Studying the Effect of Adding Titanium Dioxide (TiO_2) Nanoparticles on the Compressive Strength of Chemical and Heat-activated Acrylic Denture Base Resins

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Abstract

Problem: The commonly used acrylic resins for fabricating denture base suffer from poor mechanical properties.

Aim: This study aimed to assess the effect of incorporating Titanium Dioxide (TiO_2) nanoparticles (NPs) as a reinforcement agent on the compressive strength of different acrylic denture base materials.

Materials and methods: Thirty-two cylindrical specimens (22 mm in height and 12 mm in diameter) were prepared from PMMA resins with and without TiO_2 NPs. They were allocated into two main groups according to the materials used such as cold cure and heat cure denture base resins and then divided into two subgroups each containing eight specimens: control (without nanoparticles) and experimental (with 2 wt.% TiO_2 NPs). TiO_2 NPs were synthesized via a chemical processing route. Particle morphology and size distribution were assessed using SEM and AFM while XRD technique was employed to determine the crystalline structure of the NPs. Compression test was performed on the specimens using a universal Instron testing machine to compare the compressive strength.

Results: Size of crystalline TiO_2 NPs varied between 40-80 nm. The mean compressive strength for the cold cure acrylic resin (control group) and its nanocomposite (experimental group) were found to be 15.37 MPa and 17.42 MPa while for the heat cure acrylic resin and its nanocomposite were 23.04 MPa and 24.30 MPa. A statistically significant difference was recorded between the compressive strengths of cold cure acrylic resin and its nanocomposite. However, the difference was non-significant in the case of heat cure acrylic resin.

Conclusion: The compressive strengths of both cold cure and heat cure acrylic resins increased after the incorporation TiO_2 NPs.

Keywords

PMMA acrylic resin; Heat cure; Cold cure; TiO_2 Nanoparticles, Compressive strength, Nanocomposite; Denture base.

1. INTRODUCTION

Acrylic resin is the most commonly used material for the construction of denture bases because of its advantages such as ease of treating, inexpensive, lightweight, aesthetic properties, and stability in the oral cavity [1]. On the other hand, it has poor surface properties and questionable mechanical properties [2, 3]. In dentistry, many efforts have been made by reinforcing polymers with different materials to improve its properties like the addition of glass, polyurethane, aramid fibers [4, 5], metal wires [6], in the forms of particle, flake, fiber or fabric. These efforts significantly enhanced the mechanical properties of the acrylic resin; however, further enhancement is still demanded by the dentistry professionals.

Nowadays, new researches in the field of dentistry are aiming to introduce nanotechnology, study its application potentials and understand how to gain benefits in future treatments [7]. The principle behind the usage of nanoscale reinforcing agents is creating a new class of material termed as nanocomposite by altering the filler size to nanometer level, which is responsible for developing improved material with new mechanical and physical properties. The nature of the incorporated nanoparticles, their size, and morphology play an important role in determining the properties of new materials [5]. Various types of nanomaterials such as zirconium oxide [3, 8], carbon nanotube [9], aluminium dioxide [10], silver [11], zinc oxide [12] and widely used titanium dioxide [13-16] were used to increase the mechanical properties of Acrylic Denture Base Resin (ADBR). Titanium dioxide (TiO_2) acts as a coloring agent and it can bring additional benefits to ADBR such as antimicrobial properties [17]. Furthermore, it enables improving toughness properties and other associated mechanical properties of the acrylic resin [18].

Several investigations were carried out using various shapes of nanoparticles like nanotubes, or nanofibers [19]. Artificial denture encounters a combination of different types of forces during mastication. A recent review on the TiO_2 based PMMA nanocomposite for denture base demonstrated that mechanical tests such as flexural, impact and tensile tests and physical tests such sorption, solubility, and color stability were studied [20]. The artificial denture is compressed under high occlusal forces during mastication, which could cause failure of the denture through fracture. The denture base materials must show resistance against compressive loads. However, the compressive strength of PMMA + TiO_2 nanocomposite has not been studied extensively [21].

Therefore, it is essential to understand how the nanocomposite behaves under compressive loading condition.

Thus, this study was focused on synthesizing TiO_2 nanoparticles in a chemical processing route and evaluating the impact of adding of TiO_2 NPs on the compressive strength of ADBRs. The null hypothesis was that adding TiO_2 NPs would not significantly affect the compressive strengths of both cold cure and heat cure ADBRs.

