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# Impact of the Addition of Salinated Nano Aluminum Silicate Fillers to Cold Cured Acrylic as a Repair Material

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# ABSTRACT

**Introduction:** Repairing of fractured heat cured denture base is a clinically significant measure, either as a temporary or permanent treatment. Many attempts were conducted to increase the strength of the repaired material by reinforcing with various composite fillers. **Objective:** The aim of this study was the use of nano-aluminum silicate fillers for improving some properties of cold cure acrylic resin that is used in repairing fracture heat cure denture base material. **Material and methods:** Total 4 groups of specimens were prepared for testing the surface roughness and the surface hardness. The 1<sup>st</sup> group of specimens was constructed from heat cured acrylic, 2<sup>nd</sup> group of specimens was constructed from cold cure acrylic only, while the 3<sup>rd</sup> and the 4<sup>th</sup> group were constructed from cold cure acrylic reinforced with 3% and 5% nano-aluminum silicate composite fillers. The mode of failure was examined with the aid of a light microscope. Statistical analysis of data was performed using SPSS version 24. **Results:** In the results, the addition of filler to cold cure acrylic resin increases the transverse strength mean value and these value increases as the concentration of fillers compared with unreinforced groups. Meanwhile, there was a statistically none significance among the tested group for surface roughness, hardness and impact strength. **Conclusion:** Addition of salinated aluminum silicate nanofillers (3%, 5%) to cold cured acrylic resin improves the transverse strength of material without changing other properties when tested as a repair material for heat cure acrylic resin.

Keywords: Nano aluminum silicate fillers, Cold cured acrylic, Repair material

## INTRODUCTION

One of the inevitable situations in the prosthodontic clinic is the fracture of the denture base that is made of heat cure polymethyl methacrylate. There are multiple causes for such a condition which is either inside the patient's mouth under occlusal load or impact failure due to accidental drop [1,2]. New denture fabrication is not always feasible, it is expensive and time-consuming. Denture repair provides a good solution in most instances [3]. The most common materials used for repairing fractured heat cured denture base are the heat cured acrylic, light cure acrylic, and cold cure acrylic.

Although various materials were used for repairing fractured denture base made of polymethyl methacrylate resin, the repaired area still represents the weakest area that is prone to recurrent fracture [4]. So attempts to improve the repairing conditions still continued. Either by modifying the design of the fractured joint, treating the fractured surface with various materials, or reinforcing the repaired materials with various fillers [4-6].

The cold cure acrylic resin is the widely used repaired material because of its easy handling, color matching, and convenience cost, but the shortcoming of this material is its low mechanical and physical properties compared to heat cure acrylic [7,8]. It was stated that the strength of cold cure acrylic is only 80% of heat cure polymer [9]. It was supported by studies conducted on transverse strength and impact strength of cold cured compared to heat cured resin [10,11]. The ideal repair materials should replace the deficiency in the mechanical and physical properties of heat cure denture base material [12].

So by improving the properties of cold cure acrylic to cope with heat cure acrylic still represent the subject of growing researches, especially by reinforcing the material with various fillers [4]. Inorganic fillers have their share in conducted studies [4,13]. Aluminum silicate is one of the inorganic filler, its biocompatibility was proved, its aesthetic value has a white color, in addition to its ability to strengthen the heat polymerized polymethyl methacrylate [14,15].

The aim of this study was to evaluate the transverse strength, impact strength, surface hardness, and surface roughness of cold cured acrylic as a repair material after the addition of nano aluminum silicate filler in two concentrations 3% and 5% by weight.

