandpapers of grit sizes of 100 and 120 respectively were used to smoothen the surface, mounted on a sandpaper mandrel attached to a dental lathe (Suguna Industries Ltd). A total of thirty three heat polymerized acrylic blocks were obtained in a similar manner. The two sides of 14mm×14mm dimensions, one of the sides were randomly selected as the test surface. After smoothening, the blocks were not subjected to further polishing procedures.

**h. Storage of acrylic blocks :**

The prepared acrylic resin blocks were stored under distilled water in an air tight container for  $50\pm 2$  hours for the denture base polymer to reach water saturation.

**VI. Grouping of acrylic resin blocks: (Fig.43)**

All the thirty three acrylic resin blocks thus obtained were divided into three groups of ten blocks each according to the type of surface treatment rendered to the test surface of the acrylic resin blocks.

The acrylic resin blocks were divided randomly into three groups as follows:

1. Group A (n=11) untreated surface of acrylic resin blocks (Control Group).
2. Group B (n=11) for sandblasting surface treatment of acrylic resin blocks (Sandblasted Group).
3. Group C (n=11) for laser irradiation surface treatment of acrylic resin blocks (Laser irradiation Group).

One surface treated acrylic resin block from each group was used for 3-D profilometer study. The remaining surface treated ten acrylic resin blocks of each group was used for bonding with the silicone liner and for shear bond strength testing.

#### **V. Preparation of test surfaces (Bonding surfaces) of acrylic resin blocks:**

##### **a. Preparation of Group A (Control Group/Untreated group) acrylic resin blocks: (Fig.44)**

The test surfaces of the acrylic resin blocks of this group (n=11) were designated as control and hence no surface treatment was performed on these test surfaces. These untreated samples were stored as obtained after finishing under distilled water in an air tight container to avoid contamination till future use.

**b. Preparation of Group B (Sandblasted Group) acrylic resin blocks:**

**(Fig.45)**

The test surface of the ten acrylic resin blocks (n=11) in this group were subjected to sand blasting using 110µm aluminium oxide (Korox, Bego, Germany). The test surfaces of the acrylic resin blocks were air abraded by holding them at a distance of 10mm from the nozzle, maintaining the pressure at 2psi for a period of 30 seconds following which they were cleaned using a steam cleaner. The surface treated samples were stored in an air tight container to prevent contamination prior to application of silicone liner.

**c. Preparation of Group C (Laser irradiation Group) acrylic resin blocks:**

**(Fig.46)**

The test surface of the ten acrylic resin blocks (n=11) in this group were subjected to laser surface treatment using Er,Cr:YSGG laser system (waterlase iplus laser unit, Biolase Technology, CA, USA ). The test surfaces of the acrylic resin blocks were subjected to laser irradiation which was done at the wavelength of 2.78µm, pulse duration of 700µs and repetition rate of 10Hz. The power output was set at 3W according to test protocols. The air and water sprays from the handpiece were adjusted to a level of 85% air and 85% water to prevent the acrylic surface from overheating. Laser energy was delivered through a fibre optic system to a sapphire tip terminal 600µm in diameter and 6mm long. The focused laser beam was aligned to the test surface perpendicularly at a distance of 10mm. The area to be bonded was

lased manually in a circular motion for a period of 30 seconds. The surface treated samples were stored in an air tight container to prevent contamination prior to application of silicone liner.

#### **VI. 3-D Non-contact surface profilometer analysis: (Fig.47)**

One representative surface treated acrylic resin block from Groups A, B and C were subjected to 3-D surface scanning to evaluate the surface topography and surface roughness. Surface roughness was measured using 3-D Non-contact surface profilometer (TalysurfCCI, Ametek, UK). The surface roughness (Ra) value of each acrylic block was obtained. The magnification of the optical lens was 50x. Each acrylic block was placed under the objective lens and photomicrographs at 50x magnification were obtained in advanced 3-D views using Advanced Aspherics Analysis software. One photo micrograph was obtained per block to qualitatively assess the surface topography of the test surfaces.

#### **VII. Bonding of resilient liner material: (Fig.48-53)**

##### **a. Fabrication of custom made Teflon jig: (Fig.48a,b,c)**

A cylindrical Teflon jig, 20mm in diameter and 6mm in height was fabricated. The jig had a fitting surface and superior surface. Teflon jig had a central circular opening, 6mm in diameter and 3mm in height, so as to limit the soft liner to a circular area of 6mm diameter and a height of 3mm for all the samples. This was kept ready prior to the bonding procedure of the liner.

**b. Application of primer to the test surfaces: (Fig.49)**

The test surfaces of all the acrylic resin blocks of each test group were coated once with primer (GC liner, Germany). Each coating was applied to the test surface using an applicator with an application time of 30 seconds as per the manufacturer's instructions. Care was taken such that there was no contamination of test surface after application of the primer.

**c. Assembling of acrylic resin blocks and Teflon jig: (Fig.50, 51a, b)**

One block at a time was assembled with the custom made Teflon jig described before. The fitting surface of the custom made Teflon jig was placed on the primer-coated, surface treated end of the acrylic resin block. The design of the jig was such that the resin block fitted snugly into the indentation present in the fitting surface of the cylindrical jig. Thus the assembly serves the dual purpose of delineating the shape and size of the bonding area and preventing the soft liner from contacting the acrylic resin surface outside the circular bonding area.

**d. Bonding of silicone-based soft liner to heat polymerized acrylic resin blocks : (Fig.52,53a, b & c)**

The silicone based soft liner(GC liner, Germany) which is supplied in cartridges was mixed using a hand held auto mixing device(GC liner, Germany) and was introduced gently from one end into the bonding area to



avoid air entrapment till the material completely filled the central hole. An acetate sheet was placed over the material and pressure was applied until polymerization was completed. The working time for silicone liner is 2 minutes and it is allowed to set for 5 minutes. After the soft liner has set, the acetate sheet and the Teflon jig were removed and the test samples of acrylic blocks with silicone based soft liner, of height 3mm bonded to a circular area of 6mm in the center of resin blocks were obtained. This process is carried out for all the remaining resin blocks to obtain 30 test samples with ten test samples per group.

#### **VIII. Thermocycling of test samples: (Fig.54)**

All the thirty samples of the three test Groups A, B and C were subjected to thermocycling for a total of 250 cycles in a distilled water bath between 5°C and 55°C with a dwell time of 60 seconds and a dry time of 10 seconds at 27 °C between the warm and cold cycles using a thermo cycling apparatus (Haake, W15, Germany) to simulate three months of clinical use. The test samples of each group (n=10) were tied in a cloth pouch and the three sets of pouch were collectively thermocycled in the apparatus. Upon completion of thermocycling, the specimens were stored under distilled water in their respective containers until they were subjected to shear bond strength testing.

### **IX. Shear bond strength testing of the test samples: (Fig.55)**

A total of thirty samples (Groups A, B, C) were tested individually for shear bond strength in an Universal Testing Machine (Instron, Llyod instruments, UK). Each test sample was fixed to the sample fixture at the bench vice of the machine with a knife edged chisel blade positioned parallel to the material interface. Force was applied to the sample in such a way that shear load was exerted directly to the bonding interface at a cross head speed of 1 mm/min until failure of the bond occurred. The tests were conducted in an open room at room temperature. Load deflection curves and ultimate load to failure were recorded automatically and displayed by the computer software of the testing machine. Shear bond force at which the bond failed was recorded in newton (N) and shear bond strength (MPa) was calculated by dividing the force (N) at which failure of the bond occurred by the surface area of adhesion ( $\text{mm}^2$ ). The tested samples were stored in distilled water.

$$\text{Bond Strength (MPa)} = \text{Force (N)} / \text{surface area (mm}^2\text{)}$$

### **X. Qualitative analyses of bond strength and mode of failure by Scanning Electron Microscopy (SEM): (Fig.56, 57)**

Surface analysis of the tested specimen was carried out individually on one representative sample per test group selected randomly using scanning electron microscope (SA400N, Canada). The sample was secured into Cu stubs with double adhesive tapes and coated with a layer of gold using gold

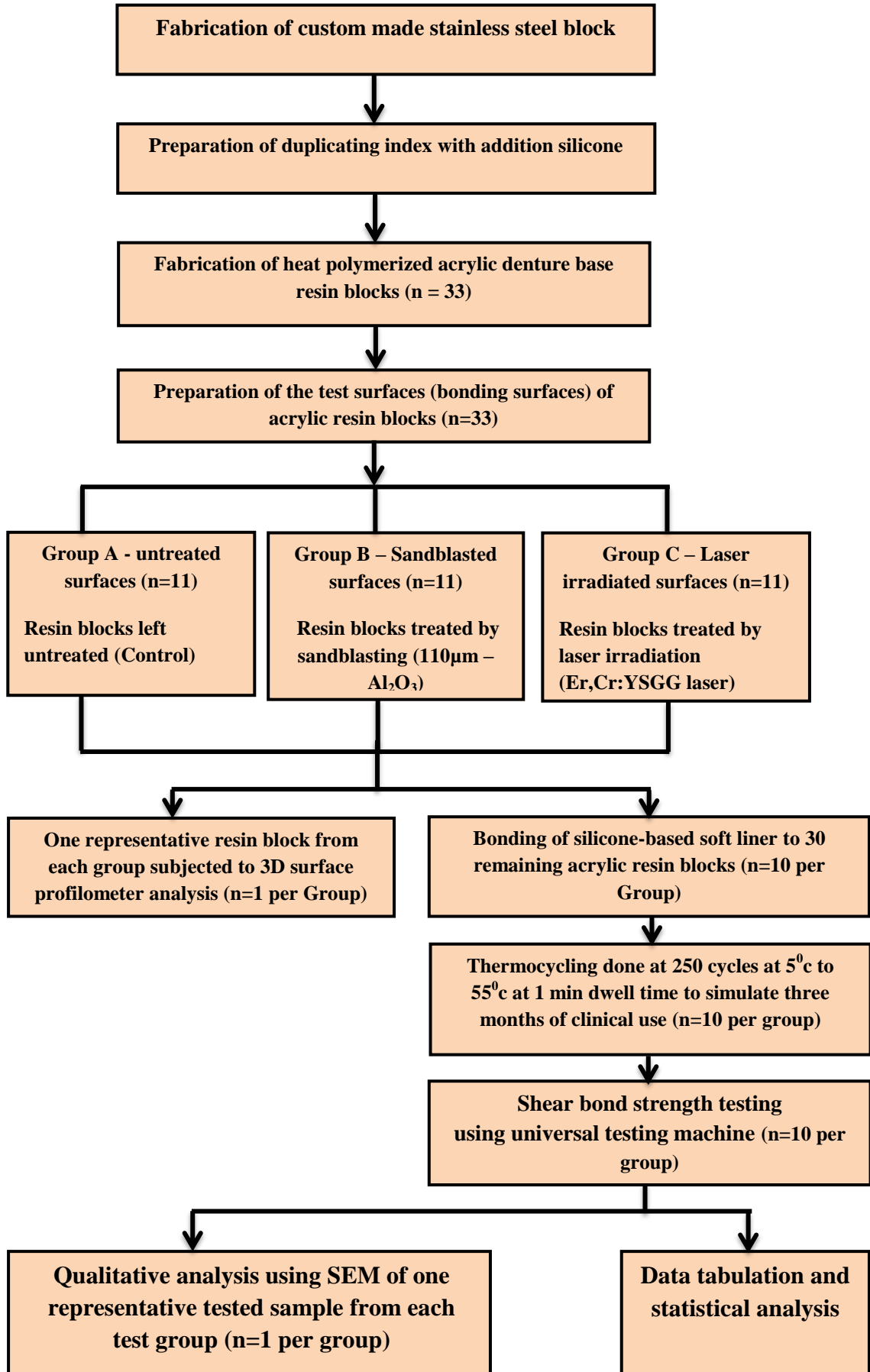
sputtering system. Coated samples were examined under SEM to qualitatively assess the surface topography of tested samples of each test group at 100x magnifications. The mode of the failure of tested samples was assessed under these magnifications

