Evaluation Of Silano Adhesive Efficiency in Improvement of Shear Binding Strength of Synthetic Teeth to Thermo-Plastic Denture Material

Ruyaa Mohammed Ridha (1) *
Alaa Ezzat Abdulmaged (2)

(1,2) Department of Prosthetic Dental Technologies, College of Health and Medical Technology, Middle Technical University, Baghdad, Iraq

Abstract
Background: One of the most serious problems concerning denture performance is the possibility of prosthetic tooth displacement due to increased chewing efficiency. The nature of the mechanical bonding between thermoplastic denture base materials and acrylic teeth has aggravated the problem of tooth debonding. The study aiming to examine the strength of shear bond between prosthetic teeth and thermoplastic base material utilizing silano (silane coupling agent) and mechanical surface modifications (Alumina-blasting, T. Shape hole) of tooth ridge lap pre and post (100 hrs.) of accelerated ageing conditions. Materials and method: The surface treatments of (120) maxillary central incisors were chosen and grouped into 6 groups of 20 samples each. Each group split into before and after (100 hrs.) according to ageing (n=10). Measurement of shear bonding strength applied by a universal testing equipment with 0.5mm/min crosshead speed. Statistical analyses were done using one-way ANOVA test and Tukey’s (HSD) test. Results: Statistics showed high significant variation in average binding force after several surface manipulations in contrast to the control group at (P < 0.000). Regarding surface manipulations, dual treatment of “Alumina-blasting + silano” revealed significant elevation of binding strength in compare to “alumina-blasting” alone (P < 0.001). Similarly, with “T. hole + silano” yielded in greatly increased bonding strength compared to using “T. hole” alone (P < 0.001). a great reduction in bonding strength value was indicated along all treated groups after ageing process (P < 0.000) except the group treated with “T. Hole + silano” show non-significant variations at (p = 0.112). Conclusion: combination treatment (chemo-mechanically) with “T. Hole + silano” improve shear binding strength and not effected with ageing process.
**Introduction:**

In the 1970s and 1980s, patients sought the “Hollywood Smile,” forcing dentists to find prosthetic treatments that were both beautiful and practical. The use of flexible partial dentures for individuals who desired excellent aesthetics and had healthy residual teeth was becoming more common (1). Flexible dentures are extremely pleasant and resistant to breakage since they are made of a specific thermoplastic substance (2). Thermoplastic resins are applied to fabricate complete and partial prosthetic dentures due to their benefits, including: low solvent solubility, elevated binding strength, hardness, flexible nature, ductile consistency, withstand to abrasion, increased temperature tolerance, and chemical impacts tolerability. Because the bond is mostly mechanical, one of the materials' disadvantages is a lower binding strength to prosthetic acrylic teeth (3,4). Synthetic acrylic teeth constitute an essential unit of denture prosthesis which must be bonded to the denture foundation to enhance the duration of the prosthesis (5). The success of the dental prosthesis depends on the appropriate attachment of artificial acrylic teeth to denture material. With implant-supported and retained prostheses, displacement of artificial teeth apart from denture base resins stands single most common prosthetic problems (3). In most flexible removable partial dentures (RPDs) acrylic teeth do not chemically bind to the thermoplastic denture base hence simply detachment from the denture base (6).

It is essential in flexible resin RPDs to present mechanically retention for synthetic teeth in order to strengthen the bonding of artificial teeth to thermoplastic base substance (7). Thus, T-shaped holes with vertical and horizontal grooves may provide better retentive mechanism within prosthetic teeth and thermoplastic resins than straight holes. Teeth composed of synthetic composite material with T-shaped canals are also present (8). Previous research has shown that adhesive compounds enhance the bonding strength of artificial teeth with denture base materials (9). One adhesive substance in common use is silane coupling agent (SCA), which uses unconverted C-C double bonds to form chemical connections with resins (10). Lang et al., 2012, evaluated that tribochemical silica covering and applying SCA for salinization process on the ridge lap side of the artificial teeth prior to denture preparation led to better strengthening of bonding to a significant degree(11). Artificial accelerated aging replicates oral environmental conditions extra-orally.

