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Effect of Nanoclay on Mechanical Properties of Two Types of Heat Cure Acrylic Resin-An In Vitro Study

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Abstract

Background and Objective: *The present study was designed to evaluate the effect of nanoclay on the mechanical properties of standard and high impact heat cure acrylic resin.*

Methods: *A sample of 80 specimens were made from standard and high impact heat cure acrylic resin for testing flexural strength and impact strength (10 in each group).The flexural strength testing was done on universal testing machine and impact strength was tested using charpys impact testing machine. The force at which the specimen break was recorded. The results were statistically analysed.*

Results and Conclusion : *The results were statistically evaluated with ANOVA. The study shows significant level p <0.001. The samples of standard heat cure acrylic resin exhibited low flexural strength upon adding nanoclay. However, it showed improved impact strength. The high strength resin showed decrease in both flexural strength and impact strength upon adding nanoclay.*

Keywords: *PMMA, nanoclay, flexural strength, impact strength*

Introduction

Polymethyl methacrylate was introduced in 1973 by Dr Walter Wright and is currently the material of choice for the fabrication of removable partial complete denture. PMMA is continued to be in use because of its favourable working characteristics, processing ease, accurate fit, stability in the oral environment and superior esthetics. Despite the excellent properties, there is a need for improvement especially in terms of strength properties (1).

Many studies were carried out to improve the properties of the denture base materials by adding suitable fillers into PMMA denture base. Till date, maximum impact strength (IS) 6.55kJ/m2 was observed by Asar et al using 2% of ZrO2 incorporated in PMMA acrylic resin(3). The most popular material currently employed as an alternative to conventional PMMA is a rubber modified acrylic polymer. (4) For example, modification of PMMA denture base using styrene butadiene rubber to enhance the impact strength has been done. Synthetic rubber acts as an impact modifier, which absorbs the energy during the application of fracture load. However, till date no reports were found on using nitrile butadiene rubber to reinforce PMMA denture base (4).

Nanotechnology is widely used in our day-to-day life including its use in medicine. Using nanotechnology, it is easy to analyse and manipulate atoms, chemical bonds and molecules present between various compounds. While choosing the nanoparticle for the use in the field of nano dentistry its chemical, physical, along with the biological aspect of nanostructures are taken into account. Often various atoms or molecules are added to form the functional structure. Nanostructures are used in either innovations or diagnosis of dentistry. Some nanoparticles are used for oral disease preventive drugs, prostheses and for teeth implantation. Nanomaterials further deliver oral fluid or drugs, preventing and curing some oral disease (oral cancer) and maintain oral health (9).

In recent years, perceiving the advances in the nanotechnology sciences, many attempts have been made to use these particles in dental acrylic resin to improve its fatigue behaviour, impact strength, thermal conductivity and achieved success (6). Polymer materials have been filled with nanometre scaled, layered silicates in order to obtain improved mechanical properties and better thermal resistivity. The effects of the filler on the composite materials depend on its size, aspect ratio, hybrid morphology, and dispersion quality. Generally, the layered silicate fillers had a thickness on the order of 1 nm along with a high aspect ratio of between 10: 1 and 1000:

1. These nanometre scaled silicate pellets with such high aspect ratios tend to produce good reinforcement and improve the mechanical properties of the polymer (9).

Review of Literature & Background

A review of the most recent research revealed the following conclusions

S. B. Sehajpal, et al (1989) studied the effect of metal fillers on some physical properties of acrylic resin. Addition of metal fillers to PMMA can result in a composite material for denture bases that will have thermal conductivity matching that of metal denture bases. It will overcome the major disadvantages of acrylic resin bases (thermal conductivity) and those of metal bases such as higher cost, tedious fabrication, the finishing process, and difficulty in relining. Additional advantages of adding metal fillers to PMMA are a decrease in the coefficient of thermal expansion of acrylic resin, reduced curing shrinkage, and the reduction of internal stresses in processed dentures. Metal fillers also reduce water sorption and thus may help in the reduction of organic growth on acrylic resin dentures (13). P. K. Vallittu et al (1995) investigated the impact strength of heat-cured acrylic resin that reinforced with 1mm diameter steel wire and continuous E glass fibres. The result showed that, compared with the unreinforced specimens, both types of reinforcement increased the impact strength.