### PATIENTS AND METHODS

### Surface Modification of Nano Aluminosilicate (Al,SiO<sub>5</sub>R) with 3-methacrlyoylpropyl trimethoxysilane (MPS)

An ethanol (99.8 vol%) (Carloerba, Rodano, Milano, Italy) was diluted with deionized water to prepare a 100 milliliter of ethanol aqueous solution (70 vol%). The pH was adjusted to 4.5 by titrating with 99.9% acetic acid (Carlo Erba, Milan, Italy), the adjusted pH was measured with the aid of a pH meter (ORION 420A, Orion Research Inc., Boston, MA, USA). Then, 1.5 g of MPS was added respectively into the ethanol aqueous solution and stirred well. This MPS solution was stored in a 100 mL polyethylene cup with a cover and allowed 10 min for hydrolysis and silanol formation. Then 30 g of nano aluminum silicate powder (The British Drug Houses LTD.B.D.H. Laberayory Chemical Group Poole England) was added into MPS solution with stirring until the solution was completely evaporated, and left for 2 weeks at room temperature for complete dryness [16]. The (FTIR) spectrophotometer (Bruker Tensor27, Europe) analysis for the characteristic vibrations of functional groups of MPS was used to ensure the attachment to aluminum silicate fillers [17].

### **Specimens' Preparation**

Two different metal patterns were constructed by cutting metal copper alloy plate in desired shape and dimension. For the impact strength test, a bar-shaped specimen with  $(80 \times 10 \times 4)$  mm in length, width, and thickness, respectively [18]. For hardness test, surface roughness test, and for transverse strength: A bar-shaped specimen with  $(65 \times 10 \times 2.5)$  mm in length, width, thickness, respectively [19]. Mold for specimens was prepared as for the conventional flashing technique for complete denture (Figure 1).

#### **Proportioning and Mixing of Acrylic**

Heat cure acrylic powder was mixed with the monomer (Vertex, Netherland) according to the manufacturer instruction. An electronic balance with an accuracy of (0.0001) was used for weighing powder and including cold cure acrylic resin (Veracril, Colombia) and fillers. Salinized alumina silicate with percentages 3%, 5% was weighted separately. Then added to cold cure acrylic powder and thoroughly mixed with a mortar and pestle until a uniform color is achieved which indicate the equal distribution of particles [13]. The proportioning of acrylic powder/liquid ratio and the percentage of added filler was revealed in Table 1. Heat cured specimens were cured by short curing cycle.

Materials	Amount of Al <sub>2</sub> SiO <sub>5</sub> (g)	Amount of PMMA (g)	Amount of monomer (ml)
Cold cure acrylic	0.00	6.00	3
Cold cure acrylic + $3\%$ Al <sub>2</sub> SiO <sub>5</sub>	0.18	5.82	3
Cold cure acrylic + 5% $Al_2SiO_5$	0.30	5.70	3

Table1 The proportioning of acrylic powder/liquid ratio and the percentage of added filler

#### **Specimens Preparation**

A total of 160 specimens were divided into 2 groups. In each group, 80 specimens were divided equally for surface roughness and surface hardness test. The other 80 specimens were used for the impact and the transverse strength with 40 specimens for each test. Every 40 specimens were subdivided into 10 specimens for each variable in this study.

The specimens for impact and transverse strength were made of heat cured acrylic, 20 specimens without addition were used as a control group, the remaining specimens were cut with the aid of separating disc in the middle into two sections, that returned back into mold space to ensure the measurements, then both edges of the sections were reduced to result in 3 mm gap, after that 2 horizontal slopes of 5 mm length and 1 mm width at the end of each section were reduced that represent the repair site [20].

The cold cure acrylic repair materials were mixed in a glass container and covered, until reach dough stage. The gap site was wetted with a drop of cold cure monomer for 1 minute, and then the dough material was applied to the gap space, with the aid of flasking nylon sheet, the two portions of the metal flask were closed together and placed under the hydraulic press period for one hour in room temperature (Figure 1).



Figure 1 Specimens' preparation A-metal pattern; B-specimens for impact and transverse strength; C-repaired specimens, 1-repaired with cold cured, 2-repaired with 3% Al<sub>2</sub>SiO<sub>5</sub> reinforced cold cured resin, 3-repaired with 5% Al<sub>2</sub>SiO<sub>5</sub> reinforced the cold cured the resin

#### **Finishing and Polishing**

Specimens were finished with the straight handpiece and acrylic bur removing the flashes, stone bur to obtain smooth edges, followed by (125) grain size sandpaper with low speed. All the specimens were finished, even specimens for surface roughness for standardizing the surfaces of the specimens [21]. All the specimens were immersed in distilled water at 37°C for 48 hours before being tested [19].

