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The Reinforcement Effect of Nano-Zirconia on the Flexural Strength and Impact Strength of Microwaved and Heat Cured Acrylic Resin

Amal A. Eid[1*], Mohamed A. Souhail[2], Heba E. Eltayeb[3]

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azhardentj@azhar.edu.eg

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ABSTRACT

Purpose: This study was conducted to evaluate the effect of adding nano-zirconia with different concentrations on the flexural and impact strength of microwaved and heat cured acrylic resin. **Materials and Methods:** The silanized nano-ZrO_2 particles (nano-zirconia) were added to PMMA powder for both heat polymerized and microwaved acrylic resin. A total of 160 specimens were prepared from both types of acrylic resin (n=80). For both types of acrylic resin, specimens for each strength test were subdivided into 4 subgroups (n=10): a control group (with no fillers) and three reinforced groups with 3 concentrations (3%, 5% and 7%) wt. % of nano-ZrO_2 fillers. Flexural strength was measured using an Instron mechanical testing machine and impact strength was measured by Charpy tester. **Results:** For the heat cured resin 3% nano-zirconia showed the highest mean flexural strength value followed by 5 % nano-zirconia. For the microwaved acrylic resin, 3% nano-zirconia showed the highest mean flexural strength value while 5% and 7% led to decrease in the flexural strength value. For the impact strength results, increasing nano-zirconia percentage led to decrease the impact strength for both types of acrylic resin. **Conclusions:** The incorporation of 3% and 5% nano-zirconia into heat cured acrylic resin improved its flexural strength but 7% of nano-zirconia addition had a deteriorating effect on the flexural strength of both types of resin investigated. Moreover, the addition of nano-zirconia to both types of acrylic resin had a negative effect on their impact strength.

KEYWORDS

Silanized (nano-ZrO2), PMMA, flexural strength, impact strength.

- Paper extracted from Master thesis titled "The Reinforcement Effect of Nano-Zirconia on the flexural strength and impact strength of Microwaved and Heat cured acrylic resin"

1. Demonstrator in Dental Biomaterials Department, Sinai University, North Sinai, Egypt.
2. Assistant Professor of Dental Biomaterials and Head of Dental Biomaterials Department, Faculty of Dentistry, Sinai University, North Sinai, Egypt.
3. Assistant Professor and Head of Dental Biomaterials Department, Faculty of Dental Medicine for Girls, Al-Azhar University, Cairo, Egypt.

* Corresponding author email: amalabdelaziz371@gmail.com

INTRODUCTION

PMMA has been widely used in construction of denture bases. In spite of the popularity of PMMA which includes aesthetic and low cost, it has some problems such as; brittleness, low modulus of elasticity, low impact strength and low flexural strength that lead to fracture of prosthesis in-situ and ex-situ(1,2).

Microwave energy processing was introduced as a substitutive technique to the traditionally cured acrylic resin(3). The American dental association specification reported that microwave energy used for polymerization of the acrylic resin produces dentures with higher mechanical strength than conventionally cured one (4).

In comparison, the curing of heat-cured acrylic resin is usually done in a water bath for at least 9 hours, while using of the microwave energy decreased the time up to three minutes so this method is fast and clean, and the final denture has better accuracy with better adaptation(5). Regardless of the benefits of microwave polymerization, it isn't regularly used because of its difficult availability and cost of the material (6).

Recently, several attempts have been made to incorporate nano-fillers into PMMA to improve its mechanical properties. Nano-zirconia has recently been noted for their excellent biocompatibility, good aesthetics, good wear and corrosion resistance. In addition to these features, nano-zirconia has good toughness and mechanical strength properties (7, 8).

Hence, the purpose of this study was to evaluate and compare the flexural and impact strength of heat cured acrylic resin and microwaved acrylic resin after its reinforcement with nano-zirconia in different concentrations.

