

# The Impact of Incorporating Zirconium Oxide and Iron Oxide Nanoparticles into PMMA on Various Mechanical Properties

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Polymethyl methacrylate (PMMA) is extensively employed as a denture base material owing to its biocompatibility, antimicrobial qualities, and advantageous physical characteristics. However, its natural brittleness and limited mechanical strength make it less effective in the clinic. This study aims to enhance the mechanical properties of PMMA by incorporating zirconium oxide (ZrO<sub>2</sub>) and iron oxide (Fe<sub>2</sub>O<sub>3</sub>) nanoparticles at different concentrations (1 wt%, 2 wt%, and 3 wt%) using hydrothermal autoclave. Two groups of nanocomposites were produced and underwent post-curing heat treatment to examine the impact of nanoparticle type and concentration on the mechanical properties of the material. Mechanical studies indicated that PMMA reinforced with nano-ZrO<sub>2</sub>/Fe<sub>2</sub>O<sub>3</sub> displayed lower impact resistance, especially post-heat treatment, in contrast to PMMA reinforced purely with ZrO<sub>2</sub> nanoparticles. The inclusion of Fe<sub>2</sub>O<sub>3</sub> appears to decrease toughness instead of boosting it. Of the studied formulations, heat-treated PMMA with 3 wt% nano-ZrO<sub>2</sub> displayed the most advantageous balance of strength and durability, indicating excellent nanoparticle distribution and interfacial adhesion. The findings underscore the potential of regulated nano-ZrO<sub>2</sub> integration to enhance the mechanical dependability of denture base materials, but hybrid reinforcement with Fe<sub>2</sub>O<sub>3</sub> offers no supplementary advantages under the assessed conditions.

**Keywords:** Polymethyl methacrylate (PMMA), zirconium oxide nanoparticles (ZrO<sub>2</sub>), hardness, impact strength, iron oxide nanoparticles (Fe<sub>2</sub>O<sub>3</sub>), nanocomposite

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Tooth loss is a major concern among patients, for esthetic as well as functional reasons, so replacing them by artificial substitutes like dentures is of great benefit [1]. The denture base serves as a medium between teeth and jaw transmitting forces of mastication to the adjacent tissues [2]. For many decades, the most commonly used material for building dentures has been the poly methyl methacrylate acrylic resin (PMMA) which provides a number of advantages, including precise fit, stability within the mouth environment, affordable equipment, easy clinical and laboratory manipulation, and good aesthetics [3,4]. Denture base materials are a type of biomaterials that must possess desirable mechanical properties including stiffness [5], toughness [6], hardness [7] and resistance to wear [8] and abrasion [9], and good thermal properties such as thermal conductivity, thermal diffusivity, and Dimensional stability for example should not expand, tissue compatibility (non-toxic or allergic), Color stability, good chemical stability, in addition to esthetical pleasing and use in the oral cavity [10-12].

Nanotechnology has proven useful in many areas of knowledge, especially in dentistry. Nowadays,

new commercial dental materials are available, and nanoparticles used in their production have improved their quality. This great potential for nanotechnology to address dental health issues is because it can be used against pathogenic bacteria or to repair dental damage [13].

Metallic nanoparticles such as iron oxide, gold and silver nanoparticles have been utilized and modified for dentistry applications as a result of their intrinsic properties as diagnostic and/or therapeutic agents for diseases [14]. Magnetite (Fe<sub>3</sub>O<sub>4</sub>), hematite (α-Fe<sub>2</sub>O<sub>3</sub>), maghemite (γ-Fe<sub>2</sub>O<sub>3</sub>), and mixed ferrites are considered to be the main representative of iron oxide nanoparticles (IONPs) because of their high biocompatibility, low biodegradability, low toxicity, strong magnetism, and efficient removal from the body, making them appropriate for clinical use. IONPs have also been utilized in biomedical fields, such as magnetic resonance imaging (MRI), targeted drug delivery, cancer immunotherapy and hyperthermia mediators. The readily-modifiable size and surface chemistry of SPIONs allow cellular targeting and permit them to move across physiological barriers [15,16].

