

Research Article

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Assessment of surface hardness and impact strength of denture base resins reinforced with silver–titanium dioxide and silver–zirconium dioxide nanoparticles: *In vitro* study

<https://doi.org/10.1515/eng-2024-0064>

received May 12, 2024; accepted July 05, 2024

Abstract: Polymethyl methacrylate (PMMA) is frequently utilised for fabricating denture bases due to its perfect qualities. However, a significant issue with this resin is the occurrence of frequent fractures caused by heavy chewing power, resulting in early cracks and fractures during clinical use. This study investigates the influence of silver, titanium dioxide, and silver zirconia nanoparticles on the surface hardness and impact strength of self-cured denture base. The samples were categorised into four categories according to the incorporation of different nanoparticles. The samples were divided into three subgroups based on the nanoparticle content: 0.1, 0.3, and 0.5% of TiO_2 and ZrO_2 . Each group had a set ratio of 0.3% Ag as an antibacterial agent. Except for the fourth group (Group D), a combination of 0.05, 0.15, and 0.25% of TiO_2 and ZrO_2 , along with 0.3% Ag, was utilised to investigate their collective impact. The Shore D hardness and Charpy test were employed to quantify the surface hardness and impact strength, respectively. The samples were subjected to X-ray diffraction analysis and field emission-scanning electron microscopy to characterise nanoparticles and ascertain the structure of acrylic samples. All nanoparticle-modified samples showed a substantial improvement in surface hardness compared to the control group. The maximum hardness value was seen in the samples containing 0.3% Ag–0.3% TiO_2 and 0.3% Ag–0.5% ZrO_2 . The samples treated with 0.3% Ag and 0.3% TiO_2 showed the maximum impact strength. The incorporation of Ag and

ZrO_2 hinders the ability to withstand impact strength. The samples treated with 0.3% Ag, 0.15% TiO_2 , and 0.15% ZrO_2 exhibited an augmentation in impact strength. Modified samples in all groups showed a colour change, which required colour modifiers.

Keywords: denture base, impact strength, nanoparticles, PMMA, surface hardness

1 Introduction

Polymethyl methacrylate (PMMA) is extensively used to construct removable complete and partial dentures, making it one of the most commonly employed materials in this field [1]. This could be due to several reasons, such as its ease of manipulation, low cost, biocompatibility, oral environment stability, and aesthetic characteristics [2]. Despite all of the above, PMMA often encounters denture failures while being used due to its unfavourable properties, including its inherent susceptibility to fracture when dropped or exposed to significant occlusal stresses; this could generate several problems, especially for those who wear full dentures [3,4]. Recently, nanotechnology has benefited dentistry due to the growing demand for improved materials used in denture bases [5,6]. Nanoparticles such as silver, alumina, titanium oxide, and zirconia are used to improve the mechanical properties of denture resins [7]. The unique physicochemical properties of nanoparticles, such as ultra-small sizes, increased chemical reactivity, and high surface-area-to-mass ratios, are harnessed in diverse fields, including drug delivery, sensing, and antimicrobial applications [8,9]. By incorporating nanoparticles into base materials, advanced and specially designed materials can be created with unique mechanical and physical characteristics that are impossible with just the base material [10]. The addition of silver nanoparticles to resin composites greatly enhances their ability to effectively inhibit the growth

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of oral streptococci, providing strong antibacterial properties. The enhancements render AgNP-infused resin composites exceedingly auspicious for dental restorations and other clinical applications, providing robustness and enduring safeguards against bacterial infections [11,12]. Furthermore, scientists have an increasing interest in nano-compounds that include nano-titanium dioxide. This substance is inexpensive, biocompatible, chemically stable, non-toxic, highly refractive, and resistant to corrosion. Additionally, it has demonstrated antibacterial capabilities [13]. Recently, there has been significant interest in incorporating ZrO₂ nanoparticles into PMMA to improve its strength and enhance its characteristics. The study indicates that nano-ZrO₂ nanoparticles have favourable characteristics that can enhance the mechanical properties of PMMA, such as elevated surface hardness, fracture toughness, and flexural strength [14,15].