MATERIALS AND METHODS

Two types of acrylic resins were used in this study (Bracon Ltd , UK), heat cured acrylic resin (Oracryl heat cured) and microwaved acrylic resin (GC Acron MC). Nano-zirconia particles were prepared by the Nano technology center (Nano-tech Egypt for photo electronics, city of 6th of October, Egypt). Nano-zirconia with different concentrations 3wt%, 5wt% and 7wt% each were added to the heat cured and the microwaved resins then the flexural strength and impact strength were measured.

Characterization of nano-zirconia particles

Transmission Electron Microscope (TEM) at the Regional Centre for Mycology and Biotechnology (RCMB) Al- Azhar University was used to determine the average particle size of nano-zirconia. The nano-zirconia powder was dispersed in distilled water. A drop of the solution was placed on the carbon coated copper grids (CCG) and then dried by evaporating the water at room temperature. The nano-zirconia particles were then deposited onto the TEM grid, having a carbon support film. The grid was then dried before mounting on the TEM.

Scanning electron microscope (SEM) examination

The surface of the specimens was examined by utilizing a high vacuum mode of SEM (Model: JSM-5500 LV; JEOL Ltd -Japan) at the Regional Center of Mycology and Biotechnology, Cairo, Egypt. SEM evaluation was done with an acceleration voltage of 20 kV and the working distance was ~10 mm with a 3.5 spot size. A sputter coating machine (SPI-Module, USA) was used to apply a gold coating to the specimens to overcome its non-conductive nature. Then by using a double sided adhesive tape; the specimens were mounted on a metallic stub to be tested.

Surface modification of nano-zirconia

In the present study, nano-zirconia powder has been prepared. Reactive groups were introduced onto the fillers surface by a reaction of 3 Trimethoxysilylpropyl methacrylate (TMSPM), (SIGMA-ALDRICH, Germany) with nano-zirconia of 99.5% purity (Nano-tech Egypt for photo electronics, city of 6th October, Egypt), this treatment was done to promote adequate adhesion between the nanoparticles and the resin matrix.

This was done by dissolving TMSPM in acetone to ensure that it would coat the surfaces of the nano-zirconia particles uniformly. The prepared solution was then mixed with the nano-zirconia powder by in a magnetic stirrer for 60 min. Moreover, the solvent was removed by a rotary evaporator under vacuum at 60°C and 150 rpm for 30 min. After the sample was dried, it was heated at 120°C for 2 hours and left to cool to obtain the surface-treated nano-zirconia (9).

Nano-zirconia incorporation into acrylic resin

The silanized nano-zirconia powder was incorporated to both types of acrylic resins separately with the same procedure. Nano-zirconia and PMMA were pre-weighed using an electronic balance (Scaltec, SBC series, India), so that the nano-zirconia concentrations were 3%, 5% and 7% by weight. Pre-weighted nano-zirconia powder were separately added to both acrylic resins powder and thoroughly mixed using a mortar and pestle to promote an equal distribution of particles then the powder was mixed with the liquid with a ratio of 2.5:1 by weight as listed below in (Table 1) (9,10).

Table (1) *Percentages of polymer, monomer and nano-zirconia powder used in the study:*

nano-zirconia percentage	Amount of nano-zirconia	Amount of polymer	Amount of monomer
0%	0	16g	6.4g
3%	0.48g	15.52g	6.4g
5%	0.8g	15.2g	6.4g
7%	1.12g	14.88g	6.4g

Specimen preparation

160 specimens were prepared from both types of acrylic resin (n=80) then they were equally divided for the flexural and impact strength tests (n=40). For both types of acrylic resin, specimens for each strength test were subdivided into 4 subgroups (n=10): a control group (with no fillers) and three reinforced groups with 3 concentrations (3%, 5% and 7 %) of nano-zirconia fillers.

