# The Influence of Silica Dioxide and Aluminum Oxide Nano Fillers Reinforced Heat Cured Acrylic Denture Base Material and Thermocycling on Tensile and Shear Bond to Denture Soft Lining Material

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## Abstract

**Background:** Soft lining materials play an important role in modern prosthodontics treatment because of their capability to restore the inflamed and distorted mucosa. The purpose of the research was to estimate the influence of acrylic denture base reinforcement with silanted nano fillers ($Al_2O_3$ and Sio2) separately on tensile and shear bond strength of soft lining material and studying effect of thermo cycling on bonding strength.

**Materials and methods:** Total 120 specimens were prepared; it divided into 60 Specimens for shear bond strength test and 6o specimens for tensile bond strength test. Specimens were sub grouped into 30 Specimens without thermo cycling and 30 specimens with thermo cycling. Each sub group is consisted from: 10 Specimens control, 10 specimens were reinforced with 2 wt% of $Al_2O_3$ nano fillers and 10 specimens were reinforced with 5 wt% of $SiO_2$ nano fillers. Samples were processed depending on test applied. Soft lining material was applied for each testing group. Samples were immersed in distal water for 24 hours at 37°C before testing. For thermo cycling test, specimen were thermo cycled in thermo cycling device. Bonding strength test was done using INSTRON universal testing machine.

**Results:** reinforcement of acrylic denture base with nano-fillers was significantly increase both tensile and shear bonding of lining material and thermo cycling decreases both bonding strength.

**Conclusion:** reinforcement of acrylic denture base with nano fillers could improve bonding strength of lining material, while thermo cycling had a deleterious effect on bonding strength.

**Key words: nano-fillers, soft lining, bonding strength.**

## Introduction

The increased demand for removable prosthesis to treat the partial and complete edenutulism cases as a result of the rapid growth of population age all around the world, and an important goal of prosthodontic treatment is to provide a comfortable conventional acrylic removable denture [(1)]. Since the gradual oral tissues changes, allows certain parts of the alveolar ridge to be sensitive to the functional pressure applied during normal functional activities [(2)] ; Therefore the use of soft lining material for relining of a removable prosthesis will provide an absorbing layer on the part of denture that being in contact with the tissue resulting in decreased traumatic occlusal forces transmission [(3)], because of the viscoelastic properties of denture soft lining material which redistribute and reduce the functional loads over the denture bearing area [(4)], these beneficial properties of soft liner material make it useful for treating patients with residual ridge resorption or a trophy, therefore, wearing a prosthesis relined with soft lining materials resulting a more comfortable for the patient[(3)].

The denture resilient lining materials could be divided basically in two types: the plasticized acrylic resin based and silicone based resilient lining materials[(6)], and according to polymerization techniques, these materials could further divided into heat polymerization and room temperature polymerization denture lining materials [(3)].

The resilient denture liners have many problems related to their clinical implication as the material hardening and loss softness, Candida albicans growth and colorization and low tear strength, but one of the most serious problem is the failure of soft denture liner adhesion to acrylic denture base material, this failure will create a potential surfaces for plaque accumulation, calculus formation and bacterial growth[(7)], therefore it became imperative that bond strength of the soft lining material and denture base being optimized using different mechanical surface treatments as denture base roughening using sand blasting [(5)], laser [(6)], or plasma treatment [(7)].

Also it has been found that the clinical etching of the denture base material as using monomethyl methacrylate, methyl chlorides or acetones, could be responsible for the increase in the bond strength between soft liner and denture base material [(9)].

Now day, there will be a great attention directed to the reinforcement of acrylic denture base material as using glass fibers which improve the mechanical properties [(10)], and improve the bonding strength to soft linear [(11)], and the reinforcement using nano fillers as aluminum oxide and silicone dioxide which improve the mechanical properties of polymethyl methacrylate denture base material [(12- 14)].

Hence, this study was designated to compare and evaluate the addition of silaneted $Al_2O_3$and $SiO_2$ nanofiller separately to denture base acrylic resin on tensile and shear bonding strength of soft liner.

the most critical effect is temperature changes during chewing and drinking of hot and cold food and fluids, therefore the study was also evaluate the effect of thermocycling on both tensile and shear bond strength of soft liner to denture base resin.

