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Nanoclay-reinforced polymethylmethacrylate and its mechanical properties

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ABSTRACT

Background: Incorporation of extra fillers into dental resins might enhance their physical properties. In this study, the tensile and impact strengths of modified heat-curing acrylic resin reinforced with nanoclay were investigated.

Materials and Methods: In this experimental study, nanoclay-acrylic resin composite was prepared by mixing 0.5, 1, and 2 wt% of nanoclay with methacrylate monomer in an ultrasonic probe, followed by mixing with the polymethylmethacrylate (PMMA) powder. 24 cubic 20 mm × 20 mm × 200-mm specimens for each test, 18 samples containing nanoclay and 6 samples for the control group and a total of 48 samples were prepared. The tensile and impact strengths of the samples were tested according to ISO 527 and 179, respectively. One-way ANOVA was used for statistical analysis, followed by multiple comparison tests (Scheffé's test). Statistical significance was set at $P < 0.05$.

Results: The maximum mean tensile and impact strengths were recorded in the control group, and an acrylic resin containing 2% of nanoclay demonstrated the minimum mean in all the tests. Increasing the percentage of nanoclay in PMMA compromised the tensile strength ($P < 0.05$) with no effect on its impact strength.

Conclusion: Incorporation of nanoclay particles into acrylic resins can adversely affect the mechanical properties of the final products, and this effect is directly correlated with the concentration of nanoparticles.

Key Words: Impact, nanoparticles, polymethylmethacrylate, strength, tensile strength

INTRODUCTION

Polymethylmethacrylate (PMMA) is generally used as a common component of acrylic materials due to its optical properties, biocompatibility, and esthetics.[1] However, low mechanical properties against impact, bending and fatigue are important issues to be addressed to improve the properties of acrylic polymers for removable acrylic appliances.[2] Various methods have been used for improving mechanical properties, including chemical correction of polymeric structure by additives like polyethylene glycol dimethacrylate.[3] Another useful method is to reinforce acrylic base composite with materials

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such as fibers and particles.[4-6] These reinforcement agents have been added to the polymerizing matrix to improve the fatigue properties and fracture resistance of the PMMA. These include fibers made of Kevlar, polyethylene, carbon, hydroxyapatite, bone mineral, high-strength PMMA fibers and titanium as well as particles of glass, alumina, and acrylonitrile-butadiene-styrene.[7-13]

Along with significant developments in the field of nanotechnology, nanoparticles (NP)' use is growing rapidly in many dental materials. The role of these particles in improving the mechanical properties of composite materials has been proved.[14-16]

Nanoclay particles have been used for enhancement of flexural and tensile strength properties of nanocomposites.[17] Montmorillonite (MMT) is one of the available forms of nanoclay, and it has been shown that it greatly increases the mechanical properties of polymers.[14,18] MMT consists of platelets with an inner octahedral layer interposed between two tetrasilicate layers. It has been claimed that this structure prevents the formation of cracks, and therefore, it can improve flexural strength.[19] Furthermore, the lower density of nanoclay particles compared to other NP can significantly decrease the overall weight of resin phase, which is a great advantage.[20] Use of NP in modified form could enhance mechanical properties of dental adhesives[21] and nanocomposites.[16]

Literature has also showed that the organoclay loading at a concentration of 1% and the effect of surface preparation of NP on the dimensional stability, flexural strength, and tensile strength of different nanocomposites can be increase the flexural strength and tensile toughness up to 12%–27%.[17]

It was reported that incorporation of 0.5 wt% of nanoclay into the acrylic resin increased its yield strength and shear strength. Incorporation of higher concentrations of this material increased the shear modulus of the material.[22]

In addition, the flexural strength reached its maximum after incorporation of 2 wt% of PMMA-grafted nanoclay particles, which improves the mechanical properties of fiber reinforced composites and allows them to be used in more complex restorations.[23] The study of clay-based nanocomposites is still underway, and much research remains to be carried out to explore improved synthesis techniques, yielding different nanocomposite structures, and to fully understand the actual structure/properties relationships.

This study aimed to investigate the hypothesis that tensile and impact strengths of heat-curing acrylic resin can be enhanced with nanoclay particles.

MATERIALS AND METHODS

In this experimental research (PMMA; SR Triplex Hot, Ivoclar Vivadent, Liechtenstein, Germany) was used as a heat-curing acrylic resin and Cloisite 20A (Southern Clay Products Inc, Austin, USA), with a diameter of <6 μm and a density of 1.77 g/mL, was used as nanoclay. It was a MMT modified with dimethyl dihydrogenated tallow ammonium to increase the layer spacing of Na + MMT. Hereafter, Cloisite 20A is referred to as the nanoclay.

