



Impact of Silanized Nano-Alumina on Flexural Strength of Auto-Polymerized Acrylic Resins

Duangjai Uraivichaiikul^{*1}, Issarawan Boonsiri¹, Kamolporn Wattanasirmit², Kittithaworn Aranda¹, Pornmanee Amornviriyakul¹, Jennipit Chiayot¹, Narisara Pokong¹, and Nareekarn Sangprapanun¹

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

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

*Corresponding author, E-mail: Duangjai.j@rsu.ac.th

Abstract

The purpose of this study was to evaluate impact of 0.5, 1.5 and 10 weight % silane-treated nano-aluminum oxide (Al_2O_3) particles (20 nm) reinforcement on the flexural strength of auto-polymerized acrylic resins. Sixty rectangular acrylic resins ($65 \times 10 \times 3 \text{ mm}^3$) (ISO standard 1567) were equally divided into 5 groups ($n=12$) and there were 2 control groups including heat-polymerized acrylic resin group and auto-polymerized acrylic resin group. The others were experimental groups reinforced with 0.5, 1.5 and 10 weight % silane-treated nano-alumina. The flexural strength was measured by a three-point bending test. Statistical analysis used independent t-test, One-way ANOVA and Tukey Honestly Significant Difference (HSD). The results showed that the mean flexural strength of silane-treated nano-alumina reinforced auto-polymerized groups (SA0.5, SA1.5 and SA10) were significantly higher than the control auto-polymerized group (SC) (136.63 MPa, 127.99 MPa, 121.53 MPa vs. 111.49 MPa, respectively) ($p < 0.05$). Group SA0.5 had the highest mean flexural strength. Also, group SA0.5 had the mean flexural strength as the same as group HC (138.35) ($p > 0.05$). In conclusion, the study suggests that auto-polymerized acrylic resins with low weight percentages of silane-treated nano-alumina may have higher flexural compared to those with higher weight percentages. Specifically, the results indicate that auto-polymerized acrylic resins reinforced with 0.5 weight% silane-treated nano-alumina had a comparative flexural strength to that of unreinforced heat-cure acrylic resins.

Keywords: Alumina, Aluminum Oxide, Auto-Polymerized Acrylic Resin, Acrylic Resins, Flexural Strength, Nano-Alumina, Reinforcement, Silane-Treated Nano-Alumina

1. Introduction

A study of Johnston and colleagues showed that 68% of acrylic resin dentures break within a few years after fabrication because of the flexural fatigue due to low flexural strength caused by repeatable intraoral force (Johnston, 1981). Fractured acrylic resin denture bases are commonly repaired instead of making new dental prostheses due to cost and time saving. After being repaired, the dentures' lifespan did not last long. It was found that the dentures were more often broken in auto-polymerized repair dentures than heat-polymerized repair dentures. Denture repair depends on several factors: material type, surface design, material reinforcement and surface treatment (Seó, Neppelenbroek, & Filho, 2007).

Various materials are used to reinforce and repair the denture base, including heat-polymerized, auto-polymerizing, and light-polymerized acrylic resins. Heat-polymerized repairs are rarely performed (Alkurt, Duymuş, & Gundogdu, 2014) because they require a difficult laboratory process, longer polymerization time, and higher laboratory fees. Therefore, fractured dentures are often repaired with auto-polymerizing acrylic resins due to their lower cost and greater practicality (Foo et al., 2001). However, when heat-polymerized acrylic resins are used, the ratio of the strength of the repaired area to the strength of the original denture base is 75% to 80% (Leong, & Grant, 1971). When auto-polymerizing acrylic resins were used, the percentage decreased to 60 to 69% (Stanford, Burns, & Paffenbarger, 1955). There are many recommended methods for reinforcing the material used to fabricate denture, adding Aluminum oxide is one of the methods that helps reinforcing the strength of dentures. The aluminum oxide referred to as alumina. Addition of 5 weight % aluminum oxide nanoparticles to acrylic resin was improve thermal properties and transverse strength of acrylic resin, and at the same time decreased its water sorption and solubility (Safi, 2014). On the other hand, addition of aluminum oxide nanoparticles to acrylic resin was increase in surface

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roughness of acrylic resin, but did not significantly change if the concentration of aluminum oxide nanoparticles were increased (Kul, Aladağ, & Yesildal, 2016). The reinforced particle has various shape that affects the mechanical properties of PMMA. Elongated particles are more efficient to increase the properties of acrylic resin than spherical particles (Ben Hasan et al., 2014).