This study aims to investigate the surface hardness and impact strength of self-cured acrylic resins that contained a constant percentage of 0.3 wt% Ag, and three different percentages, 0.1–0.3 wt%, and 0.5 wt%, of TiO₂ and ZrO₂ nanoparticles and compared with the control group.

2 Materials and methods

Self-curing PMMA powder (static S.A, Antioquia-Colombia) and methyl methacrylate (MMA) liquid monomer (static S.A, Antioquia-Colombia) were used as denture base material in this study. Silver nanoparticles 50 nm (Sky Spring Nanomaterials, USA), titanium dioxide, and zirconium dioxide nanoparticles 10 nm (Sky Spring Nanomaterials, USA) were used as inorganic fillers to prepare the nanocomposite. A total of 60 specimens were prepared for this work, using silicone moulds in dimensions 55 mm × 10 mm × 3 mm for assessing the surface hardness and impact strength. The self-curing denture base resin polymer was mixed with the monomer at a ratio of 2:1, according to the manufacturer's instructions, to create the control (group A).

The silver and titanium dioxide nanoparticles with a percentage of 0.3% Ag and 0.1–0.3, and 0.5 wt% TiO₂ were added to prepare specimens (group B). They were added to the monomer MMA contained in a test tube. To achieve homogeneous MMA/Ag/TiO₂ mixtures with equal distribution of particles, this mixture was magnetically stirred for 30 min, followed by ultrasound in a cold-water bath for 50 min. PMMA powder was added to the mixture and immediately packaged into the mould. The samples were set aside in the mould until the temperature dropped and the reaction was complete. After that, it is removed from the moulds, left at room temperature for 24 h, and immersed in distilled water for 48 h before each test.

Table 1: Symbols of each type of material in groups being examined

No.	Groups of specimens	Code of specimens	Percentage of nanoparticle (wt)
1	Group B	B1	0.3% Ag–0.1% TiO ₂
		B2	0.3% Ag–0.3% TiO ₂
		B3	0.3% Ag–0.5% TiO ₂
2	Group C	C1	0.3% Ag–0.1% ZrO ₂
		C2	0.3% Ag–0.3% ZrO ₂
		C3	0.3% Ag–0.5% ZrO ₂
3	Group D	D1	0.3% Ag–0.05% TiO ₂ –0.05% ZrO ₂
		D2	0.3% Ag–0.15% TiO ₂ –0.15% ZrO ₂
		D3	0.3% Ag–0.25% TiO ₂ –0.25% ZrO ₂

In the same proportion as titanium oxide, zirconium oxide was added with silver to the PMMA powder to prepare specimens (group C). It was stirred manually before subjecting the mixture to ultrasound. Then, the monomer was added as a final step before being poured into the moulds [16]. Group D combines the three nanomaterials to study how they collectively affect the PMMA's characteristics. Ag was added at a fixed ratio of 0.3% with each mixture. The proportion was 0.05% TiO₂ + 0.05% ZrO₂, 0.15% TiO₂ + 0.15% ZrO₂, and 0.25% TiO₂ + 0.25% ZrO₂ (Table 1 and Figure 1).

To characterise the nanoparticles, they were examined using X-ray diffraction (Malvern Panalytical, The Netherlands). The morphology was investigated using a field scanning electron microscope (FE-SEM INSPECT 50FEI, The Netherlands).

2.1 Surface hardness (shore D) test

Hardness (shore D) was performed using a handheld shore ASTM D2240-05(2010) device. To assess the depth of indentation, the values were modified according to the content of the specimen materials. With the digital screen of the device, the final value of the hardness was detected. Three different points were tested, and the average value was finally considered for each specimen.