Artificially aged prosthetic materials have been subjected to UV radiation, variable temperatures, and moisture (12). Aging induces periodic contraction and swelling, which creates tension within the materials, reducing prosthesis lifetime and degrading binding of denture material to artificial teeth. Application of stressful condition exposes materials used in denture preparation to influence of heat that leads to (cracks)(13). The study employed innovative thermoplastic monomer free semi-rigid micro crystalline polymer denture foundation materials (Karadent by TCS, INC, USA). So, it can replace PMMA in allergic people (14). The goals under investigation in the study was guided to identify most effective mode for surface modification strategies for maximizing the shear of binding strength created within attachment surface between artificial acrylic teeth and thermoplastic denture substance.

**Materials and Methods:**

**specimen’s preparation**

Total 120 cylinder-shaped specimens’ dimension (35mm length, 12mm diameter) with acrylic teeth were constructed. The laser-cutting equipment (TIL-6090, India ,2009) prepared the custom-made acrylic discs and (7 shaped) piece (Sumi-pex acrylic sheets (2mm), India). Auto CAD 2015 software was used to create the final model. The specimen molds were created using an acrylic discs and copper tubing as follows(15)

**Acrylic discs designing:**

A pair of rounds, custom-made acrylic discs with a hole in the center for custom
copper tubing were fabricated. A slot was created in the discs for a (7 shaped) piece. The discs are secure a copper tube mould and a (7 shaped) piece. A seven (7 shaped) piece with 45° angle end essential for accurate acrylic tooth placement according to Japanese standards for acrylic teeth (JIST 6506, 1989) (16). The dimensions are shown in Fig.(1a).

**Copper Tube designing:**
A turning machine was employed to create a unique tubing mold for a test specimen wax design with outside diameter of 24.5mm, inner hole diameter of 12mm, and length of 35mm (15). Maxillary central incisor acrylic teeth (Sinalident, Hencheng Shuzhi) were utilized for testing. The tooth was only embedded into the wax cylinder to the neck of the central incisor. The mold portions of the shear binding with acrylic teeth test are shown in the Fig.(1b), (1c).

**Construction of SBS test specimens mold:**
Standard flasking method for flexible denture fabrication was followed Using aluminium flask (IRIS, china). The specimens were coated with separating medium (sinalident, china) and dried. Then type III dental stone (Syna Rock, Bulgaria), mixed according to supplier's guidance 100gm/20ml. The specimens were placed in the slurry, half submerged, half uncovered, and let to set fully.. Special sprues were selected (6-8mm) in diameter and adhered to the wax pattern specimens which permits material to be injected(13). After that the upper metal flask was screwed onto the lower metal flask and filled with slurry stone. Vibratory dental device (Quayle Dental, England) was utilized to eradicate air bubbles during stone pouring. Prior to wax removal and flask disclosing, they left wholly to set. The metal flasks were placed in a boiling water bath (XMTE-205, Germany) for 15 minutes to eliminate wax (17) then the mold cavity was gently cleaned. Before injection, the specimens were categorized into 6 categories according to modification in their (n=20 per group)

**Surface Treatment of The Acrylic Teeth Ridge Lap:**

- **Control group:** without any surface treatment.
- **T. Holes group:** To ensure proper material flow, daitoric perforations must be T-shaped. using a (0.06) inch twist drill on the ridge lap surface(16). Fig.(2a).
- **Alumina-blasting group:** Surfaces of teeth's ridge laps were roughened up using aluminum oxide particles (250µm) for 30 seconds using a sandblasting machine at 2.5 bar pressure(5). A custom-made fixture was designed to uniformly space the teeth's ridge lap surface from the sandblasting machine's nozzle (10 mm)(17) Fig.(2b). Following by a clean-up of loose debris with an air-water spray for 10 seconds, then drying with compressed air about 3 seconds.
- **Silano (SCA) group:** was applied as a thin single coating with a paintbrush on the ridge lap surface of acrylic teeth and allowed about 20 minutes to enable acetone to evaporate before injection (18) Fig.(2c).
- **T. Hole + silano group:** Teeth treated with preparation T. Shape daitoric and Silano(SCA).
- **Alumina-blasting + silano group:** Teeth treated with aluminum- oxide abrasion and Silano (SCA).

