Effect of Silanized Nano-alumina Reinforcement on Flexural Strength of Auto-polymerized Acrylic Resins

Uraivichaikul Duangjai[1], Boonsiri Issarawan[1], Wattanasirmkit Kamolporn[2]

[1]Department of Prosthodontics, College of Dental Medicine, Rangsit University, Pathum Thani, Thailand

[2]Department of Prosthodontics, Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand

Abstract

Denture base fracture is often found as a primary clinical mode failure because of low flexural strength and repetitive stress. The fabrication of new dentures incurs high expenses and is time-consuming. Purpose: The aim of this study was to evaluate the flexural strength of auto-polymerized acrylic resins reinforced with 0.5, 1, 3, 10 weight % silane-treated nano-aluminum oxide particles. Seventy-two rectangular (ISO standard 20795-1) acrylic resins were fabricated and divided into six groups (n=12). Two control groups were an unmodified heat-polymerized acrylic resin group and auto-polymerized acrylic resin group. The others were experimental groups incorporated with 0.5, 1, 3 and 10 weight % silane-treated nano-aluminum oxide particles. The flexural strength was measured by a three-point bending test. One-way ANOVA and Tukey Honestly Significant Difference (HSD) were used for statistical analysis. The means flexural strength of silane-treated nano-alumina reinforced auto-polymerized groups (Groups SA0.5, SA1, SA3 and SA10) (136.63, 128.46, 125.61 and 121.53 MPa) were significantly improved compared to that of the control self-cured group (Group SC) (111.49 MPa) ($p<0.05$). Group SA0.5 showed the highest mean flexural strength compared to group SA10 and SC. Group SA0.5 also had the mean flexural strength almost as high as group HC. The flexural strength of auto-polymerized acrylic resins with low weight percentages of silane-treated nano-alumina tended to be higher than those with high weight percentages of silane-treated nano-alumina. Flexural strength of auto-polymerized acrylic resins reinforced with nano-alumina at 0.5 weight percent was comparable to that of heat-cured acrylic resins.

Keywords: Auto-polymerized acrylic resins, Flexural strength, Reinforcement, Alumina, Nano-alumina

Correspondence to:

Uraivichaikul Duangjai, Department of Prosthodontics, College of Dental Medicine, Rangsit University, Pathum Thani, Thailand.
Email: Duangjai.j@rsu.ac.th

Introduction

A study by Johnston and colleagues showed that 68 % of acrylic resin dentures broke within a few years after fabrication because of the flexural fatigue due to low flexural strength caused by repeatable intraoral force.[1] Fractured acrylic resin denture bases are commonly repaired instead of making new dental prostheses due to cost and time saving. After being repaired, dentures' durability does not last long. Dentures are more often

broken in auto-polymerized repaired denture than in heat-polymerized repaired denture. Denture repair depends on several factors: material type, surface design, material reinforcement and surface treatment.[2]

Different materials are used in reinforcement and repair of denture base, including heat-polymerized, auto-polymerizing, and light-polymerized acrylic resins.[3] Heat-polymerized repairs are rarely performed due to difficult laboratory processes and long polymerization time compared to auto-polymerized repairs. Therefore, auto-polymerized repairs are commonly used.[4] There are many surface designs that influence flexural strength or transverse strength of the repaired denture base. A previous study showed that rounded joint provided more transverse strength than rabbeted joint and butt joint.[5] In addition, the 45° bevel of the repaired surface had a significant impact on the transverse strength of the repaired denture base, resulting in the highest strength value.[6]

There are many recommended methods to reinforce the material used for denture base fabrication, such as chemical modification to prepare high impact resin and mechanical reinforcement with glass fibers, sapphire whiskers, carbon fibers, nylon polyethylene fibers, aramid fibers and zirconia.[7] Glass fiber addition leads to higher impact strength and improved flexural strength of PMMA.[8] The current meta-analysis showed that reinforcement of PMMA considerably enhanced flexural strength and impact strength.[9] Titanium, zirconia and nano-zirconia reinforcement also contributed to an increase in flexural and impact strength of PMMA.[10-12] A recent study showed that the use of nano-zirconia reinforcement could enhance the transverse strength of repaired acrylic denture bases.[13] The incorporation of 0.75%-ZrO_2 or 0.25%-SiO_2 into repaired resin increases repair strength.[14] Compression strength and hardness are also increased with increasing the volume fraction of nano-aluminum oxide and nano-silicon dioxide particles in PMMA. The addition of nano-aluminum oxide particles has more considerable effect than the addition of nano-silicon dioxide.[15] However, the use of metal powder filler has caused the dentures to be unaesthetic. Ideally, metal powder should be incorporated into the resin and

increases strength without affecting other properties. Ceramic filler is more preferable for reinforcement of acrylic resins than metal filler because of its low density and being white. Thus they are less likely to alter the finished appearance of the denture base material compared to metal powders.[16] Furthermore, previous studies demonstrated an increase in flexural strength by adding metal oxides (alumina, tin, silver and zirconia) and sapphire whiskers.[2, 17] Addition of alumina improves the properties of acrylic resin. For instance, an increase in thermal conductivity results from alumina reinforcement on PMMA.[18] Reinforcing PMMA with alumina also contributes to better flexural strength, impact strength, compressive strength, tensile strength, and surface hardness of the acrylic resin.[2,19] A previous study reported that current silane coupling agents promoted the bonding of resin composite to dental restorative materials.[20] Besides, the addition of 3-(trimethoxysilyl) propyl methacrylate in hydroxyapatite (HA) reinforced poly methyl methacrylate (PMMA) could enhance the mechanical properties by strengthening the chemical bonding and increasing mechanical interlocking between HA and PMMA.[21] Therefore, the application of silane on nano-aluminum oxide particles reinforced in self-cured acrylic resins was used in the study.