2.2 Impact strength test

The Charpy impact test was conducted to determine the fracture toughness of PMMA rein before and after the reinforced process. A Charpy-type impact test apparatus was used (Impact Tester N. 43-1, TMI, USA). The principle of the

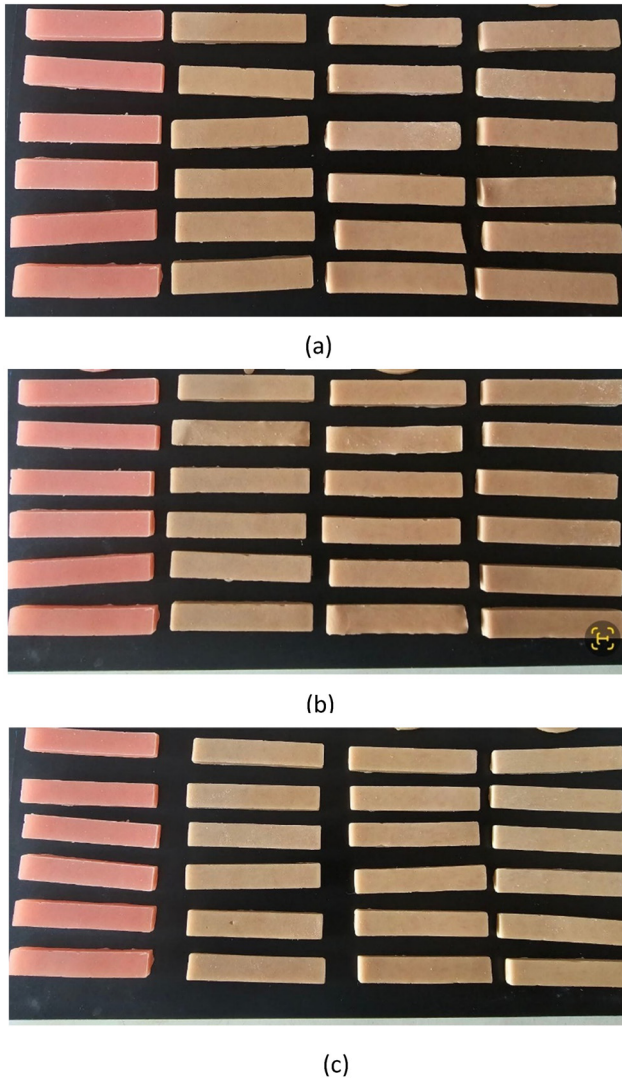


Figure 1: Nanoparticle-enforced specimens: (a) silver and titanium dioxide nanoparticles, (b) silver and zirconium dioxide nanoparticles, (c) silver, titanium dioxide, and zirconium dioxide nanoparticles.

Charpy impact test is to determine the amount of energy absorbed by a material sample during the fracture, where a pendulum of energy (2 J) is calculated from the following equation:

$$\text{Impact Strength (kJ/m}^2\text{)} = \frac{\text{Energy of fracture (kJ)}}{\text{Cross - sectional area (m}^2\text{)}} \quad (1)$$

3 Results

3.1 X-ray diffraction

The crystal structure of the prepared samples was determined using X-ray diffraction, as shown in Figure 2(a)–(c).

The XRD patterns of the silver nanoparticles, shown in Figure 2(a), exhibit three peaks at $2\theta = 38.2^\circ$, 44.1° , and 64.3° , which correspond, respectively, to the (111), (200), and (220) planes face-centred cubic (FCC) metallic silver, and all peaks are compatible with JCDPS 04-0783. In Figure 2(b), the XRD patterns of TiO_2 nanoparticles exhibited diffraction peaks at 25° , 37.9° , 47.6° , and 54.4° , which correspond to the (101), (004), (200), and (105) planes in the anatase phase and the diffraction data were consistent with JCPDS 21-1272 [17]. The XRD results confirmed the monoclinic structure of zirconium dioxide nanoparticles. In Figure 2(c), the XRD patterns of ZrO_2 nanoparticles exhibited diffraction peaks at $2\theta = 24.1^\circ$, 28.2° , 31.4° , and 40.7° corresponding to the (110), (-111) , (111), and (-121) and the peaks were compatible with JCPDS 37-1484. It also showed a peak at 50.8° , which corresponds to (200) and belongs to the tetragonal phase of ZrO_2 [JCPDS 17-0923] [18].