2. MATERIALS AND METHODS

2.1. Synthesis of Titanium Dioxide nanoparticles

At first, 15 g of Trigonella Foenum leaves was washed with distilled water more than once to eradicate soil, dirt and pores. After that, the leaves were cut into small fragments to be homogenized with 50 mL distilled water. They were boiled in a water bath for 30 min at 80 °C. The resulted extract was filtered through 0.6 μm filters. For the synthesis of TiO_2 NPs 9 ml of the prepared leaf extract was mixed with 1 ml titanium trichloride ($TiCl_3$) solution, dissolved in deionized water and stirred for 20 min. The resulted solution was sintered at high temperature (80 ^oC) to produce well-crystallized nano TiO_2 particles [22].

2.2. Specimen preparation and grouping

Commercially available PMMA (polymethyl methacrylate) heat cure (powder and liquid, Vertex, Netherlands) and cold cure (powder and liquid, Vertex, Netherlands) acrylic resins were selected as the denture base materials. TiO_2 NPs weighing 1.0 gm was added separately to 22 ml of chemical (cold cure) and heat (heat cure) activated monomers. The mixtures were ultrasonically processed (Soniprep150, England at120 W, 60 KHz) for 2 hrs to ensure that individual NPs were well dispersed in the monomers. An electronic balance (Sartorius, Germany with an accuracy of 0.0001 gm) accuracy was employed to weigh the required quantity of TiO_2 NPs and acrylic resins. The suspensions of the monomers and TiO_2 NPs were immediately mixed manually with acrylic powders (49 gm in both cases) to minimize the possible aggregation of the particles and phase separation following the manufacturer's instructions. The mixing continued for approximately 20 min until the mixture reached a dough-like stage, which was suitable for handling. In all the cases the powder to monomer ratio was maintained to 2:1 (49g/22ml) to obtain 2.0 wt.% NP concentration within the nanocomposite. Based on a pilot study and existing literature [23], it was

found that 2.0 wt.% NP concentration would be appropriate for the resultant naocomposite with improved strength. The mixture was poured into a mould with a dimension of 22 mm in height and 12 mm in diameter to produce specimens for compression testing (Figure1 and Figure 2). In terms of colour, there was not much difference after adding ZrO_2 for the heat cure resin. However, bright pink colour of the cold cure resin was changed to slightly whitish pink after adding ZrO_2. The cylindrical specimens were prepared with a milling machine (imesicore/450i, Germany) in order to get a consistent dimensions for all specimens. The mould was then closed and placed in hydraulic press under a pressure of 15 MPa. Then compression technique was used for flasking with type IV dental stone (Elite stone, Zhermack, Germany). In the case of a chemical activated denture base resin, clamped flasks were left for 30 min bench curing until the process was finalized. While in case of heat-activated denture base resin, the clamped flasks were sent to the processing unit for final curing. After processing, the flasks were cooled to room temperature before mould opening and the specimens were removed gently from the mould.

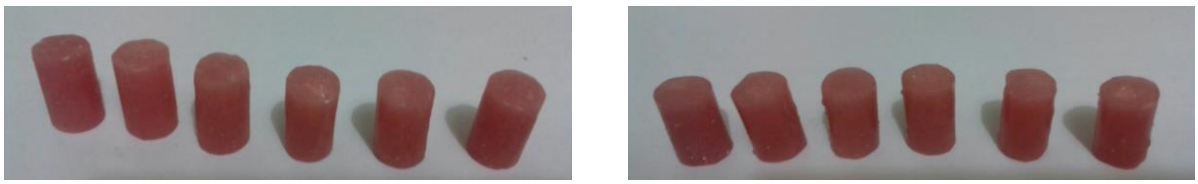
A total number of thirty-two specimens were used in this study. These specimens were divided into two main groups according to the materials used (chemical and heat-activated denture base resins) then further divided into two subgroups (control and experimental) as follows:

Group A-control group: eight specimens each for the two types of acrylic resins without any nanoparticles.

Group B-experimental group: eight specimens each for the two types of acrylic resins containing 2 wt.% of TiO_2 NPs.

(a) (b)

Figure 1. Cold cure acrylic resin (a) without (b) with adding TiO_2 NPs.