#### **Mechanical Properties**

**Surface roughness test:** The profilometer device (Surface roughness tester SRT-6210, England) was used to study the effect of  $Al_2SiO_5$  filler reinforcement on the microgeometry of the test surface. This device is supplied with a sharp stylus made from the diamond to measure the surface. An average of 3 readings was calculated from the readings on a standardized area of each specimen.

**Surface hardness test:** The durometer hardness tester shore D type (hardness tester-Th210, time group Inc. Italy) which is suitable for an acrylic resin material. The instruments consist of blunt-pointed indenter attached to the calibrated scale that is graduated from 0 to 100 units. An average of 3 readings was calculated from measurements on standardized areas of each specimen.

**Transverse strength:** The Instron testing machine (Laryee, WDW-50, CHINA) to measure a 3 point bending test for specimens were used. The full-scale load was 50 KN, and the load was applied by a rod placed centrally on the specimens placed between 2 parallel supports until fracture.

**Impact strength:** The Charpy type impact testing instrument (Impact tester N. 43-1, INC. USA.) was used. Each specimen was fixed at its ends, and then stricken by 2 joules free-swinging pendulum which was released from a fixed height in the middle. The Charpy impact strength was calculated in KJ/m<sup>2</sup>.

**Mode of failure:** The specimens of the impact strength and transverse strength were examined at the fractured side to declare the mode of failure with aid of a light microscope (Sinwon, China). The magnification used was 50X.

#### RESULTS

FTIR spectrum obtained from aluminosilicate composite indicated that FTIR test before and after silanation evaluate the differences in the curve which represent the presence of functional groups, as seen in Figure 2. Another FTIR examine the cold cured resin before and after the addition of the silanated aluminosilicate to evaluate the reaction of fillers particles with a cold cured polymer, as seen in Figure 3.



Figure 3 FTIR spectrum of cold cured resin before and after the addition of silanated aluminum silicate

271.5

100

In this study, the comparison was done between heat cured acrylic resin specimens as a control group (A), repaired with cold cured acrylic resin without addition (B), and reinforced cold cured acrylic containing salinized aluminum silicate composite fillers in concentration 3% (C), and 5% (D). All data were subjected to one-way analysis of variance (ANOVA) and followed by multiple comparisons by the Bonferroni test method.

Table 2 shows the mean and standard deviation values of transverse strength, impact strength, surface hardness, and surface roughness. The highest mean values were for heat cured acrylic specimens among all tested groups. While for the surface hardness test surface roughness test, and transverse strength tests there is an increase in the mean values as the concentration of fillers increase when compared to the unreinforced cold cure groups. In the ANOVA test, there are a highly significant difference among the tested groups of transverse strength while the remaining tested groups have a statistically non-significant value (p=0.000).

Groups		No.	<b>Surface Hardness</b>		Surface Roughness		<b>Impact Strength</b>		<b>Transverse Strength</b>	
			Mean	SD	Mean	SD	Mean	SD	Mean	SD
He	eat cure	10	76.3	3.53	0.001	0.024	7.074	60.4	0.047	0.195
Repair	Cold cure	10	73	4.97	0.004	0.0246	7.423	24	0.044	0.164
	Cold and 3% Al <sub>2</sub> O <sub>3</sub>	10	73.3	3.04	0.004	0.0255	5.719	31	0.042	0.148
	Cold and 5% Al <sub>2</sub> O <sub>3</sub>	10	75.5	3.57	0.005	0.0266	5.811	30.4	0.035	0.131
ANOVA Test	F-test		61.7	49	4.	013	0.6	7	2.	.12
ANOVA Test	p-value		0.00	0*	0.	015	0.57	76	0.	048

In Tables 3 and 4 for surface hardness and surface roughness tests, the multiple comparisons between groups show there is the non-significant difference.