**Injection process**
Injection is carried out with the AX-YD manual Dental injection system machine (ISO 9001, CN) as in fig.(3). The procedure starts by heating the cylinder in an electrically driven furnace until it reaches the desired temperature of (288°C). As per the manufacturer's instructions then the thermoplastic resin cartridge (Karadent by TCS, USA) was then positioned in the heated cylinder, crimped top first (labelled part facing out). Set a timer for 15-16 minutes (per manufacturer's instructions). After the resin has melted, gently withdraw the cylinder from the furnace, maintaining it horizontal until it reaches the sprue hole of
the flask, then verticalize it. Turn handles with both hands once piston is in cylinder. When the cartridge explode, keep rotating the handle with both hands until the springs are gently compressed. Let injection to compress for 3 minutes before removing (14).

**The Specimens’ Finishing and Polishing**

Sprues of specimens have been cut using a cutoff disc under mild pressure and water cooling to avoid overheating, and the samples’ borders were cut with a grinding stone bur spun at (1500 rpm) (17). The samples were then sanded using (220) grain sandpaper. Polishing was done in lathe polishing machine (SD-La54, China) by using bristle brush and rouge wheel with pumice. Digital Vernier (Vaster, China) was used to check the specimen’s dimension (15).

**Storage of Specimens**

Specimens per category were placed in a plastic container as a group and filled with distilled water. They were incubated for (48 hrs.) at (37°C). (ADA 1999 Specification No.12 in 1999). Afterwards, half of the samples were submitted to shearing tests utilizing a universal testing equipment (INSTRON 1195, USA). The second half of the samples were aged using an Accelerated Weathering Tester (Model QUV/Spray, Q-Lab Corps, USA) for about (100 hrs.) of alternated periods of ultraviolet radiation (UVA-340), elevated temperature, and darkness with condensation of filtered water. Each aging cycle, according to ASTM G154/cycle 7 (2006), was accomplished in (12 hrs.) (19,20).

**Shear Bond strength (SBS)test**

In an attempt to best simulate the clinical forces on the maxillary central incisor, loading directed at 45 degrees from the longitudinal axis of every denture tooth by a wedge-shaped end metal stud at a cross head speed of 0.5mm/min until fracture using a universal testing machine (Instron 1195, USA, 50 kg full scale) (13, 15, 21) as in Fig.(4). The load at failure was recorded in (kg) multiplication to 9.81 to obtain the bond strength in (N) in accordance with the Japanese Standard for acrylic teeth (JIST 6506, 1989) (22) (24).

**Fracture mode Analysis**

All tested specimens were checked by eye to specify the fracture type occurred. As shown in fig.(5) the joint failure was categorized as adhesive type, cohesive type, or a mix of both types. (17)

**Adhesive:** when the bonding interface fractures (No residues of denture base material could be seen on the tooth’s surface after the fracture.).

**Cohesive:** When the denture base or tooth was fractured (any residue of denture base resin on the tooth surface or any fragments of tooth remaining on the denture base).

**Mixed:** When the fracture included both adhesive and cohesive.

**Scanning Electron Microscope (SEM) Evaluation:**

Scanning Electron Microscope (SEM) was performed on one sample from each group at random to test the sample’s topography. The Scanning Electron Microscope (SEM) photomicrographs was done by Electro scanning microscope (Inspect S50, FEI company, Netherlands, 2013), under 3mm and 250µm magnification with working distance of 10 mm and an acceleration voltage of 20 KeV for studied groups. As shown in fig.(6)

**Statistical Analysis**

This study used Analysis of variance (ANOVA) test to identify the differences among different groups before and after ageing process and to analyze treatment groups and control group all together. After that, the Tukey Honestly Significant Differences (HSD) multiple comparison test was used to make the post hoc comparisons and identify significant differences at $\alpha =0.05$.