For the flexural strength test, a specially fabricated split teflon mold was used to prepare a wax pattern in the dimensions of 65 mm × 10 mm × 2.5 mm following the ISO/DIS 1567 international standard. Wax patterns were used to prepare eighty specimens for the test; forty specimens for each type of the acrylic resin, while specimens for the impact strength test were prepared using a split teflon mold with dimension 65.5 mm × 12.7 mm × 3.75 mm, according to ADA specification NO.12.The wax patterns were used to prepare both the heat cured and microwaved acrylic resin specimens (11).

Heat cured Acrylic resin processing

The wax patterns were invested in a metal dental flask filled with dental stone (GC Fujirock EP, Belgium). Once the wax pattern was completely set, it was removed from the flask. Separating medium (cold mold seal; Dental products of India (DPI)) was applied to the negative dental stone impression.

According to manufacturer's instructions, the powder of the heat-cured acrylic resin was mixed with the monomer with a stainless steel spatula in a sealed glass jar to avoid the loss of the monomer by evaporation. The mix was then packed with slightly overfilling the molds cavity to compensate for polymerization shrinkage. The flask was compressed under manual machinery press and processed for 8 hours in a water bath at 74 °C and then the temperature was increased to 100 °C for 1 hour in the thermal curing unit (UGIN, Polymerisateur A97, France).

After curing, the flask was removed from the water bath and was allowed to bench cool at room temperature to avoid the development of thermal internal stresses within the specimens upon premature opening of the flask. After deflasking, the specimens were finished using finishing stone and polished with wet sand paper of 60 and 80 grit. The final dimensions of the cured specimens were ensured using a digital caliper. Specimens were then stored in distilled water at 37°C for 48 hours before testing (9).

Microwaved acrylic resin curing

According to the manufacturer's instructions, microwaved acrylic resin powder was added to the liquid without mixing to prevent entrapment of air bubbles. The mixture was kept in a sealed glass jar during the reaction to avoid monomer evaporation .When the mix was packed in the dough stage into the mold cavity in special non-metal flask. Then the non-metal flask was closed under manual machinery press and inserted into the microwave oven (Galanz, China) with a rotating table for 3 minutes at a power of 500 W (12).

Prior to deflasking the flasks were bench cooled at room temperature to avoid deterioration of the samples. Premature opening may cause development of thermal internal stresses within the specimens. The specimens were then deflasked, finished and polished with the same procedure previously mentioned with the heat-cured acrylic resin. Specimens were inspected for any irregularity. Faulty specimens were discarded and final specimens were selected for each group. Specimens were then stored in distilled water at 37°C for 48 hours and then tested (10).

Flexural strength test

Instron testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA) was used for testing the specimens at across head speed of 5mm / min. The specimens were placed between 2 parallel supports 50 mm apart, with a load-cell of 5kN. The force was centrally applied to the specimen until fracture. The flexural strength was then calculated by computer software using the following formula:

$$\mathbf{FS = 3FL/ 2wh^2}$$

Where, **F** is the maximum load; **L** is length of span; **w** is the width of the specimen; **h** is the height of the specimen (9).

Impact strength test

The impact strength of the specimens was measured by CHARPY impact testing machine (Digital Izod/Charpy Impact tester, BEIJING JINSHENGXIN, India).At the midline of the samples, a V-shaped notch of 2 mm was prepared with notch cutter (VEB Frasmaschinenwerk Leipzig, Germany) .The samples were clamped horizontally between two metal fixtures so that the middle of the sample at the notch coincided with the striking pendulum but from the opposite side. The pendulum of (2 joule) was dropped from a specific height to record the zero reading, the pendulum dropped again to impact the specimen. The energy absorbed by the material was obtained by difference between these reading values. Impact strength (IS) in KJ/m^2 was then calculated using the following formula(13, 14):

$$\mathbf{Impact\ strength\ (IS) = E/t.w}$$

Where, **E** is the absorbed energy, **t** is the sample thickness; **w** is the remaining width at the notch base.