3.2 Field emission-scanning electron microscopy (FE-SEM)

The morphology of the prepared sample was characterised by FE-SEM, as depicted in Figure 3(a)–(c). The morphology exhibited a compact and uniform structure, as shown in Figure 3(a) for a sample modified with silver and titanium oxide and Figure 3(c) for a sample modified with silver, titanium oxide, and zirconium dioxide. These figures demonstrate the formation of nanostructures characterised by compact arrangements and uniform dispersion of nanoparticles. Conversely, Figure 3(b) shows samples modified with silver and zirconium dioxide, demonstrating less nanoparticle distribution.

3.3 Surface hardness

The samples from the control (group A) exhibited a surface hardness of 81.2. We significantly improved surface hardness by incorporating silver and titanium dioxide nanoparticles into group B. The highest recorded value was 85.3, with a 0.3% Ag–0.3% TiO_2 composition. The results demonstrated that the surface hardness of the modified samples in group C increased gradually with the addition of silver and zirconium dioxide nanoparticles. Specifically, the surface hardness value increased as the percentage of zirconium dioxide nanoparticle increased. The highest hardness value recorded was 85.5 at 0.3% silver and 0.5% zirconium dioxide. The modified samples showed that the addition of

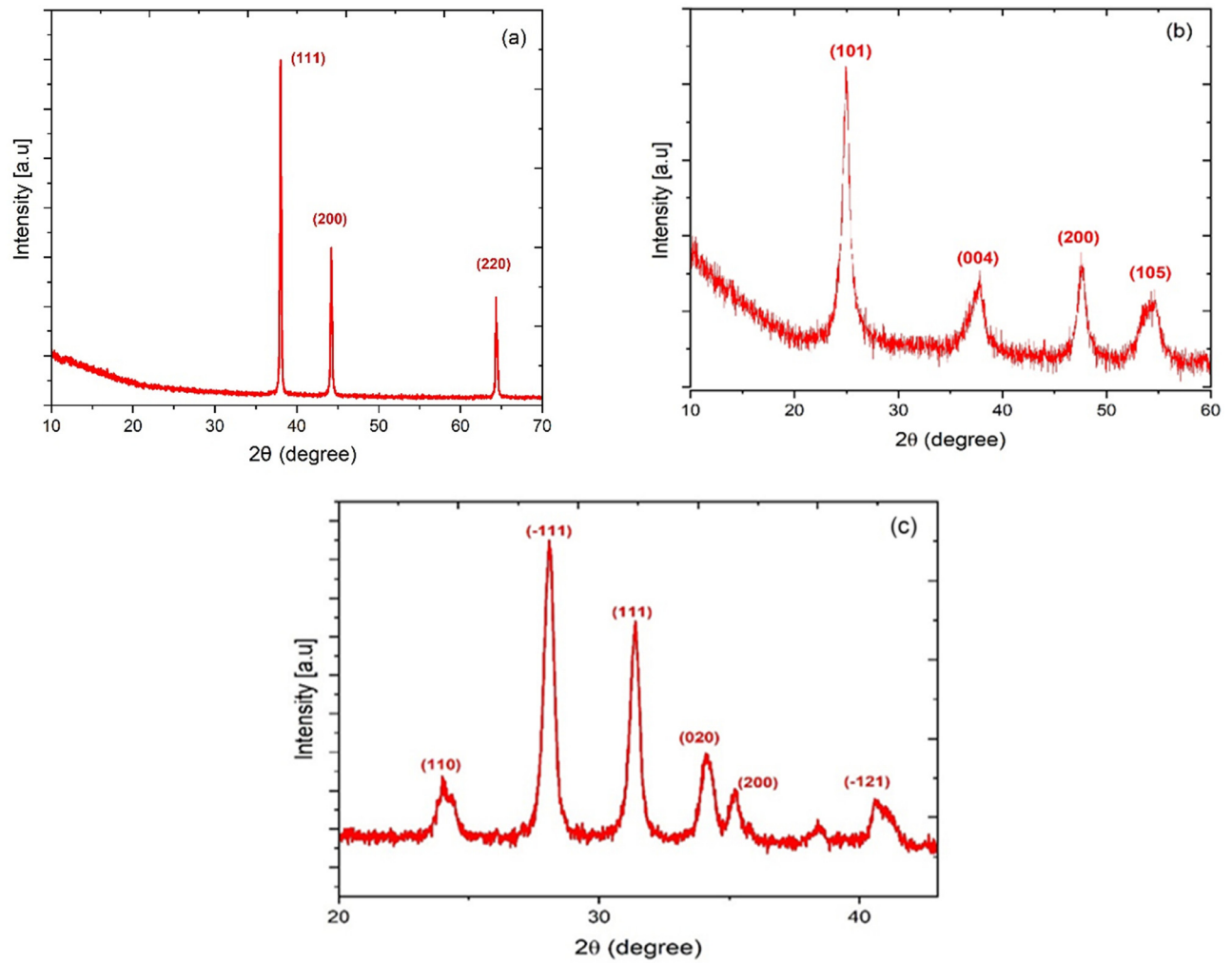


Figure 2: XRD patterns of the nanoparticles: (a) silver nanoparticles, (b) titanium dioxide nanoparticles, and (c) zirconium dioxide nanoparticles.

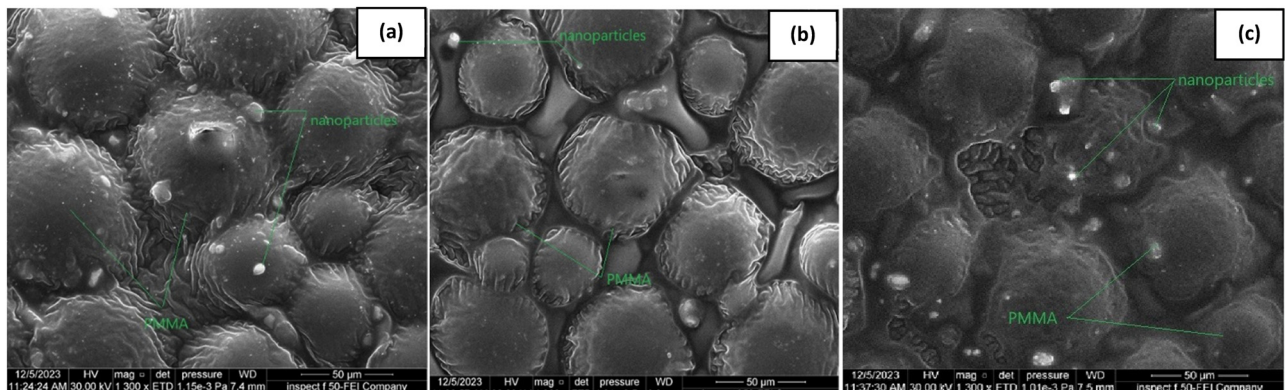


Figure 3: FE-SEM images showing nanoparticle distribution in a polymer matrix: (a) 0.3% Ag-0.3% TiO₂, (b) 0.3% Ag-0.3% ZrO₂, and (c) 0.3% Ag-0.15% TiO₂-0.15% ZrO₂.