Nanoclay in three concentration groups at 0.5, 1, and 2 wt% was mixed with heat-curing acrylic resin.

The sample size was determined based on the results of a pilot study and 24 specimens for each test (tensile and impact strengths), and a total of 48 samples were prepared. The specimens of each test were divided into four groups as follows:

- Group A – 6 specimens of pure acrylic resin were used as the control group
- Group B – 6 specimens of PMMA were mixed with 0.5 wt% of nanoclay
- Group C – 6 specimens of PMMA were mixed with 1 wt% of nanoclay
- Group D – 6 specimens of PMMA were mixed with 2 wt% of nanoclay.

Before acrylic resin packing procedures, for best distribution, the monomer containing the specified wt% of nanoclay was placed in an ultrasonic probe (Hielscher Ultrasonics Gmbh, UP200H, Germany) for 5 min,[24,25] followed by mixing with the powder. After the paste achieved a doughy consistency, it was packed into steel molds, and the specimens were removed from the molds after curing.

Based on ISO 527 recommended by the measurement device manufacturer, 24 tensile test samples were prepared [Figure 1]. Another 24 notched rectangular cubic specimens were prepared for the impact test with standard dimensions according to ISO 179 [Figure 2].

All the samples were measured by a digital caliper (Guanglu, Strikhlu, Germany) and an error of ±0.03 mm was considered insignificant.

The specimens were polished to 400-grit emery paper (Grades 320, 500, 800, Nippon Coated Abrasive, Aichi, Japan) to get the correct size of samples.

Scanning electron microscopy (SEM, VEGA/TESCAN, Czech Republic) was used to study the distribution of NP and the cross-sectional morphology of the samples.

The specimens were conditioned in the standard laboratory environment for 24 h before performing the tests (temperature = 23°C ± 2°C, humidity = 50% ±5%).

Based on ASTM D638-10, the specimens were placed in relevant points on a universal tensile strength measurement apparatus (20 [kN] Cell Load Capacity, Zwick Z100, Germany) and fixed by levers. The apparatus exerted a tensile force (N) at a strain rate of 1 mm/min on each specimen until the specimen fractured. Force at the time of fracture was recorded. Considering the cross-sectional area of each sample, the tensile bond strength was calculated in MPa.

The impact test was carried out based on ASTM D6110-10 using a Zwick material pendulum impact testing machine (Model Z100, Germany). This instrument measures the energy required to fracture the specimen by recording the reduced swing, and hence, the reduced kinetic energy of the pendulum.

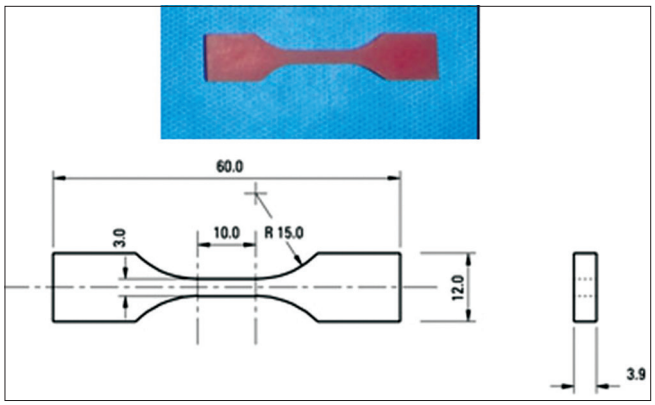

Figure 1: Exact shape and dimension of tensile test sample.

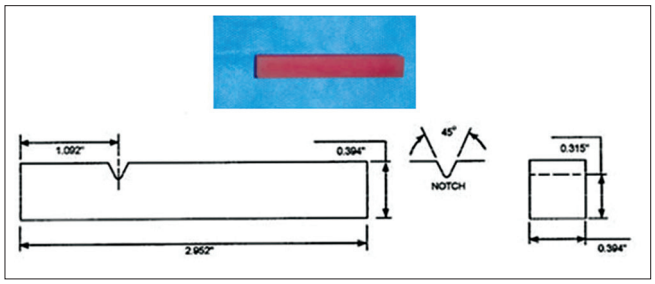

Figure 2: Exact shape and dimension of impact test sample.

The tests were carried out with a pendulum of the testing capacity of 2 J and specimen supports with a separation of 55 mm.

After testing, data were analyzed with statistical methods. Mean, average and mode in each group were calculated, and normal distribution curve was evaluated. Kolmogorov–Smirnov test was used to evaluate normal distribution. Statistical analysis of the results for each test group was conducted using one-way ANOVA, followed by multiple comparison tests (Scheffé's test). Statistical significance was set at $P < 0.05$.