Statistical analysis

Data management and statistical analysis were performed using the Statistical Package for Social Sciences (SPSS) version 18. Numerical data were summarized using means, standard deviations, minimum, maximum and confidence intervals. Data were explored for normality by checking the data distribution and using Kolmogorov-Smirnov and Shapiro-Wilk tests. Comparisons between the 2 groups with respect to normally distributed numeric variables were compared by independent t test. Comparisons between different concentrations were done by one way analysis of variances (ANOVA) test and pairwise difference were detected by the Tukey's post hoc test. All p-values are two-sided. P-values ≤ 0.05 were considered significant.

RESULTS

Scanning electron microscope

SEM examination was done under (3000X) Mm. This test was done for a random sample from each tested group (control (0%), 3%, 5% and 7%) of both heat cured and microwaved acrylic resin as shown in (Fig. 1) and (Fig. 2) . These images showed that the tendency for agglomeration increased with increase the concentration of nanoparticles. At higher concentrations, homogeneous distribution of nano-zirconia became difficult and this explains weaker mechanical properties at high concentrations.

Flexural strength test results

The flexural strength results for the heat cured and the microwaved acrylic resin specimens are summarized in (Table 2). For the heat cured resin, the results revealed that the mean flexural strength values of 3% (80.16±2.24) and 5 % (78.20±4.74) nano-zirconia groups were statistically significantly higher than the control group (72.86±7.40) (p=0.00). However, there was no significant difference between the two groups 3% and 5%. Moreover, the flexural strength of 7% nano-ZrO$_2$ group (70.49±3.16) showed non-significant difference with the control. For the microwaved acrylic resin, the mean values for 3% concentration (116.55±30.18) and 5% concentration (83.40±3.83) showed non-significant difference with the control (101.32±19.26) whereas; the 7% concentration (77.49±8.51) showed statistically significant lower mean value than the control. Moreover, 5% concentration was not significantly different than 7% concentrations. Microwaved acrylic resin showed significant increase in the flexural strength mean values for the same concentrations than the heat cured acrylic resin (p<0.05).

Impact strength test results

The impact strength results for the heat cured and the microwaved acrylic resin specimens are summarized in (Table 3). For the heat cured acrylic resin, the results showed that the mean impact strength values of 5% (5.91±1.44) and 7 % (4.66±.50) nano-ZrO2 groups were statistically significantly lower than the control group (7.71±.70) (p=0.00).With respect to microwaved acrylic resin, the mean impact strength values for all concentration of nano-ZrO2 (3% (8.61±.37), 5% (7.25±.65) and 7% (5.40±.83)) were statistically significantly lower than the control group (10.63±1.47) (p=0.00). Microwaved acrylic resin showed significant increase in the impact strength mean values for all concentrations than the heat cured acrylic resin (p<0.05) .

Table (2) *Comparison between the Flexural strength (MPa) of the microwaved and heat cured acrylic resin at each concentration.*

Concentration	Microwaved		Heat cured		Mean Difference	t-value	P-Value between group
	Mean	Std. Dev.	Mean	Std. Dev.			
0%	$101.32^{a,b}$	19.26	72.86^{b}	7.40	28.46	4.36	0.00*
3%	116.55^{a}	30.18	80.16^{a}	2.24	36.40	3.80	0.004*
5%	$83.40^{b,c}$	3.83	78.20^{a}	4.74	5.20	2.69	0.015*
7%	77.49^{c}	8.51	70.49^{b}	3.16	7.00	2.43	0.032*
P-Value within the same group	*0.00		*0.00			-	

*Significance level p≤0.05, *significant Tukey's post hoc test: within the same column, means sharing the same superscript letter are not significantly different*

Table (3) *Comparison of the impact strength (KJ/m^2) of microwaved and heat cured acrylic resin at each concentration.*

Concentration	Microwaved		Heat cured		Mean Difference	t-value	P-value between group
	Mean	Std. Dev.	Mean	Std. Dev.			
0%-	10.63^a	1.47	7.71^a	.70	2.91	5.66	0.00*
3%-	8.61^b	.37	7.33^a	.75	1.28	4.82	0.00*
5%-	7.25^c	.65	5.91^b	1.44	1.34	2.68	0.020*
7%-	5.40^d	.83	4.66^c	.50	0.74	2.41	0.029*
P-value within the same group	*0.00		0.00*			-	