## Materials and methods

A heat polymerized acrylic soft liner (vertex –soft /Netherlands), a heat cured poly methylmethacrylate acryclic resin (superacyl .Sopofa dental, Czech), and aluminum oxide ($AL_2$ $O_3$) and Silicon dioxide ($SiO_2$) Nano fillers (Sigma – Aldrich/ Germany) were used to perform this study.

### Sampling:

Total 120 specimens were prepared; it divided into 60 Specimens for shear bond strength test and 6o specimens for tensile bond strength test.

For each test, the specimens were sub grouped into 30 Specimens without thermo cycling and 30 specimens with thermo cycling.

Each sub group is consisted from:
10 Specimens which considered as control without any reinforcement for acrylic resin denture base.
10 specimens were the acrylic resin is reinforced with 2 wt% of $Al_2O_3$ nano fillers.
10 specimens were the acrylic resin is reinforced with 5 wt% of $SiO_2$ nano fillers.
Specimens design:

**Shear bond strength test:**

For the evaluation of soft lining material bonding to nano reinforced acrylic resin material, an acrylic blocks with the dimensions of 75mm x 25mm x 5mm, length, and width and depth respectively with a stopper of 3mm depth [24].

Two acrylic blocks were put over each other to produce one specimen with a space of 25mm x 25mm x 3 mm length, width and depth respectively between blocks.

**Tensile bone strength test:**

A rectangular acrylic resin blocks of dimension 8mm x 10 mm x 30mm as width, height and length respectively were prepared[8] ,each two blocks were arranged in such away that a 3mm space in between blocks was created for soft lining material placing.

**Mould preparation:**

In order to obtain the acrylic blocks for both shear and tensile bond strength tests, a plastic patterns were constructed in accordance with the dimension of each test and invested in a flexible silicon duplicating material (Addition Silicon, putty consistency Zhermach- Italy) to produced a silicon moulds, which were invested in dental stone in flask for production of silicone-stone mould for standardized production of specimens.

**Acrylic resin proportioning and mixing:**

The acrylic resin was proportioned and mixed in accordance with manufacturer instructions; 2.2g power/ 1ml monomer in a clean and dry glass container till reaching the dough stage, then packed into mould. After flask closure, and compressed in hydraulic press, this then transferred to water bath for curing at 70 $C^o$ for 30 minutes and then boiled at 100 $C^o$ for 30 minutes. Finishing of the resin blocks was done and polishing all surfaces except the surface was the soft liner is being placed. The finished acrylic specimens were conditioned and stored in distilled water at 37$C^o$ for 48 hour according to ADA Specification No. 12, (1999).

**Nano fillers addition**

The addition of both salinated $AL_2O_3$and $Sio_3$nanofillers were done by weight to the experimental groups.

The powder of both nano $AL_2O_3$and $Sio_2$ were weighted using electronic balance( Sartorius BP 30155, Germany) with(0,0001 g) accuracy.

A 2wt% of $AL_2O_3$ and 5wt% of $Sio_2$ were added to acrylic resin monomer separately and to achieve the proper dispersion of Nano particles in monomer the sonicating apparatus (Soniprop 150 England) was used for 3 minutes in order to reduce tendency of nano particles aggregation, then mixed immediately with polymethayl methacrylate powder, and packing and curing was done as for control acrylic specimen production.

**Soft liner mixing and Applications**

**Shear bond strength:**

The acrylic blocks for each Specimen were placed over each other and being invested in Silicon duplicating material to products the silicon mold; which then invited in dental stone that filled the lower part of custom flask designed for soft liner application and curing (figure 1).

### Tensile bond strength test:

Two acrylic blocks were invested is silicon duplicating material with 3mm space in between. Thin space is required for soft liner application. This silicon mold was then invested in dental stone is custom flask fabricated for soft liner application and curing (figure 2).

For both shear and tensile bond strength, the soft lining material was proportioned in accordance to manufacturer instruction (p/l ratio: 1,2g/1ml) and mixed in a dry and clean glass container till reaching the dough stage which then applied in custom flasks with gradual application in order to achieve the even flow of the lining material and after flask closure , the curing process was done using thermostatically controlled water bath by heating up to 70C$^{o}$ for 90 minute and then boiled up to 100C$^{o}$ for 30 minutes. The specimen were finished by cutting the excess materials with sharp blade and then stored in distilled water at 37C$^{o}$ for 24 hours before testing.