#### **XI. Data tabulation and Statistical analysis:**

The data obtained from shear bond strength testing were tabulated and subjected to statistical analysis. The SPSS (SPSS 16 for Windows 8.0, SPSS Software Corp., Munich, Germany) software package was used for statistical analysis. Mean and standard deviation were estimated from the results obtained from each sample for each study group. The data were analyzed with One Way Analysis Of Variance (ANOVA) for overall significance. Further pair-wise multiple comparisons of mean values of the test groups were done by Post-Hoc test (Tukey's HSD Analysis). Statistical significance was considered at 5% significance level.

# ANNEXURE I

## METHODOLOGY – OVERVIEW



## ANNEXURE II

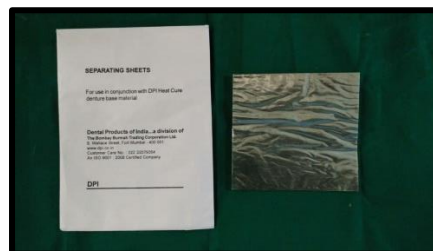
### MATERIALS



**Fig.1: a: Putty consistency -Polyvinylsiloxane impression material**  
**b: Light body consistency -Polyvinylsiloxane impression material**  
**c: Dispensing gun**  
**d: Auto mixing spiral**



**Fig.2: Tray adhesive**



**Fig.3: Acetate sheet**



**Fig.4: Modelling Wax**



**Fig.5: Type-II Dental Plaster**



**Fig.6: Separating medium**



**Fig.7: Heat cure acrylic resin**



**Fig.8: Sand paper 100 and 120 grit sizes**



**Fig.9: Silicone-based chair-side soft resilient liner**

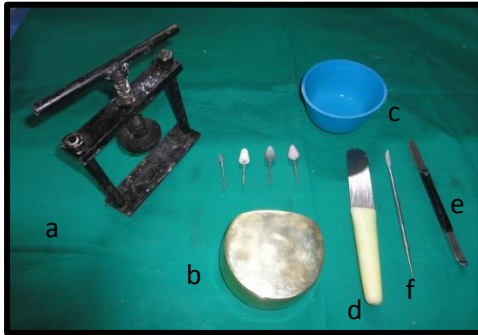


**Fig.10: Petroleum Jelly**



**Fig.11: Distilled Water**

## INSTRUMENTS



**Fig.12: : a. Clamp**  
**b. Denture flask**  
**c. Rubber bowl**  
**d. Spatula**  
**e . Wax Knife**  
**f. Wax carver**



**Fig.13: a. Acrylic Trimmers**  
**b. Sand paper mandrel**

## EQUIPMENTS



**Fig.14: Acrylizer**



**Fig.15: Dental Lathe**





**Fig.16: Sand blaster**



**Fig.17: Er,Cr:YSGG laser unit**



**Fig.18: 3-D Surface Profilometer**



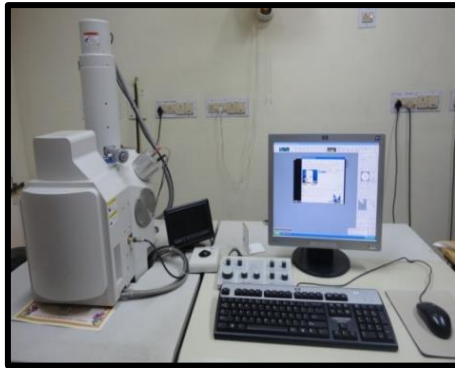
**Fig.19: Automated Thermocycling Unit**



**Fig.20: Universal testing Machine**



**Fig.21: Scanning Electron Microscope- Sputtering Machine**



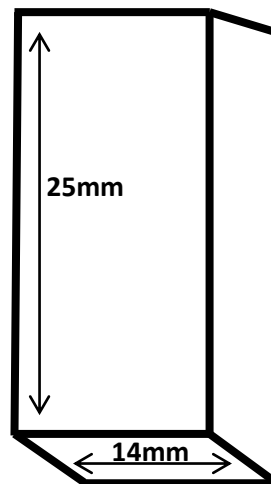
**Fig.22: Scanning Electron Microscope**

## **METHODOLOGY**

### **I. Fabrication of custom made stainless steel mold**



**Fig.23 Stainless steel block**

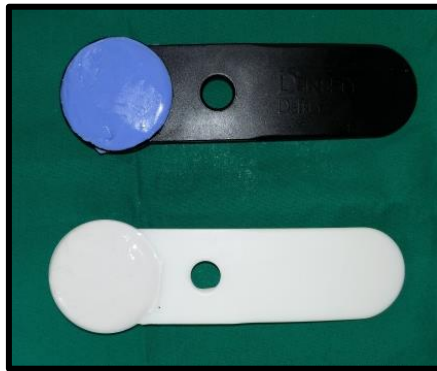


**Fig.24: Schematic diagram of stainless steel block**

## II. Preparation of duplicating index



**Fig.25: Application of tray adhesive**



**Fig.26: Proportioning of putty base and catalyst**



**Fig.27: Hand mixing of putty base and catalyst**



**Fig.28: Placing acetate sheet**



**Fig.29: Impressing stainless steel block in putty**



**Fig.30: Removal of block and acetate sheet**



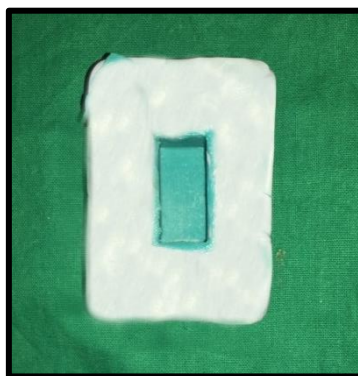
**Fig.31: Light body injecting into the mold**



**Fig.32: Reinserting stainless steel block into the injected mold space**



**Fig.33: Seated stainless steel block into the mold space**



**Fig.34: Fabricated putty index**

### III. Fabrication of heat polymerized acrylic denture base resin blocks



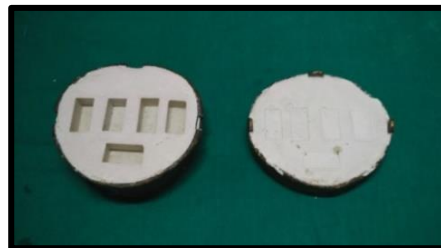
**Fig.35: Modelling Wax poured into mold space**



**Fig.36: Prepared wax block**



**Fig.37: Flasking procedure**



**Fig.38: Dewaxed mold**



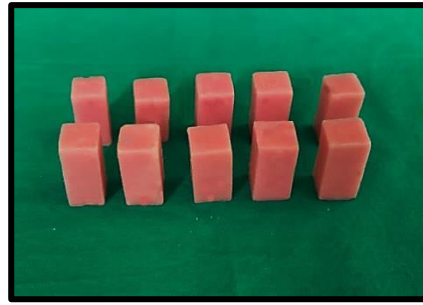
**Fig.39: Packing of acrylic resin**



**Fig.40: Deflasking**



**Fig.41: Finishing of the acrylic resin block**



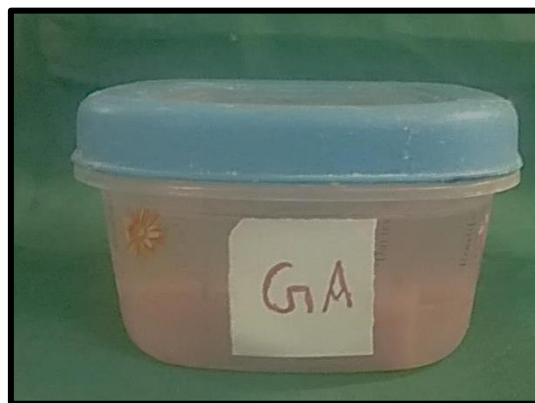
**Fig.42: Finished acrylic resin blocks**

#### **IV. Grouping of acrylic resin blocks**



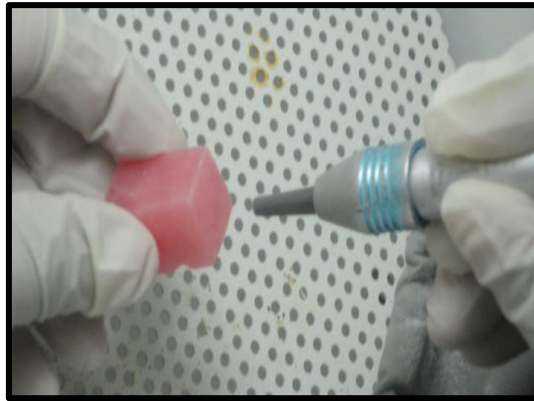
**Fig.43: Grouped test samples stored in different containers**