Zirconia is widely known in the ceramic industry for its properties of hardness and ability to resist fractures under normal circumstances. Additionally, the fine grain size of the material, that is less than a micron, permits achieving remarkable surface finishes and the while maintaining a sharp edge. Many complex formulations have been developed to avoid the spread of cracks. These include yttria-stabilized tetragonal zirconia polycrystal (Y-TZP; zirconia) as a common ingredient. The arrival of zirconia ceramics, together with electronic technology, has permitted dental research and business to achieve their goals. Zirconia's popularity has grown in biomedical applications, especially surgical implants, for its aesthetic characteristics and biocompatibility. It is also commonly used in dentistry for crowns, bridges, implants, and veneers; this substance is highly biocompatible and can tolerate the long-term effects of the mouth's thermal, chemical, and mechanical stresses. There has been a huge breakthrough in dental industry in the last decade when it comes to production of zirconia for dental uses [17].

Among these materials,  $\text{TiO}_2$  and  $\text{ZrO}_2$  are the choice of many investigators to test various mechanical characteristics such as maximum flexural load, flexural strength, and load at the break. Adhershitha, A. R., & Viswambharan, P. concluded that addition of nano  $\text{TiO}_2$  and nano  $\text{ZrO}_2$  provided enhanced flexural strength. 1% of nano  $\text{TiO}_2$  and nano  $\text{ZrO}_2$  was found to be sweet spot for better properties. In this context, one weight% of nano  $\text{TiO}_2$  and nano  $\text{ZrO}_2$  to PMMA powder was added for the purpose of testing and comparing thermal conductivity [18]. Ata, S. O. Ata *et.al* was to assess the flexural strength of acrylic resin base material combined with iron, copper, and titanium nanoparticles. Within the limitations, adding nanoparticles to acrylic resins could improve the mechanical properties; but, when

the percentage of nanoparticles added increases, the flexural strength values of the acrylic resins decrease [19]. S Acosta-Torres, *L., et al.* developed the mechanical properties of new prostheses by using nanopigments serving as antimicrobial reagents and reinforcing fibers [13].

This study aims to enhance the mechanical properties of two types of denture materials and compare their performance.