**Results:**

One-way ANOVA findings revealed extremely significant variation between groups prior ($F = 400.091, P< 0.01$) and post ($F = 431.929, P < 0.01$) of artificial ageing as shown in Table (1).

Table (2) illustrated the mean readings in (N) and standard deviations (SD) of
binding strength, and their significance as shown in fig.(7). The results reveal the influence of ageing on all groups was very clear after (100hrs.), which there were a noticeable drop in all groups investigated at (P<0.05) except the group treated with “T. Shaped hole+silano” show non-significant variations at (P= 0.112).

The bond strength readings were greater in all groups that had been treated with a different surface treatment than control group at (p< 0.01). Among all test groups, both aged and non-aged, when treatment type was considered, there were substantial differences between all groups for each type of treatments. The findings indicated that combining T. Hole + silano (silane coupling agent) produced substantially higher shear bond strength than T. Hole alone, and alumina-blasting+ silano (silane coupling agent) produced much higher shear bonding than alumina-blasting alone. all groups had considerably decreased shear bond strength levels after ageing at (P < 0.01). The control group showed the greatest percentage decrease in Shear strength (25.80%), whereas the (T. Hole + silano) group gave the lowest percentage decrease in shear strength in just (6.13%), as shown in fig. (8). Scanning Electron Microscope (SEM) images of fractured surfaces were showed in Fig. (6), the control group’s surfaces appeared smooth with few scratches as in Fig. (6a), the surface was pitted and uneven as a result of the alumina blasting group show in Fig. (6b), silano group treatment produced a smoother surface than alumina blasting, with shallow troughs and hollows as in Fig. (6c), Alumina-blasting + silano produced a variety of surface topography and a microporous surface appearance shows in Fig. (6d). For control specimens with brittle fracture characteristics in Fig (6e). While in the alumina-blasted, silano and alumina-blasted + silano groups, exhibiting ductile features, as in Fig. (6f), (6g) and (6h).

Visual inspection of the characteristics of failure revealed two distinct forms of failures: adhesive and mixed failures in all tested groups as depicted in Fig. (5), while cohesive failure was absent in all study groups. The control and alumina-blasting groups failed primarily adhesively, whereas the T. Hole, Silano, and all other chemically treated groups failed in a mixed manner.

**Discussion:**
SBS refers to bonding property between two substances and the amount of load they can withstand before fracturing or separating (18). It is essential for evaluating two-material interfaces (23). Because the bonding strength of artificial teeth might be affected by the foundation material used in dentures, Karadent thermoplastic resin ((by TCS, INC, USA) was employed in this study (6). Artificial aging was used to replicate the intraoral environment that will subject the prosthetic to fluctuating conditions of temperature and stressful situations within patient using time (3). Our method for preparing specimens and determining bond strength was based on JIST 6506 (1989), that considered the most current testing method and requires one maxillary or mandibular anterior tooth, and debonding occurring more commonly in anterior maxillary teeth(22) (24). Based on the findings, the null hypothesis that varied ridge lap surface modifications had no influence on the shear bond strength between thermoplastic denture base resin and acrylic teeth was rejected.

**I-Mechanical treatment**
According to the findings of this research, it showed a statistically highly important variation between T. hole group and control group. This is attributed highly efficient mechanically designed interlocking connection of the artificial acrylic teeth with the thermo-plastic resin material used in injection molding process. In order to properly bind prosthetic teeth, the hole diameter must be big. A too-small opening may weaken the binding strength of the prosthetic teeth, causing ridge lap tension. However, due to the high viscosity of karadent thermoplastic resin, the manufacturer recommends drilling bigger holes. This result was aligned with that of (Tashiro, S., et al.,2020). They observed that T. shape designed holes can
be a good mechanical jointing design for creating the high binding strength value of acrylic artificial teeth to thermo-plastic denture material during injection (8).