Aluminum oxide particles are commonly used as a sandblasting media especially in dentistry in order to increase mechanical retention of dental materials such as porcelain and full metals. Generally, aluminum oxide referred to as alumina, possesses strong ionic interatomic bonding, offering many interesting material characteristics. It can exist in several crystalline phases, all of which revert to the most stable hexagonal alpha phase at elevated temperatures. This is the phase of particular interest for structural applications. Alpha phase alumina is the strongest and the stiffest of the oxide ceramics. It has high hardness, excellent dielectric properties, refractoriness, and good thermal properties which result in a wide range of its applications in dentistry.[7, 15] Antimicrobial properties are considered a desirable characteristic that nanoparticles should obtain. Aluminum oxide nanoparticles have wide range of dental applications

and are also known to possess antimicrobial properties in the dental aspect. Alumina shows a mild bacterial growth-inhibitory effect, but only at very high concentrations.[22] Another research presented that the antimicrobial properties of nanoparticles containing formulations were increased.[23]

Furthermore, there have been many studies presenting the effect of particle size and percentage of concentration of particles on flexural strength of acrylic resin. Addition of 10 weight % and 15 weight % of alumina powder to heat cure acrylic resin improves the flexural strength.[24-26] Saritha MK.., *et al.* 2012 also found that 5 weight % of 5-22 μm. untreated alumina particles improved flexural strength by 13.99 %.[27] Jasim BS, Ismail IJ, 2014 found that 1 weight % treated nanofillers of alumina increased flexural strength by 24%.[28,29] Improved flexural strength can also be explained via the phenomenon of transformation. Al_2O_3 is found in many crystalline phases, and all filler particles revert to the most stable hexagonal alpha phase at elevated temperatures. Its structural application is interesting.[16] Transformation phenomenon appears and reduces crack propagation when enough stress develops and microcracks begin to propagate. As a result, even distribution of the filler within the matrix can stop or deflect cracks.[30,31] Furthermore, 0.2 % of 20 nm TiO_2 nano particles content increases the flexural strength of cold cured PMMA.[21] Nanofillers are more effective than microfillers at lower concentrations.

In addition, the use of alumina particle treated with silane coupling agent leads to considerable increase in properties of acrylic resin as compared to untreated particles because it improves the surface bonding of filler and resin matrix. Silane coupling agent playes an important role in improving repair bond strength because it increases bonding surface area and wets the repair area and fills microspores interlocking the repair material within irregularities.[31,32] Silane coupling agents contain inorganic reactive groups on silicon and bond well to most inorganic substrates, especially if the substrate contains silicon, aluminum, or most heavy metals in its structure.

Once a silane coupling agent is attached to an inorganic surface, it assumes the surface reactivity properties of the attached organic group. Consequently, the treated surface acquires the surface energy characteristics of the organic group, thus transforming into a reactive surface based on the reactivity of the organofunctional group present in the silane coupling agent. A silane coupling agent can be selected to give the surface whatever property is desired to transform the nature of the surface into whatever is needed to allow optimization of the inorganic material for the intended use. Previously, a study presented that 10 weight % treated particle with silane coupling agent 18-23 μm particles led to 23.86 % increase in flexural strength whereas non-silanized resulted in 5.46 % increase in the flexural strength.[26] Therefore, the application of silane on nano-aluminum oxide particles reinforced in self-cured acrylic resins was used in the study.

The objective of the study was to compare the effects of silane-treated nano-aluminum oxide reinforcement with weight percentages of 0.5, 1, 3, and 10 on the flexural strength of auto-polymerized acrylic resins. The hypothesis of the study was that different weight percentages of silanized nano-alumina particles did not significantly affect the flexural strength of auto-polymerized acrylic resins.

Materials and methods

1. Specimen preparation

Seventy-two rectangular (64*10*3.3 mm.[3])[33] (ISO standard 20795-1) acrylic resin specimens were fabricated and divided into six groups (n=12). Two control groups were an unmodified heat-polymerized acrylic resin group and an unmodified auto-polymerized acrylic resin group. The others were experimental groups which were auto-polymerized acrylic resin group reinforced with 0.5, 1, 3 and 10 weight percent silane-treated nano-aluminum oxide particles. The materials used in this study were listed in table 1 whereas coding according to reinforcement material used for reinforcing process of each group was shown in table 2.