(a) (b)

Figure 2. Heat cure acrylic resin (a) without, (b) with adding TiO_2 NPs.

2.3. Nanoparticle characterization

The sintered TiO_2 NPs was characterized in a Scanning Electron Microscope (SEM) to observe the particle morphology (Inspect S50, FEI company, Netherlands) at an accelerating voltage of 20 kV. Crystalline structure of the NPs were charatcterised by an X-ray Diffraction (XRD) machine (Shimadzu - XRD6000, Shimadzu Company /Japan) with Cu-Kα X-rays of wavelength (λ) = Å, 2θ range of 10° to 70° and a step of 0.1972°. The NPs were also characterized by an Atomic Force Microscopy (micrographs were taken with a digital instruments, Inc. Nanoscope III and Dimension 3100). The grain size and distribution of synthesized nano particles was obtained from Granularity accumulation distribution charts.

2.4. Compression test

Compressive testing concludes the behaviour of materials against crushing loads. Compressive strength of the nanocomposite specimens was evaluated using a universal Instron testing machine (Tmi,testing machine Inc. Amity Ville, NewYork, USA) where each specimen was placed on the test platform and compressed to record the deformation at various loads as shown in Figure 3 [24].

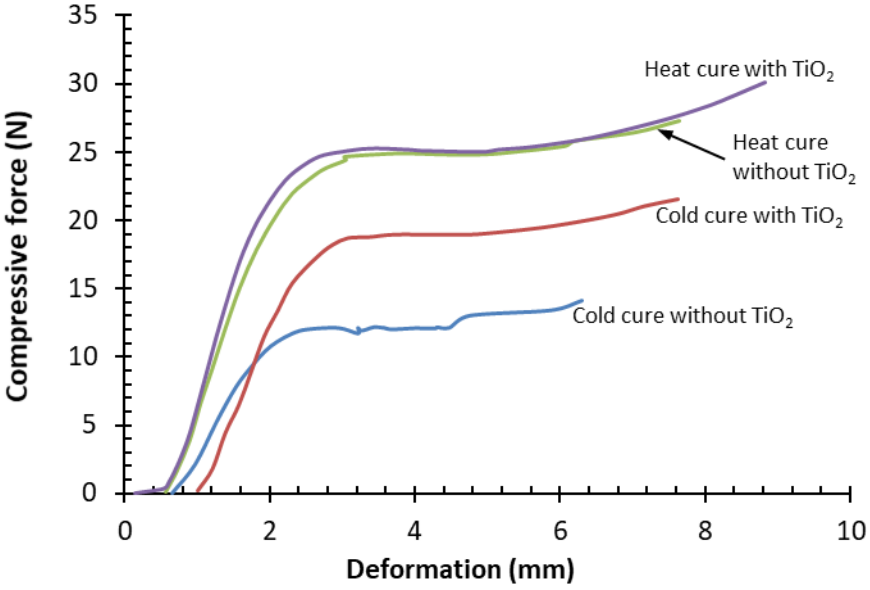


Figure 3. Typical force vs deformation curves for different resins

The maximum capacity of the load cell was 80 N and the crosshead speed was 1 mm/min (Figure 4). Before the compression test cross-sectional area of each specimen was determined. The specimens were preserved in a distilled water bath at 37 ºC for two days before testing.