In this investigation, the alumina-blasting group sandblasted with (250μm Al2O3 particle size) showed importantly more strength full binding than the specimens without surface treatment. This elevation in this strength might be referenced to the abrasive influence created by blasting particles of aluminum oxide by pummeling the surface which increase over all surface energy and roughens it by generating undercuts and dissimilarities, as a result micromechanical retaining pits creation., and ultimately improving bond strength. This group finding confirms with (Krishna, V.P., et al., 2014). They discovered that sandblasting the teeth’s ridge lap elevated the shear binding strength with the denture resin material(9).

II-Chemical treatment
Surface modification using silane coupling agent adhesive enhanced acrylic tooth-denture bonding strength (5). A thin single coating of silano that applied to the ridge lap area using a micro brush that enhance covalent bonding between the denture base and teeth (18,24). Silane coupling agent characteristics increase the wettability of restorative materials, increasing mechanical, physical, and chemical adhesion between two distinct materials, causing this group to demonstrate a substantial difference compared to the control group. Objecting to (Melo, M.A. 2011). It shown that those utilizing silane had similar results to those not using silane (20).

III-Combination treatment (chemo-mechanical)
Shear bond strength was greatly improved by the (chemo-mechanical) treatment when compared to the control and other individual treatments, the groups treated with T. holes + silano exhibited significantly high binding force within the prosthetic acrylic teeth and denture base material. When compared to the groups treated with T. holes alone. The results are consistent with those of (Meng GK, et al., 2010), who indicated that employing diatomic recess with adhesives prior to injection elevates the binding strength between acrylic artificial teeth and denture material (25).

The results show alumina blasting + silano group had high a significant difference in bond strength than alumina blasting group alone, this improved in shear bond strength might be attributable to the micro-retenve areas created by aluminum particles, which increased the total adhesion area. These micro-retenve areas were filled by adhesives that functioned as a bridge between the teeth ridge lap area and the denture base material, leading to an extra elevation in the shear binding strength. The results from this treated group support the findings of (AlZaher, Zahra A., et al., 2020). They discover that chemo-mechanical treatment of the denture base and teeth by the application of alumina blasting and a silane adhesive enhances the tooth-denture base shear attachment (18).

IV-Evaluating the impact of accelerated aging on SBS of all groups
The results shown the influence of aging on all groups was extremely clear after (100 hrs.) of artificial ageing. All study groups show a reduction in SBS except “T. Hole +silano” group was non-significantly affected. This reduction in shear bond strength post (100 hrs.) of simulated ageing process can be ascribed to degradative influence on the interface within the binding different materials, that leads to loss in bond strength due to recurrent expansions and contractions produced by temperature changes (26). Both the T. Hole and Alumina-blasting mechanical treatment groups showed identical percentage reductions of (12.36% and 13.24 %), respectively. Water molecules can concentrate in the site of binding and leak into the openings in the binding site, this results in increased tension growing at the binding site due to sample expansion (13). Our results contradicted the findings of (Pande N, et al., 2018) researchers who found that temperature cycling had no influence on shear bond strength between teeth and denture bases (26). The combined surface
treatments resulted in the lowest percentage reductions in SBS, with “T. Hole + Silano” resulting in just a (6.13 %) drop in SBS. Unlike (AlZaher et al., 2020), they discover that heat cycling reduces shear strength of bonding in total groups specifically those presenting both chemical and mechanical modifications in their surfaces (18).

**Conclusion:**

• Within the constraints of this investigation, it was discovered that modifying the ridge lap surface enhanced bond strength when compared to the unmodified surface.

• The influence of artificial ageing process was clear on all studied groups except “T. Shaped hole + silano” group not affected by ageing process.