From previous studies, the particle size and percent weight concentration of aluminum particles have an effect on the flexural strength of acrylic resin. Addition 10 weight % and 15 weight % of alumina powder to heat cure acrylic resin can improved the flexural strength (Dhole et al., 2017). Reinforcement by 15% weight aluminum oxide powder to the conventional heat-polymerized acrylic resin and high impact heat cured acrylic resin significantly improved its flexural strength and hardness without adverse effects (Lavanya Pentapati, Shankar, & Swetha, 2021). Addition of 10 weight % treated alumina particles sized 18-23 μm . resulted in 23.86% increase in flexural strength (Chaijareenont, Takahashi, Nishiyama, & Arksornnukit, 2012). Jasim and Ismail (2014) conducted that 1 weight % treated nanofillers of alumina increased 24% of flexural strength (Jasim & Ismail, 2014) also found that 5 weight % of 5-22 μm untreated alumina particles improved flexural strength by 13.99% (Saritha, Shadakshari, Nandeeshwar, & Tewary 2012). 1 and 1.5 weight % nano- Al_2O_3 increased flexural strength of repaired denture (Gad, & Al-Thobity, 2021). 0.2% of 20 nm TiO_2 nano particles content increased the flexural strength of cold cured PMMA (Balos et al., 2020). At lower concentrations nanofillers are more effective than microfillers.

Silane-treated alumina particle leads to a considerable increase in the properties of acrylic resin compared to untreated particles. Silane coupling agents contain inorganic reactive groups on silicon that 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, the treated surface will exhibit the surface energy of the organic group, and the surface can become a reactive surface. 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. A previous 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 strength (Lavanya Pentapati, Shankar, & Swetha, 2021).

Thus, this study was conducted to compare the effect of 0.5, 1.5 and 10 weight % of silane-treated nano-aluminum oxide (20 nm) reinforcement on the flexural strength of auto-polymerized acrylic resins. The null hypothesis was that there were no statistically significant differences in the flexural strength of auto-polymerized acrylic resins amongst the different weight percentages of nano-aluminum oxide particles and two control groups.

2. Objective

To compare the impact of 0.5, 1.5 and 10 weight % of silane-treated nano-aluminum oxide (20 nm) reinforcement on the flexural strength of auto-polymerized acrylic resins.

3. Materials and Methods

Twenty-four rectangular (65x10x3 mm^3) (ISO standard 15666) PMMA specimens were equally divided and fabricated from auto-polymerized (Tokuso Cure fast, Tokuyama Dental Corporation, Japan) and heat-polymerized (Meliodent Heat cure, KULZER MITSUI Chemicals group, Heraeus Kulzer GmbH, Tokyo, Japan) as two control groups. The other PMMA specimens were fabricated as nano-alumina reinforced auto-polymerized groups. The materials used in this study were listed in Table 1. All specimens were prepared by using silicone mold for custom-made acrylic blocks. The pink wax was added into the silicone mold, a glass slide was used to control the thickness of the pink wax. Then packed in metal flask with dental stone, and boil-out (100 $^{\circ}\text{C}$, 5 minutes), later applied auto-polymerized and heat-polymerized at the rectangular shaped mold cavity in the dental stone.



3.1 Specimen preparation

All PMMA specimens were equally divided into five groups: two control groups (heat-polymerized and auto-polymerized specimens) and three experimental groups (addition of nano-alumina) as shown in Table 2 and Figure 1.

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	Meliudent KULZER MITSUI Chemicals group, Heraeus Kulzer GmbH, Tokyo, Japan
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
320-grit silicon carbide paper	Finishing and polishing	C 0.07%, SiC 93.9%, Fe ₂ O ₃ 0.64%, Si 0.79%, Al ₂ O ₃ 0.25%, CaO 0.20%, SiO ₂ 4.11%	TOA Paint Public Company Limited, Thailand
Silane coupling agent (MPS)	Silanization	1% 3-methacryloxypropyl trimethoxysilane ethanol/water-based solvent, acetic acid	KBM 503, Shin-Etsu Chemical, Tokyo, Japan
Nano-alumina particles with diameter 20 nm.	Reinforcement	Al ₂ O ₃ , gamma, 99+%, 20 nm.	US Research Nanomaterials, Inc., Houston USA

Table 2 Controlled groups and experimental groups were categorized according to reinforcement material used for reinforcing process.

