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Research Paper

Investigating the Mechanical, Physical and Biological Properties of PMMA/TiO_2 Composites with Nanoclay for Denture Applications

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Prosthodontics uses permanent or removable dentures to replace missing teeth, with partial dentures made from cobalt chromium and full dentures from acrylic resin. This study presents Veracril self-curing acrylic, a material for hybrid artificial denture composites reinforced with montmorillonite (MMT) and titanium oxide (TiO_2) particles. It is softer, lighter, and more stable than previous materials. Several experiments (mechanical, physical, and biological) have been carried out to examine the novel composite at various MMT and TiO_2 weight ratios. The findings indicate that these additives significantly improve the mechanical properties of the matrix material, making it suitable for denture manufacturing. The addition of MMT as a filler enhances bonding between the matrix and additives. The additives' success in biological tests, including antibacterial activity and toxicity assessments, further supports their suitability for dental applications.

Keywords: biological properties; denture applications; nanoclay; TiO_2; nano-composites.

1. Introduction

Dental materials science studies the physical, chemical, and mechanical properties of dental materials and their manipulation. It focuses on managing these properties to aid dentists in the appropriate selection and application of dental materials. Understanding dental materials helps dental professionals comprehend their behavior, select appropriate equipment (the right materials) for patients, and use them effectively. In prosthodontics, missing teeth are replaced with either permanent or removable dentures. Partial dentures typically consists of clasps, occlusal bases, and surrounding natural teeth. Cobalt chromium is commonly used for partial dentures, while acrylic resin is used for full dentures [1, 2]. Dental prostheses quickly adopted the translucent polymer poly (methyl methacrylate) (PMMA), which was introduced in the 1930s. PMMA rapidly replaced vulcanite as the most widely used denture base material, becoming a critical component in dental prostheses [3].

Furthermore, the market share of PMMA was not significantly threatened by the discovery of various thermoplastic polymers starting in the mid-1960s for use as denture base materials. These included polycarbonates, polystyrene, polyvinyl acrylic, polyamides, and polyoxymethylene [4]. Although PMMA polymers can be used as a replacement for acrylic resins, each of these materials has its own benefits. Despite this, PMMA resin remains the recommended material for denture bases, as none of the alternatives have shown to surpass poly (methyl methacrylate) in terms of accuracy or performance [5].

Al-Karagholi [6] developed a radiopaque denture base material by adding Kevlar fiber and barium sulphate to acrylic resin. The material was polymerized using microwave and water bath curing systems, and the study found that the addition of Kevlar fiber significantly improved its mechanical properties. Both Kevlar fiber and barium sulphate helped to preserve the material's good mechanical properties.

Vallittu *et al.* [7] examined the impact of various resin matrix compositions containing aramid, glass, and carbon fiber on the fracture resistance of test specimens made of acrylic resin. The results indicated that adding fiber to acrylic resin enhances the fracture resistance of PMMA test specimens. This improvement was more pronounced at higher fiber concentrations.

Using the monsoon and antimonsoon theories, Hamid [8] employed two-dimensional finite element modeling (FEM) to investigate the stress distribution in upper full dentures. The results showed that when these theories were applied, stress concentration occurred at the palatal sides, and these stresses gradually decreased as cusp angulations decreased.

Barão *et al.* [9] applied a vertical force of 100 N to the central incisors, and employed finite element analysis via the Ansys program to analyze stress distribution in complete dentures and implant-retained dentures with different attachment techniques. The application of in-situ produced poly (methyl methacrylate) and TiO_2 nanocomposites dental materials was also explored. PMMA was combined with TiO_2 nanoparticles in varying weight percentages (1wt% and 2wt%) using a melt compounding technique [10].

The synthesized nanocomposites were evaluated mechanically using a micro-indentation test, a scratch test, and field emission scanning electron microscopy

(FESEM) analysis. The effects of varying volumes and percentages of TiO_2 on the composites' mechanical properties were examined, and the results demonstrated that the polymer was strengthened by TiO_2 as a reinforcing agent. With a homogeneous distribution of TiO_2 in the polymer matrix, morphological observations revealed that considerable adhesion between TiO_2 and the polymer matrix. The mechanical properties were enhanced due to the appropriate compatibilization between TiO_2 and the polymer matrix [10].