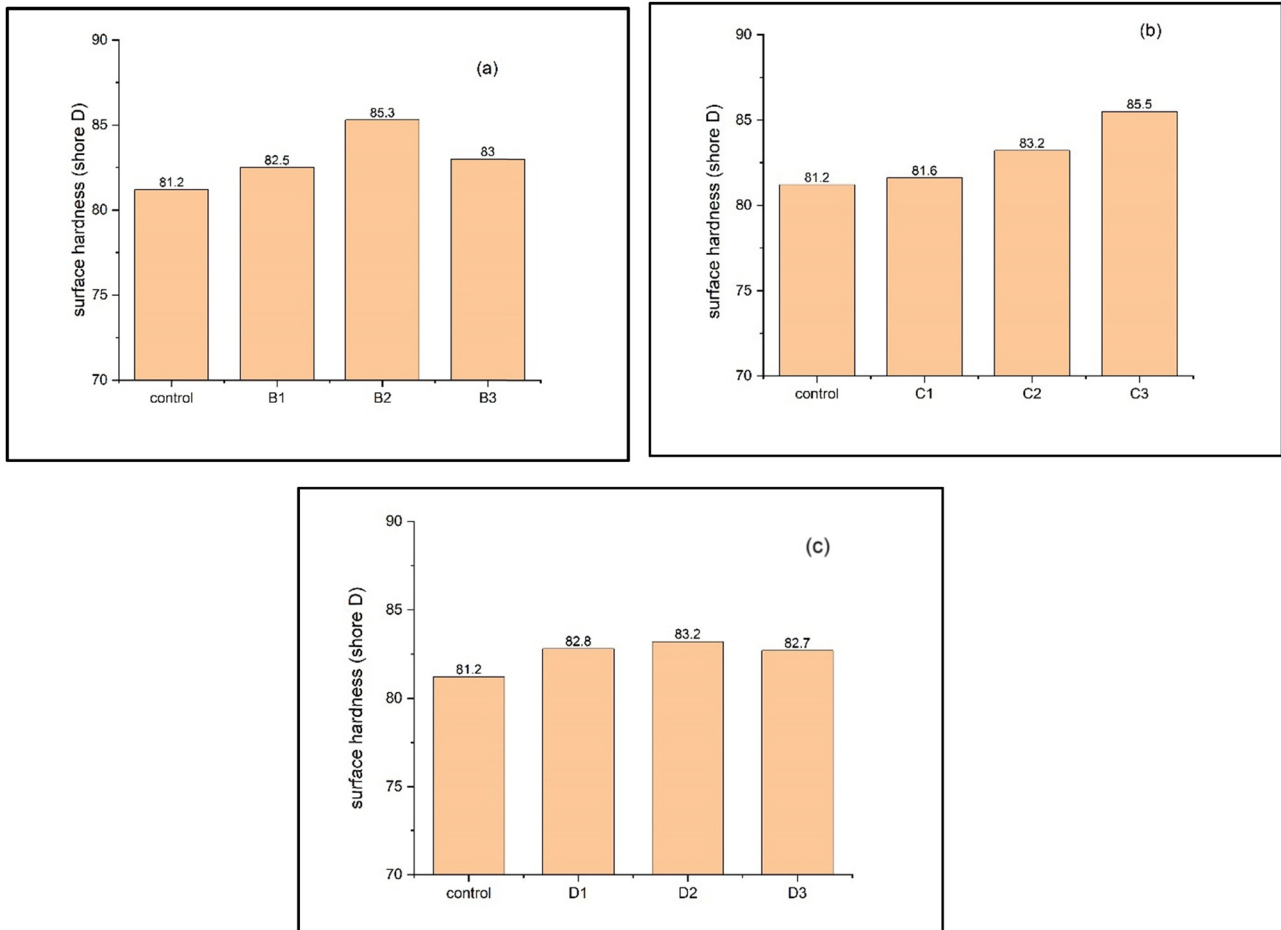


Figure 4: Surface hardness rate with the percentage of addition of (a) silver and titanium dioxide nanoparticles, (b) silver and zirconium dioxide nanoparticles, and (c) silver, titanium dioxide, and zirconium dioxide nanoparticles.

a combination of nanoparticles in group D resulted in the most excellent hardness value of 83.2 when 0.3% Ag, 0.15% TiO₂, and 0.15% ZrO₂ were used (Figure 4).

3.4 Impact strength

The control (group A) attained an impact strength value of 6.81 kJ/m². Group B exhibited a positive development with a 0.3% Ag–0.3% TiO₂ composition (group B). Its impact strength value reached 7.54 kJ/m². We observed a progressive increase in the impact strength value in group C as the addition percentage increased. However, the impact strength value in this group was lower than that of the control group. In group D, a similar pattern of behaviour was seen as in group B. Specifically, the impact strength value increased to 7.14 kJ/m² with an addition of 0.3% Ag–0.15% TiO₂–0.15% ZrO₂. However, the impact strength value decreased as the percentage increased (Figure 5).