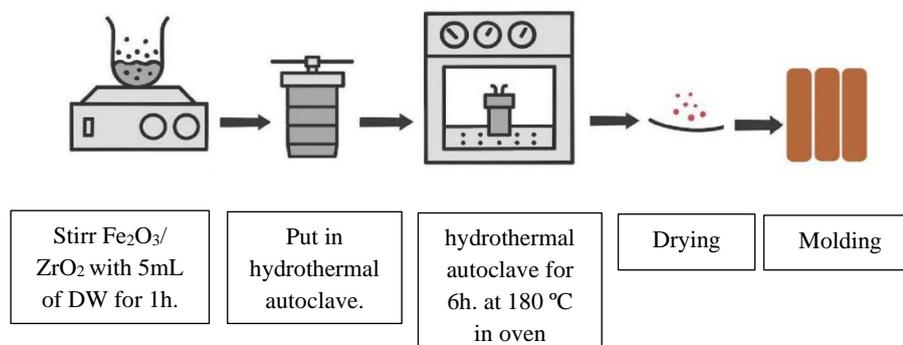
## EXPERIMENTAL

### Chemicals and Materials

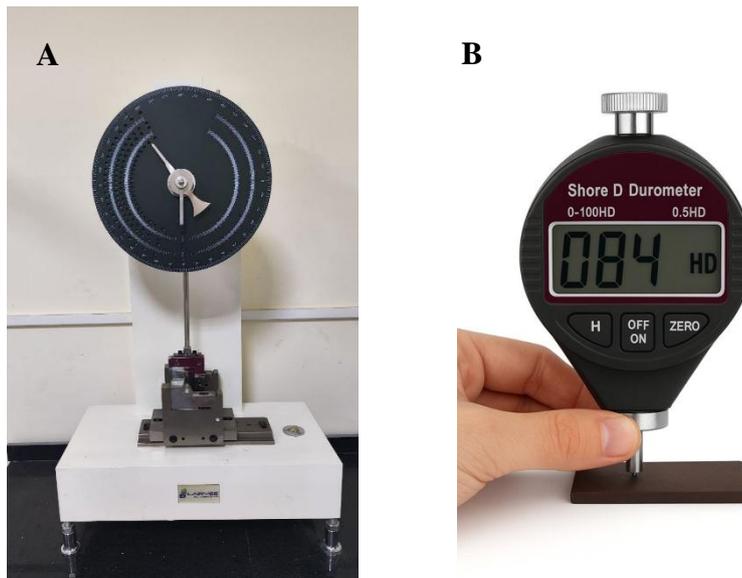
Zirconium oxide ( $\text{ZrO}_2$ ) from Research Nanomaterials, USA, CAS number 1314-23-4 (Assay 99.5 % purity, 20 nm particle size;  $\text{Fe}_2\text{O}_3$ -gamma from Research Nanomaterials CAS number 1309-37-1(99.5 % purity, 20 nm particle size; cold-cure and heatt-cure denture base polymers from MEGA.

### Methods

Fourteen samples were categorized into four categories. Acrylic specimens were fabricated in accordance with the manufacturer's guidelines. Initially,  $\text{ZrO}_2/\text{Fe}_2\text{O}_3$  nano mixture synthesized using hydrothermal autoclave were incorporated at a ratio of 1 wt% of  $\text{ZrO}_2$ , alongside 1 wt%, 2 wt%, and 3 wt% of  $\text{Fe}_2\text{O}_3$  respectively with 5 mL distilled water and stir for 1 h, then put in stainless-steel hydrothermal autoclave in oven at 180 °C for 6 hours (Figure1). These nano mixtures were included into the heat-polymerized, cold-polymerized acrylic resins and then molded. The characterization methods were Hardness Test Using Shore Durometer was used for 3-point, impact strength using Charpy test (Figure 2) and Fe-sem.



**Figure 1.** The Preparation of  $\text{ZrO}_2/\text{Fe}_2\text{O}_3$  Nano composites.



**Figure 2.** (A) Charpy test for impact strength measurement, (B) Shore D hardness measurement using a digital durometer.

### Sample Preparation

The sample was prepared using a stainless-steel mold that was 10 mm thick, 55 mm long, and 10 mm wide. As directed by the manufacturer, the heat-cure acrylic resin samples were made by combining (additive and initiator) and (monomer and cross linked) in a 25 gr/10 ml ratio. Regarding the groups that incorporated nanoparticles in a proportion of  $ZrO_2$  1wt%, and  $Fe_2O_3$  nanoparticles 1wt%, 2wt% and 3wt%, each time.

In a mixer (President Dental, Germany), the powdered acrylic resin including nanoparticles (1wt%, 2wt%, and 3wt%) was fully homogenized for 30 seconds at 2900 rpm. The resins has been prepared and placed in the mold, left in water at  $74 \pm 1$  °C for 8 hours, and then boiled for 2 hours to complete the polymerization process. Following their removal from the molds, the nanocomposites samples were polished using 200, 400, and 600 grits, respectively, for five minutes under water cooling to achieve a standard surface. To guarantee uniformity for the test procedures, sample sizes were measured. For a week, the samples were stored in distilled water.

The cold-cure acrylic resin (self-curing PMMA) samples were made by combining polymethyl methacrylate (PMMA) and methyl methacrylate (MMA) monomer with benzoyl peroxide as initiator. Regarding the groups that incorporated nanoparticles

in a proportion of  $ZrO_2$  1wt%, and  $Fe_2O_3$  nanoparticles 1wt%, 2wt% and 3wt%, each time.