Group code	Reinforcement material used for reinforcing process
HC	Intact heat polymerized acrylic resin specimens (control)
SC	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.).
SA1.5	Auto-polymerized acrylic resin specimens reinforced with 1.5 weight % silane-treated nano-aluminum oxide particles (20 nm.).
SA10	Auto-polymerized acrylic resin specimens reinforced with 10 weights % silane-treated nano-aluminum oxide particles (20 nm.).

Nano-aluminum oxide particles (20 nm) (US Research Nanomaterials, Inc., Houston USA) were pre-weighed into 0.5 weight %, 1.5 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 μ L size, SCILAB) according to Arkle's equation (Arkles, 1987).

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

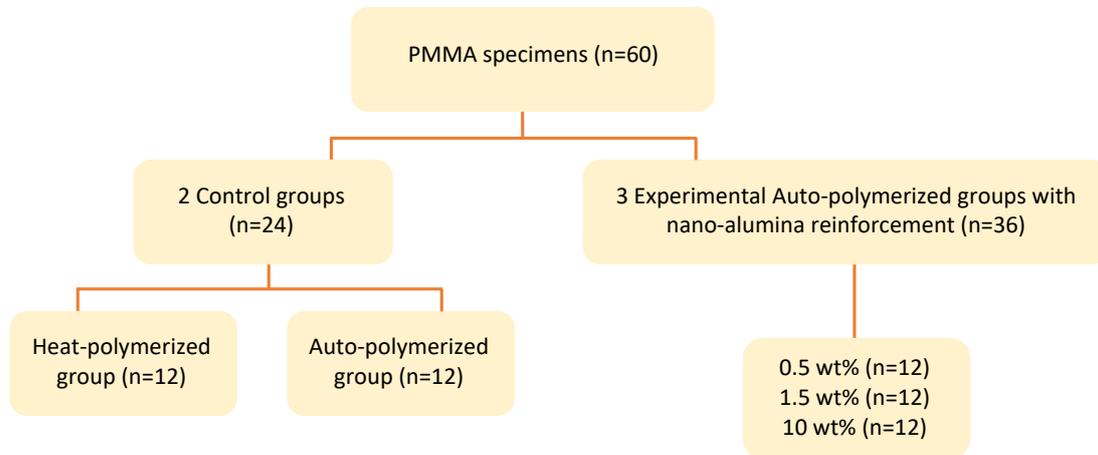


Figure 1 Sample groups

Silane was coated with the pre-weighed nano-aluminum oxide particles using a microbrush and left to dry for 1 minute. With a magnetic stirrer, mix the pre-weighed 0.5 weight %, 1.5 weight % and 10 weight % nano-aluminum oxide powder with resin polymer powder (50 mg) (IKA C-MAG HS7) to achieve an equal distribution of particles and uniform consistency (Vojdani, Bagheri, & Khaledi, 2012). Figure 2 illustrates the packing and processing of specimens, which were carried out following the manufacturer’s instructions. The upper and lower flasks were closed and maintained under 200 lbs of compression for 30 minutes. The flasks were then 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 (Sakaguchi, Ferracane, & Powers, 2018). Flasks were allowed to cool to room temperature overnight before opening and deflasking. The specimens were removed from the mold, finished, and polished with 320-grit silicon carbide paper (TOA, Thailand) using a polishing machine (Presi Minitech 233). The specimens were subsequently stored in an incubator at 37°C for 2 days before flexural strength testing (Alhotan, Yates, Zidan, Haider, & Silikas, 2021). The reinforced and unreinforced samples were shown in Figure 3 and Figure 4, respectively.