Samples		Mean Difference	Std.	Sig.
Heat cure	Cold cure	3.33	1.814	0.049
	Cold with 3% Al <sub>2</sub> O <sub>3</sub>	3.00	1.814	0.048
	Cold with 5% Al <sub>2</sub> O <sub>3</sub>	0.78	1.814	0.671
Cold cure	Cold with 3% Al <sub>2</sub> O <sub>3</sub>	-0.33	1.814	0.855
	Cold with 5% Al <sub>2</sub> O <sub>3</sub>	-2.56	1.814	0.048
Cold with 3% Al <sub>2</sub> O <sub>3</sub>	Cold with 5% Al <sub>2</sub> O <sub>3</sub>	-2.22	1.814	0.049

#### Table 3 Multiple compressions (Bonferroni test) among all tested groups for indentation hardness mean values

Table 4 Multiple compressions (Bonferroni test) among all tested group for surface roughness

San	Mean Difference	Std.	Sig.	
Heat cure	Cold cure	0.0006	0.00196	0.761
	Cold with 3% Al <sub>2</sub> O <sub>3</sub>	0.0015	0.00196	0.448
	Cold with 5% $Al_2O_3$	0.0026	0.00196	0.192
Cold cure	Cold with $3\% \text{ Al}_2\text{O}_3$	0.0009	0.00196	0.648
	Cold with 5% Al <sub>2</sub> O <sub>3</sub>	-0.0020	0.00196	0.314
Cold with 3% Al <sub>2</sub> O <sub>3</sub>	Cold with 5% Al <sub>2</sub> O <sub>3</sub>	-0.0011	0.00196	0.578

In Table 5 for impact strength, although shows a decrease in impact strength mean values of test groups compared to the control group, there is a significant decrease in mean difference between the reinforced  $3\% \text{ Al}_2 \text{SiO}_5(\text{C})$  compared to control group (A) and highly significant decrease in mean difference between the reinforced  $5\% \text{ Al}_2 \text{SiO}_5(\text{D})$  compared to control group (A). But there are non-significant differences in mean value comparisons between tested groups.

Table 5	5 Multiple	compressions	(Bonferroni	test) among	all tested	group fo	r impact s	trength
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Sam	ıples	Mean Difference	Std.	Sig.
Heat cure	Cold cure	0.031	0.0192	0.116
	Cold with 3% Al <sub>2</sub> O <sub>3</sub>	0.047*	0.0192	0.02
	Cold with 5% Al <sub>2</sub> O <sub>3</sub>	0.064*	0.0192	0.002
Cold cure	Cold with 3% Al <sub>2</sub> O <sub>3</sub>	0.016	0.0192	0.411
	Cold with 5% Al <sub>2</sub> O <sub>3</sub>	0.033	0.0192	0.049
Cold with 3% Al <sub>2</sub> O <sub>3</sub>	Cold with 5% Al <sub>2</sub> O <sub>3</sub>	0.017	0.01923	0.382

In Table 6 it was clear that there is a highly significant decrease in all tested groups in the mean values of transverse strength compared to control one. Also, there is a significant increase in the mean values of reinforced cold cure test groups compared to the unreinforced group, while there is a non-significant difference between the reinforced test groups as in Table 6.

<b>Table 6 Multiple</b>	compressions	(Bonferroni test	) among all tested	l group for tran	sverse strength
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Sample	Samples		Std	Sig.
Heat cure	Cold cure	36.400*	2.92954	0.000
	Cold with 3% Al <sub>2</sub> O <sub>3</sub>	30.000*	2.92954	0.000
	Cold with 5% Al <sub>2</sub> O <sub>3</sub>	29.400*	2.92954	0.000
Cold cure	Cold with 3% Al <sub>2</sub> O <sub>3</sub>	-6.400*	2.92954	0.036
	Cold with 5% Al <sub>2</sub> O <sub>3</sub>	-7.000*	2.92954	0.022
Cold with 3% Al <sub>2</sub> O <sub>3</sub>	Cold with 5% Al <sub>2</sub> O <sub>3</sub>	-0.600	2.92954	0.839

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# Mode of Failure

The mode of failure as examined by light microscope reveals rough surface in all fractured repaired groups which indicate adhesion failure type as shown in Figures 4 and 5.