#### **V. Preparation of test surfaces (Bonding surfaces) of acrylic resin blocks**

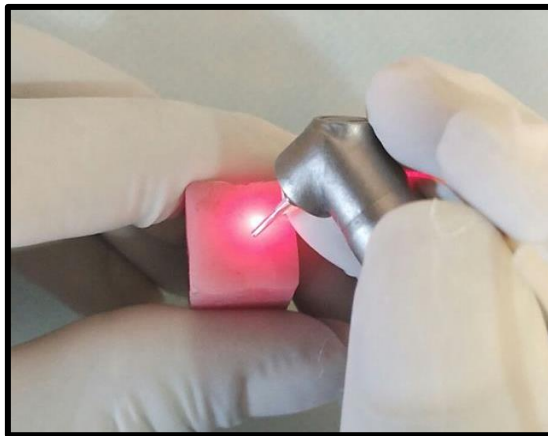


**Fig.44: Group A (Control/Untreated Group) acrylic resin blocks**



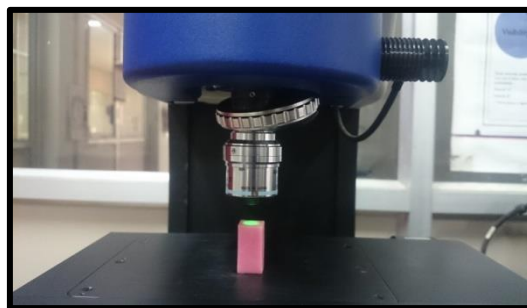


**Fig.45: Sandblasting of Group B (Sandblasted Group)  
acrylic resin block**



**Fig.46: Laser irradiation of Group C (Laser irradiation Group)  
acrylic resin block**

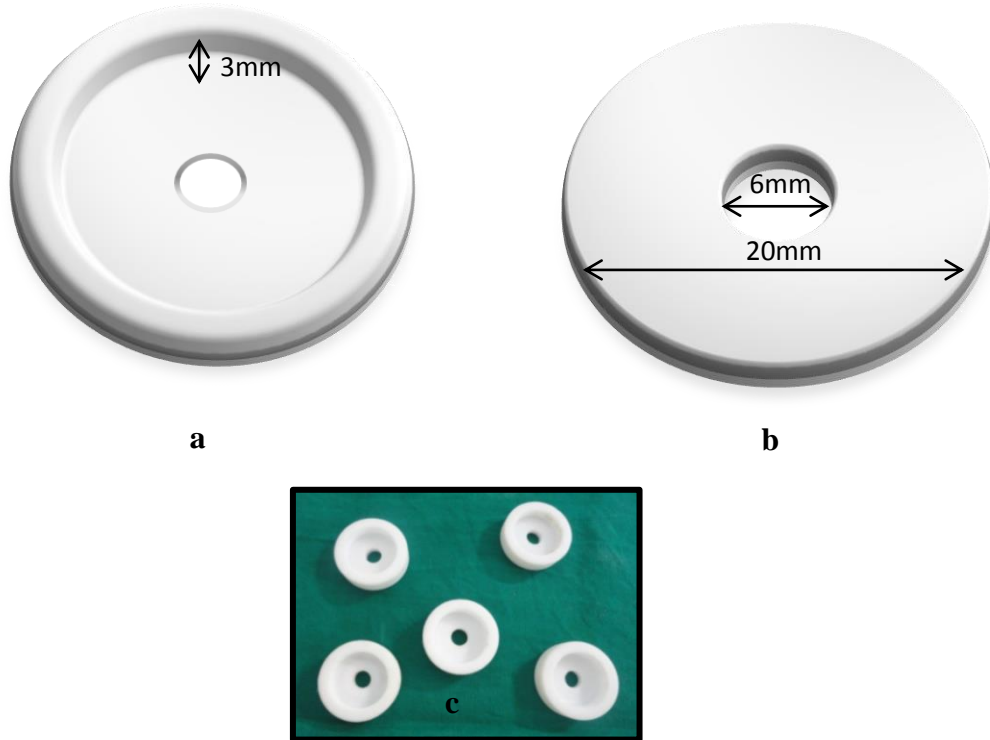
**VI. 3-D Non-contact surface profilometer analysis of Group A, Group B, Group C**



**Fig.47: Performing surface texture analysis using 3D Profilometer**

## VII. Bonding of resilient liner material

### a. Fabrication of custom made Teflon jig

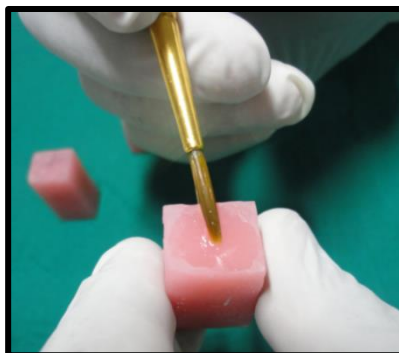


**Fig. 48a: Custom-made Teflon jig (Fitting surface)**

**b: Custom-made Teflon jig (Superior surface)**

**c: Fabricated Teflon jig**

### b. Application of primer to the test surfaces

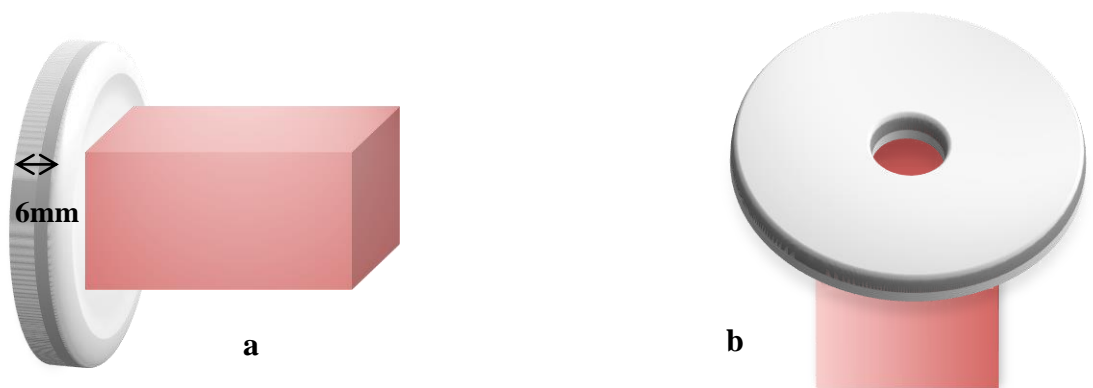


**Fig.49: Primer application prior to bonding with silicone liner**

**c. Assembling of acrylic resin blocks and Teflon jig**

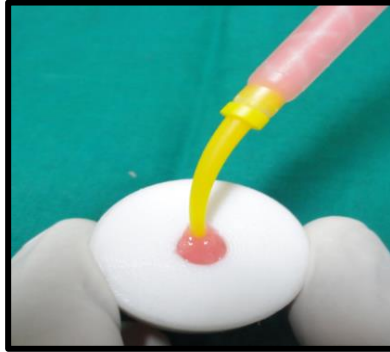


**Fig.50: Assembly of Teflon jig- Acrylic resin block prior to adding silicone liner**

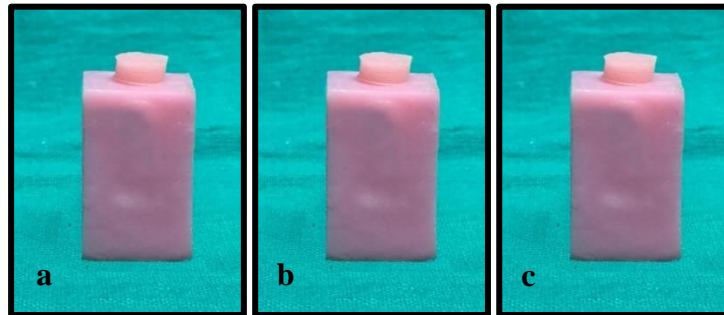


**Fig.51a & b: Schematic representation of Assembly of Teflon jig – Acrylic resin block prior to adding silicone liner**

**d. Bonding of silicone based soft liner to heat polymerized acrylic resin blocks**



**Fig.52: Incorporation of silicone-based chair-side soft liner**



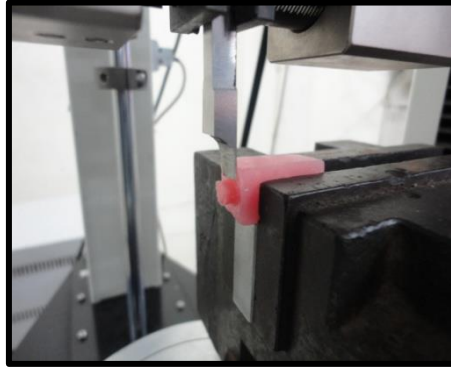
**Fig.53a, b & c: Test samples of Group A, Group B & Group C respectively**

**VIII. Thermocycling of test samples**



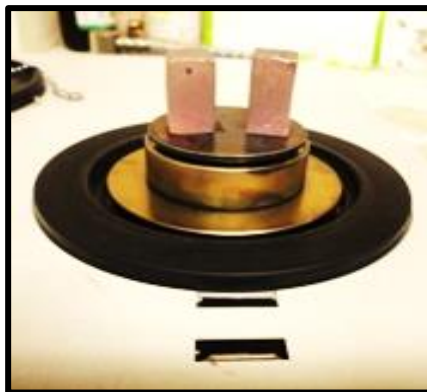
**Fig.54: Thermocycling of test samples**

**IX. Shear bond strength testing of the test samples**

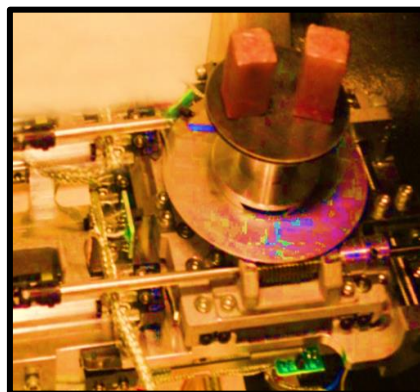


**Fig.55: Shear bond strength testing of test samples in Universal Testing Machine**

**X. Qualitative analyses of bond strength and mode of failure by Scanning Electron Microscopy (SEM):**



**Fig.56: Gold sputtering of debonded test samples prior to SEM analysis**



**Fig.57: Surface analysis of debonded test samples using Scanning Electron Microscopy**

## RESULTS

The present in-vitro study was conducted to comparatively evaluate the effect of two different surface treatments on the shear bond strength between silicone soft liner and heat polymerized denture base resin after thermocycling.