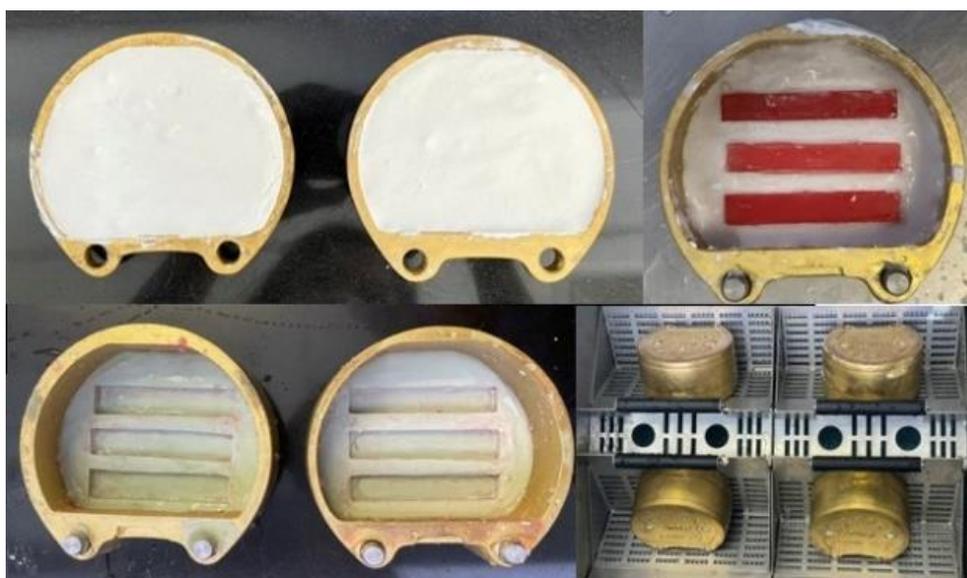


Figure 2 Packing and processing

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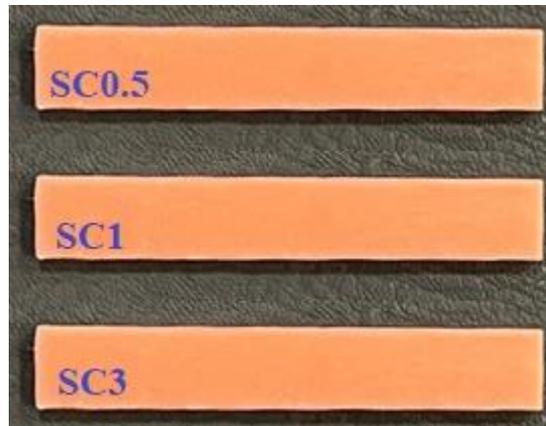


Figure 3 Polished silane-treated aluminum oxide reinforced auto-polymerized acrylic resin specimen (65x10x3 mm³)



Figure 4a Polished unreinforced heat-polymerized acrylic resin specimen (65x10x3 mm³)



Figure 4b Polished unreinforced auto-polymerized acrylic resin specimen (65x10x3 mm³)

3.2 Flexural strength testing

Before flexural strength testing, the size of each specimen was examined to make sure the accuracy of the specimens using a digital micrometer (minimum reading: 0.001 mm, Digimatic Micrometer Mitutoyo Corp., Kanagawa, Japan). The flexural strength of the specimens was determined using a 3-point bending testing device in a universal testing machine (EZ test, Shimadzu, Japan) (Figure 5). The device comprised 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 and set a crosshead speed of 5 mm/min, engaged the center of the upper surface of the specimens. Specimens were loaded until fracture occurred. According to the formula below, the flexural strength was calculated (Alhotan et al., 2021).

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

S is the flexural strength (N/mm²), 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 5 EZ test, Shimadzu, Japan

3.3 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 experimental specimens. It was also used to analyze the mode of failure of each experimental specimen. Definitions of the modes of failure are described in the following sentences. 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 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 using Gold Coater Jeol Model JFC-1200 and visualized at 1500x magnification (Figure 6). 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.



Figure 6 Gold Coater Jeol Model JFC-1200

3.4 Statistical analysis

Data analysis was performed using SPSS software version 23, SPSS Inc, IRM corp, Chicago IL, USA at 95% confidence of level. A $p\text{-value} \leq 0.05$ was considered significant. After gaining results from the experiment, it was used to test for normality using Shapiro-Wilk test as the degree of freedom of each group which was less than 50. The results were well-modeled by normal distribution and parametric statistical tests were proceeded.

The hypotheses were analyzed using one-way ANOVA since there were six sets of data, a single independent variable, and one dependent variable. Afterwards, 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.

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Tukey's Honestly Significant Difference (HSD) test was selected for pair-wise comparison due to the equal number of sample sizes in each set of data.