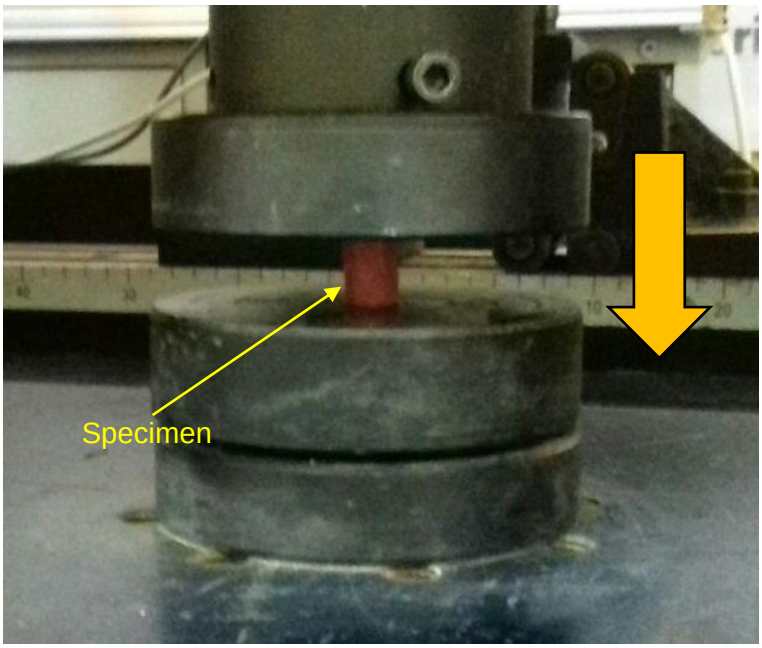


Figure 4. Compression testing machine with specimen

The compressive strength (σ) in MPa of different specimen groups were calculated by Equation 1.

$$\sigma = \frac{F}{A} \quad (1)$$

where F is the maximum applied load in N collected from the load-deformation curve and A is the cross-sectional area of the specimen in mm^2.

The data obtained from the compression tests were statistically analyzed using independent t-test and ANOVA (Analysis of Variance) after adjusted for multiple comparisons using Bonferroni Correction. Statistical tests were performed using SPSS 21.0 (Statistical Package for Social Science; IBM Statistics) for which the level of significance was fixed at 5%.

3. RESULTS

The results from this investigation could be divided into two main parts. The first part studied characteristics of the TiO_2 NPs using XRD, SEM and AFM while the second part measured compressive strength of the cold and heat cure acrylic resins with and without adding TiO_2 NPs.

3.1. Characterisation of TiO_2 NPs

3.1.1. *Crystalline structure of TiO_2 NPs*

The crystalline nature of the synthesized TiO_2 NPs was recognized by the XRD spectrum presented in (Figure 5) Five major peaks were detected in the XRD spectrum at $2\theta = 25.64°$, 36.95°, 48.44°, 56.1° and 64.15°, which corresponded to the crystal planes (101), (004), (200), (105) and (204) respectively. The broad bottom and sharp peak indirectly confirmed the smaller size and crystallite nature of the synthesized nanoparticle [21].

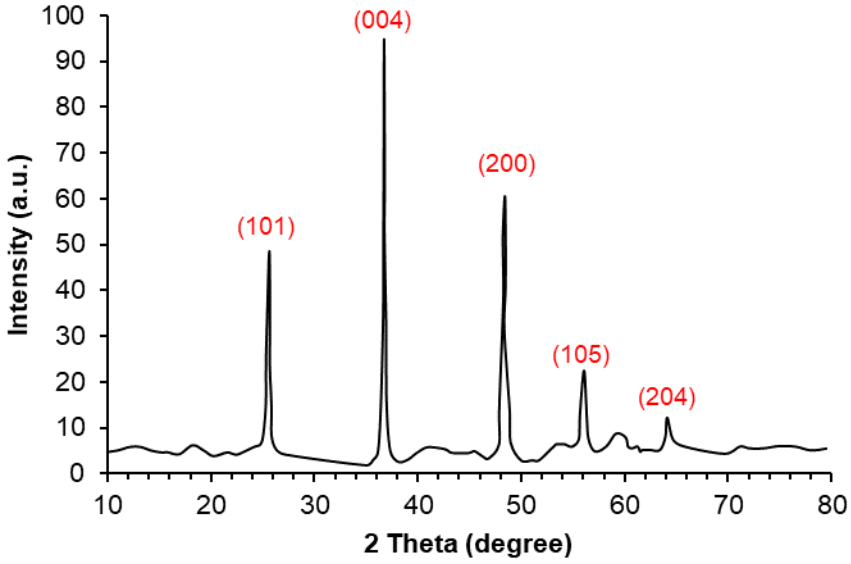


Figure 5. XRD Analysis of TiO_2 NPs

3.1.2. *2-D surface morphology and particle size*

Figure 6 illustrates an SEM image of the prepared TiO_2 NP. According to the micrograph, the particles appeared spherical in shape with a fairly uniform size distribution ranging from 37.7 nm to 81.65 nm. However, the larger particles might indicate aggregations of the smaller particles [23].