Thirty three acrylic resin blocks were grouped into three groups of eleven blocks each and were subjected to the following surface treatments:

1. Group A (n=11) untreated surface of acrylic resin blocks (Control Group).
2. Group B (n=11) for sandblasting surface treatment of acrylic resin blocks (Sandblasted Group).
3. Group C (n=11) for laser irradiation surface treatment of acrylic resin blocks (Laser irradiation Group).

One surface treated acrylic resin block from each group was used for 3-D surface roughness analysis. The remaining surface treated ten acrylic resin blocks of each group were used for bonding with the silicone liner and subjected to shear bond strength testing. These were divided into three groups as follows:

Group A – silicone-based soft liner bonded to untreated test surface of heat polymerized acrylic block (n=10, Control group).

Group B – silicone-based soft liner bonded to sandblasted test surface of heat polymerized acrylic block (n=10, Sandblasted Group).

Group C – silicone-based soft liner bonded to laser irradiated test surface of heat polymerized acrylic block (n=10, Laser irradiation Group).

One representative tested sample from each test group (Group A, Group B and Group C) was randomly selected and qualitatively analyzed using Scanning Electron Microscopy (SEM) under 100x magnification. The mode of failure was analyzed using the SEM data.

**The following results were drawn from the study:**

**Fig.58, Fig.59 and Fig.60** show surface textures and surface roughness values (RA values) for one representative surface treated Group A, Group B and Group C acrylic resin blocks respectively.

**Tables 1** shows the Basic data and Mean shear bond strength value between silicone soft liner and Control acrylic resin block (Group A, Untreated).

**Tables 2** shows the Basic data and Mean shear bond strength value between silicone soft liner and sandblasted acrylic resin block (Group B, Sandblasted group).

**Tables 3** shows the Basic data and Mean shear bond strength value between silicone soft liner and laser irradiated acrylic resin block (Group C, Laser irradiated group).

**Table 4** shows comparative evaluation of the mean shear bond strength value of untreated samples (Group A), sandblasted samples (Group B) and laser irradiated samples (Group C) using One Way Analysis of Variance (ANOVA).

**Table 5** shows multiple comparisons of mean shear bond strength values of untreated samples (Group A), sandblasted samples (Group B) and laser irradiated samples (Group C) using Post-hoc Tukey's HSD analysis.

**Graph 1** shows the Basic data of shear bond strength between silicone soft liner and Control acrylic resin block (Group A, Untreated).

**Graph 2** shows the Basic data of shear bond strength between silicone soft liner and sandblasted acrylic resin block (Group B, Sandblasted group).

**Graph 3** shows the Basic data of shear bond strength between silicone soft liner and laser irradiated acrylic resin block (Group C, Laser irradiated group).

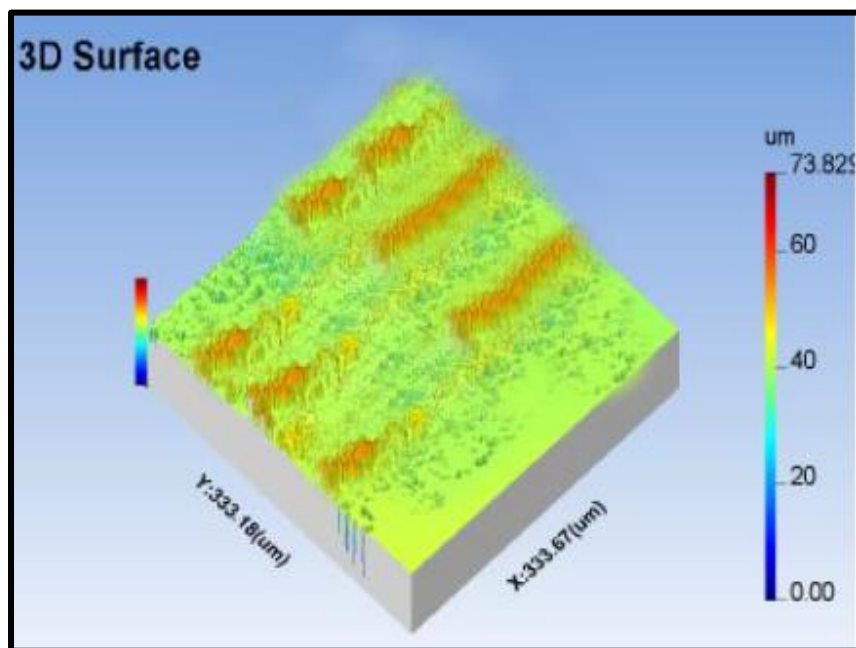
**Graph 4** shows the Comparisons between the mean shear bond strength values of Untreated (Group A), Sandblasted (Group B) and Laser irradiated (Group C) test groups.



### ANNEXURE III

**Fig.58: 3-D surface texture analysis photomicrograph of Group A (Control/Untreated Group) representative acrylic block by 3-D Surface Profilometer**

Advanced 3-D view

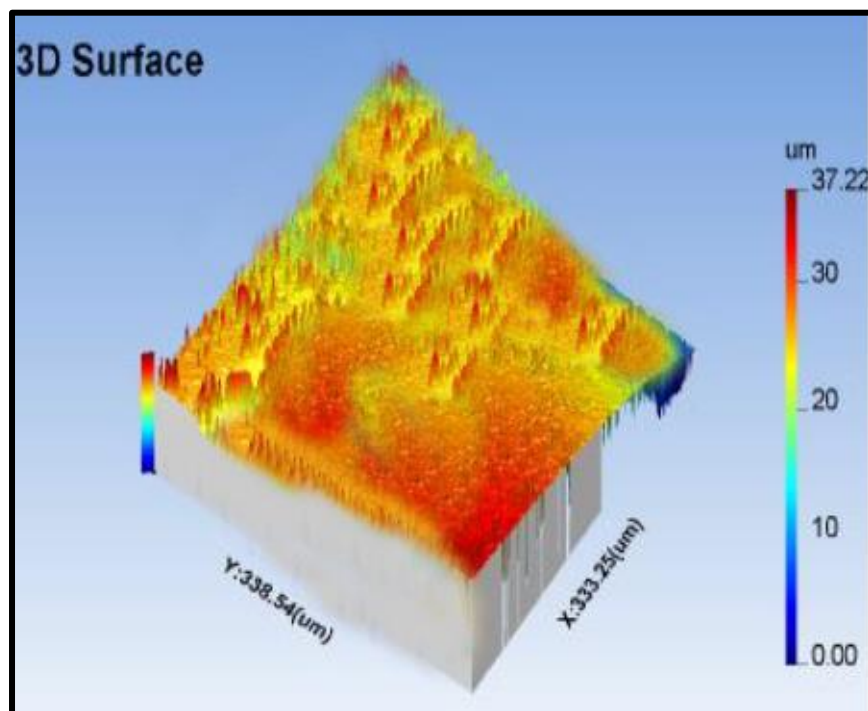


Parameters	Values in $\mu\text{m}$
Rp	2.03
Rv	2.10
<b>Ra</b>	<b>0.449</b>

**Inference:** Advanced 3-D images for Group A representative control sample exhibits sparsely distributed peaks and valleys throughout the test surface.

**Fig.59: 3-D surface texture analysis photomicrograph of Group B (Sandblasted Group) representative acrylic block by 3-D Surface Profilometer**

Advanced 3-D view

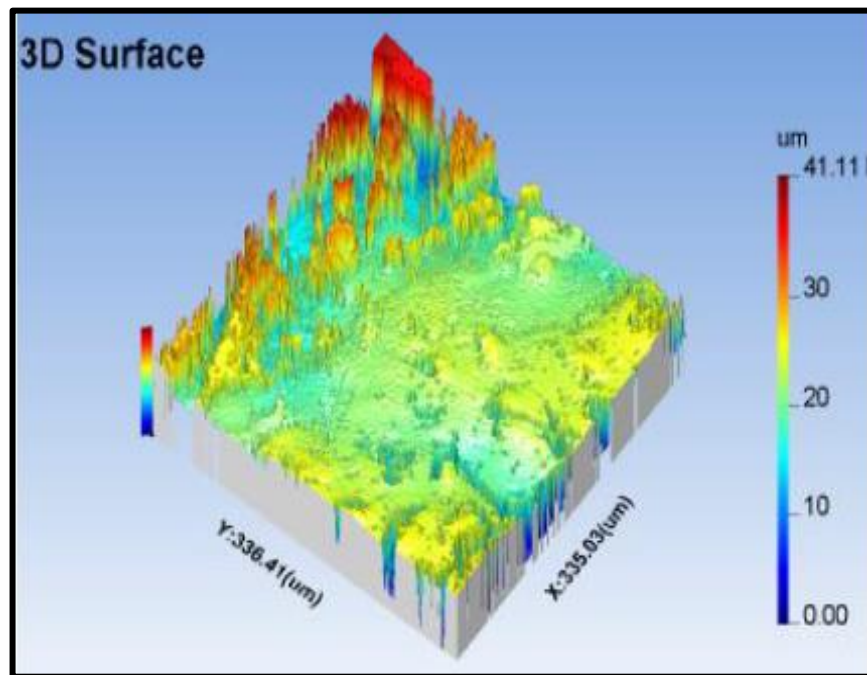


Parameters	Values in $\mu\text{m}$
Rp	5.32
Rv	2.45
<b>Ra</b>	<b>1.40</b>

**Inference:** Group B representative surface treated sample showed well defined peaks and valleys confined to minor portion on the test surface. Major portion of the bonding surface demonstrated uneven distribution of peaks and valleys.

**Fig.60: 3-D surface texture analysis photomicrograph of Group C (Laser irradiated Group) representative acrylic block by 3-D Surface Profilometer.**

Advanced 3-D view



Parameters	Values in $\mu\text{m}$
Rp	3.45
Rv	3.56
<b>Ra</b>	<b>1.59</b>

**Inference:** Group C representative surface treated sample showed evenly distributed and well defined distribution of peaks and valleys on the test surface.

**1: Basic data and Mean shear bond strength value between silicone soft liner and Control acrylic resin block (Group A, Untreated)**

<b>Sample No</b>	<b>Shear Bond Strength in MPa</b>
1	0.36455
2	0.34415
3	0.38011
4	0.27199
5	0.35779
6	0.29529
7	0.25677
8	0.31799
9	0.33103
10	0.28590
<b>Mean</b>	<b>0.320557</b>

**Inference**

Group A exhibited maximum shear bond strength value of 0.38011MPa and minimum shear bond strength value of 0.25677Mpa. The mean shear bond strength was 0.320557 MPa.