Table 1 *Materials used in this study*

Materials	Use of materials	Compositions	Manufacturer
Heat-polymerized acrylic resins	Denture base processing	Powder: 95% PMMA, 4% plasticizer, 1% benzoyl peroxide Liquid: 90% MMA, 10% dimethacrylate, catalyst	Meliodent KULZER MITSUI Chemicals group, Heraeus Kulzer Gmbh, Tokyo, Japan LOT no. U010174
Auto-polymerized acrylic resins	Denture base processing	Powder: PMMA, benzoyl peroxide, Di-isobutyl phthalate Liquid: Methyl methacrylate, Hydroquinone, N, N-dimethyl-para-toluidine, Butyl or Octyl methacrylate Glycol dimethacrylate	Tokuso® Cure fast, Tokuyama Dental Corporation, Japan LOT no. 128E62
Silane coupling agent (MPS)	Silanization	1% 3- methacryloxypropyl trimethoxysilane ethanol/water-based solvent, acetic acid	KBM 503, Shin-Etsu Chemical, Tokyo, Japan LOT no. Z02CFS
Nano-alumina particles with diameter 20 nm.	Reinforcement	Al_2O_3, gamma, 99+%, 20 nm.	US Research Nanomaterials, Inc., Houston USA LOT no. US3023
320-grit silicon carbide paper	Finishing and polishing	C 0.07%, SiC 93.9%, Fe_2O_3 0.64%, Si 0.79%, Al_2O_3 0.25%, CaO 0.20%, SiO_2 4.11%	TOA Paint Public Company Limited, Thailand LOT no. 202112

Table 2 *Controlled groups, experimental groups and coding according to reinforcement material used for reinforcing process*

Group code	Reinforcement material used for reinforcing process
HC	Unmodified heat polymerized acrylic resin specimens (control)
SC	Unmodified auto-polymerized acrylic resin specimens (control)
SA0.5	Auto-polymerized acrylic resin specimens reinforced with 0.5 weight % silane-treated nano-aluminum oxide particles (20 nm.)
SA1	Auto-polymerized acrylic resin specimens reinforced with 1 weight % silane-treated nano-aluminum oxide particles (20 nm.)
SA3	Auto-polymerized acrylic resin specimens reinforced with 3 weight % silane-treated nano-aluminum oxide particles (20 nm.)
SA10	Auto-polymerized acrylic resin specimens reinforced with 10 weight % silane-treated nano-aluminum oxide particles (20 nm.)

Nano-aluminum oxide particles (20 nm.) (US Research Nanomaterials, Inc., Houston USA) was used as a reinforcement material in this study. It was preweighed into 0.5 weight %, 1 weight %, 3 weight %, and 10 weight % of auto-polymerized acrylic resin particles using an electronic weighing machine. Then, 0.1% silane coupling agent MPS (3-methacryloxy propyl trimethoxysilane) (KBM 503, Shin-Etsu Chemical, Tokyo, Japan) was measured using micropipette (10-100 µl. size, SCILAB) according to Arkle's equation.[30]

$$\text{amount of silane (g)} = \frac{\text{amount of filler (g)} \times \text{surface area (m}^2\text{/g)}}{\text{minimum coating area of silane coupling agent (m}^2\text{/g)}}$$

Silane was applied to coat the preweighed nano-aluminum oxide particles using a microbrush and waited to dry for 1 minute. Then, mixing of preweighed 0.5 weight %, 1 weight %, 3 weight %, and 10 weight % nano-aluminum oxide powder with resin polymer powder (50 mg.) was done using a magnetic stirrer (IKA C-MAG HS7), in order to achieve an equal distribution of particles and uniform consistency.[34]

All PMMA specimens were prepared using a heavy-body condensation silicone mold made of rectangular custom-made acrylic blocks. For auto-polymerized acrylic resin specimens, the simple mixing of auto-polymerized acrylic resin powder with a liquid monomer was done, according to the manufacture's recommendations. First, measured liquid monomer was poured into the rubber cup. Then, prepared auto-polymerized acrylic resin powder for unmodified auto-polymerized acrylic resin specimens and prepared nano-alumina reinforced auto-polymerized acrylic resin power for reinforced specimens was mixed with the liquid monomer within 15 seconds of working time. Later, the mixture was poured into the mold slightly in excess in order to compensate for the polymerization shrinkage and the glass slide was put on the top of the mold to stabilize the mold until it was set within 2 minutes and 30 seconds. After that, the mold was placed in water at a temperature of 50°C for 5 minutes to ensure a complete polymerization reaction.

For unmodified heat polymerized acrylic resin specimens, pink wax was melted and placed into the silicone mold where glass slides were used to control the thickness of the rectangular wax pieces. The rectangular wax pieces were invested in a metal flask with dental stone. Then, the flasks were placed in a scalding unit for wax boil-out (100°C, 5 minutes). After that, the flasks were left the rectangular shaped mold cavity in the dental stone, which was used as matrix for the fabrication of specimens. Separating medium was applied to the stone mold. Heat-polymerized acrylic resin powder was mixed with prepared liquid monomer. The mixture was then poured into the stone mold. Packing and processing of specimens was done according to manufacturer's instructions. The upper and lower flasks were closed and maintained under 200 lbs of compression for 30 minutes. The flasks were removed from the hydraulic pressure and cooled over the bench for 150 minutes. The curing procedure was processed by placing the flasks in the water bath at 71°C for 9 hours.[35] Flasks were allowed to cool to room temperature overnight before opening and deflasking. The specimens were removed from the mold. Finally, all specimens were finished, and polished with 320-grit silicon carbide paper (TOA, Thailand) using a polishing machine (Presi Minitech 233). They were later stored in distilled water in an incubator at 37°C for 2 days before flexural strength testing.[36]