### Thermo cycling test:

The specimen were thermo cycled in thermo cycling device by subjecting the specimen to 60 seconds cycle for three days at temperature range from 5C$^{o}$-55C$^{o}$ using special device **(Haackt, Germany)**.

Shear and tensile bond strength testing

All the specimens were subject to a shear load with across head speed of 0.5 mm/min using load cell capacity of 100kg using universal testing machine **(Instron Corporation, canton mass)**. The maximum load required for the failure was recorded to calculate the value of bond strength of each specimen according to ASTM Speech chain D-638mm 1986 formula:

Bond strength (

$$N/mm^2 = (\text{Maximum } load)/(\text{Cross sectional area}) = F/A)$$

The collected data were statistically analyzed.

## Results

The statistical analysis of the results gained, indicated that the addition of silanted $Al_2O_3$and $SiO_2$ nanofillers separately to acrylic denture base causes an improvement in both tensile and shears bonding strength of the soft lining material to denture base material (table 1).

### Effect of Silanted Al2O3 Nano filler on bonding strength:

addition of silanted $Al_2O_3$nanofillers to the acrylic denture base increases the bonding strength of soft lining material to the denture base, as cleared in table 1, and the tensile bond strength was increased more than shear bond strength, as shown in figure 3 and 4. The analysis of variance ANOVA, indicated a highly significant difference of both tensile and shear bond strength as compared to control group (table 2), and there was a highly significant difference in tensile bonding strength as compared to shear bonding strength of soft lining material to denture base (table 3).

### Effect of silanted SiO2 Nano fillers on bonding strength:

the silanted $SiO_2$ nanofillers had an improving effect on both bonding forces of soft lining materials to denture base material, and as shown in table and figure 1 and2, the silanted $SiO_2$nanofillers increase the tensile bonding strength of soft lining material to acrylic denture base more than the shear bonding strength and there was a highly significant difference of both bonding strength as compared to control group (table 2) also there was a highly significant difference of tensile bonding strength as compared to shear bonding strength as indicated in table 2.

### Comparison of effect of silanted Al2O3 and SiO2 nano fillers on bonding strength of soft lining material.

There was a highly significant difference in bonding strength between the $Al_2O_3$nano fillers group as compared to $SiO_2$ nano fillers as illustrated in table 2 and 3

### Effect of thermo cycling on bonding strength

the thermo cycling having deleterious effect on tensile bond strength, for all experimental group, although the addition of silanted $Al_2O_3$and $SiO_2$nano fillers would not decrease the bonding strength below the control group, the shear bond strength was decreased more than the tensile bonding strength of both $Al_2O_3$ $SiO_2$nano fillers as compared to control group (table 4) and there was a highly significant difference between silanted $Al_2O_3$and $SiO_2$nano fillers on both tensile and Shear bonding strength after thermo cycling (table 3).

## Discussion

In prosthodontics treatment, the use of denture soft lining material gain an important interest for both patients and prosthodontist in spite of the problem related to bonding failure between denture base acrylic resin and the soft lining material.[1]

The improvement in acrylic resin mechanical properties after incorporation of nano particles as $Al_2O_3$and $SiO_2$and the reinforcement of polymer used in prosthodontics with metal composite system having a prime importance and interest.

### Effect of silanted Al2O3 nano fillers bonding strength

The increase in bonding strength of soft lining material to silanated $Al_2O_3$nano filler reinforced denture base material (table 1), may be attributed to the strong inter atomic ionic bonding which be related to the most stable phase that is alpha hexagonal phase [15] which had a high dialectical properties resulting in cross linking formation and high bonding forces between the nano fillers and the resin material which limit the polymer molecules mobility forming a dense polymer matrix composite [16, 17], in addition, the presence of silane coupling agent which is based on thiophosphoric acid methacrylate which had a similar chemical structure with acrylic resin, allowing a greater bonding between the nano fillers and resin matrix producing a more dense composite, and reducing the amount of water reaching the inner layers of resin matrix [18, 19],and since the water absorption had a direct damaging effects on the bonding strength by water peculation directly into the bonding site leading to swelling and consequently a stress buildup at the interface leading to decrease in bonding strength, in addition, the leaching out of plasticizer and other soluble impurities into water which leaving a empty spaces and gaps that resulted in reduction of cushioning effect and leading to transmission of internal loads to the bonding interface which decrease the bonding strength [20, 21,28].