*Significance level p≤0.05, *significant Tukey's post hoc test: within the same column, means sharing the same superscript letter are not significantly different*

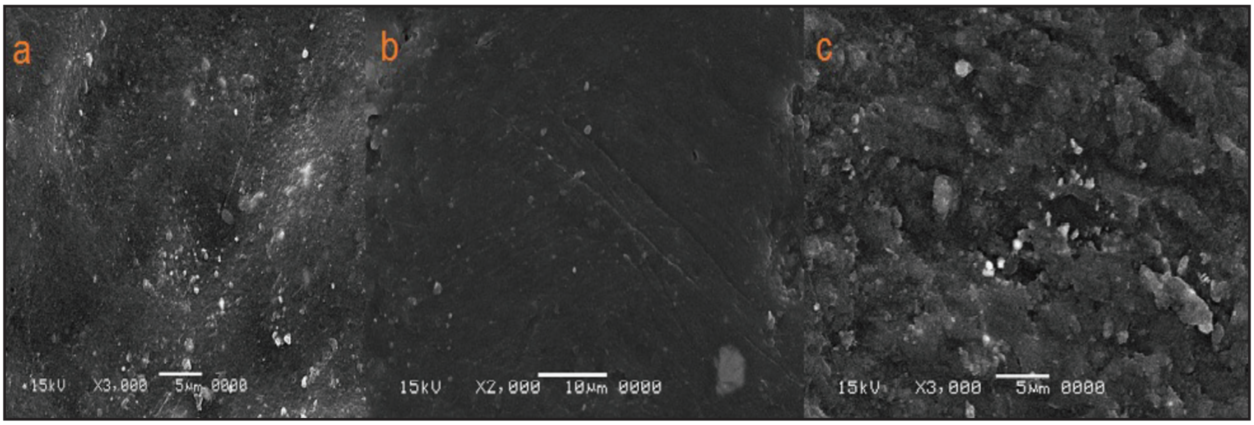

Figure (1) SEM micrographs of heat-polymerized acrylic resin specimens. (a) 3% nano-zirconia group. (b) 5% nano-zirconia group. (c) 7% nano-zirconia group.

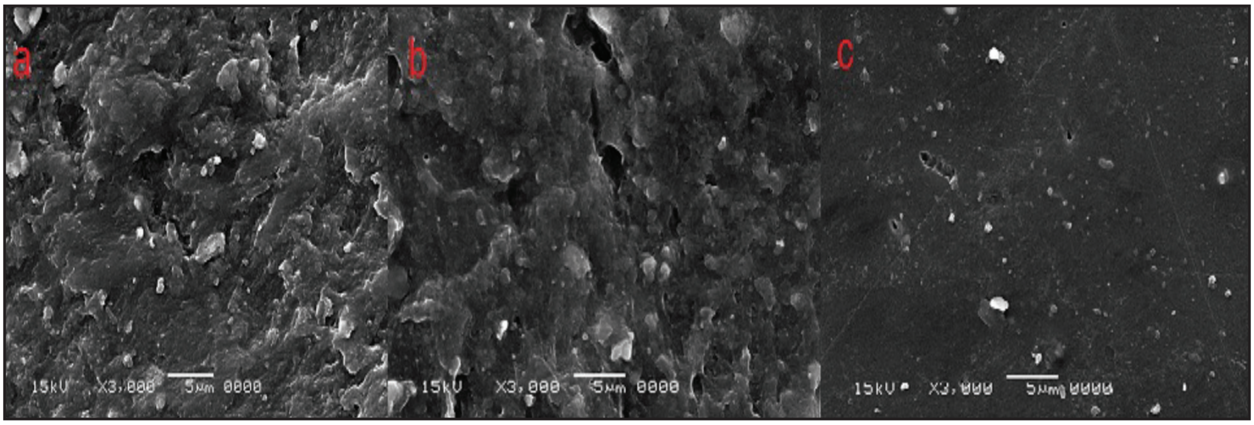

Figure (2) SEM micrographs of microwaved acrylic resin specimens. (a) 3% nano-zirconia group. (b) 5% nano-zirconia group. (c) 7% nano-zirconia group.