Research into the development of new TiO_2-based blends for dental use is crucial for advancing modern dentistry. PMMA remains essential and valuable material in the dental field, and ongoing studies aim to unlock its full potential. Researchers are investigating synergistic combinations of materials such as titanium oxide and montmorillonite clay to improve treatment outcomes and enhance patient experiences. These materials offer opportunities to enhance durability, strength, biocompatibility, and both mechanical and biological properties. By understanding their interactions and their impacts on dental applications, we can drive the development of the next generation of dental materials and therapies.

2. Materials and methods

The current study developed a new liquid resin matrix, Veracril self-curing acrylic, using PMMA cold curing to create hybrid artificial denture composites. This multipurpose acrylic is softer, has a lower molecular weight, maintains color stability, shrinks less, and undergoes a flawless polymerization cycle. However, it also has some drawbacks, such as low hardness and strength, as well as greater production difficulties.

This study uses titanium oxide (TiO_2) particles and montmorillonite (MMT) particles as reinforcement materials. TiO_2, a commonly used ceramic oxide in medical applications, was supplied by Shanghai Jyota Chemicals Company. MMT, a widely used clay that improves composite mechanical properties, was supplied by Sigma-Aldrich. The materials are homogenized using a ball mill device (type NOM-0.4 Model Planetary Ball Mill) of American origin for mixing powders at the Nanotechnology Center, University of Technology for 30 min. Both materials have broad applications as reinforcement materials. Three types of mixtures between TiO_2 and MMT are used, as follows:

1) (95% PMMA-5% TiO_2), noted as S1;
2) (90% PMMA-10% TiO_2), noted as S2;
3) (85% PMMA-15% TiO_2), noted as S3.

Next, MMT is added to improve the properties of the mixtures. The addition is made in the following proportions: 1%, 2%, and 3%, as shown in Table 1.

TABLE 1. The percentage of MMT added to the PMMA/TiO_2 mixture.

1% MMT	2% MMT	3% MMT
S1 + 1% MMT	S1 + 2% MMT	S1 + 3% MMT
S2 + 1% MMT	S2 + 2% MMT	S2 + 3% MMT
S3 + 1% MMT	S3 + 2% MMT	S3 + 3% MMT

The mixtures were then placed into an electric mixer (type NOM-0.4 Model Planetary Ball Mill) of American origin for powder mixing, for a period of 45 min at a speed of 850 rad/min, to obtain the optimal mixture of materials.

2.1. *Mechanical tests*

This study evaluates the physicomechanical properties of PMMA through various mechanical and physical tests on composite and hybrid dental base materials.

2.1.1. Tensile test. The tensile test was performed following ASTM D638 [11], using a tensile machine (Computer Control Electronic Universal Testing Machine – Laryee Technology – Model UE34300 – University of Technology) with a crosshead speed (strain rate) of 5 mm/min and a unit load of 5 kN applied until fracture occurred. Figure 1 shows the sample used for the tensile test.

a) b)

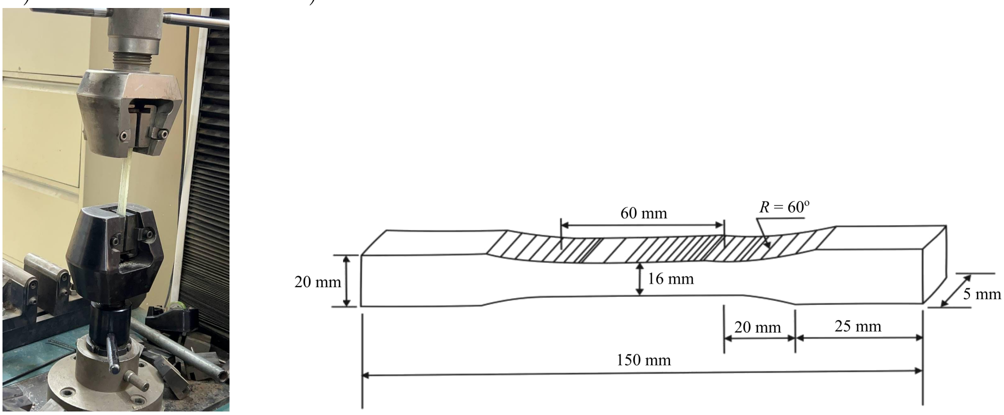

FIG. 1. a) Tensile test machine; b) and tensile test sample.