• The greatest best results for improvement of shear strength of bonding were obtained in total groups specifically those presenting both chemical and mechanical modifications “T. shaped hole + silano” in their surfaces it had the highest shear bond strength value of between all groups treated either before or after 100 hours of artificial ageing.

![Fig.(1): Testing Shear Bond Strength](image)
Fig. (2): surface treatments.
(A): T. Hole perforations,
(B): abrading teeth ridge lap of with AL₂O₃ (250 µm),
(C): applying silane (SCA)

Fig. (3): AX-YD manual Dental injection system machine
Fig. (4): Testing shear bond strength

Fig. (5): mode of failure (a) control group (b) T. Hole group (c) alumina-blasting group (d) silano group (e) T. Hole+ silano group (f) alumina-blasting + silano group.
Fig. (6): SEM images of fractured surfaces of the studied groups

Fig. (7): cluster bar chart shows the means of SBS before and after 100 hrs. of artificial ageing
Fig. (8): percentage reduction in SBS after accelerated artificial ageing

Table (1): Levene’s and one-way ANOVA tests

<table>
<thead>
<tr>
<th>Test's Situation</th>
<th>Levene Statistic</th>
<th>Sig. (*)</th>
<th>F-test</th>
<th>Sig. (*)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ageing Groups Before</td>
<td>4.975</td>
<td>0.000</td>
<td>400.091</td>
<td>0.000</td>
</tr>
<tr>
<td></td>
<td>(HS)</td>
<td></td>
<td>(HS)</td>
<td></td>
</tr>
<tr>
<td>Groups After 100hrs of ageing</td>
<td>5.586</td>
<td>0.000</td>
<td>431.929</td>
<td>0.000</td>
</tr>
<tr>
<td></td>
<td>(HS)</td>
<td></td>
<td>(HS)</td>
<td></td>
</tr>
</tbody>
</table>

(*) HS: Highly sig. at P<0.05
Table (2): mean shear bond strength ±SD (N) and significance of specimens with different surface treatments at 0 and 100 hrs of ageing

<table>
<thead>
<tr>
<th>Treatment groups</th>
<th>Before aging (0 hours) n=10</th>
<th>After aging (100 hours) n=10</th>
<th>Mean differences</th>
<th>p-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>80.63±12.76 aE</td>
<td>59.83±11.66 bE</td>
<td>25.80%</td>
<td>0.001 (HS)</td>
</tr>
<tr>
<td>T. Hole shape</td>
<td>237.79±10.37 aC</td>
<td>206.4±10.49 bC</td>
<td>13.20%</td>
<td>0.0001 (HS)</td>
</tr>
<tr>
<td>Aluminablasting</td>
<td>140.56±12.9 aD</td>
<td>119.94±10.18 bD</td>
<td>14.66%</td>
<td>0.001 (HS)</td>
</tr>
<tr>
<td>Silano</td>
<td>169.9±12.68 a</td>
<td>148.05±11.82 b</td>
<td>12.86%</td>
<td>0.001 (HS)</td>
</tr>
<tr>
<td>T. Hole +Silano</td>
<td>388.77±32.15 aA</td>
<td>364.93±31.67 aA</td>
<td>6.13%</td>
<td>0.112 (NS)</td>
</tr>
<tr>
<td>Sandblast +Silano</td>
<td>265.55±10.37 aB</td>
<td>244.65±11.38 bB</td>
<td>7.87%</td>
<td>0.0001 (HS)</td>
</tr>
</tbody>
</table>

Different small letters significant differences between two groups (before and after) at p-value <0.05. ns: non-significant. Horizontally identical superscripted different capital letters significant differences among treatment groups in each column at p-value <0.05. ANOVA and Tukey’s test.

References

11. Lang, R., et al., Bond of acrylic teeth to different denture base resins after various surface-conditioning methods. Clinical


16. Der Poel, V. and N. Olive, Mechanical retention of acrylic teeth onto a pure nylon base. 2014, Cape Peninsula University of Technology.