RESULTS

SEM was used to verify the homogeneous distribution of particles. Figure 3 presents the morphology of the samples in cross-section and mapping of samples with different contents of nanoclay. As shown, the samples that loaded with 0.5 wt% of nanoclay (B) exhibited more homogeneous dispersion compared to samples C and D. This figure shows that an increase in nanoclay resulted in aggregation of NP in the sample by extra content, leading to changes in the fractured surface. Microcracks became visible at higher concentrations.

The mean, standard deviation, minimum and maximum stress values of tensile strengths for each experimental group are presented in Table 1. One-way ANOVA revealed a statistically significant difference between the mean values ($P < 0.05$). Scheffé's test showed no significant differences between the test group with 0.5% nanoclay and the control group ($P > 0.05$). On the other hand, the tensile strength significantly decreased in the 1% and 2% nanoclay test groups in comparison with the control group. There were no significant differences between the 1% and 2% nanoclay test groups ($P > 0.05$) [Table 2]. Figure 4 presents the results of comparisons of tensile strengths between all the groups.

Table 1: Means and standard deviations of tensile strengths for the test groups (MPa)

Group	*n*	Mean	SD	Minimum	Maximum	ANOVA	
						F	*P*
A (control)	6	28.8183	4.06522	21.00	33.00	13.001	<0.001
B (0.5%)	6	18.7150	13.24596	8.00	40.00		
C (1%)	6	8.5000	1.76068	7.00	11.00		
D (2%)	6	6.3333	1.50555	4.00	8.00		

SD: Standard deviation

Table 2: Multiple comparison test (Scheffé's test) results to compare the tensile strengths between various groups

Group (I)	Group (J)	The mean difference (I-J)	*P*	95% CI	
				Lower limit	Upper limit
Acrylic resin	Nanoclay 0.5 wt%	10.10333	0.137	−2.2605	22.4672
	Nanoclay 1 wt%	20.31833*	0.001	7.9545	32.6822
	Nanoclay 2 wt%	22.48500*	0.000	10.1211	34.8489
Acrylic resin with nanoclay=0.5 wt%	Acrylic resin	−10.10333	0.137	−22.4672	2.2605
	Nanoclay 1 wt%	10.21500	0.130	−2.1489	22.5789
	Nanoclay 2 wt%	12.38167*	0.049	0.0178	24.7455
Acrylic resin with nanoclay=1 wt%	Acrylic resin	−20.31833*	0.001	−32.6822	−7.9545
	Nanoclay 0.5 wt%	−10.21500	0.130	−22.5789	2.1489
	Nanoclay 2 wt%	2.16667	0.962	−10.1972	14.5305
Acrylic resin with nanoclay=2 wt%	Acrylic resin	−22.48500*	<0.001	−34.8489	−10.1211
	Nanoclay 0.5 wt%	−12.38167*	0.049	−24.7455	−0.0178
	Nanoclay 1 wt%	−2.16667	0.962	−14.5305	10.1972

CI: Confidence interval; *: 0.001.

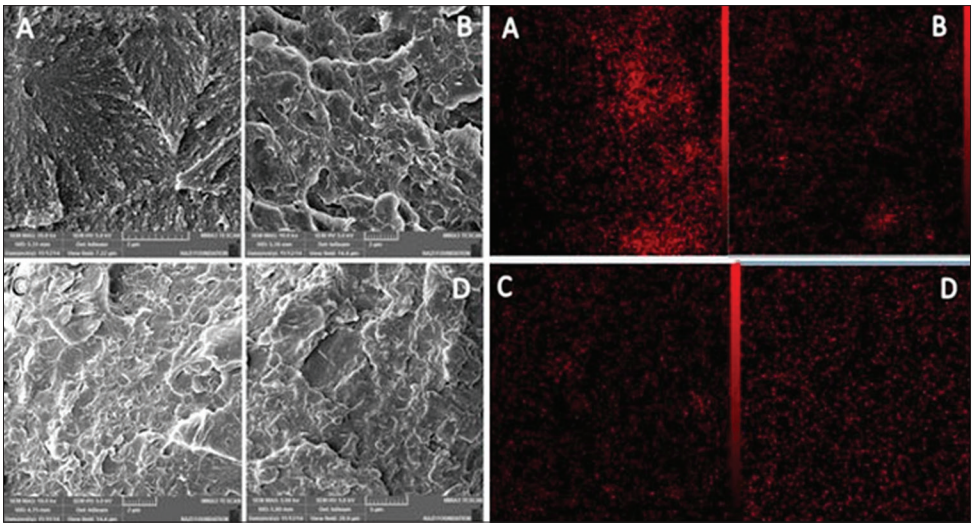

Figure 3: Scanning electron micrograph and mapping of samples (A) Pure acrylic resin, (B) 0.5% nanoclay, (C) 1% nanoclay, (D) 2% nanoclay.