DISCUSSION

Flexural strength

The occurrence of denture fracture has been reported in the literature with high rate which compromises the life span of the prosthesis (15). Flexural strength test has been helpful in numerous studies that evaluate denture base acrylic resins, as it is easy to perform and simulates the stress that is applied on the prosthesis during chewing (16).

For the heat cured acrylic resin, addition of nano-zirconia fillers 3% and 5% increased the flexural strength significantly compared to the control. Addition of more nano-zirconia to 7% showed the least flexural strength mean value with a non-significant difference compared to control group (Table 2). These results were in accordance with previous studies which attributed the increase in the flexural strength to the good dispersion of the nanoparticles. As their very fine size allows them to penetrate in between the chains of the polymer and occupy spaces between them. This restricts the movement of the macromolecular chains leading to an increase in the strength and rigidity of the resin. Therefore, the fracture resistance and flexural strength are improved (10, 17, 20).

In the present study, the particle size of nano-zirconia fillers used was 9 nm as detected by TEM which was much smaller than that of the powder resin particles confirming previous explanation. However, the reduction in the flexural strength at 7% might be because the addition of nano-zirconia fillers till 5% filled all the spaces between the PMMA chains so any excess fillers added would cause detachment of PMMA chains and the force between them is weakened. This would lead to decrease in the fracture resistance and mechanical properties of the polymer. Moreover, aggregation of nano-zirconia particles due to its high surface area might have led to a decrease in the flexural strength because of the micro fractures that weakened the acrylic resin at this percentage as detected by the SEM (Fig. 1).

The present study was in disagreement with other previous studies which reported that the increase in concentrations of the nano-fillers increased mechanical properties of the heat cured acrylic resin due to the high interfacial shear strength between the nano-fillers and the resin matrix which in turn prevented crack propagation, also complete wetting of the nano-fillers by the resin might have led to an increase in the flexural strength (15, 18).

For the microwaved acrylic resin, the results of the present study showed that there was a non-significant increase in the flexural strength at 3% and a non-significant decrease at 5% and significant decrease at 7% concentrations compared to the control. Moreover, the flexural strength decreased with the increase in concentration of nano-zirconia which was in agreement with a study that attributed this result to the nano-filler ratio which acted as impurities within the resin causing decrease in the degree of conversion and leading to reduction in the flexural strength of the polymer (19). This was in disagreement with another study which stated that the flexural strength increased with the increase in the concentration of nanoparticles (4).

As seen in the SEM micrographs (Fig. 2), the percent of aggregation increased with increasing the concentration of nano-zirconia. Therefore, addition of low percentage of the nano-zirconia should be used to ensure that they would be homogenously distributed in the resin without aggregation. Moreover, the size, shape and distribution of filler particles in the polymer matrix play a major role on its mechanical properties (17).

In the current study, the microwaved cured acrylic resin showed higher flexural strength in comparison to the conventionally cured one. This agrees with the Specification of American Dental Association (ADA) which reported that, curing of acrylic resin with microwave energy produces dentures with higher strength properties and greater resistant to mechanical loading than conventionally cured materials (4). Results of the present study agreed with previous researches which stated that the microwaved acrylic resin had higher flexural strength than conventional heat polymerized acrylic resin (12, 19, 3). Another research observed an increase in the flexural strength of the evaluated resin in both added ratios of zirconia nanoparticles (1% & 3%) whether the specimens were processed in conventional water-bath or microwave energy, the increase in the flexural strength was explained by transformation toughening mechanism of ZrO_2 (4).