2.1.2. Compression test. The compression test was performed according to ASTM D695, with a crosshead speed (strain rate) of 5 mm/min, and a load of 25 kN was applied until fracture occurred [12]. Figure 2 illustrates the sample used in the compression test.

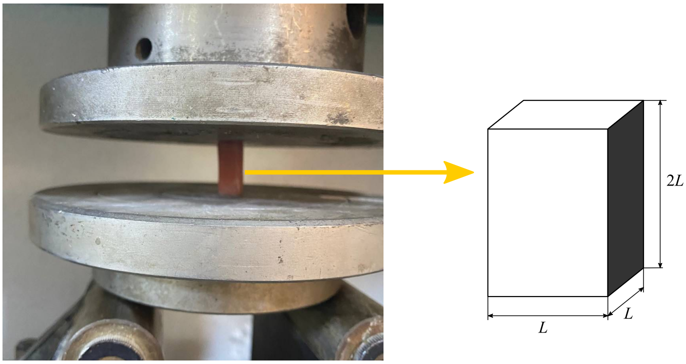

FIG. 2. Compression test sample.

2.1.3. Hardness test. In accordance with (ASTM D2240), this test was carried out using a hardness tester (Dorumeter Shore D Larry Company) with a force of 50 N applied for 15 s. The sample must have a diameter of 30 mm and a thickness of 3 mm, as shown in Fig. 3. Three tests were conducted simultaneously on each sample at different positions. The figure shows the standard sample [13].

a) b)

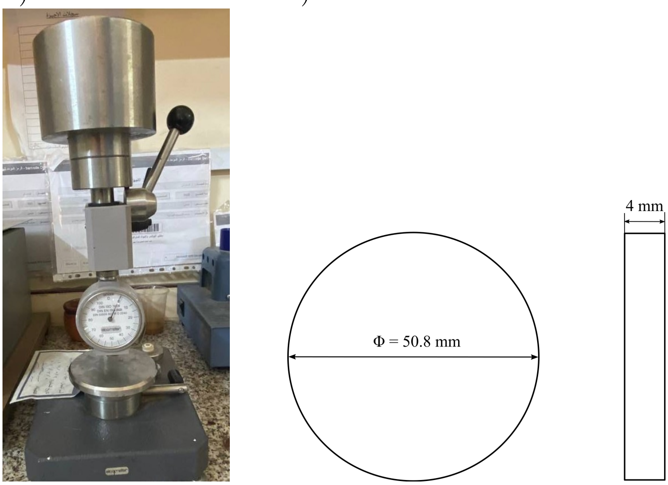

FIG. 3. a) Dorumeter device; b) and the sample dimensions.

2.2. Biological tests

Biological testing is one of the most crucial assessments to determine whether materials are suitable for use as biological materials. These materials are described in the following subsections.

2.2.1. Anti-bacterial activity (Pseudomonas and Staphylococcus aureus). The antibacterial susceptibility of bacterial isolates to various antibiotics was tested using disc diffusion methods. Mueller–Hinton (MH) Agar was prepared by dissolving 38 grams of dried powder in 1000 ml of distilled water, mixing, and boiling. The medium was sterilized by autoclaving at 121°C for fifteen minutes, then distributed into sterile plates. The disc diffusion test involved suspending a pure culture of bacteria in a PBS buffer (pH 7.4), to produce a standard bacterial turbidity of 0.5 McFarland. A volume of 100 µl of bacterial inoculum was streaked onto the MHA plate, which was then incubated overnight at 37°C. The zone of inhibition around each antibiotic disk was measured with a specific ruler. Afterward, the plates were allowed to dry for approximately 5 minutes, and the discs were gently pressed to the agar to ensure firm attachment [14].