**Table 2: Basic data and Mean shear bond strength value between silicone soft liner and sandblasted acrylic resin block (Group B, Sandblasted group)**

<b>Sample No</b>	<b>Shear Bond Strength in MPa</b>
1	0.47911
2	0.45155
3	0.51365
4	0.41987
5	0.41009
6	0.53349
7	0.57755
8	0.49165
9	0.55565
10	0.51855
<b>Mean</b>	<b>0.495116</b>

### **Inference**

Group B exhibited maximum shear bond strength value of 0.57755Mpa and minimum shear bond strength value of 0.41009MPa. The mean shear bond strength was 0.495116MPa.

**Table 3: Basic data and Mean shear bond strength value between silicone soft liner and laser irradiated acrylic resin block (Group C, Laser irradiated group)**

<b>Sample No</b>	<b>Shear Bond Strength in MPa</b>
1	0.51554
2	0.55518
3	0.53611
4	0.58831
5	0.52335
6	0.58206
7	0.54245
8	0.58939
9	0.52663
10	0.57911
<b>Mean</b>	<b>0.553513</b>

**Inference**

Group C exhibited maximum shear bond strength value of 0.58939Mpa and minimum shear bond strength value of 0.51554MPa. The mean shear bond strength was 0.553513MPa.

**Table 4: Comparative evaluation of the mean shear bond strength value of untreated samples (Group A), sandblasted samples (Group B) and laser irradiated samples (Group C) using One Way Analysis of Variance (ANOVA)**

<b>Groups</b>	<b>Mean shear bond strength (Mpa)</b>	<b>Standard Deviation</b>	<b>'p' Value</b>
A	0.32	0.042	0.000*
B	0.50	0.056	
C	0.55	0.029	

**Note:** 'p' value < 0.05 denotes statistical significance.

### **Inference**

One way analysis of variance (ANOVA) shows overall statistically significant difference between the test groups at 5% level. Group C showed the highest mean shear bond strength followed by Group B and least by Group A.

**Table 5: Multiple comparisons of mean shear bond strength values of untreated samples (Group A), sandblasted samples (Group B) and laser irradiated samples (Group C) using Post-hoc Tukey’s HSD analysis**

<b>Groups</b>	<b>Mean shear bond strength (Mpa)</b>	<b>‘p’ value</b>
Group A	0.32	0.00*
Group B	0.50	
Group A	0.32	0.00*
Group C	0.55	
Group B	0.50	0.01*
Group C	0.55	

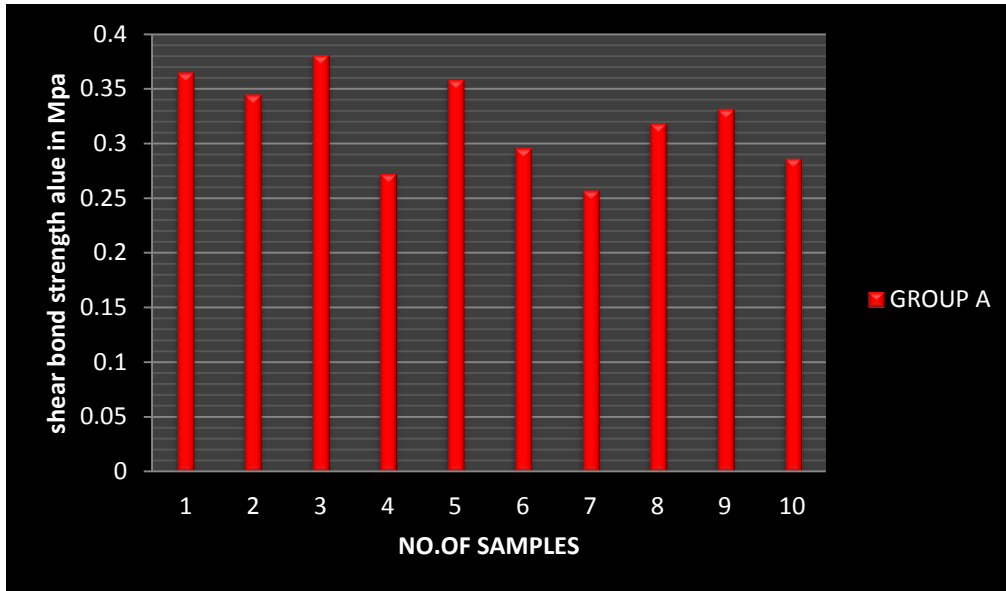
‘p’ value < 0.05 denotes statistical significance.

Multiple comparisons of the mean shear bond strength values using Post-hoc Tukey’s HSD analysis, statistically significant differences were revealed between all the test groups. Group C (Laser irradiated group) exhibited significantly higher shear bond strength compared to Groups A and B, followed by Group B (Sandblasted group). Group A (Control/Untreated surface group) was significantly lesser than both Groups B and C.

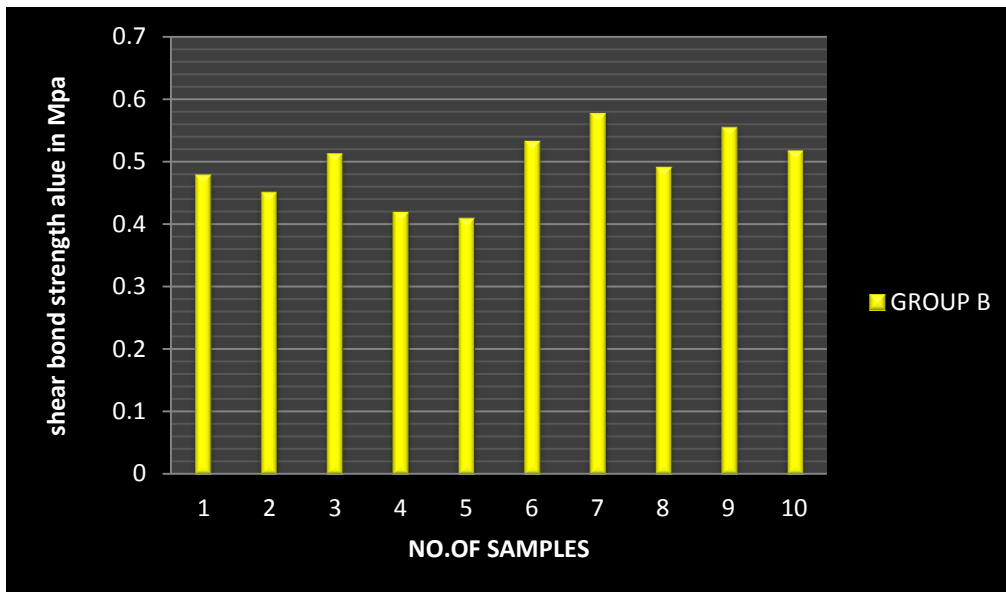


## ANNEXURE IV

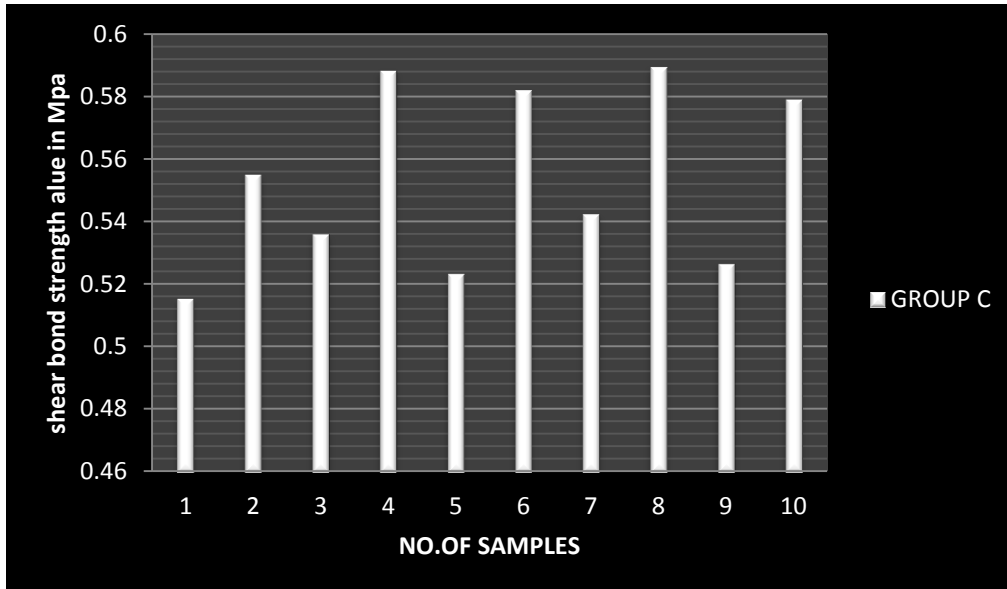
**Graph 1: Basic data of shear bond strength between silicone soft liner and Control acrylic resin block (Group A, Untreated)**



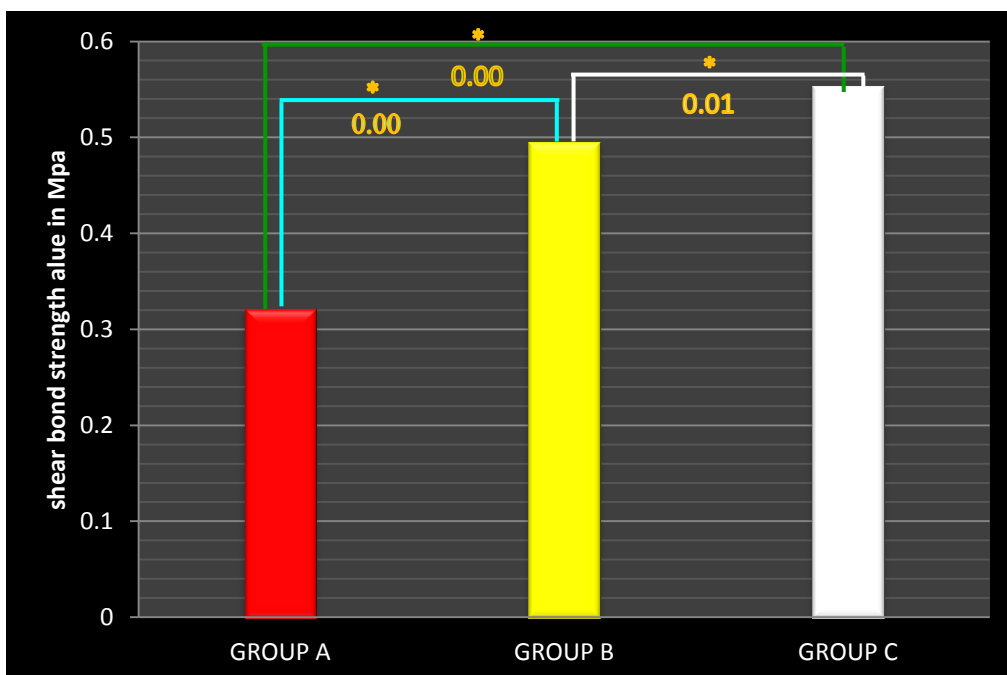
**Graph 2: Basic data of shear bond strength between silicone soft liner and sandblasted acrylic resin block (Group B, Sandblasted group)**