4. Results and Discussion

Figure 7, table 3a and table 3b present the investigation of differences in the flexural strength of auto-polymerized acrylic resins among different weight percentages of nano-aluminum oxide particles and two control groups. The alternative hypothesis was accepted. There were statistically significant differences in the flexural strength amongst different amount of nano-alumina reinforced and unreinforced auto-polymerized acrylic resins and unreinforced heat-polymerized acrylic resins. The mean flexural strength of all reinforced auto-polymerized groups (group SA0.5, SA1.5, SA10) were 136.63, 127.99 and 121.53 MPa, respectively, which were significantly higher than the control auto-polymerized group (group SC) ($p < 0.001$). For reinforced auto-polymerized groups, the highest mean flexural strength was 136.63 MPa which found in 0.5% particle reinforced group (Group SA0.5) while the lowest mean flexural strength was 121.53 MPa which found in 10% particle reinforced group (Group SA10). However, unreinforced heat-polymerized acrylic resin had the highest mean flexural strength compared with other groups. The mean flexural strength of 0.5% particle reinforced group (group SA0.5) was statistically significantly higher than the mean flexural strength of 1.5% and 10% particle reinforced group (group SA1.5 and SA10) ($p < 0.001$). In contrast, the mean flexural strength between group SA1.5 and SA10 was not significantly different ($p > 0.05$) as shown in table 3b. Besides, the mean flexural strength of group SA0.5 was 136.63 MPa which was not statically significant ($p > 0.05$) lower than that of group HC which was 138.35 MPa.

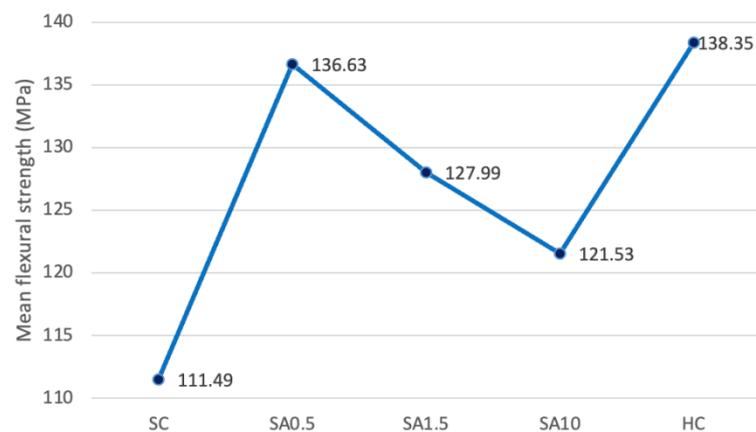


Figure 7 The effect of difference weight percentages of aluminum oxide particle reinforcement on the flexural strength of acrylic resins

Table 3a The statistic results of descriptive analysis

Groups	N	Mean	Std. Deviation	Std. Error
SC	12	111.49	3.43	0.99
SA0.5	12	136.63	4.88	1.41
SA1.5	12	127.99	7.98	2.30
SA10	12	121.53	3.73	1.08
HC	12	138.35	9.49	2.74
Total	60	127.20	11.75	1.52

**Table 3b** Multiple comparison of different weight percentages of nano-alumina reinforced auto-polymerized acrylic resins and two control groups

Group		Mean Difference (I-J)	Std. Error	Sig.
SC	SA0.5	-25.141*	2.602	0.000
	SA1.5	-16.494*	2.602	0.000
	SA10	-10.041*	2.602	0.003
	HC	-26.853	2.602	0.000
SA0.5	SC	25.141*	2.602	0.000
	SA1.5	8.647*	2.602	0.013
	SA10	15.100*	2.602	0.000
	HC	-1.712	2.602	0.964
SA1.5	SC	16.494*	2.602	0.000
	SA0.5	-8.647*	2.602	0.013
	SA10	6.453	2.602	0.110
	HC	-10.359*	2.602	0.002
SA10	SC	10.041*	2.602	0.003
	SA0.5	-15.100*	2.602	0.000
	SA1.5	-6.453	2.602	0.110
	HC	-16.813*	2.602	0.000
HC	SC	26.853*	2.602	0.000
	SA0.5	1.712	2.602	0.964
	SA1.5	10.359*	2.602	0.002
	SA10	16.813*	2.602	0.000

*p-value less than 0.05 was considered to be statistically significant

In short, multiple comparisons were conducted to investigate whether each combination was significant difference in flexural strength with other combinations or not. As presented in table 3b, the mean flexural strength of the unreinforced auto-polymerized group (group SC) had the lowest strength (111.49 MPa) compared with other groups ($p < 0.001$) whereas the mean flexural strength of the heat control group had the highest strength (138.35 MPa) ($p < 0.05$), which was similar to the 0.5% nano-alumina reinforced auto-polymerized group (group SA0.5) (136.63 MPa).