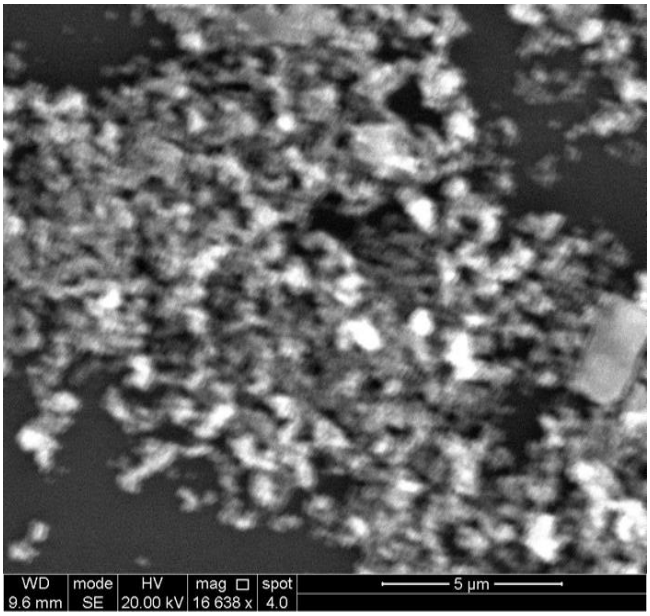
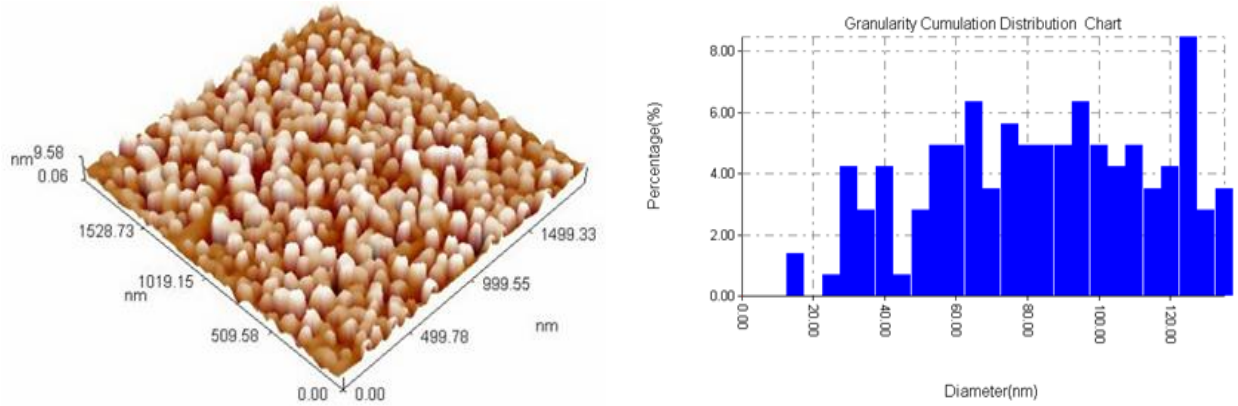


Figure 6. SEM image of TiO_2 NPs

3.1.3. *3-D surface morphology and granularity*

Figure 7 shows three-dimensional surface area plots (1.5×1.5 μm^2) and granularity accumulation distribution chart of TiO_2 NPs deposited by drop-casting technique on a glass substrate. The results showed that the root mean square value, average roughness, and average grain size of the NPs were approximately 0.441 nm, 0.36 nm, 81.65 nm respectively and the latest value agreed with the value obtained from SEM observation. The granularity cumulation chart showed that the particle size distribution followed normal distribution to some degree with larger standard deviation with majority of the values fell in the range between 50 nm to 130 nm.