Weigh the required amount of nanoparticles according to the desired weight percentage (1%, 2%, or 3% of PMMA powder weight). Add the nanoparticles to the MMA liquid monomer and sonicate the mixture for 15–20 minutes in an ultrasonic bath to ensure uniform dispersion and prevent agglomeration. Mix PMMA powder with the produced nanoparticle-dispersed monomer using the manufacturer's specified powder-to-liquid ratio (usually 3:1 by volume). Stir gently with a spatula until a uniform dough-like consistency is produced. Allow the mixture to reach the dough stage, which is optimal for molding (the surface becomes non-sticky). Pack the dough mixture into the mold cavity slightly in excess and shut it under modest pressure to remove air bubbles and surplus material. Until polymerization is finished, which should take 15 to 20 minutes, keep the mold at room temperature (23 to 25 °C) with gentle pressure. In the absence of external heat, benzoyl peroxide initiates a free-radical reaction that results in polymerization. Gently remove the specimen from the mold once it has polymerized. Use a rotary cutter or fine sandpaper to remove glare and uneven edges. To remove any remaining monomer and replicate oral and ambient conditions, store the specimens in distilled water at 37 °C for a full day before to testing.

**Table 1.** lists the control PMMA, PMMA/ZrO<sub>2</sub>, and PMMA/ZrO<sub>2</sub>/Fe<sub>2</sub>O<sub>3</sub> nanocomposites' mechanical characteristics for both heat and cold curing.

Sample	Hardness (HD)	Impact strength (kJ/m <sup>2</sup> )
Control cold cure	77.5	3.50
Cold PMMA/ ZrO <sub>2</sub> (1%)	89.05	4.80
Cold PMMA/ ZrO <sub>2</sub> (2%)	91.4	5.20
Cold PMMA/ ZrO <sub>2</sub> (3%)	92.5	6.30
Cold PMMA/ ZrO <sub>2</sub> (1%)/ Fe <sub>2</sub> O <sub>3</sub> (1%)	89.8	1.20
Cold PMMA/ ZrO <sub>2</sub> (1%)/ Fe <sub>2</sub> O <sub>3</sub> (2%)	79.0	1.80
Cold PMMA/ ZrO <sub>2</sub> (1%)/ Fe <sub>2</sub> O <sub>3</sub> (3%)	86.4	1.50
Control heat cure	80.6	4.50
heat PMMA/ ZrO <sub>2</sub> (1%)	90.7	5.60
heat PMMA/ ZrO <sub>2</sub> (2%)	93.9	6.90
heat PMMA/ ZrO <sub>2</sub> (3%)	94.5	8.10
heat PMMA/ ZrO <sub>2</sub> (1%)/ Fe <sub>2</sub> O <sub>3</sub> (1%)	84.6	1.35
heat PMMA/ ZrO <sub>2</sub> (1%)/ Fe <sub>2</sub> O <sub>3</sub> (2%)	89.9	2.10
heat PMMA/ ZrO <sub>2</sub> (1%)/ Fe <sub>2</sub> O <sub>3</sub> (3%)	87.2	1.80

## RESULTS AND DISCUSSION

### Mechanical Properties

When zirconium oxide (ZrO<sub>2</sub>) nanoparticles were added to PMMA composites, the results, as displayed in Table 1, showed a significant improvement in both hardness and impact strength. In contrast, the addition of iron oxide (Fe<sub>2</sub>O<sub>3</sub>) resulted in a decrease in both properties when compared to ZrO<sub>2</sub>-only composites.

The hardness values for the cold-cured PMMA grew gradually as the ZrO<sub>2</sub> concentration increased from 1% to 3%, reaching their maximum value at 92.5 HD. The strong interfacial connection between the hard ZrO<sub>2</sub> nanoparticles and the polymer matrix, which function as efficient stress-transfer sites and limit the mobility of the PMMA chains, is responsible for this improvement. As a result, this limitation increases the material's ability to withstand surface deformation.

Nevertheless, the injection of Fe<sub>2</sub>O<sub>3</sub> in conjunction with ZrO<sub>2</sub> resulted in a decrease in hardness, suggesting a likely aggregation of particles or insufficient interfacial interaction between Fe<sub>2</sub>O<sub>3</sub> and the polymer matrix. This could have resulted in areas of stress concentration that undermine the mechanical integrity of the composite.