2.2.2. Toxicity and biocompatibility. Toxicity and biocompatibility refer to the ability of biological materials implanted in the body to perform their intended functions during medical evaluation. This research uses physical toxicity testing to assess the safety of the materials used, following the ISO 10993-5 test code and the MMT test method. The MMT test accurately measures the effect of drugs and implants on the body and diagnoses toxicity to the original cells.

The MMT test procedure involves planting cells in 96-well plates and placing them in 200 µl dishes. The dishes are covered with sterile film, stirred gently, and incubated at 37°C for 24 hours. The medium is then removed, and the dishes are placed in a special incubator. Afterward, a 5% CO solution is added to each well, and the dishes are returned to the incubator for another 48 hours [15].

2.3. Morphology test

This test was performed on the samples to understand their nature and explain their behaviors. The scanning electron microscope (SEM) – type Inspect S50, in this study – is an essential device in industrial fields, providing high-resolution images and information about materials, as shown in Fig. 4. SEM images are taken at the microscopic level to analyze the nature of materials in terms of compactness and homogeneity [16].

2.4. Physical test

2.4.1. Water absorption test. According to ASTM D570, the samples were submerged in distilled water at a specified time and temperature. After being fully submerged in a bowl of distilled water at room temperature (often 23°C) for 24 hours, the samples were removed of the water and weighed using a digital scale, ensuring all surfaces were cleaned with dry towels beforehand [17].

Fig. 4. Inspect S50 device.

Figure 5 shows the samples at various ratios produced according to each test standard, as mentioned previously in Sec. 2.

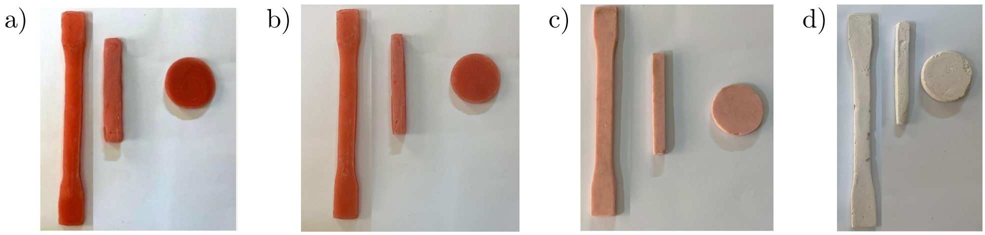

Fig. 5. PMMA/TiO_2 samples at various ratios: a) pure (PMMA), b) 95%PMMA+5%TiO_2, c) 90%PMMA+10%TiO_2, d) 85%PMMA+15%TiO_2.

3. Results and discussion

3.1. Mechanical test

3.1.1. Tensile strength. The tensile test is crucial for assessing the mechanical behavior of composite materials, as it indicates their resistance to failure. The test is conducted at room temperature and 20% humidity. Table 2 clearly

Table 2. Tensile test results.

Sample	Tensile strength [MPa]			
	Pure (without MMT)	1% MMT	2% MMT	3% MMT
100% (PMMA)	53 ±2	57 ±1	58.5 ±1	61 ±1.5
S1	60 ±2	62 ±1	67 ±1	71 ±1.5
S2	66 ±2	72.5 ±1	74 ±1	80 ±1.5
S3	68.5 ±2	73 ±1	74 ±1	65 ±1.5

shows that the results vary with the addition ratios of the base material. The tensile strength of the composites with ratios S1, S2 and S3 increases by about 13%, 24%, and 29%, respectively.

The results in Fig. 6 and Table 2 clearly show that certain modifications occurred when MMT was added to the composites in accordance with the specified ratios. For example, 1% MMT was added to the base material or composites containing both PMMA and TiO_2. The sample (100% PMMA + 1% MMT), composite (S1 + 1% MMT), composite (S2 + 1% MMT), and composite (S3 + 1% MMT) all show increases in tensile strength of around 8%, 22%, 36%, and 38%, respectively.