Specimens with fibre concentrations greater than 25 wt % yielded to the higher impact strength more readily than those with the metal wire reinforcement (14). J. John et al (2001). Performed a study to determine the flexural strength of commercially available heat polymerised acrylic denture base material improved by reinforcement with glass, aramid or nylon fibres, and concluded that the reinforcement of denture based resin with glass, aramid or nylon fibres improved the structural strength of the resin. Type of fibre depends on the type of prosthesis fabricated. Glass or aramid fibres appear to be suitable for long term use in complete dentures and distal extension partial denture bases, which is considered prone to fracture. Glass fibre reinforcement may also help to prevent fracture in provisional fixed partial dentures by strengthening them at the connector sites (15). DC Jagger et al; (2002) investigated transverse and impact strength of five high strength acrylic denture based materials. The aim of the study was to find out high strength denture-based material among them. The five high strength material compared with a conventional heat cure acrylic resin. Among the five materials, three of them showed significantly high impact strength (16). Solhi L.et al., in 2012 ,in the study on a novel dentin bonding system containing poly (methacrylic acid)grafted nanoclay: synthesis, characterization and properties, concluded that the incorporation of PMAA-g-nanoclay to the adhesive resulted in a significant increase in micro shear bond strength ,DTS and FS. Higher PMAA-g-nanoclay contents resulted in increased flexural modulus (8). Mowade TK et al (2012), investigated the effect of reinforcement with different fibres on impact strength of heat polymerized polymethyl methacrylate denture base resin and the effect of surface treatment of the fibres on the impact strength. The study showed that reinforcement with 2% by weight of glass, polyethylene and polypropylene fibres on the impact strength of PMMA resin and the surface treatment of the fibres further increased the impact strength significantly.

Reinforcement with plasma treated polypropylene fibre showed the highest impact strength, hence can be used clinically to reinforce the denture base to minimize the denture fracture (22).

Methodology

The study was done to evaluate the effect of nanoclay on the mechanichal properties of standard and high impact heat cure acrylic resin. The materials and methods used in the study are described under the following subheadings

1. Study subject
2. Dependent Variable
3. Armamentarium

4. Sample size
5. Procedure
6. Statistical analysis

Study Subjects

1. High impact heat cure acrylic resin **[**Trevalon H**]** **[**ISO 9001;ISO 13485 Densply India Pvt. Ltd.
2. Standard heat cure acrylic resin **[**Trevalon**]** **[**ISO 9001;ISO 13485 Densply India Pvt. Ltd**]**

Dependent Variable

1. Flexural Strength in Mega-Pascal's**[**MPa**]**.
2. Impact Strength in Joules**[**J**]**.

Armamentarium

- Acrylic mixing silicon cup
- Mixing spatula
- Digital weighing scale
- Dental flask
- Acrylizer
- Metal die
- Vaseline
- Dental stone
- Acrylic stone bur
- Emery paper
- Universal testing machine
- Charpys impact testing machine

Sample Size

Study sample was calculated using comparison of Two Mean

$$N = \frac{2(Z_{\alpha} + Z_{\beta})^2 \sigma^2}{\Delta^2}$$

Where;

$Z\infty = 1.96$ for $\infty = 0.05$

$Z\beta = 0.84$ for $\beta = 0.20$

$\Delta = \mu T - \mu C$ (difference in mean)

σ = Standard deviation

In this study (12): -

Pooled standard deviation of flexural strength (σ) = 11.5

Mean difference in flexural strength between methods (Δ) = 17

Refer study

$$N = \frac{2\,(1.96 + 0.84)^2 \times 11.5^2}{(17)^2}$$

$$= 8$$

Minimum sample size is 8 in each group

	Group A		Group B	
	Control	Test	Control	Test
Flexural Strength	10	10	10	10
Impact Strength	10	10	10	10
Total		80		