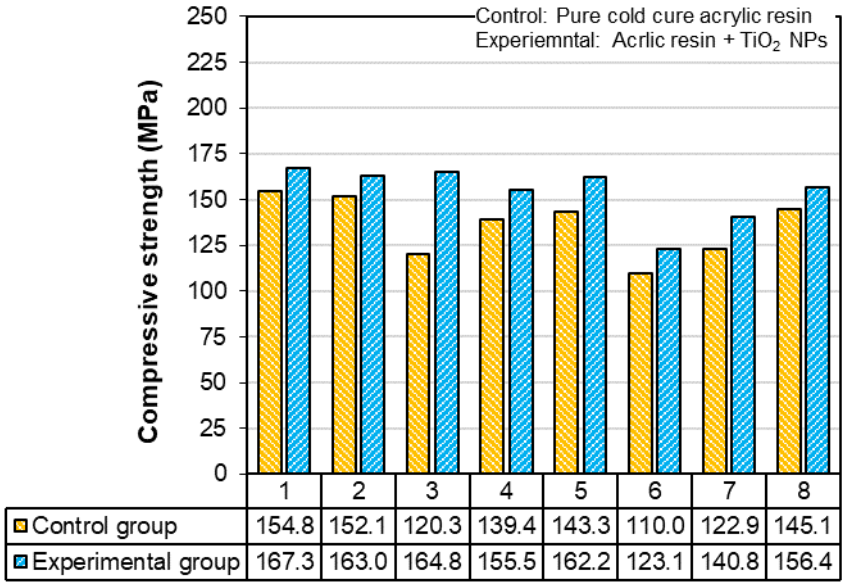


(a) (b)

Figure 7. (a) 3D AFM image and (b) granularity distribution chart of TiO_2 NPs.

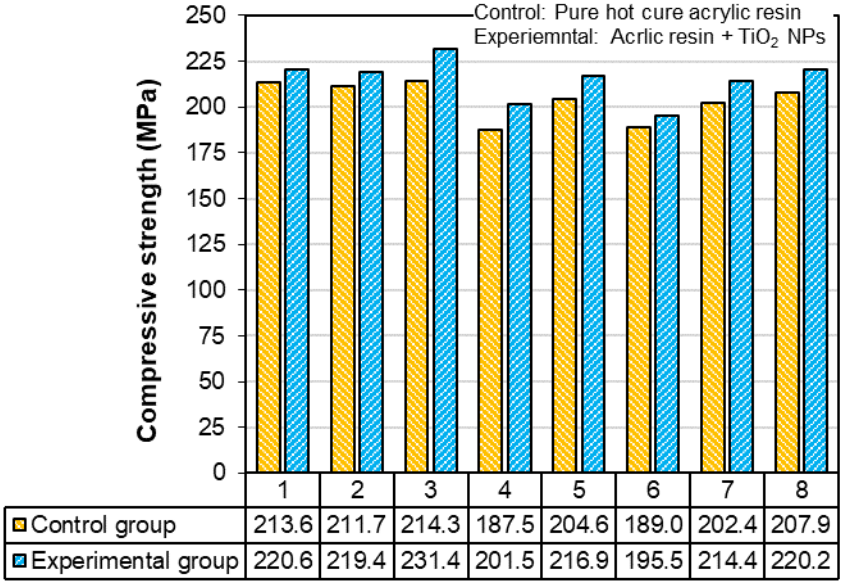
3.2. Compressive strength

Figures 8 and Figure 9 present compressive strengths of the heat cure and cold cure ADBR resins without and with TiO_2 NPs. Analysis of the compressive strength data indicated that the addition of 2 wt.% TiO_2 NPs to chemical and heat activated ADBRs increased the compressive strengths when compared with the ADBRs without any nanoparticles for all specimens in the two groups.



	1	2	3	4	5	6	7	8
Control group	154.8	152.1	120.3	139.4	143.3	110.0	122.9	145.1
Experimental group	167.3	163.0	164.8	155.5	162.2	123.1	140.8	156.4

Figure 8. Compressive strength of cold cure ADBR before and after incorporation TiO_2 NPs.



	1	2	3	4	5	6	7	8
Control group	213.6	211.7	214.3	187.5	204.6	189.0	202.4	207.9
Experimental group	220.6	219.4	231.4	201.5	216.9	195.5	214.4	220.2

Figure 9. Compressive strength of heat cure ADBR before and after incorporation TiO_2 NPs.

Statistical analysis of the mean compressive strength values showed significant difference for the cold cure ADBR and a non-significant difference for the heat cure ADBR (Table 1). The results were presented graphically in Figure 10. However, the compressive strength of the cold cure nanocomposite was significantly lower than the heat cure nanocomposite.

Table 1. Statistical data of compressive strength (MPa) of cold cure and heat cure acrylic resins.