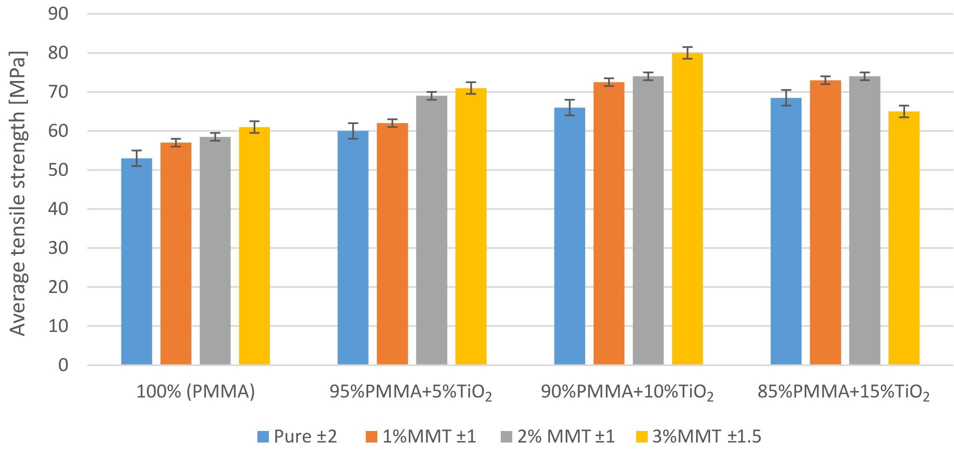

Fig. 6. Tensile test results.

As the MMT addition ratio increase, the tensile strength further improves. For samples with (100% PMMA + 2% MMT), (S1 + 2% MMT), (S2 + 2% MMT), and (S3 + 2% MMT), the increases were 10%, 30%, 43%, and 41%, respectively.

Finally, the tensile strength increases following the addition of 3% MMT were 15%, 33%, 50%, and 22% for 100% PMMA, S1, S2, and S3, respectively.

Analyzing and graphing the findings reveal the beneficial influence of the additives on the base material, with an improvement in mechanical qualities as the weight fraction increases. This behavior can be attributed to the nature of the physical bond between the additives and the base material, as well as the strengthening/reinforcement mechanism via dispersion. This mechanism impedes the sliding in PMMA resin chains, which requires high energy for sliding or movement, ultimately increasing the stress values. Additionally, the role of nanomaterials is significant in reducing the spaces between molecules and increasing the obstacles that hinder molecular movement [18].

3.1.2. Compression test. Compression tests are important for dentures, as they naturally expose to compressive forces during chewing and tooth movement. The test is performed at room temperature and 20% humidity.

According to Table 3 and Fig. 7, the compressive strength of 100% PMMA is 76 MPa and it increases with the addition of TiO_2, by about 4%, 9%, and 12% for S1, S2 and S3, respectively. When 1% MMT was added to the samples of 100% PMMA, S1, S2, and S3, the compressive strength increased by 8%, 8%, 5% and 4%, respectively.

TABLE 3. Compression test results.

Sample	Compressive strength [MPa]			
	Pure (without MMT)	1% MMT	2% MMT	3% MMT
100% (PMMA)	76 ±2	82 ±1.5	86.8 ±1.5	91.3 ±1
S1	79.2 ±2	85.42 ±1.5	88.35 ±1.5	93.13 ±1
S2	82.8 ±2	87.17 ±1.5	92.25 ±1.5	96.4 ±1
S3	85.31 ±2	88.95 ±1.5	93.47 ±1.5	98.95 ±1

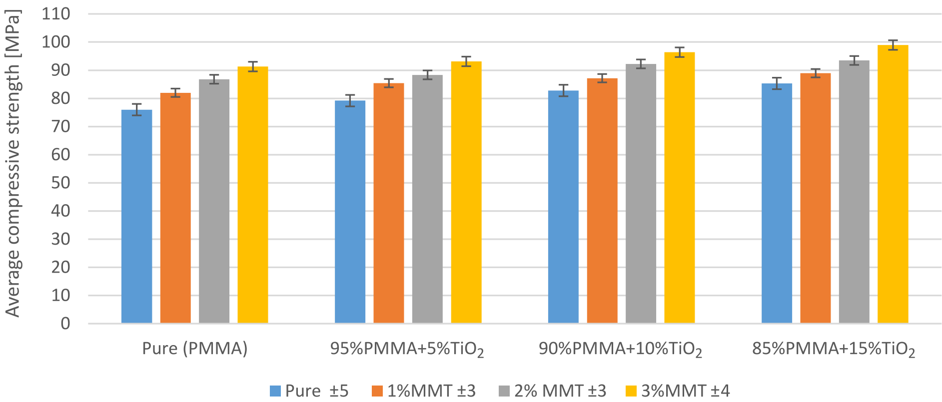

FIG. 7. Compression test results.