Procedure

Specimen preparation: Metal dies are made for sample preparation as per ADA specification no:12 and ISO/DIS 1567:1998 for denture base resins in 1999,with specified length, width and thickness respectively(1)

I. Flexural strength: specimen with 65mm x 10mm x 2.5mm
II. Impact strength: specimen with 50mm x 6mm x 4mm

Preparation of mould: Metal dies were fabricated. Type III Dental Stone was poured to dental flask base and metal dies were invested. A second layer of type III Dental Stone was poured. After the setting of stone, dental flask was opened and patterns were removed. The resulting shape of the metal die obtained in the flask was used as a mould for packing acrylic for specimen fabrication.

Fig1.Mould space obtained with die

Figure 02 Montmorilonnite nanoclay

Addition of nanoclay to polymer : Nanoclay 0.5 wt% of the W/P ratio of the acrylic resin according to manufacture was proportioned using electronic weighing balance. A measuring jar was used to measure monomer. Nanoclay was added to the polymer by mechanical mixing for 3 minutes. Monomer was mixed with modified polymer on a clean, dry porcelain vessel with a clean mixing spatula for 30 seconds. Compression moulding technique was used to pack the acrylic. The flask assembly was placed on hydraulic press and pressure was applied incessantly until the flask was firmly closed. The flash was teased away gently with spatula. Flasks were immersed in water bath at 100ºC for 2 hours.

Deflasking and removal of specimens: Flasks were allowed to cool for 30 minutes. Then it was immersed in water bath for 15 minutes at room temperature before deflasking.

Finishing and polishing of specimens: The specimens were trimmed with tungsten carbide burs and acrylic stone burs. The specimens were ground to final dimension with silicon carbide abrasive paper. Pumice was used for final polishing.

Testing of specimens: As per ADA specification No.12 and ISO/DIS 1567:1998 for denture base resins. (1)

Flexural strength: The specimen were mounted on the designed part of Universal Testing machine (three point loading and testing equipment). The load of 1 Newton was applied on the centre of the specimen. The flexural strength of the specimen was calculated using the standard formula. The measuring unit is Mega-Pascal's. **[**MPa**]**

$$S= 3LP / 2WT^2$$

Where S=Flexural strength, P=Maximum load before fracture, L=Distance between supports, W=Width of the specimen (10mm), T=Thickness of the specimen (2.5mm).

Impact strength-The samples were tested with Pendulum impact tester using Charpys method. The specimens were placed horizontally, and the swinging pendulum is used to break the specimens. The load at which the specimen fractured was noted and values obtained were tabulated for statistical analysis.

Impact strength is calculated using the following formula. The measuring unit of is J/mm2 .

IS =E/wt where IS is the impact strength, E is the energy required to break the specimen w is the width (6 mm) and t is the thickness of the specimen (4 mm)