2. Flexural strength testing

Before conducting flexural strength testing, the size of each specimen was examined using a digital micrometer (minimum reading: 0.001 mm, Digimatic Micrometer Mitutoyo Corp., Kanagawa, Japan) to ensure the accuracy of the specimens. The flexural strength of the specimens was determined using a 3-point bending testing device in a universal testing machine (EZ test, Shimadzu, Japan) (Fig. 1). The device was composed of a loading wedge and a pair of adjustable supporting wedges placed 50 mm apart. The specimens were centered on the device in such a way that the loading wedge engaged the center of the upper surface of the specimens and a crosshead speed was set at 5 mm/min. Specimens were loaded until fracture occurred. According to the formula below, the flexural strength was calculated.[36]

$$S = \frac{3Pl}{2bd^2}$$

S is the flexural strength (N/mm^2), P is the fracture load (N), I is the distance between the supporting wedges (mm), b is the specimen width (mm), and d is the specimen thickness (mm).

Figure 1 *EZ test, Shimadzu, Japan*

3. Visual surface analysis

A scanning electron microscope (FEI QUANTA FEI 200, FEI Company, Oregon, USA) was used to visualize the surface topography and examine the filler distribution in the polymer matrix of the control and the experimental specimens. It was also used to analyze the mode of failure of each experimental specimen. Definition of each mode of failure is described as follows. Adhesive failure means that fracture occurs between the interface of silane-treated aluminum oxide particles and PMMA. Cohesive failure means that fracture occurs between PMMA and PMMA. Mixture of adhesive and cohesive failure means that fracture occurs between the interface of silane-treated aluminum oxide particles and PMMA (adhesive failure) and between PMMA and PMMA (cohesive failure). One sample from each group was coated with gold by Gold Coater Jeol Model JFC-1200 and visualized at 1500x magnification. An attempt was done to focus on a presenting area and adjustment of the higher magnification while remaining on the same area was also done.

4. Statistical analysis

Data analysis was performed using SPSS software version 23, SPSS Inc, IRM corp, Chicago IL, USA at 95% confidence of level. A *p*-value ≤ 0.05 was considered statistically significant. The test of normality was achieved by using Kolmogorov-Smirnov's test. Mean flexural strength values were compared between different groups using one-way ANOVA. Then, the Post-Hoc or Multiple Comparison test was used to determine which of the paired means amongst a set of means was significantly different from the rest. Tukey's Honestly Significant Difference (HSD) test was selected for a pair-wise comparison due to the equal number of sample sizes in each set of data.

Results

As illustrated in figure 2 and table 3, while investigating differences in the flexural strength of auto-polymerized acrylic resins amongst the different weight percentages of nano-aluminum oxide particles and the two control groups, the alternative hypothesis was accepted. There were statistically significant differences in the flexural strength amongst different amount of nano-alumina reinforced auto-polymerized acrylic resins groups and unreinforced auto-polymerized acrylic resins and unreinforced heat-polymerized acrylic resins.

The mean flexural strength of group SA0.5 was 136.63 MPa which was not statically significant (*p*>0.05) lower than that of unmodified heat-cured group (group HC) which was 138.35 MPa. On the contrary, the mean flexural strength of other reinforced auto-polymerized groups (group SA1, SA3 and SA10) and unmodified self-cured group (SC) were 128.46, 125.61, 121.53 MPa, and 111.49 MPa respectively, which were statistically significant lower than that of unmodified heat-cured group (group HC) (*p*<0.001).
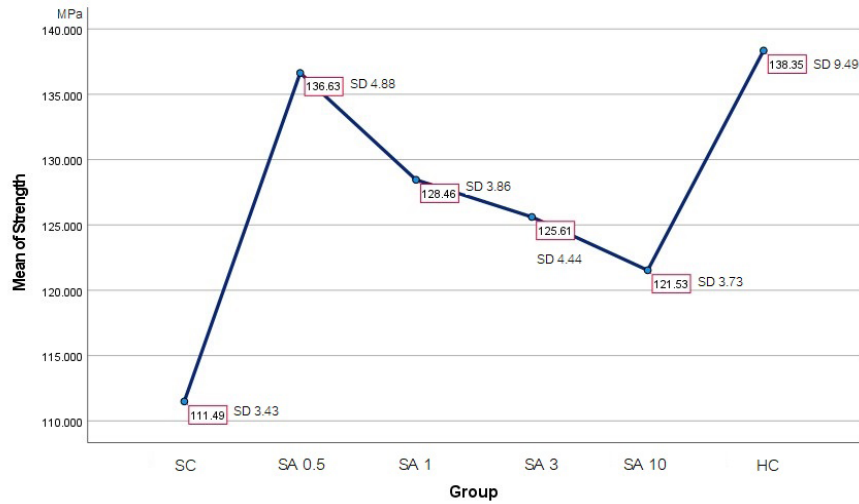
Figure 2 The effect of difference weight percentages of nano-aluminum oxide particle reinforcement on the flexural strength of acrylic resins

Table 3 The statistic results of descriptive analysis

Group	N	Mean	Std. Deviation
SC	12	111.49[a]	3.43
SA0.5	12	136.63[b]	4.88
SA1	12	128.46[c]	3.86
SA3	12	125.61[c,d]	4.44
SA10	12	121.53[d,e]	3.73
HC	12	138.35[b,f]	9.49
Total	72	127.01	10.53

*Groups with the same lowercase superscripted letter indicated no significant differences between groups at p-value<0.05.