The compressive strength further increases when 2% MMT is added with the following percentage increases: 11%, 12%, 11%, and 10% for samples 100% PMMA, S1, S2, and S3, respectively. Finally, adding 3% MMT results in increases of 20%, 17%, 16%, and 16% for samples 100% PMMA, S1, S2, and S3, respectively.

The results above indicate that, additives have an impact on improving compressive properties because they help form strong interfacial bonds between matrix materials, which has been shown to improve this property [19, 20]. Additionally, nanoparticles play a crucial role in impeding crack movement and deflecting their paths, which requires greater forces for failure to occur [21, 22].

3.1.3. Hardness test. Hardness is one of the most crucial and necessary qualities in denture manufacturing due to the friction and wear dentures are subjected to. The hardness test is conducted to ensure that the materials used exhibit good mechanical qualities that are appropriate for use in dental applications. Following the examination, the results obtained, as shown in Table 4 and Fig. 8, illustrate the impact of the additives on the base material.

TABLE 4. Results of hardness tests.

Sample	Hardness (Shore D)			
	Pure (without MMT)	1% MMT	2% MMT	3% MMT
100% (PMMA)	60.00 ±5	67.00 ±3	73.00 ±3	80.00 ±4
S1	65.40 ±5	70.20 ±3	77.43 ±3	86.65 ±4
S2	69.00 ±5	76.25 ±3	83.30 ±3	90.12 ±4
S3	73.50 ±5	81.35 ±3	88.90 ±3	94.18 ±4

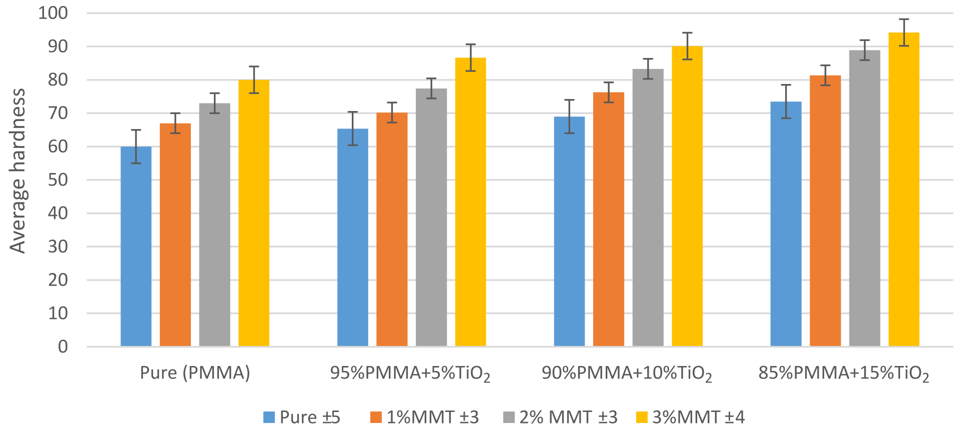

FIG. 8. Hardness test results.

The findings above demonstrate that the direct effect of the additives on both the base material and the composite compositions. The hardness value of 100% PMMA increases with the addition of TiO_2, by about 9%, 15%, and 22.5%, in the ratios of S1, S2 and S3, respectively. When 1% of MMT was added, the hardness values for the base material and composites (100% PMMA, S1, S2, and S3) increased by about 11.7%, 7.3%, 10.5%, and 10.6%, respectively.

When the addition rate of MMT is increased by 2%, the hardness increases by 22%, 20%, 20%, and 21% for samples 100% PMMA, S1, S2, and S3, respectively. Finally, adding 3% MMT increases hardness by 33%, 32%, 30%, and 28% for 100% PMMA, S1, S2, and S3, respectively.