**Graph 3: Basic data of shear bond strength between silicone soft liner and laser irradiated acrylic resin block (Group C, Laser irradiated group)**



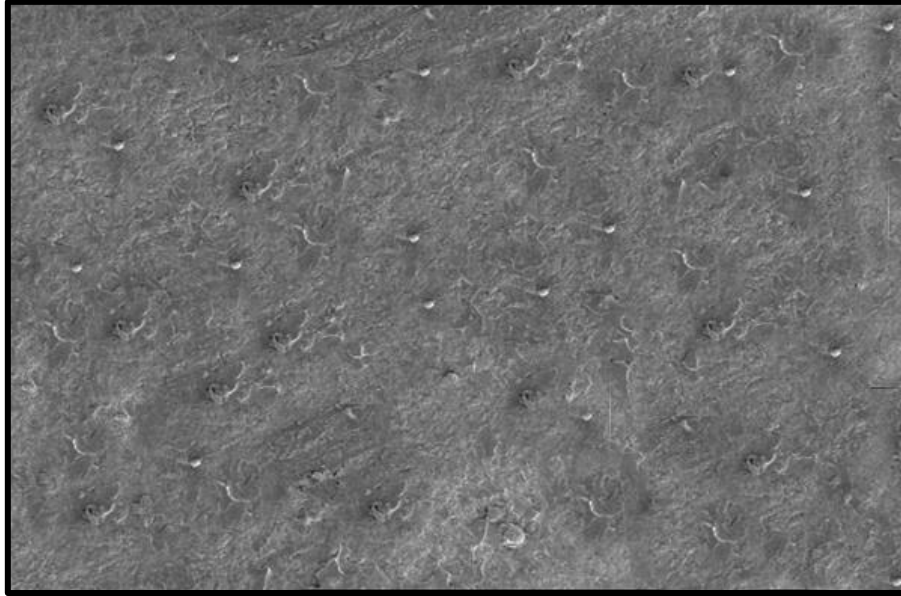
**Graph 4: Comparisons between the mean shear bond strength values of Untreated (Group A), Sandblasted (Group B) and Laser irradiated (Group C) test groups.**



\*p < 0.05, statistically significant

## ANNEXURE V

### QUALITATIVE ANALYSIS OF MODE OF FAILURE BY SCANNING ELECTRON MICROSCOPY (SEM)



**Fig.61 SEM photomicrograph of representative Group A  
(Control/Untreated Group) debonded sample under 100x magnification**

#### **Inference:**

Under 100x magnification, few, thin and isolated areas of soft liner material on the resin surface were visible indicating a mixed mode of failure. More of the resin surface was exposed, indicating a predominantly adhesive pattern within the overall mixed failure observed.

**QUALITATIVE ANALYSIS OF MODE OF FAILURE BY SCANNING  
ELECTRON MICROSCOPY**

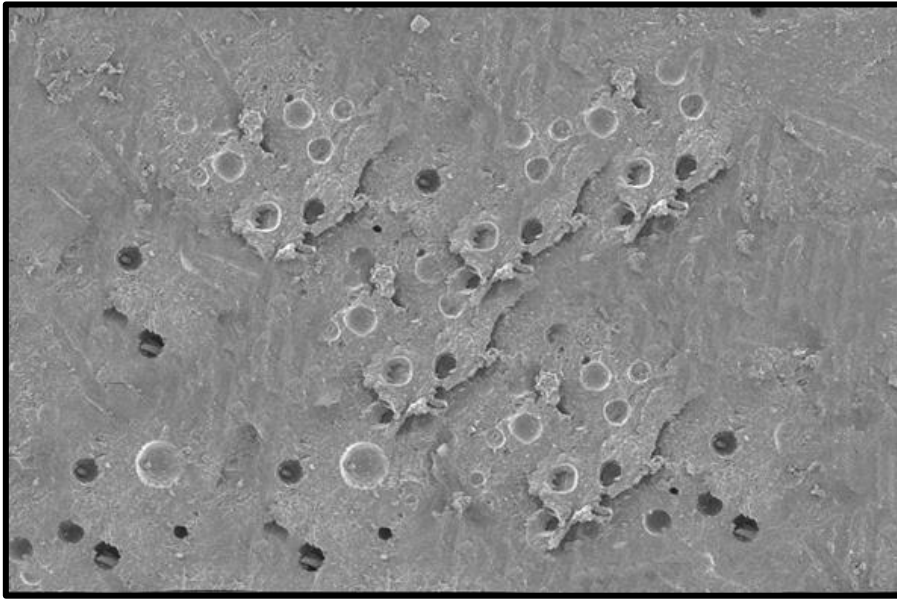


**Fig.62 SEM photomicrograph of representative Group B (Sandblasted Group) debonded sample under 100x magnification**

**Inference:**

Under 100x magnification, increased areas of soft liner material of greater thickness were visible. Lesser surface of exposed acrylic resin was visible. This pointed towards a mixed mode of failure. The observed pattern indicated a predominantly cohesive pattern of failure of the silicone liner within the overall mixed failure observed.

**QUALITATIVE ANALYSIS OF MODE OF FAILURE BY SCANNING  
ELECTRON MICROSCOPY**



**Fig.63 SEM photomicrograph of representative Group C (Laser irradiated) debonded sample under 100x magnification**

**Inference:**

Under 100x magnification, increased areas of soft liner material of much greater thickness were visible. The exposed resin surface was sparse. This indicated a mixed mode of failure. The observed pattern indicated a predominantly cohesive pattern of failure of the silicone liner within the overall mixed failure observed.

## DISCUSSION

The present in-vitro study was conducted to comparatively evaluate the effect of two different surface treatments on the shear bond strength between silicone soft liner and heat polymerized denture base resin after thermocycling.

Soft denture liners have been recognized as a valuable adjunct in Prosthodontic practice since they were introduced by Mathews in 1945 in the form of plasticized polyvinylchloride<sup>12,13</sup>. They have been used to provide comfort for patients who cannot tolerate occlusal pressure or present with alveolar ridge resorption, chronic soreness, knife-edge ridges, bony undercuts, thin and non-resilient mucosal tissues, bruxing tendencies, congenital and acquired oral defects requiring obturation, xerostomia and also to modify transitional implant prosthesis<sup>12,22,23,36</sup>.

Liners act as a cushion for the denture bearing mucosa by absorbing and evenly distributing functional loads, thereby improving patient's comfort. Currently available soft liners can be categorized as plasticized acrylic and silicone elastomers. Both of these can be either autopolymerizing (chair-side reliners) or heat polymerizing (laboratory reliners)<sup>12,13,22,20,40</sup>.

Autopolymerizing soft lining materials are usually used for direct, chairside relining, as they are easy to manipulate and require no laboratory procedures. They also serve as economical and time saving procedure to

resurface the tissue surface of an existing denture<sup>1,12,20,22,33,35</sup>. Although acrylic resin-based soft liners are similar in chemical structure to polymethyl methacrylate (PMMA) denture base resins, silicone liners are reported to keep their softness for the longer periods than acrylic resin liners<sup>12</sup>. Previous drawbacks with silicone reliners have been reduced and currently available silicone reliners have improved properties like low water sorption, low solubility and low surface roughness.

However some of the reported drawbacks with the use of soft liners are separation between the materials at the bond interface leading to colonization by microorganisms, poor tear strength and loss of softness. Further, when exposed to the moist oral environment these materials absorb water leading to leaching out of plasticizers from the liner thereby reducing their resilient potential<sup>12,20,28,31</sup>. Moreover, unlike acrylic soft liners, silicone liners do not bond chemically with the underlying acrylic resin which may also render this joint susceptible.

The fluctuations in temperatures in the oral cavity to which these materials are also exposed can also significantly compromise the bond strength<sup>12,20,28,31</sup>. Hence bond strength is considered to be very important with regards to clinical outcome<sup>12</sup>. According to Glossary of Prosthodontic terms, bond is the force that holds two or more units of matter together<sup>50</sup>. Bond strength is the force required to break a bonded assembly with failure occurring in or near the adhesive/adherens interface<sup>50</sup>. Lack of durable bond

between the resilient liner and the denture is reported to be a common clinical problem<sup>6,12,28</sup>.

Several methods have been advocated to enhance the bonding of acrylic denture base to silicone soft liner. They can be broadly categorized into mechanical and chemical modifications or a combination of both<sup>6</sup>. Mechanical methods are reported to produce an improvement in bond strength<sup>2,22,23,25,27</sup>.

Among these methods, sand blasting (Air abrasion) involves spraying a stream of aluminium oxide particles against the material surface intended for bonding under high pressure, since alumina particles employed produce micromechanical retention by producing surface irregularities. Aluminium oxide of various particle sizes has been employed to enhance the bond between the silicone based soft liner and denture base resin<sup>2,23,26</sup>. The role of sandblasting in improving the bond strength between soft liners and acrylic resin remains controversial and has been recommended for further investigation in the previous studies<sup>6,22,23,35,38</sup>.

Progress in laser technology has shown a quick adoption in the field of dentistry since the development of the first working laser by Maiman in 1960. Recently, lasers have been found to provide relatively safe and easy means of altering the bonding surface of materials. Theoretically, it should benefit the bonding interface and result in stronger bond. Laser irradiation with various lasers like Er:YAG, Er,Cr:YSGG, Nd:YAG, KTP lasers have been reported to modify the intaglio surface of the denture before application of liner materials.



It has been indicated in one study that Er,Cr:YSGG laser treatment may significantly enhance the shear bond strength between silicone soft liner and denture base resin<sup>27</sup>. However, literature using Er,Cr:YSGG laser as a surface modification method of denture base to yield better bond strengths is sparse<sup>27</sup>.