Lastly, multiple comparisons were conducted to investigate whether every combination was significantly different in flexural strength from other combinations or not. As presented in table 3, the mean flexural strength of unreinforced auto-polymerized group (group SC) was the lowest (111.49 MPa) compared to other groups ($p<0.001$) whereas the mean flexural strength of unreinforced heat-polymerized group was the highest (138.35 MPa) ($p<0.05$) which was comparable to that of 0.5% silane-treated nano-alumina reinforced auto-polymerized group (group SA0.5) (136.63 MPa).

From previous data, it can be concluded that different percent by weight of silane-treated nano-alumina reinforcement also influenced flexural strength of auto-polymerized acrylic resins. In auto-polymerized acrylic resins, 0.5% by weight of silane-treated nano-alumina

reinforcement could improve flexural strength and was superior to 3% and 10% by weight of silane-treated na-no-alumina reinforcement. Furthermore, 0.5% by weight of silane-treated nano-alumina reinforced self-cured acrylic resins had the flexural strength as high as unreinforced heat-cured acrylic resins.

SEM images shown in figure 3 were taken from the fractured surface of nano-aluminum oxide reinforced self-cured PMMA specimens. Results of the SEM study showed that the nano-aluminum oxide particles in reinforced self-curing PMMA specimens were widely and evenly distributed within the resin matrix. The presence of reinforced particles also increased as the weight percentage of reinforcement increased. Moreover, the SEM images showed that the shape of nano-aluminum oxide particles was spherical and the size of nano-aluminum oxide particles was equal

among groups. As shown in figure 4, the images also revealed that a mode of failure for the specimens was a mixture of cohesive and adhesive failures. Fracture occurred between the interface of silane-treated aluminum oxide particles and PMMA (adhesive failure) and between PMMA and PMMA (cohesive failure). Mode of failure of each group was presented in table 4. Control groups (Group HC and SC) experienced 100% cohesive failure while the other experimental groups (Groups SA0.5, SA1, SA3, SA10) experienced a 100% mixture of cohesive and adhesive failures.
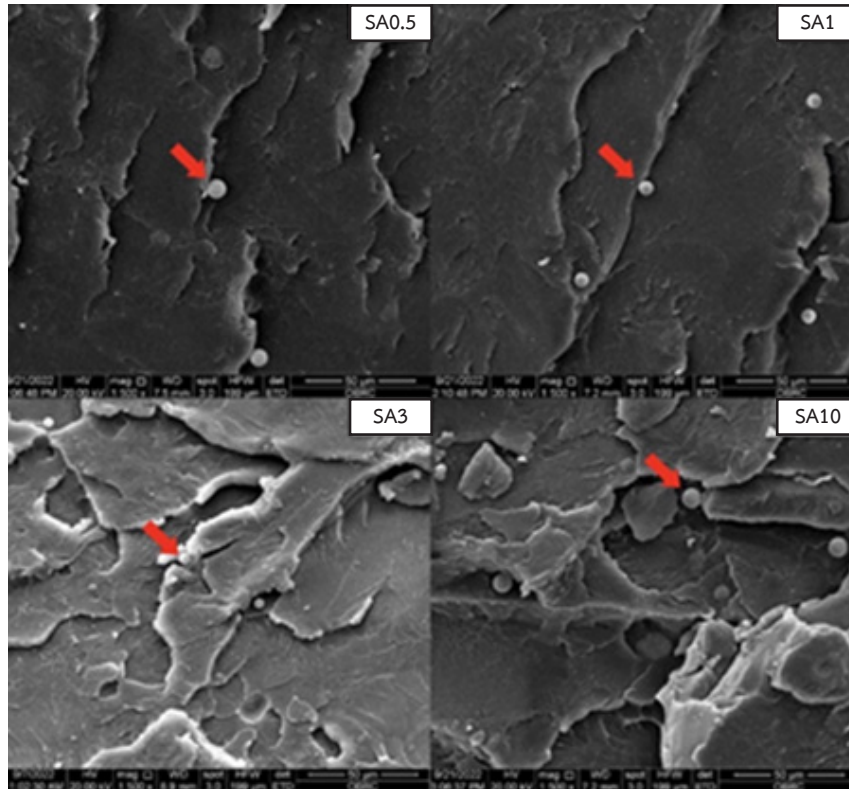

Figure 3 *Scanning electron microscope images of PMMA specimens (1500 magnification). The red arrow indicates the reinforced particle. SA0.5: fractured surface of 0.5% nano-alumina reinforced self-cured PMMA specimen, SA1: fractured surface of 1% nano-alumina reinforced self-cured PMMA specimen, SA3: fractured surface of 3% nano-alumina reinforced self-cured PMMA specimen, SA10: fractured surface of 10% nano-alumina reinforced self-cured PMMA specimen*