The equation for impact strength is expressed as:  $a = E/bh$

Where  $a$  represents the impact strength value in joules per meter,  $E$  denotes the absorbed energy in joules,  $b$  indicates the width of the test piece in millimeters, and  $h$  signifies the thickness of the test piece in millimeters.

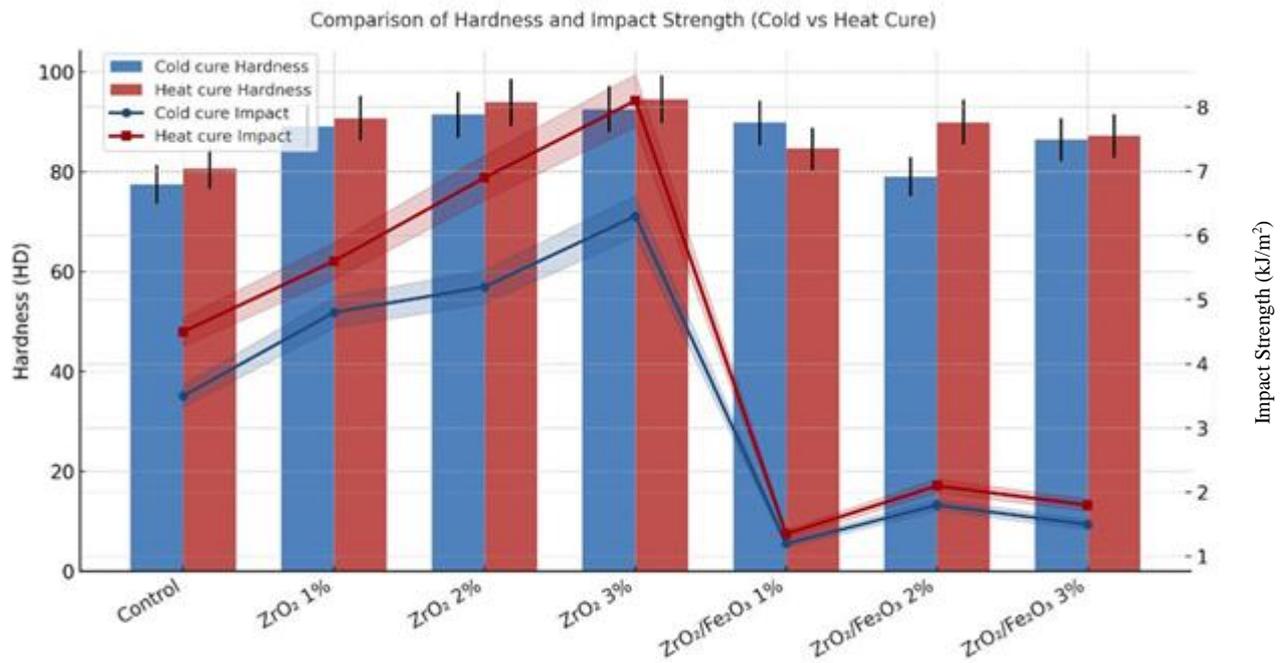
The heat-cured PMMA samples showed a similar pattern, but their overall hardness and impact strength values were higher than those of the cold-cured samples. Increased polymerization and cross-linking density are made possible by the thermal curing process, which improves load distribution and decreases internal voids, both of which increase the stiffness and toughness of the composite. The heat-cured PMMA reinforced with 3% ZrO<sub>2</sub> showed the highest hardness (94.5 HD) and impact strength (8.10 kJ/m<sup>2</sup>), confirming the effectiveness of zirconia nanoparticle reinforcement.

Conversely, Fe<sub>2</sub>O<sub>3</sub>-containing composites performed worse mechanically. This decline can be explained by the difference in particle morphology and interfacial compatibility between Fe<sub>2</sub>O<sub>3</sub> and PMMA, which may hinder effective stress transfer and energy absorption during impact.

The values obtained from the impact strength tests were compared by using one-way ANOVA test. The means, standard deviation values are shown in Table 2. Chart comparison for hardness and impact strength for two types of dentures are shown in Figure 3.