The findings above clearly demonstrate an increase in hardness results, indicating the direct effect of the additives. It is well known that the hardness is measured by the resistance needed to remove a material with a harder substance. The results show that the resistance value increased as the number of additives increased. This can be attributed to the stronger bond formed between the base material and the additives, which enhanced interfacial friction. As a result, the mechanical properties, such as hardness, were improved, leading to an increased resistance to material disintegration [23].

3.2. Physical tests

Physical tests are crucial in demonstrating the impact of additives on the base material, providing insights into the behavior of target materials.

Water absorbency is crucial in denture manufacturing as it prevents material cracking and implant failure. The porosity test is another key physical test that determines the bulk density of selected materials and their real mass, both of which are crucial for selecting the appropriate material for dentures. The results of these tests help determine the material's real/ overall mass and suitability for the intended application mass [10].

As shown in Table 5 and Fig. 9, a decrease in water absorption values is observed when TiO_2 is added to PMMA. Additionally, when 1% MMT is added, the absorbance value decrease by around 6%, 5%, 2%, and 5% for 100% PMMA, S1, S2, and S3, respectively. With the addition of 2% MMT, the decrease in absorption rates is 10%, 10%, 8%, and 9% for 100% PMMA, S1, S2, and S3, respectively. Finally, at 3% MMT, the water absorption decreases by about 18%, 17%, 14%, and 16% for 100% PMMA, S1, S2, and S3, respectively.

Table 5. Water absorption values.

Sample	Water absorption values			
	Pure (without MMT)	1% MMT	2% MMT	3% MMT
100% (PMMA)	0.430 ±0.02	0.405 ±0.05	0.386 ±0.02	0.352 ±0.03
S1	0.412 ±0.02	0.391 ±0.05	0.369 ±0.02	0.341 ±0.03
S2	0.391 ±0.02	0.382 ±0.05	0.361 ±0.02	0.335 ±0.03
S3	0.384 ±0.02	0.364 ±0.05	0.350 ±0.02	0.319 ±0.03

From the findings above, it can be concluded that the additions have an impact on the material's physical behavior in terms of absorbency. These improvements enhance the material's ability to maintain a consistent volume and reduce water retention. The production of dentures can benefit from this phenomenon. The SEM images of the samples, shown in Fig. 10, reveal a homogenous distri-

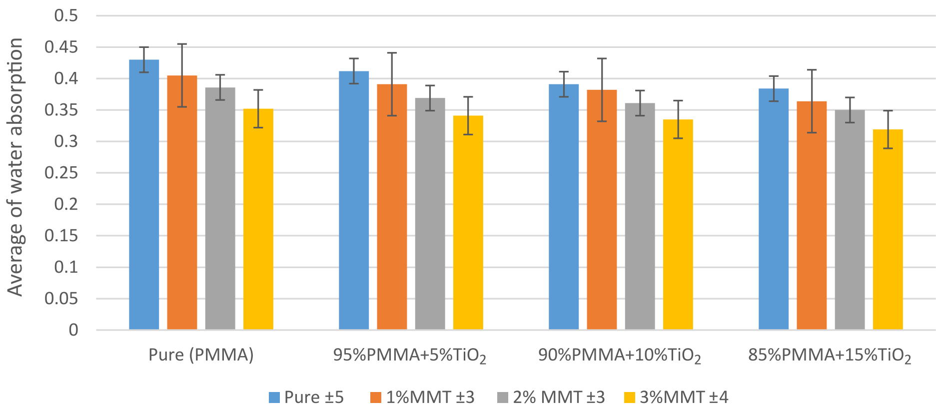

Fig. 9. Results of water absorption tests.

a) b) c)

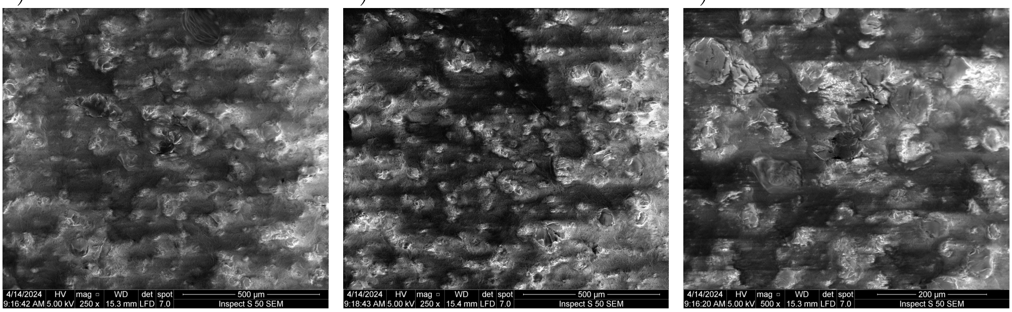

Fig. 10. SEM images of PMMA/TiO_2 samples: a) S1, b) S2, and c) S3.