**Effect of Salinated Nano SiO2 fillers**

The result indicated an improvement in bonding strength of soft lining material to salinated nano silica composite denture base material (tab 1). This increased bonding strength could be explained on the bases, that the polymerization reaction of methylmethacrylate in the presence of modified nano SiO2 particle with epoxy silane characterized by un saturated end groups which can provide a helpful method of good dispersion of nano particles within the composite[22] by nano particles leading to no pullout phenomena are clear and absence of voids resulting in a strong polymer phase[23] that characterized by strong interfacial shear strength between the resin materials the nano fillers leading to develop a dense matrix composite that will increase the bonding strength to soft lining material by preventing water peculation directly to the interface and preventing leaching out of plasticizer of the lining material [20,21] , with the reduction of overall volume of water absorbing properties of the results composite polymer [18] and since the water could directly leading to swelling of lining materials and buildup of stresses at the bonding interface which reducing the bond strength [24].

**Effect of thermocycling on bonding strength**

Although the thermocycling decrease the bonding strength for all tested groups tab (), the Nano filler reinforced acrylic resin showing a high bonding strength than the control groups.

the decrease in bonding strength could be explained on the bases that the huge amount of water that is absorbed during the thermocycling process leading to swelling and concentration of stresses at the interface between the lining and denture base material or could related to the viscoelastic properties changes of the lining material [25], the nano fillers reinforced denture base exhibit a dense matrix with a little water absorbing abilities [17], these explain the significant difference with control groups, and the decrease in bonding strength is due to huge amount of water absorbed by the hydrophilic acrylic relining material which lead to internal damage of the polymer by growth of water droplets and leading to irreversible break down of polymer matrix and formation of cracks by continual growth of water droplets [26].

**Comparison between tensile and shear bonding strength**

The tensile bond strength test was used since it gives the required information on strength of bonding and its considered to be an important guide to the quality of rubber [27] and its useful in testing different adhesives and processing procedure [29], while the shear bond strength is more nearly represent the force that the soft lining material is receiving during clinical functions [30].

In the present study, for all tested groups the tensile bond is higher than the shear bonding strength and these could be indicated that the adhesive interfaces were less resistant to shear loading than to tensile loading [25].

## Conclusion

Within the limitation, the conclusions are:

1. The reinforcement of acrylic denture base material with salinated Al2O3 and SiO2 nano fillers separately could improve the bonding strength of soft lining material to denture base.

2. The tensile bonding strength increased more than the shear bonding strength.
3. The thermo cycling had a deleterious effect on bonding strength of lining materials to denture base materials.

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Table 1: Mean shear and tensile bond strength among different states and groups

| Test | State | Groups | N | Mean | S.D. | Min. | Max. |
|---|---|---|---|---|---|---|---|
| Shear bond strength (N/mm$^2$) | Before thermocycling | Control | 10 | 0.709 | 0.007 | 0.697 | 0.718 |
| | | $Al_2O_3$ | 10 | 0.942 | 0.016 | 0.921 | 0.961 |
| | | $SiO_2$ Nanofillers | 10 | 0.777 | 0.011 | 0.763 | 0.790 |
| | After thermocycling | Control | 10 | 0.402 | 0.009 | 0.384 | 0.412 |
| | | $Al_2O_3$ | 10 | 0.603 | 0.010 | 0.582 | 0.612 |
| | | $SiO_2$ Nanofillers | 10 | 0.590 | 0.010 | 0.574 | 0.603 |
| Tensile bond strength (N/mm$^2$) | Before thermocycling | Control | 10 | 1.124 | 0.021 | 1.087 | 1.150 |
| | | $Al_2O_3$ | 10 | 1.214 | 0.027 | 1.150 | 1.237 |
| | | $SiO_2$ Nanofillers | 10 | 1.176 | 0.011 | 1.162 | 1.187 |
| | After thermocycling | Control | 10 | 0.818 | 0.024 | 0.787 | 0.850 |
| | | $Al_2O_3$ | 10 | 0.940 | 0.016 | 0.912 | 0.962 |
| | | $SiO_2$ Nanofillers | 10 | 0.846 | 0.018 | 0.812 | 0.862 |