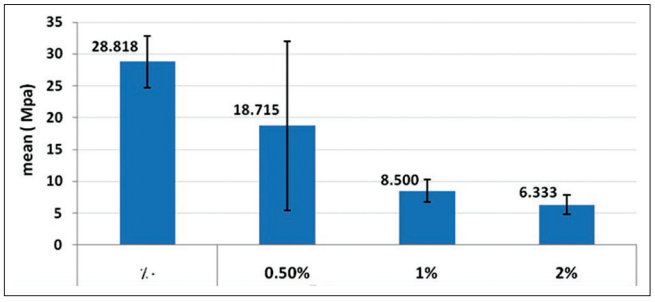

Figure 4: Mean values and standard deviations of tensile strengths calculated for all the specimens.

According to the results of ANOVA, followed by Scheffé's test summarized in Tables 3 and 4 for comparison of impact strengths in various groups, there were significant differences between the control group and other study groups, but the differences in strength between the groups containing 0.5%, 1%, and 2% nanoclay were not significant ($P > 0.05$). The results of comparisons of impact strengths between all the groups are presented in Figure 5.

DISCUSSION

Incorporation of Cloisite 20A commercial nanoclay material into acrylic resin significantly decreased the mechanical properties of the specimens compared to the control group, so the hypothesis of the study was rejected. As indicated in Table 2, the acrylic resin samples with 1% and 2% concentrations of nanoclay exhibited a significantly lower tensile strength compared with the control group, with no significant differences in mean tensile strength values between the acrylic resin samples with 0.5% and 2% nanoclay.

Based on the impact strength values obtained in the present study, incorporation of nanoclay at concentrations of 0.5, 1, and 2 wt% into acrylic resins (AR) adversely affected the mechanical properties of the polymerized material, and impact strength values decreased significantly with an increase in the concentration of nanoclay.

It should be reminded that one of the major factors affecting the mechanical properties of composite samples is the extent of interfacial interaction. In other words, a poor interfacial interaction prevents an efficient stress transfer between the components. In such cases, incorporation of filler particles is expected to increase the number of weak links, with a negative effect on the strength.[26]

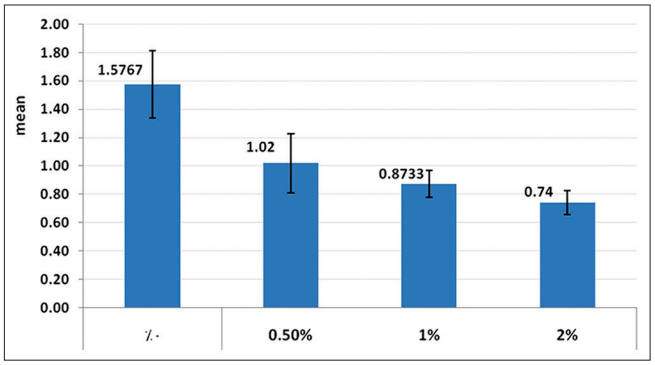

Figure 5: Mean values and standard deviations of impact strengths calculated for all the specimens.

Table 3: Means and standard deviations of impact strengths for the test groups (MPa)

Group	*n*	Mean	SD	Minimum	Maximum	ANOVA	
						F	*P*
A (control)	6	1.5767	0.23838	1.37	1.98	27.71	<0.001
B (0.5%)	6	1.0200	0.20928	0.86	1.43		
C (1%)	6	0.8733	0.09606	0.70	0.94		
D (2%)	6	0.7400	0.08485	0.58	0.80		

SD: Standard deviation

The increase in nanoclay content causes these particles to agglomerate. The agglomerated compounds can act as stress concentrating centers in the matrix, adversely affecting the mechanical properties of the polymerized material.[27] SEM was carried out for all the samples to study this effect [Figure 3]. The agglomeration of nanoclay probably gives rise to some micropores and microcracks as structural defects. Microcracks and micropores are caused by stress concentration sites and loss of mechanical properties. It has been a major challenge to prevent agglomeration in nanocomposite production, and different techniques have been suggested to solve this inherent problem during mixing with resin matrix,[17,22,28,29] including the use of an amalgamator or the probe of an ultrasonic device, the latter being used in the present study, which resulted in better separation of NP and distribution within the resin matrix. In studies by Ghaffari *et al*.[28] and also Shirkavand and Moslehifard,[29] an amalgamator was used, which does not appear to be able to properly disperse NP. A decrease in mechanical properties might be related to surface preparation and modification of NP, affecting the hydrophilicity of these particles. It seems that the additional nanoclay particles act as impurities and the mechanical properties decrease as a result of the extra additive.