Type	Group	Number of specimens	Mean Strength (MPa)	Standard deviation, SD (MPa)	t-Test	P-Value
Cold cure	Control group	8	135.992	15.213	-2.318	0.036 Significant
	Experimental group	8	154.138	14.055		
Heat cure	Control group	8	203.866	9.815	-2.025	0.062 Non-significant
	Experimental group	8	215.001	10.684		

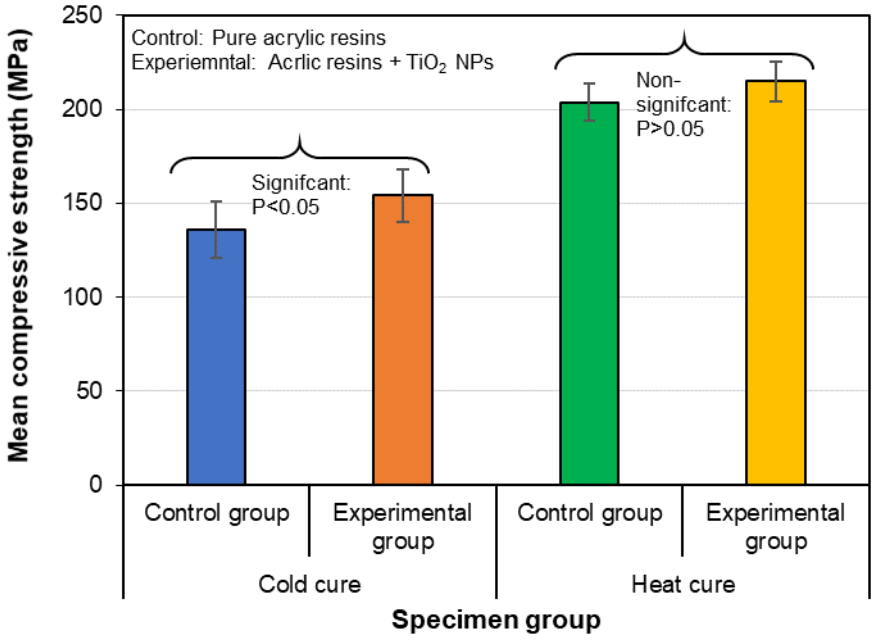
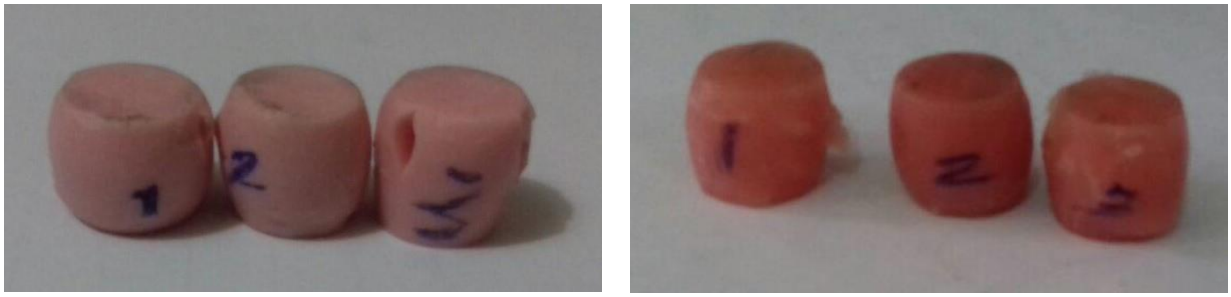


Figure 10. Mean compressive strength (MPa) values of cold cure and heat cure ADBR before and after incorporation TiO_2 NPs.

3.3. Observation of failed specimen

Examples of failed specimens after the compression tests are presented in Figure 11. It can be observed that the diameter in the middle of the samples increased forming a bulged shape. In general, the cold cure specimens deformed by vertical splitting whereas the heat cure ones showed shear type failure. However, no significant difference was observed between the specimens with and without the nanoparticles.



(a) (b)

Figure 11. Failed specimens made of acrylic resins with TiO_2 nanoparticles: (a) cold cure and (b) heat cure.

4. DISCUSSION

The most common material used in a prosthodontics field is Polymethyl methacrylate (PMMA), but it is characterized by weak physical and mechanical properties. Many attempts were made previously to overcome this limitation by adding different types and sizes of filler materials [12, 20]. Based on the idea that the decrease of filler particle size would aid in improving the mechanical properties of the resins [25], spherical nanoparticles (NPs) of titanium dioxide (TiO_2) synthesized via a chemical route was applied to improve the mechanical properties of PMMA resins [18]. In the literature, various mechanical tests including flexural, impact, fracture toughness etc. have been carried out to understand pre-clinical performance of the denture base materials. The current study was conducted to examine the impact of addition of TiO_2 nanoparticles on the compressive strength of PMMA.