The bond strength of the liner-denture base interface has been researched extensively by some authors. Al-Athel et al<sup>3</sup> studied the various bond strength assessment methods, namely, peel test, tensile bond strength and shear bond strength between the liner-denture base interfaces. He concluded that shear forces best represent the oral conditions in which the liner functions. Hence shear bond strength of the material is more indicative of its clinical longevity. The peel test is believed to simulate the horizontal component of masticatory forces as it causes lateral displacement of the denture. Tensile test on the other hand predominantly represents the vertical component of the masticatory forces.

It has also been pointed out that tensile failure was not caused by tensile forces alone because some shear forces also developed during tensile testing. This specially stands true in case of silicone lining material which has a high Poisson ratio. These materials undergo a reduction in cross sectional area on tensile load application, whereas, the bonded portion of the liner maintains a constant area. This induces shear forces at the margins of the bonded interface<sup>25,35</sup>.

Since soft denture liners function in an aqueous oral environment under rapidly changing temperature conditions, the impact of these two parameters on the bond strength is also important while conducting bond strength tests in-vitro. Thermocycling of test samples prior to testing of bond strength is done in in-vitro studies to assess the impact of these parameters and mimic oral conditions.

Cyclic thermal stress causes shear stress at the bonding interface, as it provokes repetitive shrinkage and expansion and results in a difference of thermal volumetric change between denture base and soft denture liner. During thermocycling, soft denture liner absorbs large amount of water which may result in hydrolytic degradation of the bond due to water diffusion into the interface<sup>6,31,32,35,38,39,44,48</sup>.

There is paucity of data comparing the effect of laser surface treatment with other surface treatment methods on the shear bond strength between chair-side silicone based soft liner and denture base resin, in view of its clinical impact and significance<sup>27</sup>. Further, longevity of the bond between chair-side silicone liners and denture base resin is still not clarified in the literature. Hence, in light of the above, the aim of the present in-vitro study was to comparatively evaluate the effect of two different surface treatments on the shear bond strength between silicone soft liner and heat polymerized denture base resin after thermocycling. The null hypothesis for the present study was that different surface treatments will not significantly affect the

shear bond strength between silicone soft liner and heat polymerized denture base resin.

A chair-side reliner was evaluated in the present study because of the advantages of these reliners mentioned earlier. Silicone soft reliner was selected based on its aforementioned advantages over acrylic chairside reliners.

The type of bond strength testing in the present in-vitro study was limited to shear testing because of the previously explained stress patterns that are generated during such testing.

All the steps discussed in the methodology for sample preparation were performed by a single operator to avoid operator-based errors and bias. The rectangular acrylic test blocks were made with specific dimensions to enable proper fixation of the block on the testing platform of the universal testing machine during subsequent bond strength testing. A customized stainless steel block was employed to serve as a template for obtaining acrylic blocks of uniform dimensions, as part of test standardization. Addition silicone was employed as indexing material since the material can withstand repeated handling without undergoing distortion or tearing<sup>8</sup>.

Heat cure acrylic denture base resin was the material employed for obtaining the test resin blocks since PMMA is the most widely used denture base material clinically and has been considered as an near-ideal material for

this purpose<sup>8,35</sup>. The test blocks were fabricated and stored as per standard protocols<sup>8</sup>. To limit and standardize the area of surface modification, only one 14mm×14mm surface was considered and designated as the test surface on which surface preparations as well as bonding of the silicone liner were carried out.

Since the present study was conducted to compare the effects of two different surface treatments on the bond strength between silicone liner and acrylic resin, untreated (control) group of sample were included for the purpose of comparison of test results<sup>6,27</sup>. The test surface of this group of samples was not subjected to any type of surface treatment.

Sandblasting and laser irradiation were chosen as the two test surface treatment methods in the present study based on previously outlined reasons. Sandblasting with different grits of aluminium oxide has been employed in the literature. Sandblasting the denture base area with 50µm could only remove the surface glaze on the denture base area but had no significant effect in improving the bond strength between the denture base resin and soft liner. Most of the studies reported that grit size in the range of 110-120 µm Al<sub>2</sub>O<sub>3</sub> particle is adequate to improve the bond strength<sup>2,41</sup>. Hence in the present study, Aluminium oxide of 110µm was chosen for this type of surface treatment. Laser irradiation was carried out using Er,Cr:YSGG based on that employed in a previous study<sup>27</sup>.

3-D surface profilometry was carried out for one representative of all the three types of test surfaces (Untreated, Sandblasted and Laser irradiated) to assess the surface topography and roughness of these surfaces since it may aid in interpretation of the test results as reported in a previous study<sup>36</sup>. Surface roughness (Ra value) is the arithmetic average deviation of surface valleys and peaks expressed in microns and are a measure of the finer surface irregularities in surface texture.

Silicone-based soft liners have little or no chemical adhesion to the denture base resin. Hence, manufactures supply a primer as part of the standard kit to aid in bonding of the silicone liner to the denture base resin<sup>12,22,32,35</sup>. Hence, primer was applied as per manufacturer's instructions on the designated bonding areas of each test sample of all the three test groups.

The silicone liner application was done only for the designated test surfaces of each test block in the form of cylindrical columns of 3mm height and 6mm diameter based on similar procedures followed in previous studies<sup>1,4,10,48</sup>. A custom made Teflon jig was fabricated to achieve this purpose to obtain silicone liners of uniform dimensions on each test block. Since the Teflon jig was milled, it obviated the need for making individual templates for bonding as used in source previous studies. Additionally, Teflon being an inert material does not react with the liner employed in this study and also facilitated easy retrieval of the test samples after the bonding procedure.

Bonded test specimens of all test groups were subjected to thermocycling because of reasons discussed before. In the present study, a smaller thermocycling period mimicking three months of clinical use was employed.

The test specimen interface resembled a clinical scenario of a single soft liner-denture base interface, along which parallel shear forces could be applied to evaluate the shear bond strength. The load at which the bond failed under shear stress was recorded in newton (N) and was taken as a shear load value of the particular sample. The shear bond strength values in MPa were obtained by dividing the shear load values (N) by the cross sectional area of bonding<sup>1,2,24,32</sup>. In the present study, since the bonding was confined to a circular area of 6mm diameter, the cross sectional area was calculated using the formula  $\pi r^2$  (area of a circle). The bonding area of the specimens was around 28.274 mm<sup>2</sup> which was calculated as follows:

$$\text{Bond Strength (MPa)} = \text{Force (N)} / \text{surface area (mm}^2\text{)}$$

$$\text{Bond Strength (MPa)} = \text{Force (N)} / \pi r^2 = \text{Force (N)} / 3.14159 \times 9$$

$$\text{Bond Strength (MPa)} = \text{Force (N)} / 28.274 \text{ mm}^2$$

$$\text{(Area of circle} = \pi r^2, \text{ value of } \pi = 3.14159, r = 3 \text{ mm)}$$

The basic values of shear bond strength obtained for all the test samples of the three test groups in the present study were tabulated and statistically analyzed.

Scanning Electron Microscopy (SEM) analysis of one representative debonded test sample of each test group was done to correlate the shear bond strength test values with the SEM observations of the debonded interface as done in previous studies<sup>1,5,6,9,10,12,16,20,43,45</sup>.

The results obtained by surface texture analysis, shear bond strength testing and SEM analysis were recorded and interpreted individually and also correlated to understand the test results.

The surface texture analysis of one respective sample of each test group revealed that surface treatment by both sandblasting (Group B) as well as laser irradiation (Group C) increased the surface roughness values (Ra - 1.40 $\mu$ m and Ra - 1.59 $\mu$ m respectively), compared to that of the untreated surface (Ra - 0.449 $\mu$ m) (Figs: 58, 59 & 60 respectively). The surface topography of the treated specimens also exhibited pronounced peaks and valleys indicative of a roughened surface. These peaks and valleys are more evenly distributed for the laser irradiated sample. This was in contrast to the sparse and poorly distributed peaks and valleys seen in the untreated sample.

The basic data obtained in this study showed a mean shear bond strength of 0.320557Mpa for untreated test samples (Group A), 0.495116Mpa for sandblasted test samples (Group B) and 0.553513Mpa for laser irradiated test samples (Group C) (Tables 1, 2 & 3 respectively). Overall comparison of mean shear bond strength values was done of the three test groups using One Way Analysis of Variance (ANOVA) and p value < 0.05 was considered

significant (Table 4). Since ANOVA test revealed significant difference between the three test groups, the means were subjected to multiple comparisons for pair-wise significance using Post Hoc Tukey's HSD analysis (p value <0.05 considered significant) (Table 5).

Post Hoc Tukey's HSD analysis revealed significant differences between all the three test groups: Group A- Group B; Group A- Group C and Group B- Group C (p value < 0.05). On statistical comparison, Group A (Untreated/Control Group) exhibited the least mean shear bond strength value among the three test groups and this was significantly lesser (p value < 0.05) than those of both Group B (Sandblasted Group) and Group C (Laser irradiated Group). Statistical comparison between the mean shear bond strength values of Group B and Group C revealed that Group C (Laser irradiated Group) had significantly higher shear bond strength value (p value < 0.05) than Group B (Sandblasted Group).

<p style="text-align: center;"><b>MEAN SHEAR BOND STRENGTH</b></p> <p style="text-align: center;">Group C * &gt; Group B * &gt; Group A (Laser irradiated) (Sandblasted) (Untreated)</p> <p style="text-align: center;">[&gt; - greater than; * - statistically significant]</p>
--

In a previous study by the Jacobson NL et al <sup>23</sup> both laser treatments as well as sandblasting surface treatments were shown to be ineffective in reducing the adhesive failure between soft liner and acrylic resin. This could



be attributed to the CO<sub>2</sub> laser used in that study <sup>23</sup> in contrast to the Er,Cr:YSGG laser employed in the present study. Further, the significant improvement by sandblasting observed in the present study compared to them, can be attributed to differences in study design, sample preparation and study environment.

Korkmaz FM et al<sup>27</sup> evaluated the effect of acrylic surfaces treated with laser irradiation and sandblasting on the peel strength between silicone soft liner and denture base resin. They reported a significant increase in peel strength values when surface was treated with laser irradiation than with sandblasting. The results obtained in the present are in line with those obtained for shear bond strength in that study, which has also shown that laser irradiation, could significantly improve the shear bond strength of the test samples. The type of laser used in the present study and by Korkmaz FM et al in their study was also Er,Cr:YSGG. Since the peel strength results obtained in Korkmaz FM et al<sup>27</sup> are in better correlation with the shear bond strength results obtained in the present study, it can be said that the type of laser used may also impact test results.