However, some studies have reported an improvement in mechanical properties with an increase in the filler

Table 4: Multiple comparison test (Scheffé's test) results to compare the impact strengths between various groups

Group (I)	Group (J)	The mean difference (I-J)	*P*	95% CI	
				Lower limit	Upper limit
Acrylic resin	Nanoclay 0.5 wt%	0.55667*	<0.001	0.2556	0.8578
	Nanoclay 1 wt%	0.70333*	0.000	0.4022	1.0044
	Nanoclay 2 wt%	0.83667*	0.000	0.5356	1.1378
Acrylic resin with nanoclay=0.5 wt%	Acrylic resin	−0.55667*	0.000	−8578	−0.2556
	Nanoclay 1 wt%	0.14667	0.543	−0.1544	0.4478
	Nanoclay 2 wt%	0.28000	0.075	−0.0211	0.5811
Acrylic resin with nanoclay=1 wt%	Acrylic resin	−0.70333*	0.000	−1.0044	−0.4022
	Nanoclay 0.5 wt%	−0.14667	0.543	−0.4478	0.1544
	Nanoclay 2 wt%	0.13333	0.618	−0.1678	0.4344
Acrylic resin with nanoclay=2 wt%	Acrylic resin	−0.83667*	0.000	−1.1378	−0.5356
	Nanoclay 0.5 wt%	−0.28000	0.075	−0.5811	0.0211
	Nanoclay 1 wt%	−0.13333	0.618	−0.4344	0.1678

CI: Confidence interval; *: <0.001

content of AR. Acosta-Tores and Lopez Marin[30] showed that titanium oxide is an appropriate filler to improve the mechanical properties of AR. In addition, Solhi *et al*.[22] reported that incorporation of nanoclay filler reinforced with PMMA into adhesive resin improved its flexural modulus, and it was observed that higher filler content increased the flexural modulus. A study by Chisholm *et al*.[31] showed that increasing NP relative to microfillers resulted in a higher increase in flexural strength. Similar results were achieved in a study by Labella *et al*.[32] in relation to hydroxyapatite filler: flexural strength, tensile strength, and Vickers hardness increased significantly in reinforced composite resin.

It is noteworthy that the content of nanoadditives is of critical importance. In addition, the type of the NP and the applied AR used affects the results of studies.[8] Nanoclay is available in different commercial forms. One unmodified type of it is NA-MMT, which was used in a study by Solhi *et al*.[22] Other modified commercial forms are 15A, 20A, 25A, and 30B, each with a different effect on the mechanical properties. With regard to the acrylic resin composition, just one brand was applied in this study that was not identical with above investigations.

On the other hand, one of the problematic issues in incorporating NP into AR is pertained to the lack of chemical bond between inorganic material such as nanoclay and PMMA. To improve bonding between metal and resin some chemicals such as 4-methacryloxyethyl trimellitate anhydride and g-methacryloxypropyltrimethoxysilane have been used.[1,33] Accordingly, we can extrapolate that by identifying more appropriate substances as coupling agents between nanoclay and AR, its deleterious effects on mechanical properties might decrease.

An important problem with the use of metallic particles, including nanosilver or other metallic particles, is a change in acrylic polymer color, which limits its use in the esthetic zone.[14,23,24] However, the advantage of PMMA reinforced with nanoclay is the absence of color changes in the polymer at each of the concentrations, making its use possible in all the areas of prosthetic appliances. Another important advantage of this material is its low weight; therefore, the samples produced with it did not differ from control samples in weight.

In the present study, a decrease in mechanical properties at higher concentrations might be attributed to the quality of dispersion of NP with different surface characteristics within the resin matrix. Undoubtedly, unmodified NP or particles modified with polar or nonpolar amine derivatives exhibit different effects within the resin matrix with an increase in concentration.

Finally, it should be mentioned that this study was limited to just one brand of AR which was commercially available. In addition, we could not achieve our goal with the use of other forms of nanoclay or with other concentrations by some executive limitations. Thus, longer follow-ups are suggested for further studies on similar issues.

CONCLUSION

Within the limitations of this study, it was concluded that incorporation of nanoclay particles into AR can adversely affect the mechanical properties of the final products, and this effect is directly correlated with the concentration of NP.

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Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or nonfinancial in this article.

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