4 Discussion

In the present study, three nanoparticles (Ag, TiO₂, and ZrO₂) were selected due to their excellent mechanical properties [19,20]. As shown in Figure 2(a)–(c), X-ray diffraction confirmed the absence of impurities and the nanoparticle purity [18]. The morphology of the sample surfaces described by FE-SEM was uniform and regular, with a good distribution of nanoparticles, as shown in Figure 3(a)–(c).

The surface hardness rate is determined by the percentage of silver and titanium dioxide nanoparticles added (Figure 4(a)). The addition of silver nanoparticles to the polymer matrix increases the surface hardening value. This is because the large surface area of the nanoparticles allows for a strong adhesive bond with the polymer, enabling it to resist external forces effectively [21]. The addition of TiO₂ positively affected the surface hardness of the PMMA denture base, which increased as the percentage of titanium dioxide nanoparticles added to the

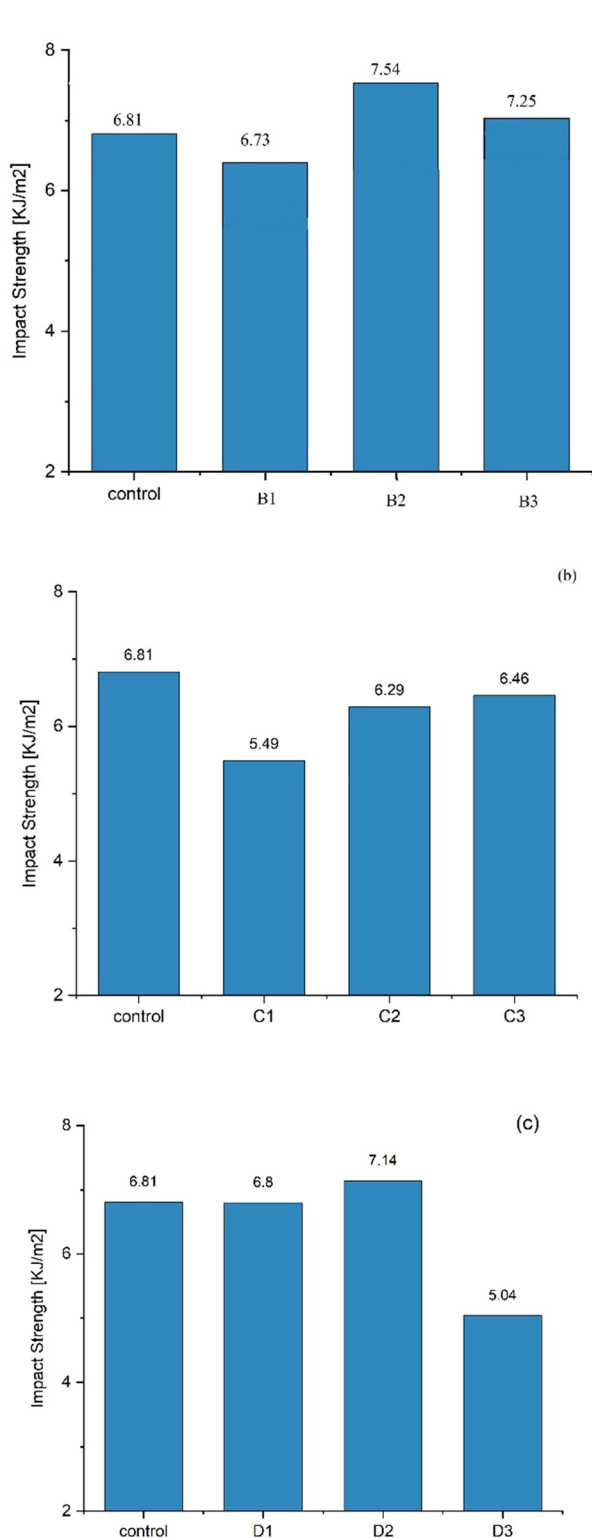


Figure 5: Impact strength with the percentage of addition of (a) silver and titanium dioxide nanoparticles, (b) silver and zirconium dioxide nanoparticles, and (c) silver, titanium dioxide, and zirconium dioxide nanoparticles.