The mean flexural strength of reinforced auto-polymerized groups significantly increased compared to unreinforced auto-polymerized groups ($p < 0.05$). The mean flexural strength of 0.5% particle auto-polymerized group (group SA0.5) had the highest strength among auto-polymerized group (136.63 MPa). It was 1.72 MPa insignificantly lower, which was not significantly different from the heat-polymerized control group (group HC). This showed that 0.5% reinforced auto-polymerized acrylic resins was as strong as unreinforced heat-polymerized acrylic resins.

In conclusion, the different percent by weight of silane-treated nano-alumina reinforcement also influenced the flexural strength of auto-polymerized acrylic resins. In auto-polymerized acrylic resins, 0.5% by weight of silane-treated nano-alumina reinforcement had the flexural strength superior to that of 1.5% and 10% by weight. Furthermore, 0.5% by weight of silane-treated nano-alumina reinforced auto-polymerized acrylic resins had the same flexural strength as unreinforced heat-polymerized acrylic resins.

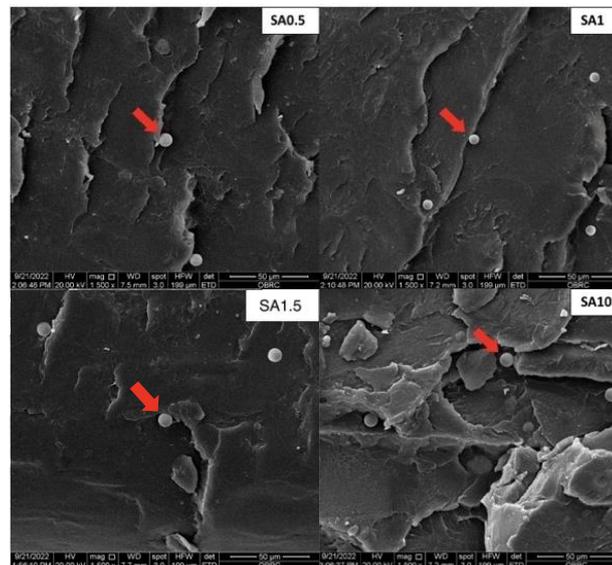


Figure 8 Scanning electron microscope images of PMMA specimens (1500 magnification).

The red arrow indicates the reinforced particle. SA0.5: fractured surface of 0.5% nano-alumina reinforced auto-polymerized PMMA specimen, SA1.5: fractured surface of 1.5% nano-alumina reinforced auto-polymerized PMMA specimen, SA10: fractured surface of 10% nano-alumina reinforced auto-polymerized PMMA specimen.

In Figure 8, SEM images showed the fractured surface of nano-aluminum oxide reinforced auto-polymerized PMMA specimens. From the SEM study, the nano-aluminum oxide particles in reinforced auto-polymerized 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 9, 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). Table 4 showed mode of failure for each group of all specimens. Control groups (Group HC and SC) experienced 100% cohesive failure while the other experimental groups (Groups SA0.5, SA1.5, SA10) experienced a 100% mixture of cohesive and adhesive failures.