Table 2: ANOVA test of tensile and shear bonding of soft lining to different nano reinforced acrylic denture base material and at different states

| Test | State | ANOVA | Sum of Squares | d.f. | Mean Square | F-test | p-value |
|---|---|---|---|---|---|---|---|
| Shear bond strength ($N/mm^2$) | Before thermocycling | Between Groups | 0.287 | 2 | 0.144 | 965.938 | 0.000 (HS) |
| | | Within Groups | 0.004 | 27 | 0.000 | | |
| | | Total | 0.291 | 29 | | | |
| | After thermocycling | Between Groups | 0.254 | 2 | 0.127 | 1348.116 | 0.000 (HS) |
| | | Within Groups | 0.003 | 27 | 0.000 | | |
| | | Total | 0.256 | 29 | | | |
| Tensile bond strength ($N/mm^2$) | Before thermocycling | Between Groups | 0.041 | 2 | 0.020 | 48.735 | 0.000 (HS) |
| | | Within Groups | 0.011 | 27 | 0.000 | | |
| | | Total | 0.052 | 29 | | | |
| | After thermocycling | Between Groups | 0.081 | 2 | 0.040 | 105.358 | 0.000 (HS) |
| | | Within Groups | 0.010 | 27 | 0.000 | | |
| | | Total | 0.091 | 29 | | | |

Table 3: Multiple comparisons LSD significant difference test of tensile and shear bonding of lining material to different nano reinforced acrylic denture base at different state

| State | Groups | | Shear bond strength | | Tensile bond strength | |
|---|---|---|---|---|---|---|
| | | | Mean Difference | p-value | Mean Difference | p-value |
| Before thermocycling | Control | $Al_2O_3$ | -0.233 | 0.000 (HS) | -0.090 | 0.000 (HS) |
| | | $SiO_2$ Nanofillers | -0.068 | 0.000 (HS) | -0.052 | 0.000 (HS) |
| | $Al_2O_3$ | $SiO_2$ Nanofillers | 0.165 | 0.000 (HS) | 0.038 | 0.000 (HS) |
| After thermocycling | Control | $Al_2O_3$ | -0.201 | 0.000 (HS) | -0.121 | 0.000 (HS) |
| | | $SiO_2$ Nanofillers | -0.188 | 0.000 (HS) | -0.028 | 0.004 (HS) |
| | $Al_2O_3$ | $SiO_2$ Nanofillers | 0.013 | 0.006 (HS) | 0.094 | 0.000 (HS) |

Table 4: Descriptive statistics and effect of thermocycling on tensile and shear bonding strength in different groups

| Test | Groups | N | Before thermocycling | | After thermocycling | | Comparison | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Mean | S.D. | Mean | S.D. | Mean Difference | t-test | d.f. | p-value |
| SBS | Control | 10 | 0.709 | 0.007 | 0.402 | 0.009 | 0.307 | 85.581 | 18 | 0.000 (HS) |
| | $Al_2O_3$ | 10 | 0.942 | 0.016 | 0.603 | 0.010 | 0.339 | 55.727 | 18 | 0.000 (HS) |
| | $SiO_2$ Nanofillers | 10 | 0.777 | 0.011 | 0.590 | 0.010 | 0.187 | 38.992 | 18 | 0.000 (HS) |
| TBS | Control | 10 | 1.124 | 0.021 | 0.818 | 0.024 | 0.305 | 30.434 | 18 | 0.000 (HS) |
| | $Al_2O_3$ | 10 | 1.214 | 0.027 | 0.940 | 0.016 | 0.274 | 27.767 | 18 | 0.000 (HS) |
| | $SiO_2$ Nanofillers | 10 | 1.176 | 0.011 | 0.846 | 0.018 | 0.330 | 50.196 | 18 | 0.000 (HS) |