Based on the results obtained in this study, it was found that the compressive strength of the cold cure PMMA resin significantly affected after adding 2 wt.% TiO_2 nanoparticles. However, in the case of heat cure resin, the changes in compressive strength was not significant. Therefore, the hypothesis was partially rejected.

The results of this study showed that adding TiO_2 NPs in both chemical and heat activated (PMMA) resins (experimental groups) would improve their compressive strengths compared to the control groups (without addition of TiO_2 nanoparticles) by 13.34% and 5.46% respectively. This may be attributed to the tiny particle size used (nanoparticle of 40-80 nm according the definition [26], high surface area and the relatively low percentage of nanofiller which may assist in a good diffusion of these fillers in a polymer matrix [21, 27]. This diffusion will aid to bind the NPs with polymer matrix strongly under adhesion force that produced limitation in polymer chains movement and improve mechanical properties of the ADBRs.

Although the improvement in the case of heat-activated ADBR was statistically non-significant, it was significant for chemical activated ADBR. The reason behind this different mechanical behavior could be due to insufficient polymerization time for the chemical activated ADBR mixture which was characterized by the evaporation of the monomer during the passage of the polymer mixture through the polymerization stages up to dough-like stage leaving porosity which leads to a decrease in the compressive strength. Mixing the non-functionalized TiO_2 nanoparticles with the polymer mixture could lead them to deposit into the interstitial porosities within the polymer matrix and interrupt crack propagation inside the material [20], which increased compressive strength of the cold cure nanocomposite compared to the control group.

Moslehifard et al. [21] tested the effect of incorporation of TiO_2 NPs up to 2 wt.% in heat cure PMMA and no significant differences was found between the compressive strength of the nanocomposite and pure resin. The findings from this investigation agreed with the reported results. The current results were also in agreement with that added TiO_2 nanoparticles to the acrylic [18] resin and found that the nanoparticles would create strong interfacial bond with the acrylic resin that led to improvement of its mechanical properties. Also, the current results were supported by Harini et al. [28] who found that reinforced PMMA with different concentrations of TiO_2 NPs would produce higher flexural strength than those of traditional PMMA. Several studies have added nanomaterials such as ZrO_2 [3] and Ag NPs [29] in deferent ratios to heat cure acrylic resins; they concluded that nanomaterials could improve the acrylic resins mechanical properties without causing any adverse effect and is highly recommended in the palatal portion of the acrylic base of maxillary dentures.

In this *in vitro* study, even though significant improvement was obtained for the case cold cure nanocomposite, but its overall compressive strength (17.423 MPa) was even lower than that of the pure heat cure resin (23.045 MPa) by approximately 32%. In terms of strength test, only compressive test results are shown here. Other tests such as flexural strength are being conducted to be published in a future paper.

Further studies could be conducted to understand the distribution of nanoparticles within the matrices and the failure modes and mechanisms of the two nanocomposite materials.

The development of TiO_2 reinforced PMMA (cold cure) nanocomposite with significantly improved compressive strength than the pure resin could pave the way for new class of denture base material with longer clinical life. Functional life tests such as equivalent flexural strength [30] and tooth bonding strength on the actual denture can be conducted before the clinical trial. The significant improvement in compressive strength of the cold cure resin with the addition of NPs could open up the opportunities for denture base reparing [31].

5. CONCLUSION

In this investigation, the compressive strength of denture base nanocomposites made of cold and heat cure acrylic resins incorporated with TiO_2 nanoparticles (NPs) was studied compared to the pure resins. The synthesized NPs showed a size distribution ranging between 40 to 80 nm and spherical shapes. XRD results confirmed the crystallinity of the NPs. In general, all the nanocomposite specimens showed higher compressive strength than both the pure resins. However, significant difference in compressive strength was only found for the chemical activated resin. Therefore, addition of TiO_2 NPs to acrylic resin is strongly recommended during construction of denture base.

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Conflict of interest

The authors declare no conflict of interest.

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