Figure 4 *Scanning electron microscope images of poly-methylmethacrylate (PMMA) specimens (1500 magnification). The red arrow indicates the reinforced particle, the blue arrow indicates the area that reinforced particle diminished and area of adhesive failure, and the yellow arrow indicates area of cohesive failure. SA3: fractured surface of 3% nano-alumina reinforced self-cured PMMA specimen, SA10: fractured surface of 10% nano alumina reinforced self-cured PMMA specimen*
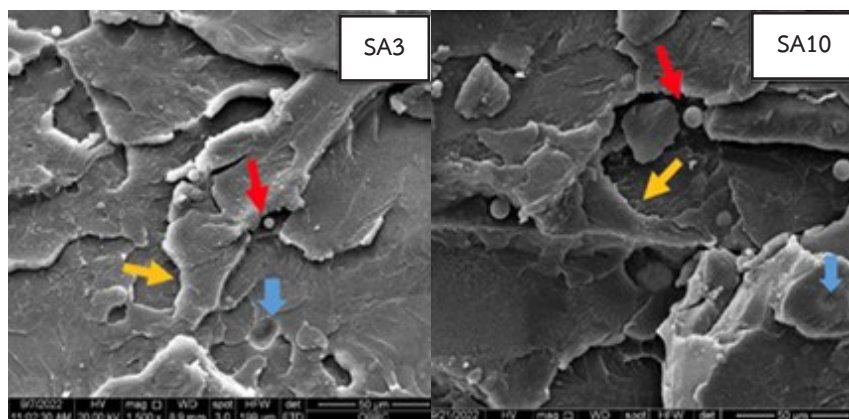
Table 4 *Mode of failure of each group*

Group	Adhesive	Cohesive	Mixed
HC	0%	100%	0%
SC	0%	100%	0%
SA0.5	0%	0%	100%
SA1	0%	0%	100%
SA3	0%	0%	100%
SA10	0%	0%	100%

Discussion

The result of the study demonstrated that unmodified heat-polymerized acrylic resin had more flexural strength than unmodified auto-polymerized acrylic resin. It could be explained that more voids which tended to reduce the flexural strength could be found in self-cured acrylic resin as the recent study mentioned.[37, 38] Furthermore, 0.5%, 1%, 3%, and 10% by weight of silane-treated nano-alumina reinforcement significantly improved the flexural strength of auto-polymerized acrylic resins. Likewise, the recent paper of Jasim BS, Ismail IJ (2014) and Mohammed M. Gad, Ahmad M. Al-Thobity (2021) presented that the flexural strength increased due to addition of small weight percentages of alumina.[18,20] From the result, 0.5% by weight of silane-treated nano-alumina reinforcement in auto-polymerized acrylic resins had flexural strength superior to 3% and 10% by weight. Similarly, Tamore SH and Jyothi KS (2018) conducted a study where they found that adding 1% and 1.5% nano-alumina to the repaired resin resulted in enhanced flexural strength compared to the unmodified resin.[31] The possible explanation could be that lower amount of nanoparticles is more effective because of their better ability of deep penetration to resin matrix. Another reason is that the more weight % of alumina reinforcement, the more void formation could be found.[39] In addition, aluminum oxide particles were pretreated with silane, which significantly improved the bond strength of alumina particles to PMMA.[39]

SEM examination of specimens reinforced with 0.5%, 1%, 3% and 10% by weight of silane-treated nano-aluminum oxide (Al_2O_3) particles reinforced self-curing acrylic resins showed a wide, equal distribution of oxide particles within the resin matrix (Fig. 3). similarly, Vojdani M, *et al.* demonstrated that specimens added with 2.5% by weight of aluminum oxide exhibited even distribution, as evaluated through SEM analysis. It confirms that the technique of mixing particles using a magnetic stirrer is acceptable and reliable. A reasonable explanation is that using the magnetic stirrer provides uniform consistency and reduces human errors.[34] In addition, the SEM examination of the fractured surface of specimens revealed that the mode of failure for most specimens was a mixture of cohesive and adhesive failures. Fracture was found between the interface of silane-treated aluminum oxide particles and PMMA (adhesive failure), and between PMMA and PMMA (cohesive failure). It could be explained that aluminum oxide reinforced particles are evenly distributed in resin specimens, so most of the fractured areas were a mixture of cohesive and adhesive failure. There was hardly any adhesive failure.

One of the limitations of this study was that surface voids could be found in tested specimens, although the specimens were preselected using exclusion criteria. PMMA has the tendency to generate submicrometer voids.[34] Therefore, fracture resistance of specimens may be influenced by void formations located within the specimens. Furthermore, material fatigue by thermocycling in water or artificial saliva with or without reinforcement was not carried out to predict the success of material in

a clinical environment, as the study was conducted in an in vitro setting. Another limitation of this study was that the SEM images revealed that some areas showed larger nano-alumina particles size compared to other areas, which was attributed to a controlled manner by means of surface tension. This resulted in an alteration of the particle-size distribution[36] Although alteration in color of alumina reinforced specimens was not obvious, it is better to be used in invisible denture areas such as lingual flange of lower dentures or the palatal area of upper dentures because discoloration and opacity of PMMA can occur when added at high concentration.[14]

Auto-polymerized acrylic resins reinforced with 0.5% by weight of silane-treated nano-alumina particles had the flexural strength almost as high as unreinforced heat-polymerized acrylic resins. Fortunately, 0.5 weight% silane-treated nano-alumina can be used for reinforcement in self-cured acrylic resins which are mostly chosen as denture repair materials. The extended longevity of dentures contributes to increased patient satisfaction. However, further research is required to examine cytotoxicity, aging effect and physical properties such as color stability and surface roughness of these reinforced denture base materials before clinical application.