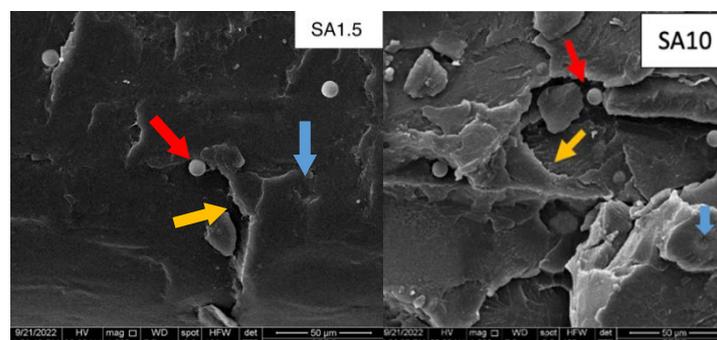


Figure 9 Scanning electron microscope images of poly-methylmethacrylate (PMMA) specimens (1500 magnification). The red arrow indicates the reinforced particle, the blue arrow indicates the area that reinforced particle diminished and area of adhesive failure, and the yellow arrow indicates area of cohesive failure. SA1.5: fractured surface of 1.5% nano-alumina reinforced auto-polymerized PMMA specimen, SA10: fractured surface of 10% nano alumina reinforced auto-polymerized PMMA specimen.

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**Table 4** Mode of failure of each group of all specimens

Group	n (specimens)	Adhesive	Cohesive	Mixed
HC	12	0%	100%	0%
SC	12	0%	100%	0%
SA0.5	12	0%	0%	100%
SA1.5	12	0%	0%	100%
SA10	12	0%	0%	100%

This study examined different amount of silanized nano-aluminum oxide particles reinforcement on the flexural strength of auto-polymerized acrylic resins. A recent study reported that the ultimate flexural strength of a material reflects its potential to resist catastrophic failure under a flexural load (Ben Hasan et al., 2014). Thus, this study selected the flexural strength to be the independent factor. It was found that many studies shown that the mechanical properties of the PMMA could be reinforced with various types of fibers and fillers such as polyaramid fibers, glass fibers, carbon fibers, ultra high modulus polyethylene fibers, metal powders, zirconia oxide, and sapphire whiskes (Heidari, Firouz, Izadi, Ahmadvand, & Radan, 2015). Due to its low density, ceramic filler is more preferable for reinforcement of acrylic resins than metal filler. For example, the density of sapphire (3.99 g/cm^3) is markedly less than that of Co (8.9) and Cr (7.1), so the light weight of acrylic resin denture bases is maintained. In addition, these ceramic powders are white, thus they are less likely to change the final appearance of the denture base material than metal powders (Foo et al., 2001). Furthermore, previous studies demonstrated adding metal oxides (alumina, tin, silver and zirconia) and sapphire whiskers increase flexural strength. An addition of alumina improves the properties of acrylic resin. For instance, alumina reinforcement on PMMA results in an increase in thermal conductivity and contributes to have better flexural strength, impact strength, compressive strength, tensile strength, and surface hardness of the acrylic resin (Ellakwa, Morsy, & El-Sheikh, 2008). A previous study reported that current silane coupling agents promote the bonding of resin composite to dental restorative materials (Ben Hasan et al., 2014). Moreover, the addition of 3-(trimethoxysilyl) propyl methacrylate in hydroxyapatite (HA) reinforced poly methyl methacrylate (PMMA) could escalate the mechanical properties by strengthening the chemical bonding and increasing mechanical interlocking between HA and PMMA (ISO, 1998). Consequently, the study used the application of silane on nano-alumina surface prior to being reinforced in heat cure acrylic resins.

The result of the study showed that based on two controlled groups, heat-polymerized acrylic resins had more flexural strength than auto-polymerized acrylic resins. It can be explained that lost-wax technique was the process of heat cure acrylic resin fabrication while that of auto-polymerized acrylic resin fabrication was not. Therefore, more voids could be present in auto-polymerized acrylic resin as the previous study mentioned (Polyzois, Handley, & Stafford, 1995). Furthermore, flexural strength of auto-polymerized acrylic resins could be significantly improved by adding 0.5%, 1.5%, and 10% by weight of silane-treated nano-alumina reinforcement. Likewise, Jasim BS, Ismail IJ, 2014 stated that 1 weight % treated nanofillers of alumina could increase up to 24% of flexural strength of acrylic resins (Jasim, & Ismail, 2014). Mohammed M. Gad, Ahmad M. Al-Thobity revealed that 1 and 1.5 weight % nano- Al_2O_3 increased the flexural strength of repaired denture as well (Gad, & Al-Thobity, 2021). Vojdani, Bagheri, & Khaledi (2012) presented that the conventional heat-polymerized acrylic resins that had reinforced with 2.5 weight % aluminum oxide powder significantly increased its flexural strength (Vojdani, Bagheri, & Khaledi, 2012). Chaijareenont et. al (2012) presented that 10% by weight of treated particles with silane coupling agent 18-23 μm . particles led to a 23.86 % increase in flexural strength. In addition, the flexural strength was increased when adding 10% and 15% by weight of aluminum fillers to the denture bases (Zhang, Zhang, Zhu, & Qian, 2011). According to the result, 0.5% by weight of silane-treated nano-alumina reinforcement in auto-polymerized acrylic resins could increase flexural strength superior to 3% and 10% by weight. The possible explanation could be that the lower amount of nanoparticles, the more effective. This is because of their better ability of penetration to resin matrix. Another reason could be explained that the more weight % of alumina reinforcement, the more