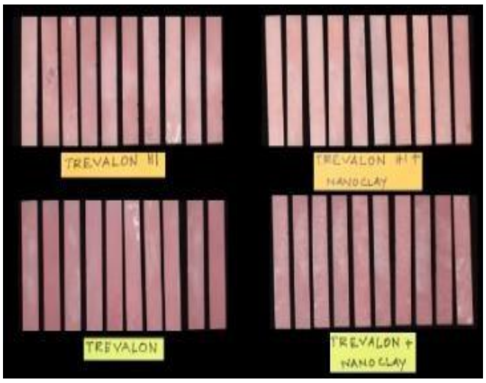


Fig 3. Samples for testing flexural strength

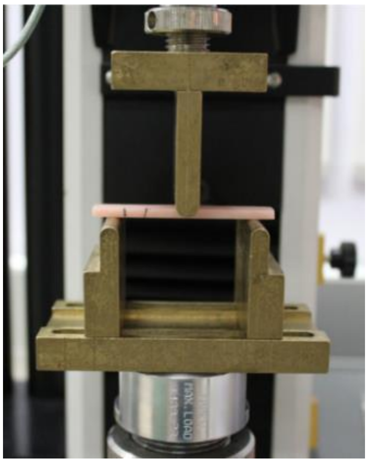


Fig 4. Sample in universal testing machine for testing

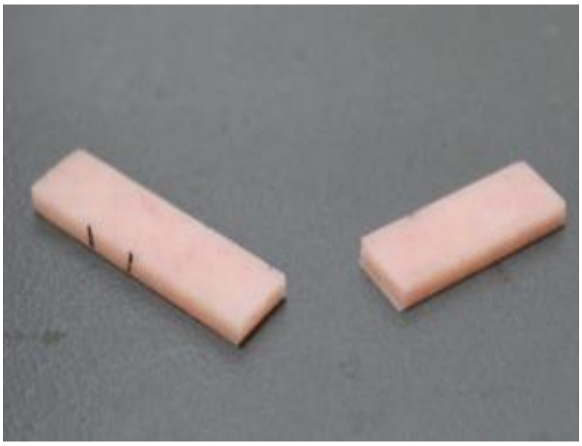


Fig 5. Broken samples after testing

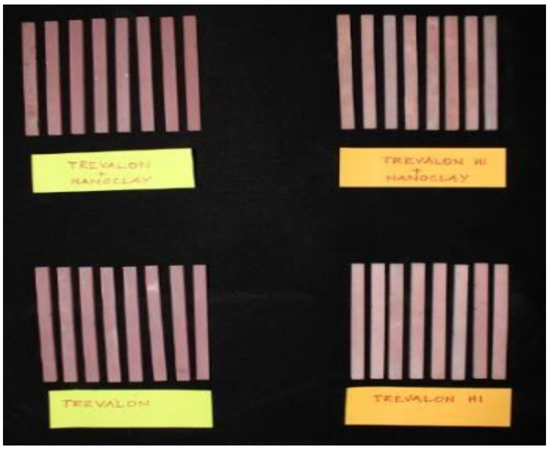


Fig 6. Samples for testing Impact strength

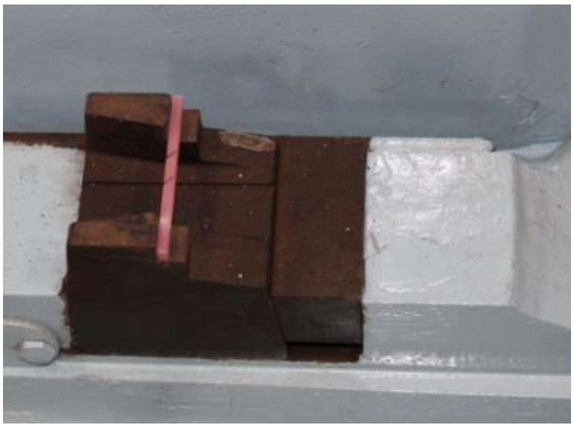


Fig 7. sample placed in the place for testing

Fig 8. Charpys Impact Testing Machine

Fig 10. Broken Samples after impact strength testing

Statistical Analysis

The data derived were analysed statistically using computer software [SPSS 17.0].The mean and standard deviation was calculated in each group and normal distribution curve was appraised. ANOVA was used for statistical analysis. The pair wise comparison was done using Tukey – Kramer test and Scheffs test. Statistical significance was set at $p<0.05$.

Results

The results include flexural strength of the standard heat cure acrylic resin (TREVALON) and high strength heat cure acrylic resin (TREVALON HI), with and without adding 0.5 wt % nanoclay . The impact strength of standard heat cure acrylic resin and high strength heat cure acrylic resin, before and after the addition of 0.5wt% of nanoclay.