bution of titanium oxide and MMT filler, the closing of interstitial spaces, and an enhanced compactness between the additives and the matrix material.

3.3. Biological test

The denture industry faces issues due to the potential toxic effects of manufacturing materials, making it essential to conduct toxicity tests to ensure their safety. A toxicity test was conducted to assess the behavior of each substance, and the results were generally positive, indicating the importance of these tests in the industry.

3.3.1. Antibacterial (Pseudomonas and Staphylococcus aureus). The antimicrobial screening procedure was conducted using all of the research materials, specifically the sample consisting of 85% PMMA + 15% TiO_2 + 3% MMT. This sample was selected to ensure that the highest concentrations of additives were present, allowing them to interact with the surrounding conditions to promote bacterial growth.

As shown in Fig. 11, the results were excellent. After the test period, no bacterial influence or growth was observed surrounding the implant.

a) b) c) d)

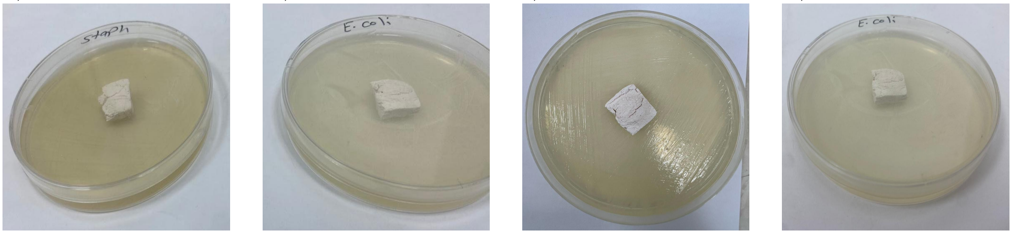

FIG. 11. The samples before the biological test (a, b) and after the biological test (c, d).

3.3.2. Toxicology. This research used an MTT test to assess the toxic effects of the target substances on cells. The test measures cell viability, which is primarily dependent on mitochondrial activity. The test is repeated three times to confirm the results, and the sample (S3 + 3% MMT) is selected for this test as it includes all components. The IC_{50} scale measures the sensitivity of the transplanted material and its toxic effect on cells. After 48 hours of incubation (see Fig. 12), the results showed that the normal cell line (HdFn) and living cells exhibited no toxicity from the substances. The values of living cells remained above 50%, confirming the material's non-toxicity.

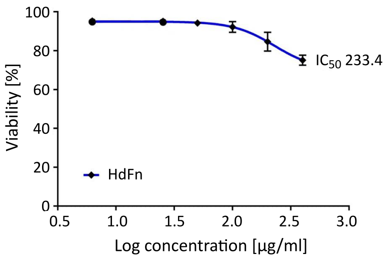

FIG. 12. The logarithmic relationship between cell validity and the concentration of the examined sample.

4. Conclusion

The research revealed that additives significantly impact the mechanical properties of matrix materials and their ratios, making them suitable for denture manufacturing applications. Mechanical tests, including tensile, compressive, and hardness tests, showed that the addition of 3% MMT as a filler directly improved these properties by about 51%, 31.5%, and 56.67%, respectively. Nanomaterials also reduced interstitial spaces between the materials, improving bonding between the matrix material and the additives. The additives' success in biological tests, such as antibacterial and toxicity assessments, supports their suitability for dental applications, indicating that they are safe for this purpose. The findings showed that there was no bacterial influence or growth surrounding the implant. Additionally, the living cell values were above 50%, confirming the material's non-toxicity. Overall, the study highlighted the potential of these additives in enhancing denture manufacturing.

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