Conclusions

According to the obtained results and the mentioned limitations, the following conclusions can be drawn:

1. Alteration of percent by weight of silane-treated nano-alumina reinforcement resulted in differences in the flexural strength of auto-polymerized acrylic resins. Self-cured acrylic resins incorporated with low weight percentages of silane-treated nano-alumina had better flexural strength than those incorporated with high weight percentages of silane-treated nano-alumina.

2. Auto-polymerized acrylic resins reinforced with 0.5% by weight of silane-treated nano-alumina paticles demonstrated the flexural strength almost as high as unreinforced heat-polymerized acrylic resins.

References

1. Johnston EP, Nicholls JI, Smith DE. Flexural fatigue of 10commonly used denture base resins. *J Prosthet Dent* 1981;46(5):478-83

2. Seo RS, Neppelenbroek KH, Filho JNA. Factors affcting the strength of denture repairs: topics of interest. *J Prosthodont* 2007;16(4):302-10.

3. Alkurt M, Yesil Duymus Z, Gundogdu M. Effect of repair resin type and surface treatment on the repair strength of heat-polymerized denture base resin. *J Prosthet Dent* 2014;111(1):71-8.

4. Foo SH, Lindquist TJ, Aquilino SA, Schneider RL, Williamson DL, Boyer DB. Effect of polyaramid fiber reinforcement on the strength of 3 denture base polymethyl methacrylate resins. *J Prosthodont* 2001;10(3):148-53.

5. Harrison WM, Stansbury BE. The effect of joint surface contours on the transverse strength of repaired acrylic resin. *J Prosthet Dent* 1970;23(4):464-72.

6. Hanna EA, Shah FK, Gebreel AA. Effect of joint surface contours on the transverse and impact strength of denture base resin repaired by various methods: an in vitro study. *J Am Sci* 2010;6(9):115-25.

7. Arora P, Singh SP, Arora V. Effect of Alumina Addition on Properties of Poly-methymethacrylate - A comprehensive review. *IJBTT* 2015;5(1):7-12.

8. Moreno MV, Acosta-Torres LS, Barcel O-Santana FH, Vanegas-Lanc RD, Plata-Rodríguez ME, Casta VM. Fiber-reinforced nano-pigmented poly (methyl methacrylate) as improved denture base. *J Appl Polym Sci* 2012;126(1):289–96.

9. Somani MV, Khandelwal M, Punia V, Sharma V. The effect of incorporating various reinforcement materials on flexural strength and impact strength of polymethylmethacrylate: A meta-analysis. *J Indian Prosthodont Soc* 2019;19(2):101-12.

10. Asar NV, Albayrak H, Korkmaz T, Turkyilmaz I. Influence of various metal oxides on mechanical and physical properties of heat-cured polymethylmethacrylate denture base resins. *J Adv Prosthodont* 2013;5(3):241–7.

11. Safi IN. Evaluation the effect of nano-fillers (TiO_2, AL_2O_3, SiO_2) addition on glass transition temperature, E-Moudulus and coefficient of thermal expansion of acrylic denture base material. *J Bagh Coll Dent* 2014;26(1):37–41.

12. Zhang XJ, Zhang XY, Zhu BS, Qian C. Effect of nano ZrO2 on flexural strength and surface hardness of polymethylmethacrylate. *Shanghai Kou Qiang Yi Xue* 2011;20(4):358–63.

13. Gad M, ArRejaie AS, Abdel-Halim MS, Rahoma A. The Reinforcement Effect of Nano-Zirconia on the Transverse Strength of Repaired Acrylic Denture Base. *Int J Dent* 2016;2016:7094056.

14. Abushowmi T, Alzaher Z, Almaskin D, Qaw M, Abualsaud R, Akhtar S, *et al*. Comparative Effect of Glass Fiber and Nano-Filler Addition on Denture Repair Strength. *J Prosthodont* 2020;29(3):261-8.

15. Hamad QA. Investigation some mechanical properties of self cured PMMA resin reinforced by different types of nano particles. *IQJMME* 2017;17(3):585-98.

16. Ellakwa AE, Morsy MA, EI-sheikh AM. Effect of Aluminum oxide addition on the Flexural strength and thermal diffusivity of heat-polymerized acrylic resin. *J Prosthodont* 2008;17(6):439-44.

17. Heidari B, Firouz F, Izadi A, Ahmadvand S, Radan P. Flexural Strength of Cold and Heat Cure Acrylic Resins Reinforced with Different Materials. *J Dent (Tehran)* 2015;12(5):316-23.