Sample size	10
Lowest value	55.2000
Highest value	83.2000
Arithmetic mean	71.3600
95% CI for the Arithmetic mean	64.9785 to 77.7415
Median	73.1500
95% CI for the median	62.0900 to78.3100
Variance	79.5804
Standard deviation	8.9208

Table 1. Statistical analysis of flexural strength in TREVALON GROUP (in MPa)

The statistical summary of flexural strength of standard heat cure acrylic resin (TREVALON) is shown in Table 1. It shows an arithmetic mean value of flexural strength as 71.36. SD is 8.9208.

Sample size	10
Lowest value	4.3100
Highest value	22.1000
Arithmetic mean	11.5130
95% CI for the Arithmetic mean	7.7225 to 15.3035
Median	11.1500
95% CI for the median	6.6995 to 14.9675
Variance	28.0761
Standard deviation	5.2987

Table 2. Statistics of flexural strength in TREVALON + NANOCLAY (in MPa)

Table 2 shows the statistical summary of flexural strength of standard heat cure acrylic resin with adding 0.5wt percentage nanoclay (TREVALON + NANOCLAY). The arithmetic mean value is 11.5130. The standard deviation is 5.2987.

Sample size	10
Lowest value	33.3000
Highest value	73.7000
Arithmetic mean	57.7100
95% CI for the Arithmetic mean	49.2390 to 66.1810
Median	60.2500
95% CI for the median	48.7925 to 67.1450
Variance	140.2254
Standard deviation	11.8417

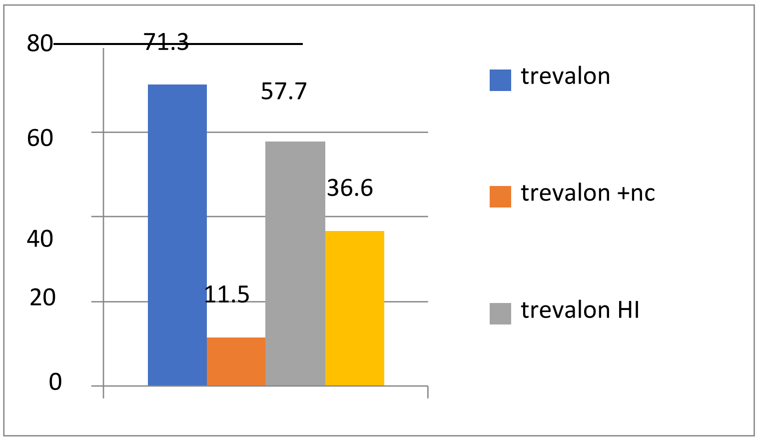
Table 3. Statistics of flexural strength in TREVALON HI (in MPa)

The table 3 is the statistical summary of flexural strength of high impact heat cure acrylic resin (TREVALON HI). The arithmetic mean obtained was 57.71. The standard deviation is 11.8417.

Sample size	10
Lowest value	19.6000
Highest value	56.8000
Arithmetic mean	36.6300
95% CI for the Arithmetic mean	27.1790 to 46.0810
Median	34.7000
95% CI for the median	23.5724 to 51.8126
Variance	174.5446
Standard deviation	13.2115

Table 4. Statistics of flexural strength in TREVALON HI+NANOCLAY (in MPa)

The table 4 shows the statistical summary of flexural strength of high impact heat cure acrylic resin with addition of nanoclay (0.5wt %) .SD is 13.2115. The arithmetic mean obtained was 36.800.



GRAPH 1. Clustered columns showing distribution of mean flexural strength (in MPa) in all groups.

The graphical representation shows that the flexural strength comparison within the group. The standard and high strength heat cure acrylic resin with the addition of nanoclay showed reduced flexural strength compared to conventional resin.

ANOVA

Source of variation	Sum of Squares	DF	Mean Square
Between groups (influence factor)	20458.8793	3	6819.6264
Within groups (other fluctuations)	3801.8388	36	105.6066
Total	24260.7181	39	

F-ratio	64.576
Significance level	P < 0.001

Tukey-Kramer test for all pairwise comparisons

Factor	n	Mean	SD	Different (P<0.001[a])from factor nr
(1) TREV	10	71.3600	8.9208	(3)(4)
(2) TREV HI	10	57.7100	11.8417	(3)(4)
(3) TREV HI NC	10	36.6300	13.2115	(1)(2)(4)
(4) TREV NC	10	11.5130	5.2987	(1)(2)(3)

Comparison between the mean flexural strength in all groups is done with ANOVA with a significant level P<0.001. Tukey- Kramar test is done for all pair wise comparison. There was significant difference between TREVALON with nanoclay and TREVALON HI with nanoclay to all other groups.