Since studies on the effect of laser surface treatment on shear bond strength between silicone liner and denture base resin are lacking, further direct correlation with the results of the present study cannot be obtained.

Air abrasion by sandblasting is said to improve the surface roughness by providing an irregular surface for the mechanical locking of the soft

material and is said to be the cause of improved bond strength<sup>6,23</sup>. However, some studies employing sandblasting as a mode of surface treatment have reported decreased bond strength values with this surface treatment. This has been attributed to stress concentration at the bond interface resulting in bond failure<sup>22,35,38</sup>. However, most of these bond strength studies are either peel or tensile strength stress tests. Studies comparing the effect of surface treatment with sandblasting on the shear bond strength are few<sup>6,25</sup>. In a study<sup>6</sup>, surface treatment by sandblasting resulted in significantly higher shear bond strength values between silicone soft liner and acrylic denture base resin as compared to the untreated (or) controlled samples. The results obtained in the present study are in line with those obtained in the above study<sup>6</sup>.

The increased surface roughness (Ra values) observed for both the surface treated groups is in direct correlation to the significantly increased shear bond strength values for these groups as against those for the untreated group in the present study. This indicates that surface treatment by either method, especially by laser irradiation, could improve the surface roughness to yield significantly higher shear bond strengths. Most studies on bond strength between soft liners (of any type) and acrylic resin have not included surface texture analysis as part of test protocol. Only one study<sup>36</sup> has included this investigation to study the surface of soft liners and found significant differences between surfaces of different liner materials. Surface texture analysis is significant in studies where the effects of different surface

treatments are tested, since the surface topography can play an important role in impacting the results.

The direct correlation between surface texture analysis and shear bond strength improvements obtained in the present study further validate this point and hence this investigation can be included in future similar studies.

Scanning Electron Microscopic analysis of the debonded specimens revealed a mixed type of failure for all three test groups (Figs. 61, 62 & 63). The untreated (Control Group) surface exhibited a predominant adhesive pattern within the mixed mode of failure. There was more of visible resin surface, with the sparsely distributed liner material. Both surface treated samples exhibited a cohesive pattern within the mixed mode of failure. There were several patches of silicone lining material distributed over the resin surface. This was more pronounced in the laser irradiated sample which showed greater cohesive pattern compared to that observed for the sandblasted group. This observation for the laser irradiated sample is in line with those observed by Jacobson et al<sup>23</sup>, who found that a majority of laser treated specimens experienced cohesive pattern of failure.

The types of failures observed under SEM in previous similar studies<sup>5,6,9,10,23,43,45</sup> revealed that silicone liners showed different failure patterns under different testing conditions. The type of bond strength testing (whether peel, tensile or shear), mode of surface treatment rendered, etc. can impact the mode of failure, and this could have resulted in the different modes

of failures observed. Previous studies have revealed a cohesive pattern of failure for silicone liners bonded to acrylic resin, which is in line with the SEM observations of the present study<sup>5,6,10,23,45</sup>. Further investigations are recommended to arrive at the exact mode of failure between silicone liners and acrylic resins following shear testing.

Since a silicone soft denture liner does not adhere chemically to denture base resins, primers are used as a part of routine bonding procedures. Therefore, the bond strength would also be impacted by the chemical composition of the primer which is not revealed by the manufacturers. This aspect also needs further evaluation.

Within the limitations of the present study, it can be concluded that surface treatment of denture base resin by either sandblasting with aluminium oxide 110µm or laser irradiation with Er,Cr:YSGG laser can significantly improve the shear bond strength between silicone soft liner and heat cure denture base resin, than if the resin surface is left untreated. Laser irradiation results in significantly higher mean bond strength as compared to surface treatment by sandblasting. Hence the null hypothesis of the present study is rejected because of significant differences in shear bond strength values between sandblasting and laser irradiation.

It has been reported that a bond strength of 0.44Mpa is a minimum acceptable measure of bond strength that is required for clinical use of soft denture lining materials<sup>12,20,22,32</sup>. When viewed in this light, the mean shear

bond strength result obtained for both the surface treated groups (Sandblasted and Laser irradiated) are within clinically acceptable limits for bond strength (0.495116Mpa and 0.553513Mpa respectively). The mean shear bond strength obtained for the control group is 0.320557Mpa, indicated less than optimal bond strength value for untreated group.

Hence, it can be recommended that surface modification of acrylic resin prior to bonding with chair-side silicone soft liner should be carried out preferably for better clinical outcomes. The choice of surface treatments between sandblasting and laser irradiation can be based on availability and/or operator's preference, though laser irradiation may yield better results.

The present study had some limitations. Only one composition of chair-side reliner was tested. The effects of other type of surface treatments mentioned in the literature were not included. Thermocycling which is used to mimic oral conditions was done for a short period, simulating 3 months of clinical use. Longer durations might impact the study results differently. Further, different intensities of laser irradiation can bring about differences in test outcomes. Further studies that include the above variables with a larger sample size are recommended to enhance the results obtained from the present study.

## CONCLUSION

The following conclusions were drawn from the results obtained in the present in vitro study, which was conducted to comparatively evaluate the effect of two different surface treatments on the shear bond strength between silicone soft liner and heat polymerized denture base resin after thermocycling.

1. Surface texture evaluation of one representative sample of the surface treated acrylic resin block of Group A, Group B and Group C revealed the following:

- The average surface roughness value (Ra) for Group A representative denture base sample was 0.449 $\mu\text{m}$ . Advanced 3-D images showed sparsely distributed peaks and valleys on the test surface of the acrylic resin block.
- The average surface roughness value (Ra) for Group B representative denture base sample was 1.40 $\mu\text{m}$ . Advanced 3-D images showed well-defined peaks and valleys on the test surface of the acrylic resin block.
- The average surface roughness value (Ra) for Group C representative denture base sample was 1.59 $\mu\text{m}$ . Advanced 3-D images showed evenly distributed and well defined distribution of peaks and valleys on the test surface of the acrylic resin block.

2. The mean shear bond strength value for Group A (Control/untreated Group) was found to be **0.320557Mpa**.
3. The mean shear bond strength value for Group B (sandblasted Group) was found to be **0.495116Mpa**.
4. The mean shear bond strength value for Group C (laser irritated Group) was found to be **0.553513Mpa**.
5. On overall comparison of the mean shear bond strength using One Way Analysis Of Variance (ANOVA), statistically significant differences were observed between the three test groups.
6. Multiple comparisons of the mean shear bond strength values of the three test groups using Post-hoc Tukey's HSD analysis revealed statistically significant differences between all the three test groups. Group C (laser irradiated group) exhibited the maximum and significantly higher shear bond strength compared to Groups A and B, followed by Group B (sandblasted group). Group A (control/untreated surface group) showed the least and significantly lesser shear bond strength than both Groups B and C.

**Group C > Group B > Group A**

7. The qualitative evaluation of the mode of failure of one representative test sample from each test group using scanning

electron microscopy under 100x magnifications revealed the following:

- Group A: Few, thin and isolated areas of soft liner material on the resin surface were visible indicating a mixed mode of failure. The exposed resin surface was more greater indicating a predominantly adhesive pattern within the overall mixed failure observed.
- Group B: Increased areas of soft liner material of greater thickness were visible. Lesser surface of the exposed resin was visible. This pointed towards a mixed mode of failure. The observed pattern indicated a predominantly cohesive pattern of failure of the silicone liner within the overall mixed failure observed.
- Group C: Increased areas of soft liner material of much greater thickness were visible. The exposed resin surface was sparse. This indicated a mixed mode of failure. The observed pattern indicated a predominantly cohesive pattern of failure of the silicone liner within the overall mixed failure observed.



## SUMMARY

The present in-vitro study was conducted to comparatively evaluate the effect of two different surface treatments on the shear bond strength between silicone soft liner and heat polymerized denture base resin after thermocycling.

A total of thirty three (n =33) heat polymerized acrylic resin blocks were fabricated from a custom made stainless steel block. The acrylic blocks were divided into three groups of eleven each and were designated as Groups A, B, and C. One of the 14mmx14mm surfaces was assigned as the test surface. Group A test surfaces were left untreated, Group B were sandblasted and Group C were laser irradiated. One representative surface treated acrylic block from each test group was subjected to surface texture analysis. Silicone based soft liner was bonded to the remaining 30 acrylic blocks of Group A (n=10), Group B (n=10) and Group C (n=10), as per the manufacturer instructions. The thirty test samples were thermocycled and later tested for shear bond strength in the Universal Testing Machine. One representative debonded sample from each test group was analysed using scanning electron microscopy at 100x magnification for mode of failure. The basic values and mean shear bond strength of the three test groups were tabulated and subjected to statistical analysis.

3-D surface texture analysis revealed well defined peaks and valleys for both Group B (Sandblasted - Ra value 1.40 $\mu$ m) and Group C (Laser

irradiated- Ra value 1.59 $\mu$ m), with the distribution being more even and well pronounced for Group C. Group A (Untreated/Control – Ra value 0.449 $\mu$ m) in contrast, exhibited sparsely distributed peaks and valleys. This indicates that surface treatment renders the surface more rougher, which may impact the bond strength.

The mean shear bond strength values for all the test groups were compared and found to be statistically significant. Both laser treated and sandblasted group showed significantly greater shear bond strength values than the untreated group. The laser treated group (Group C) (**0.553513Mpa**) showed the highest mean shear bond strength followed by sandblasted samples (Group B) (**0.495116Mpa**) and then by the untreated group (Group A) (**0.320557Mpa**). Control (Untreated) Group exhibited a significantly least shear bond strength value in present study.

Scanning electron microscopy (SEM) of the debonded surfaces of one representative test sample from each group revealed a mixed type of failure for all the three test groups. However, there was a predominant adhesive pattern of failure within the mixed failure for the control group as against a predominant cohesive pattern of failure of silicone liner within the mixed failure for both Groups B and C.

Within the limitations of the present study, surface modification by both sandblasting and laser irradiation significantly improves the shear bond strength between silicone liner and heat cure denture base resin compared to

untreated surfaces. Laser irradiation showed significant and higher bond strength values as compared to that obtained by sandblasting.

Future studies incorporating more surface treatment methods and soft liner types, subjected to longer periods of thermocycling with a larger sample size are recommended to enhance the results obtained from the present study.

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