In disagreement with the current study some researchers reported that the heat cured acrylic resin had higher flexural strength than the microwaved one with or without addition of nanoparticles. They confirmed these results by the presence of a direct relationship between the concentration of cross-linking agents and the values of the flexural strength. Thus the increase in the flexural strength of heat cured acrylic resin was due to higher concentration of ethylene glycol dimethacrylate, cross-linking agent in its composition than microwaved one (21, 22).

The microwaved acrylic resin showed significantly higher flexural strength than heat cured acrylic resin in all concentrations (Table 2). The decrease in the flexural strength of denture base resin with conventional methods might be due to the boiling of methyl methacrylate that creates porosities in the resin leading to the formation of stress and causing propagation of cracks within the acrylic resin. While for the microwave technique, the methyl methacrylate molecules orient themselves in the electromagnetic field of the microwave energy and the polymerization heat is dissipated more effectively so the polymerization has a lesser risk of porosity and therefore higher flexural strength (5).

Impact strength

The impact strength is the capacity of a material to oppose an unexpected high-level force. High impact strength is required to avoid the fracture when the patients accidentally drop their dentures(26). Charpy impact tester was chosen for this study using notched samples and a swinging pendulum to break the specimens. Regarding the impact strength testing specimens, the presence of a V-notch in its center affirmed that the specimens fractured at this point during testing as it acts as areas of stress concentration (23).

In this in vitro study, despite the silanation of nano-zirconia, results of both types of acrylic resin reinforced with nano-zirconia showed that the impact strength decreased with the increase in their concentration. This was in agreement with previous studies (18, 19, 24) which attributed this reduction to the agglomeration of nano-zirconia particles, resulting in loosely bonded cluster formations and crack propagation (18). As shown in the SEM micrographs (Fig. 1) and (Fig. 2), aggregation of nano-zirconia particles was detected which might be the cause of the reduction in the impact strength. Another study explained this reduction in the impact strength by the effect of the increase in concentration of nano-zirconia powder on the interface region which lead to lower energy dissipation per unit volume and consequently lower impact strength (25). However, the current results disagree with other previous researches (10, 26, 27, 28) which attributed that to the interaction between the silanized nano-zirconia with the resin matrix. Uniform dispersion of nano-fillers in polymer and cross-linking between the nano-fillers and resin matrix prevented crack propagation by shielding the nano-fillers which might further enhance and improve the impact strength (26).

In comparison between the microwaved and the heat cured acrylic resin, results showed that microwaved acrylic resin had higher impact strength than heat cured one. This was in agreement with previous researches which attributed the reduction of impact strength of heat cured acrylic resin to its exothermic polymerization reaction that leads to porosities and low impact strength (28, 30). The current study disagreed with previous studies which reported higher impact strength result in heat cured than microwaved acrylic resin (12, 19, 27).

Moreover, the heat cured acrylic resin showed significantly lower impact strength than the microwaved resin. This might be attributed to the high vapor pressure of the heat cured PMMA monomer. Processing temperature much beyond 100.3°C causes vaporization of the monomer, which produces porosity in the final set material. Therefore, porosity and residual monomer may act as plasticizer and decrease the impact strength Furthermore, the microwave liquid monomer contains either a triethylene or a tetraethylene glycol, which are dimethylacrylates having a reactive group on each end. Dimethylacrylates have low vapor pressures; thus, it has a high boiling point. The low vapor pressure would allow processing without the danger of porosity increasing its impact strength. (29,30). Future studies are recommended as researches in reinforcement of the microwaved acrylic resin with zirconia nanoparticles are very few in the dental literature.

CONCLUSION

Within the limitations of this study, the incorporation of 3% and 5% nano-zirconia into heat cured acrylic resin improved its flexural strength but 7% of nano-zirconia addition had a deteriorating effect on the flexural strength of both types of investigated resin. Moreover, the addition of nano-zirconia to both types of acrylic resin had a negative effect on their impact strength.

CONFLICT OF INTEREST

None declared.

FUNDING

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