Table 5. Comparison between the mean flexural strength in all groups

Sample size	10
Lowest value	0.1333
Highest value	0.1417
Arithmetic mean	0.1383
95% CI for the Arithmetic mean	0.1353 to 0.1414
Median	0.1417
95% CI for the median	0.1333 to 0.1417
Variance	0.00001852
Standard deviation	0.004304

Table 6 Statistical summary of impact strength in standard heat cure acrylic resin (TREVALON). The arithmetic mean of impact strength is 0.1383. The standard deviation is 0.004304.

Table 6. Statistics of impact strength in TREVALON group (in J/mm2)

Sample size	10
Lowest value	0.1417
Highest value	0.1500
Arithmetic mean	0.1467
95% CI for the Arithmetic mean	0.1436 to 0.1497
Median	0.1500
95% CI for the median	0.1417 to 0.1500
Variance	0.00001852
Standard deviation	0.00430

The table 7 shows the summary statistics of impact strength in standard heat cure acrylic resin added with 0.5wt percentage nanoclay (TREVALON + NANOCLAY). The arithmetic mean is 0.1467.

Table 7. Statistics of impact strength in TREVALON +NANO CLAY group (in J/mm2)

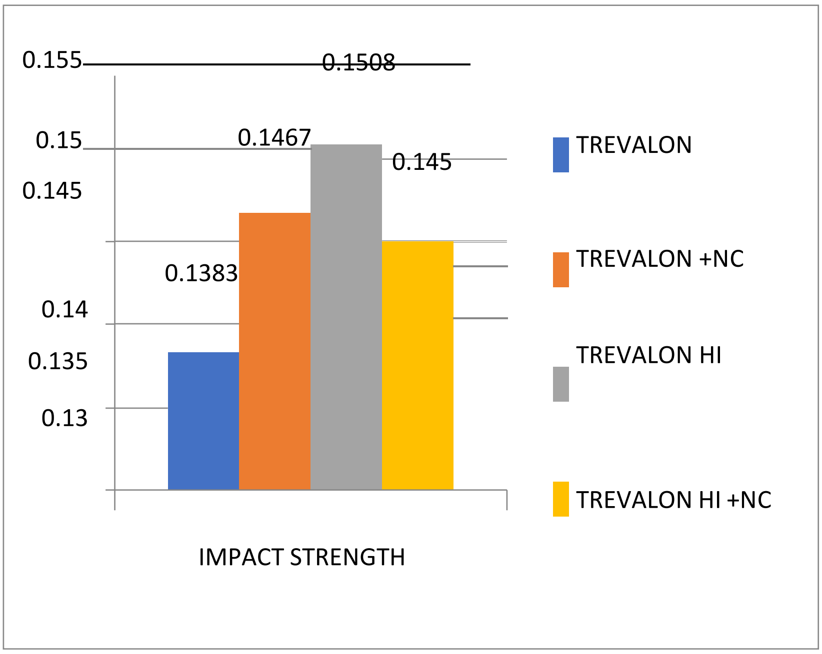
Sample size	10
Lowest value	0.1417
Highest value	0.1667
Arithmetic mean	0.1508
95% CI for the Arithmetic mean	0.1443 to 0.1574
Median	0.1500
95% CI for the median	0.1417 to 0.1588
Variance	0.00008411
Standard deviation	0.009171

The arithmetic mean of high impact heat cure acrylic resin (TREVALON HI) as shown in Table 8 is 0.1492.The standard deviation is 0.009171.