void formation could be detected. However, in the previous study, aluminum oxide particles were pretreated with silane, which significantly improved the bond strength of alumina particles to PMMA (Kul et al., 2016).

Improved flexural strength can also be explained using the phenomenon of transformation. Al_2O_3 which is found in many crystalline phases, and at elevated temperatures all filler particles revert to the most stable hexagonal alpha phase. Its structural application is remarkable (Foo et al., 2001). When enough stress develops and microcracks begin to propagate, the transformation phenomenon appears and reduces crack propagation. As a result, proper distribution of the filler within the matrix can stop or deflect cracks (Arkles, 1987). SEM examination of specimens reinforced with 0.5%, 1.5%, and 10% by weight of silane-treated nano-aluminum oxide (Al_2O_3) particles reinforced self-curing acrylic resins revealed a wide, equal distribution of oxide particles within the resin matrix (Figure 10). Likewise, Vojdani et al (2012) showed from the SEM evaluation that the aluminum oxide particles distribute evenly when specimens were added with 2.5% by weight of aluminum oxide. It proved that the technique of mixing particles using a magnetic stirrer is acceptable and reliable. A judicious explanation was that using the magnetic stirrer provided uniform consistency and reduced human errors (Vojdani et al., 2012). In addition, the mode of failure that was observed from the fractured surface in the SEM in most specimens was a mixture of cohesive and adhesive failure. Fracture was found between the interface of silane-treated aluminum oxide particles and PMMA (adhesive failure), and between PMMA and PMMA (cohesive failure). There was hardly any adhesive failure.

One of the limitations of this study was that surface voids were trapped in tested specimens even if the specimens were preselected using an exclusion criteria. PMMA has the tendency to generate submicrometer voids. Therefore, void formation in the specimens could influence the fracture resistance. Furthermore, as the study was conducted in an in vitro setting, material fatigue by thermocycling in water or artificial saliva with or without reinforcement could not be carried out to predict the success of material in a clinical environment. Another limitation of this study was that the SEM images revealed that larger nano-alumina particles size could be found in some areas due to a controlled manner by means of surface tension resulted in alteration of the particle-size distribution (Alhotan et al., 2021). The alumina reinforced specimens were suggested to use in less visible denture areas such as lingual flange of lower dentures or the palatal area of upper dentures because discoloration and opacity of PMMA particularly could happen when added at high concentration (Abushowmi et al., 2020).

Auto-polymerized acrylic resins reinforced with 0.5% by weight of silane-treated nano-alumina particles and unreinforced heat-polymerized acrylic resins had equal flexural strength. Fortunately, it is very useful in case of repairing dentures. Nano-alumina particles can be used to increase the flexural strength in auto-polymerized acrylic resins. Auto-polymerized acrylic resins, mostly chosen as denture repair materials, can be reinforced with 0.5 weight % silane-treated nano-alumina. After being repaired, the flexural strength could be able to maintain as the same level as their original flexural strength so that the further fracture can be prevented in the repaired dentures. Extended longevity of denture helps patient satisfied. However, further research is required to examine cytotoxicity, aging effect and physical properties such as color and surface roughness of these reinforced denture base materials before clinical application. Also, other mechanical properties of nano-alumina or alumina reinforced acrylic resin such as thermal conductivity should be considered.

5. Conclusion

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

- 1) Varying the percentage by weight of silane-treated nano-alumina reinforcement resulted in differences in the flexural strength of auto-polymerized acrylic resins. Auto-polymerized acrylic resins incorporated with low weight percentages of silane-treated nano-alumina could possibly have 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 particles had the flexural strength as high as unreinforced heat-polymerized acrylic resins.



6. References

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