Figure 1: Testing shear bond strength spacemen

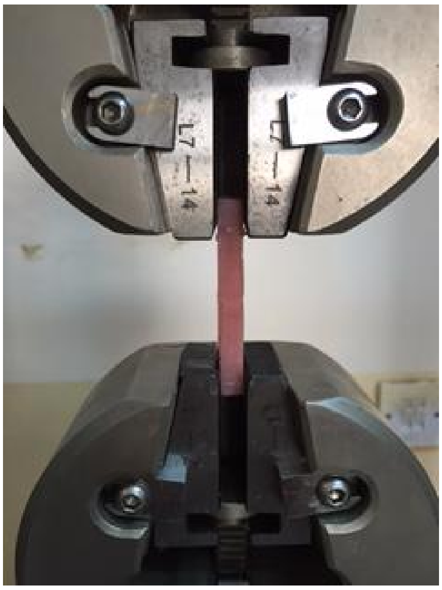


Figure 2: Testing tensile bond strength spacemen

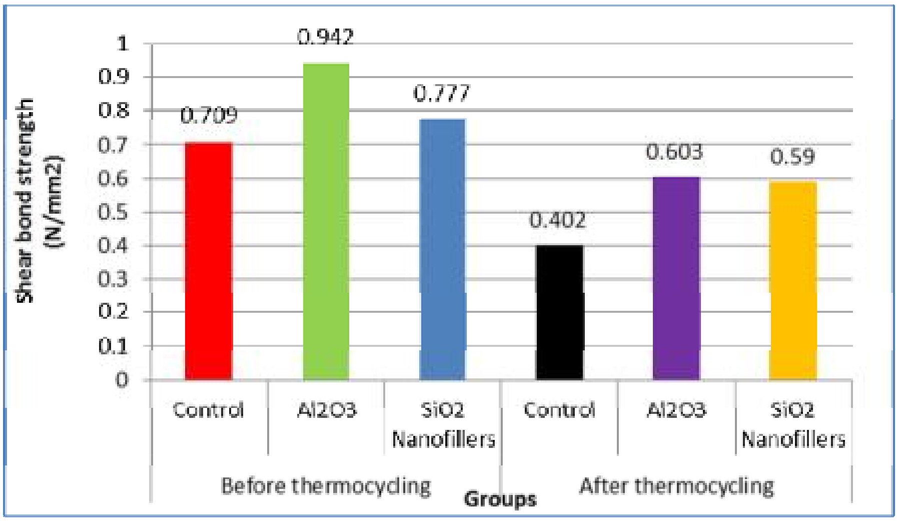


Figure 3: shear bond strength of lining material to Al2O3 and SiO2 nano filler enforcing acrylic denture base material.

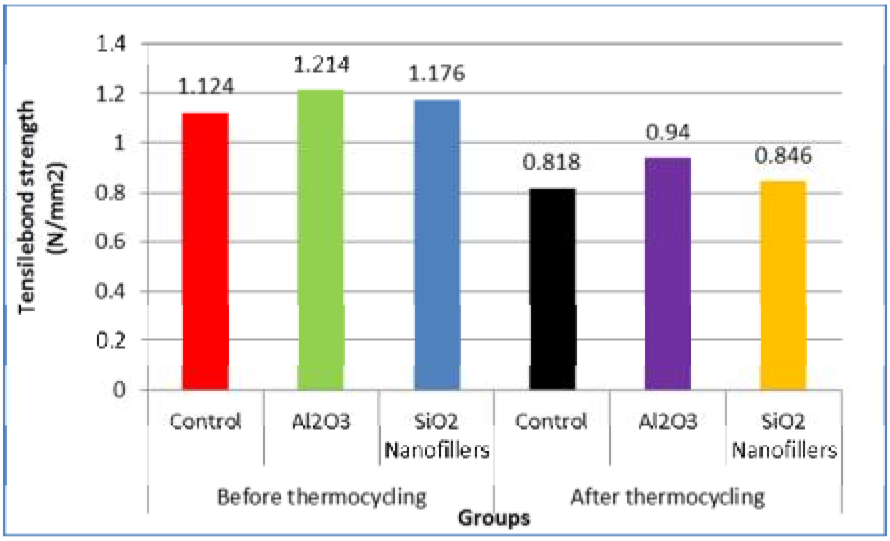


Figure 4: tensile bond strength of lining material to Al2O3 and SiO2 nano filler enforcing acrylic denture base material.