Table 9. Statistics of impact strength in TREVALON HI + NANO CLAY (in J/mm2)

Sample size	10
Lowest value	0.1417
Highest value	0.1500
Arithmetic mean	0.1450
95% CI for the Arithmetic mean	0.1419 to 0.1481
Median	0.1417
95% CI for the median	0.1417 to 0.1500
Variance	0.00001852
Standard deviation	0.004303

The table 9 shows the statistical summary of high impact heat cure acrylic resin added with 0.5wt percentage nanoclay. The arithmetic mean is 0.1450.



Graph 2. Clustered columns showing distribution of mean impact strength (in J/mm2) in all groups.

ANOVA Levene's test for equality of error variances

Levene statistic	1.304
DF 1	3
DF 2	36
Significance level	P =0.288

ANOVA

Source of variation	Sum of Squares	DF	Mean Square
Between groups (influence factor)	0.0008108	3	0.0002703
Within groups (other fluctuations)	0.001257	36	0.00003492
Total	0.002068	39	
F-ratio			7.740
Significance level			P < 0.001

Tukey-Kramer test for all pairwise comparisons

Factor	N	Mean	SD	Different ($P<0.001^a$) from factor nr
(1) TREV	10	0.1383	0.004304	(2)
(2) TREV HI	10	0.1508	0.009171	(1)
(3) TREV HI NC	10	0.1450	0.004303	
(4) TREV NC	10	0.1467	0.004303	

Levene's test done for equality of error variances. The comparison between the impact strength in all group is done with ANOVA with a significant level P<0.001. Tukey-Kramer test is done for all pair wise comparison.

Table 10 shows comparison between the mean impact strength (in J/mm2) in all groups.

Discussion

PMMA is the most commonly used denture base material, which has survived the introduction of various alternative materials such as polycarbonates and polyamides. It has a combination of both favorable and unfavorable properties.**[**8**]** Therefore, many attempts were made to enhance these properties by modifying the chemical structure of resin or by the addition of reinforcement materials. **[**11,12,36,38**]**

Various methods were tried to reinforce the acrylic resin denture bases. Metal inserts were used in the form of wires, metal oxides, metal straighteners, meshes, and plates, and the different fibres include Kevlar, glass, carbon graphite fibers, aramid fiber, ultra-high-molecular-weight polyethylene fiber, and polyethylene fibers to improve its mechanical properties. In this study Montmorillonite (MMT)nanoclay is used to reinforce the PMMA.

Nanoclay is another material which is used to improve the properties of composite and acrylic polymers. Montmorillonite (MMT) is one of the available forms of nanoclay, which consists of small layers with internal octahedral layer interposed between two tetrasilicate layers. This structure claimes to prevent formation of cracks and, therefore, it can improve flexural strength. **[**11**]**

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. **[**8**]**. Mortazavi et al. **[**11**]** evaluated the effect of pure nanoclay fillers and PMMA-grafted nanoclay fillers on the flexural strength of (fiber reinforced composite) FRC resins. The results showed that the modified nanoclay fillers increased the flexural strength of FRC. However, incorporation of unmodified particles did not significantly affect the flexural strength and even at various concentrations, none of the two above-mentioned fillers altered the flexural strength (47). Soihi et al reported that incorporation of nanoclay filler reinforced with PMMA into adhesive resin showed improved flexural modulus. (8)

Conclusion

Within the limitation of the study, it was concluded that Incorporation of 0.5 wt% nanoclay particles into a standard heat cure acrylic resin reduced the flexural strength but improved impact strength. Incorporation of nanoclay particles into high strength heat cure acrylic resin not only reduced the flexural strength but also reduced impact strength.The hydrophilic nature of montmorillonite nanoclay particles shows a tendency to agglomerate. The agglomerated sites act as stress concentration sites in the polymerised resin during force application. This can be reduced by proper dispersion of nanoclay particles. The surface modification of the nanoparticles can also change the agglomeration property. Further studies are required to find out other possible ways to improve the mechanical properties of PMMA with nanoclay.

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