**Table 2.** Statistical analysis of hardness and Charpy impact strength for cold cured and heat-cured PMMA composites.

Groups	No. of samples	mean $\pm$ SD hardness	mean $\pm$ SD impact strength
Cold-cured PMMA composites	7	86.55 $\pm$ 5.58	3.19 $\pm$ 1.86 kJ/m <sup>2</sup>
Heat-cured PMMA composites	7	88.20 $\pm$ 4.31	4.05 $\pm$ 2.34 kJ/m <sup>2</sup>



**Figure 3.** Comparison chart for hardness and impact strength for two types of dentures.

## Surface Characterization

### SEM Analysis

**Figure 4.** shows the morphology of samples which was analyzed using field emission scanning electron microscopy (FESEM) at various resolutions. The developed PMMA-based nanocomposites' surface form and particle dispersion are thoroughly revealed by the SEM micrographs.

The morphology of PMMA enhanced with ZrO<sub>2</sub> nanoparticles is shown at various magnifications in images (a) and (b). The surface is comparatively homogeneous and smooth, with tiny, evenly spaced nanoparticles that have a diameter of about 30 to 50 nm. Strong interfacial adhesion and good dispersion are shown by the homogeneous distribution of zirconia particles within the PMMA matrix, which is essential for the best possible stress transfer during mechanical loading. With minimal agglomeration and a high

degree of wetting, the nanoscale particles are successfully integrated into the polymer matrix. Because the nanoparticles operate as micro-barriers that restrict polymer chain mobility and prevent fracture propagation, the uniform dispersion increases hardness and impact strength in the mechanical results.

In contrast, the PMMA/ZrO<sub>2</sub>/Fe<sub>2</sub>O<sub>3</sub> composite's SEM pictures (c) and (d) display a less uniform shape. A few clusters of adhered nanoparticles were seen, along with regions of micro-voids and uneven surfaces. Due to differences in surface energy and polarity between Fe<sub>2</sub>O<sub>3</sub> and the PMMA matrix, the dispersion quality appears to have been adversely affected by the addition of Fe<sub>2</sub>O<sub>3</sub>.

The uniform distribution of nanoparticles in PMMA/ZrO<sub>2</sub> composites led to increased hardness and impact strength, confirming the beneficial role of well-dispersed zirconia nanoparticles as effective reinforcing agents; the increased heterogeneity and

insufficient interfacial bonding in PMMA/ZrO<sub>2</sub>/Fe<sub>2</sub>O<sub>3</sub> samples led to decreased mechanical performance; the morphological observations obtained from SEM analysis are in strong agreement with the mechanical test results. Under load, these agglomerates may serve as locations of concentrated stress and aid in the formation of microcracks.