Figure 4 Adhesive fracture under light microscope impact strength, A-specimens repaired with cold cured resin; B-specimens repaired with 3% Al<sub>2</sub>SiO<sub>5</sub> reinforce cold cured resin; C-specimens repaired with 5% Al<sub>2</sub>SiO<sub>5</sub> reinforce cold cured resin; A: conventional heat cure denture base resin, B: High-impact heat cure denture base resin, C: Glass fiberreinforced heat cure denture base resin



Figure 5 Adhesive fracture under light microscope transverse strength A-specimens repaired with cold cured resin

B-specimens repaired with 3%  $Al_2SiO_5$  reinforce cold cured resin C-specimens repaired with 5%  $Al_2SiO_5$  reinforce cold cured resin; A: conventional heat cure denture base resin, B: high-impact heat cure denture base resin, C: glass fiber-reinforced heat cure denture base resin

#### DISCUSSION

Clinical methods used for repairing fractured heat cured denture base material have a significant measure either as a temporary or permanent treatment [3]. The ideal properties of the material used as a repair material should restore the original strength and color of the fracture site [22]. Many researchers try to increase the strength of cold cure acrylic as a repaired material by reinforcing it with various composite fillers [5,22].

In this study, aluminum silicate nanoparticles were added as a filler material to cold cure acrylic to be used as a repair material. These fillers have a white color which doesn't affect the esthetic. Two concentrations 3% and 5% were selected since they improve the strength of heat cured acrylic denture base material [15].

The results of the surface hardness of the cold cure acrylic were lower than heat cured acrylic and this might be due to incomplete polymerization of cold cure acrylic compared to that of heat cure acrylic [23]. Also as the percentage of inorganic filler added to cold cure acrylic increases, it raises the mean value of surface hardness. This might be due to the higher value of hardness for the dispersed particle of aluminum silicate fillers more than polymethyl methacrylate polymer. This results in agreement with the previous studies and as the later added different inorganic filler to the polymethyl methacrylate polymer in different concentrations [15,24,25].

For the results surface roughness, mean values of heat cured acrylic specimens were lesser than that of cold cure acrylic as proved by previous authors [26]. The results of the reinforced cold cure specimens revealed higher mean values of surface roughness as compared with the unreinforced cold cure specimens. While the addition of a higher percentage of fillers to cold cure results in a statistically non-significance increase in surface roughness this might

be attributed to the differences in the size of filler particles and acrylic denture base matrix and this was agreed with Abdulhamed, et al. [25].

For impact strength, it was clear that there is a slight reduction in the impact strength between the specimens repaired with reinforced cold cured resin compared with specimens repaired with cold cured resin only. This could be explained by the presence of composite filler which makes the cold cured acrylic harder and brittle; so reduces the fracture toughness [27]. These results agreed with results of Gad, who added  $ZrO_2$  nanofillers to cold cured repaired material bevel edges at different concentrations [22].

The results of transverse strength mentioned that there was a statistically highly significant increase in the mean values for heat cure specimens (control) as compared to repaired test specimens and this was approved by a study conducted by Filho, et al., and Anasane, et al. [28,29]. The possible explanation was because of increasing the amount of residual monomer in cold cured acrylic compared to heat cured one lower the mechanical properties in cold cure acrylic including the transverse strength.

Addition of aluminum silicate nanofillers to cold cured acrylic increases the mean value of the transverse strength of the repaired specimens as compared with specimens repaired with cold cured alone, and the transverse strength mean value of cold cured acrylic increased with increasing of the number of fillers, which is in agreement with Rohit, et al., who stated that metal oxide fillers with saline coupling agent will reduce the voids between the resin matrix and the filler which add more strength to the polymer mass [30]. This result agreed with Gad, et al. and Kareem, et al. [22,31].

#### CONCLUSION

Within the limitation of this study, it could be concluded that salinated aluminum silicate nanocomposite filler could be used to improve the properties of cold cured acrylic as a repair material. Also, the addition of salinated nano aluminum silicate fillers to the cold cured acrylic resin in concentration 3% and 5% increases the surface hardness, roughness, and transverse strength significantly, but decreases the impact strength especially at 5% concentration.

#### DECLARATIONS

### **Conflict of Interest**

The authors have disclosed no conflict of interest, financial or otherwise.

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