18. Jasim BS, Ismail IJ. The effect of silanized alumina nanofillers addition on some physical and mechanical properties of heat cured polymehylmethacrylate denture base material. *J Bagh Coll Dentistry* 2014;6(2):18-23.

19. Saritha M.K., Shivamurthy Shadakshari, Nandeeshwar D.B, Shivsagar Tewary. An in vitro study to investigate the flexural strength of conventional heat polymerized denture base resin with addition of different percentage of aluminium oxide powder. *Asian J Med Cli Sci* 2012;1(2):80-5.

20. Mohammed M. Gad, Ahmad M. Al-Thobity. The impact of nanoparticles-modified repair resin on denture repairs: a systematic review. *Japanese Dental Science Review* 2021;57:46-53.

21. Balos S, Branislava P, Sarcev, Sarcev I, Janjatovic P, Pilić B, *et al.* Flexural Modulus and Strength of Cold Cured Poly(methyl-methacrylate) Reinforced with TiO2 Nano Particles. *Mater Plast* 2020;57(4):13-20.

22. Abdulkareem MM, Hatim NA. Evaluation the biological effect of adding Aluminum Oxide, Silver nanoparticles into microwave treated PMMA powder. *Int J Enhanc Res Sci Technol Eng* 2015; 10(3):172-8.

23. Acosta-Torres LS, López-Marín LM, Núñez-Anita RE, Hernández-Padrón G, Castaño VM. Biocompatible Metal-Oxide Nanoparticles: Nanotechnology Improvement of Conventional Prosthetic Acrylic Resins. *Journal of Nanomaterial* 2011;2011:941561.

24. Dhole RI, Srivatsa G, Shetty R, Huddar D, Sankeshwari B, Chopade S. Reinforcement of Aluminum Oxide Filler on the Flexural Strength of Different Types of Denture Base Resins: An *In vitro* Study. *J Clin Diagn Res* 2017;11(4):ZC101-4.

25. Lavanya P, Srinivas K, Shankar R, Swetha V, Hari Krishna M. Effects of Addition of Aluminum Oxide on Flexural Strength and Hardness of Acrylic Resins. *IOSR-JDMS* 2017;16(3):01-6.

26. Chaijareenont P, Takahashi H, Nishiyama N, Arksornnukit M. Effect of different amounts of 3-methacryloxypropyltrimethoxy silane on the flexural properties and wear resistance of alumina reinforced PMMA. *Dent Mater J* 2012;31(4):623–8.

27. Saritha M.K., Shivamurthy Shadakshari, Nandeeshwar D.B, Shivsagar Tewary. An *in vitro* study to investigate the flexural strength of conventional heat polymerized denture base resin with addition of different percentage of aluminium oxide powder. *Asian J Med Cli Sci* 2012;1(2):80-5.

28. Jasim BS, Ismail IJ. The effect of silanized alumina nanofillers addition on some physical and mechanical properties of heat cured polymehylmethacrylate denture base material. *J Bagh Coll Dentistry* 2014;6(2):18-23.

29. Mohammed M. Gad, Ahmad M. Al-Thobity. The impact of nano-particles-modified repair resin on denture repairs: a systematic review. *Japanese Dental Science Review* 2021;57:46-53.

30. Arkles B. Silane coupling agent chemistry. Pennsylvania: Petrarch Systems Catalogue; 1987.p. 59.

31. Tamore SH, Jyothi KS, Muttagi S, Gaikwad AM. Flexural strength of surface-treated heat-polymerized acrylic resin after repair with aluminum oxide-reinforced autopolymerizing acrylic resin. *Contemp Clin Dent* 2018;9(2):347–53.

32. Qaw MS, Abushowmi TH, Almaskin DF, AlZaher ZA, Gad MM, Al-Harbi FA. A novel approach to improve repair bond strength of repaired acrylic resin: an *in vitro* study on the shear bond strength. *J Prosthodont* 2020;29(4):323–33.

33. International Organization for Standardization. Specification 20795-1: Dentistry–base-polymer-part I: Denture Base Polymers. Geneva, Switzerland, 2013.p 6-7.

34. Vojdani M, Bagheri R, Khaledi A. Effects of aluminum oxide addition on the flexural strength, surface hardness, and roughness of heat-polymerized acrylic resin. *J Dent Sci* 2012;7(3):238–44.

35. Sakaguchi RL, Ferracane JL, Powers JM. Craig's Restorative Dental Materials (ed 14). St Louis, MO, Mosby, 2018.

36. Alhotan A, Yates J, Zidan S, Haider J, Silikas N. Flexural Strength and Hardness of Filler-Reinforced PMMA Targeted for Denture Base Application. *Materials (Basel)* 2021;14(10):2659.

37. Dar-Odeh NS, Harrison A, Abu-Hammad O. An evaluation of self-cured and visible light-cured denture base materials when used as a denture base repair material. *J Oral Rehabil* 1997;24(10):755-60.

38. Polyzois GL, Handley RW, Stafford GD. Repair strength of denture base resins using various methods. *Eur J Prosthodont Restor Dent* 1995;3(4):183-6.

39. Kul E, Aladag LI, Yesildal R. Evaluation of thermal conductivity and flexural strength properties of poly(methyl methacrylate) denture base material reinforced with different fillers. *J Prosthet Dent* 2016;116(5):803-10.