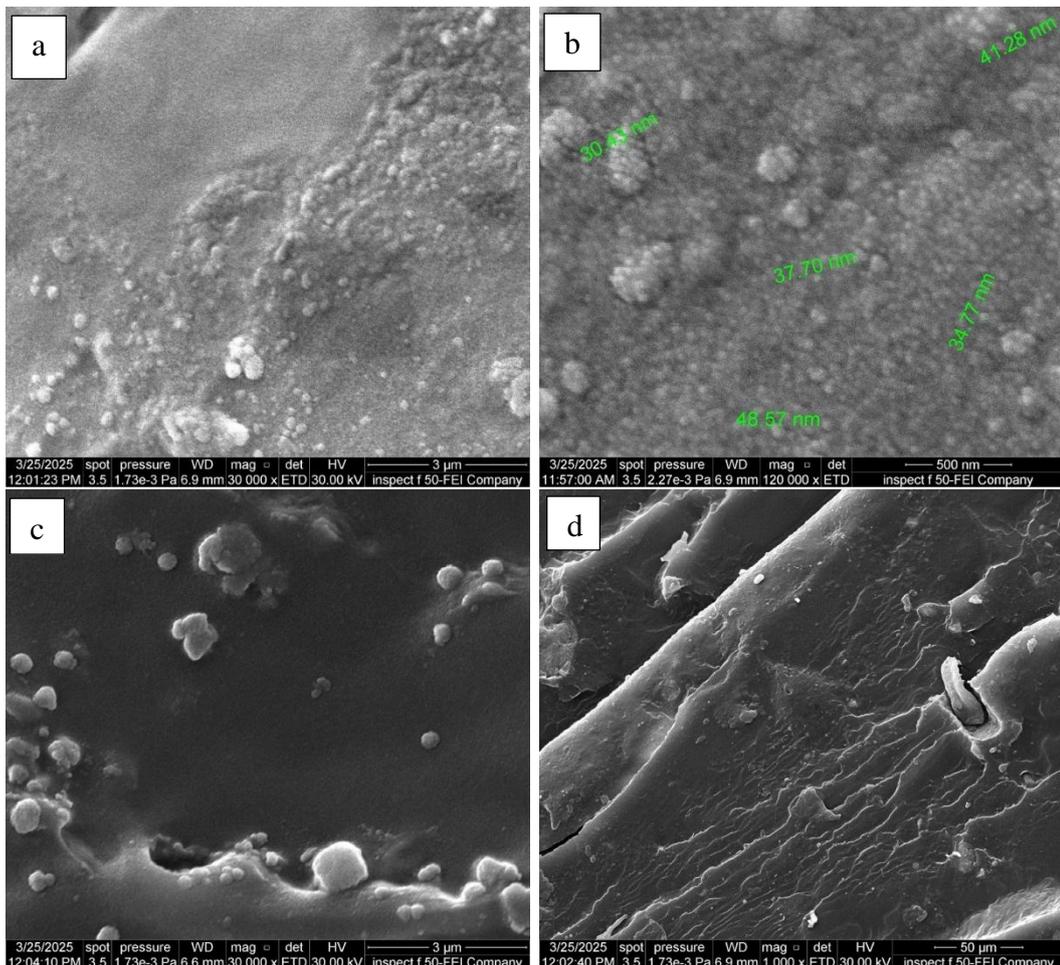
This correlation demonstrates that the primary parameters affecting the mechanical improvement of PMMA nanocomposites are the quality of dispersion and interfacial adhesion between the filler and the polymer matrix.

### CONCLUSION

This work demonstrated how nanoparticle reinforcement greatly affects the mechanical and morphological properties of PMMA composites. Zirconium oxide

(ZrO<sub>2</sub>) nanoparticles significantly improved the PMMA matrix's hardness and impact strength; the most improvement was observed at 3 weight percent ZrO<sub>2</sub>. The uniform distribution and strong interfacial adhesion between ZrO<sub>2</sub> nanoparticles and the polymer matrix was responsible for this improvement, according to SEM examination, which revealed a smooth and homogenous surface with equally dispersed nanofillers.

When iron oxide (FeO<sub>3</sub>) was added to ZrO<sub>2</sub>, mechanical performance decreased. SEM pictures of the PMMA/ZrO<sub>2</sub>/FeO<sub>3</sub> composites showed surface flaws and particle aggregation, suggesting decreased interfacial adhesion and the existence of stress concentration sites that promoted fracture initiation. These anatomical features are precisely associated with the decreased hardness and impact strength values discovered through research.



**Figure 4.** FE-SEM of the sample, (a,b) 3 μm, 500 nm for PMMA/ ZrO<sub>2</sub>/ Fe<sub>2</sub>O<sub>3</sub>, (c,d) 3 μm, 50 μm for PMMA/ ZrO<sub>2</sub>.

Additionally, a comparison of heat-cured and cold-cured PMMA composites revealed that the heat-cured specimens performed better mechanically. This might be explained by a more compact cross-linking structure and a higher polymerization efficiency.

The results show that interfacial compatibility and nanoparticle distribution have a major impact on the mechanical characteristics of PMMA nanocomposites. ZrO<sub>2</sub> is an effective reinforcing agent for increasing PMMA's stiffness and toughness, however including FeO<sub>3</sub> requires adjustment to prevent aggregation and enhance interfacial bonding.

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#### CONFLICTS OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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