(a) modified samples increased compared to the control group. The increase in polymer hardness can be explained by the structural bonds between the polymer matrix and the added nanoparticles that bear the force applied; more energy is needed to break these bonds [22,23].

As the percentage of zirconium dioxide nanoparticles increases, surface hardness increases gradually; an increase in surface hardness can be attributed to ionic solid interatomic bonding, as well as the dimensions and arrangement of the filler material within the polymer [15,24], as shown in Figure 4(b).

The study involved the integration of TiO_2 , ZrO_2 , and Ag nanoparticles into PMMA to examine their influence on the material's surface hardness. An increase in the proportion of nanoparticles resulted in a noticeable enhancement in surface hardness. The maximum value was achieved with a composition of 0.3% Ag, 0.15% TiO_2 , and 0.15% ZrO_2 , which measured 83.2. This behaviour can be attributed to the effective dispersion of nanoparticles throughout the polymer matrix [25,26]. Another factor is that inorganic compounds have superior resistance to penetration [27].

The polymer becomes saturated when filled with a greater quantity of filler. The polymer reached its maximum capacity to absorb filler, leading to a decline in its mechanical qualities [28]. This behaviour clarifies the decrease in surface hardness as the proportion of additives increased, as depicted in Figure 4(c).

The study shows that incorporating TiO_2 nanoparticles into the denture base material improves its impact strength, as shown in Figure 5(a). The enhanced impact strength can be ascribed to the strong adhesion between titanium dioxide nanoparticles and the polymer matrix [29]. Moreover, the increase in the impact strength can be related to the transfer of stress to nanoparticles incorporated into the polymer matrix. This leads to a decrease in the impact of the applied stress [30].

A study was carried out to investigate the impact of ZrO_2 nanoparticles on the strength of denture base materials. The addition of ZrO_2 nanoparticles negatively affected the impact strength of denture resin, as shown in Figure 5(b). Although the percentage addition increased, the impact strength value remained lower than that of the control group. Including ZrO_2 nanoparticles adversely affects the facade area due to insufficient adhesion between the filler particles and the polymer matrix. The reason for reduced energy dissipation results in a decline in the impact strength [7,21].

The results demonstrated an increase in the impact strength when TiO_2 , ZrO_2 , and Ag nanoparticles were

integrated into PMMA compared to the control group, as shown in Figure 5(c). The increase in the impact strength values was attributed to the strong correlation between the polymer matrix and the nanofiller particles [31]. Another contributing aspect to the increase is the minimal size of the particles. This leads to the filling of the spaces between the polymer chains and generates a significant surface area that helps in the dispersal of energy [32–34].

5 Conclusion

1. SEM images revealed a uniform dispersion of nanoparticles throughout the polymer matrix.
2. All assemblages showed a notable increase in their surface hardness. The specimens containing 0.3% Ag–0.3% TiO₂ and 0.3% Ag–0.5% ZrO₂ presented the highest hardness level.
3. The addition of silver and titanium dioxide nanoparticles at a concentration of 0.3% Ag–0.3% TiO₂ improved the impact strength of PMMA.
4. The addition of silver and zirconium dioxide nanoparticles harmed the impact strength.
5. The addition of silver, titanium dioxide, and zirconium dioxide nanoparticles at a ratio of 0.3% Ag–0.15% TiO₂–0.15% ZrO₂ improved the impact strength.
6. All samples in each group exhibited a colour change, requiring colour modifiers.

Acknowledgements: The authors thank the Technological University – Department of Applied Sciences – Medical and Industrial Materials Branch – Postgraduate Laboratories for supporting this work.

Funding information: Authors state no funding involved.

Author contributions: All authors have accepted responsibility for the entire content of this manuscript and consented to its submission to the journal, reviewed all the results and approved the final version of the manuscript. EAE is responsible for conducting the literature review, performing experiments, and analysing and interpreting data. MAA proposed the project and supervised the analysis of the work results. HTH performed manuscript revision and conducted a thorough review of the referenced papers.

Conflict of interest: Authors state no conflict of interest.

Data availability statement: The datasets generated during and analysed during the current study are available from the corresponding author upon reasonable request.

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