

RESEARCH PAPER

## Assessing Tensile and Shear Bond Strength between Artificial Acrylic Teeth and Nano-Silica Reinforced PMMA Denture Base Material

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### ARTICLE INFO

#### Article History:

Received 08 September 2023

Accepted 16 December 2023

Published 01 January 2024

#### Keywords:

Artificial Acrylic Teeth

Nano-silica

Shear Bond Strength

Tensile

### ABSTRACT

Detachment of artificial teeth from the denture base presents a common clinical issue. To manipulate this problem, silica nanoparticles ( $\text{SiO}_2\text{NP}_s$ ) strengthening was suggested, containing (0, 2.5, 5, 7 wt. %)  $\text{SiO}_2\text{NP}_s$  to form nanocomposite materials. To evaluate the tensile and shear bond strength between acrylic teeth and denture base (PMMA) reinforced with  $\text{SiO}_2\text{NP}_s$ . 60 maxillary central incisors & 60 mandibular molars were selected to be bonded to cylindrical shape specimens (diameter of 5mm and length of 20 mm) fabricated from heat-cured Poly (methyl methacrylate) (PMMA) reinforced with  $\text{SiO}_2\text{NP}_s$ . Tensile and shear bond strength tended to increase significantly ( $P < 0.001$ ) in all groups reinforced with  $\text{SiO}_2\text{NP}_s$  compared with the control group. Among the reinforced groups, the tensile bond strength increased significantly ( $P < 0.001$ ) at 2.5% followed by 5.0 %, 7.0 %, and least in the control group. Moreover, the shear bond strength values of the 2.5 wt.% and 5 wt.% modified samples were significantly higher than those of the control ( $P < 0.001$ ), while the 7wt.% group was lower compared to the control group. Incorporation of  $\text{SiO}_2\text{NP}_s$  into the acrylic resin significantly increased the tensile bond strength for all tested groups (2.5 %, 5 %, and 7%) compared with the control group, this increase was concentration-dependent.

### How to cite this article

Jaber Radha H. S., Naji S. A., Haddada. I. Assessing Tensile and Shear Bond Strength between Artificial Acrylic Teeth and Nano-Silica Reinforced PMMA Denture Base Material. J Nanostruct, 2024; 14(1):20-37. DOI: 10.22052/JNS.2024.01.003

### INTRODUCTION

The material most typically used to manufacture denture bases and artificial acrylic teeth for partial and complete prostheses is PMMA for prosthetic rehabilitation of edentate patients using removable dentures [1, 2]. The demand for prosthodontic rehabilitation continues to increase sharply despite advances in prevention and conservative treatment methods, due in particular to the high number of elderly population and the increase

in life expectancy [3, 4]. Physical and mechanical properties are of prominent significance when picking out artificial teeth used in the manufacture of dentures for patients who will be users [2]. When resin teeth are cured into denture base resin such as PMMA, the copolymerization with the cross-linking element, creates a durable bond between the denture base and the acrylic teeth owing to its chemical compatibility [5, 6]. The strength of adhesion between the acrylic teeth

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and the denture base is one of the most important factors that the denture must possess to ensure the required performance and durability [7]. Even so, tooth detachment or fracture is still a central issue in clinical applications [8]. It has been indicated that between 22% - 30% of denture failures are owing to tooth detachment, which commonly appears in the frontal region of the denture. An important factor that can have an impact on the adhesion strength between acrylic teeth and the PMMA is the chemical mismatch between diverse types of teeth and the PMMA materials or the smaller surface area of the ridge-lap available on the tooth for attachment [7 - 13]. There is no doubt that immoderate occlusal force or fatigue load on the teeth during chewing, improper use of separating medium during acrylic resin curing, surface pollution owing to wax remaining at the interface between the tooth and the base material during the manufacture of the denture, and ineffective or suboptimal treatment methods, in addition, insufficient monomer availability during polymerization are another factors that accelerate the separation of the tooth from the base of the denture [7, 8, 14, 15]. Some studies also indicated that the frequent using of chemical detergents and sterilizers can modify the physical and mechanical characteristics of denture base and teeth materials, and thus lead to a debilitating of the bond and future failure [16]. These agents may occasion the teeth to fail by either adhesion or cohesion or a combination of both. Commonly, adhesive failure happens along the point of contact between the denture base and the acrylic tooth and is distinguished by the lack of fragments of the denture base or tooth on the opposite surface [17]. A fracture that happens entirely inside the teeth or denture base is termed a cohesion failure, while it is termed a mixed mode failure if any part of the tooth material on the denture base or any part of the denture base material on the surface of the tooth [17]. The researchers worked on finding different ways to enhance this bond strength, as many of the previous studies included enhancing the chemical and mechanical preparation of the ridge-lap surface [11, 18]. Reported mechanical preparations are rivets, grooving, diatoric undercuts, glossy surface grinding, and high-energy teeth abrasives [12, 19, 20] to better mechanical interlocking during treatment. Some studies also stated that mechanical modification of the ridge-lap surfaces of acrylic artificial teeth

showed increased bond strength [8,12,20,21], while other studies found no betterment [8,18]. In the same way, chemical bonding has been boosted by investigating chemical modulation using monomers (MMA), solvents, or adhesive bonding factors applied to the contact surfaces. [12, 15]. A number of research have recorded improvements in bond strength using chemical surface handling, however, others did not find valuable improvements [8, 11, 12, 22]. The results of one study that evaluated the effects of several chemical surface-conditioning monomers on the ridge-lap surfaces of artificial teeth showed an improvement in shear bond strength. along this, monomer systems induced the dissolution of the artificial teeth [12]. Certainly, the evaluation of the bond strength of heat cure acrylic resin for acrylic teeth is still in dire need of further research and investigation. Although there are several national and international criteria used to estimate the strength of the bond, there is a great contrast in the types, sizes, and shapes of materials used, in addition to the fabrication techniques of the test samples and the testing approaches [8]. In clinical cases, the direction of the load applied to the denture teeth during chewing causes detachment by a combination of compression, shear failure, and tension [23]. Flexural strength, tensile or (partial tensile), compression, shear tests, and finite element stress analysis are the methods used to assess the strength of the adhesion bond between the artificial teeth and the denture base [23, 9]. Nevertheless, it is recognized that denture failure in clinical practice cannot be completely avoided by bettering only the mechanical properties of denture bases. The synergistic forces conferred by both the high strength of the denture base and the strong bond between the denture base and teeth are essential to ensure the proper functioning and endurance of the prosthesis. Wherefore, the potentiality of the denture base material to counter impact forces and crack propagation and increase the adhesion force between the denture base and the denture teeth is a crucial factor impacting its performance. Hence a demand to promote the mechanical features of polymethacrylate-based materials to attain dentures with more resistance to fracture and detachment [24]. Adding some fillers, fibers, and rubber-like materials, as well as chemical modification are various methods adopted to perfect the mechanical characteristics of polymethacrylate-based materials [25]. Of

late, attention has focused on reinforcing acrylic denture base materials with nanoparticles to improve their mechanical properties [26]. The unique properties of these particles (their small size, large specific surface area, strong interfacial interaction with organic polymers, and the surface area of the material being nanometers in size), when compared to bulk properties, confer different mechanical, chemical, electrical, optical, magnetic, electromagnetic and magneto-optical properties owing to their markedly large surface area compared to the size of the material [27, 28].

The type of nanoparticles included, and their sizes, give a picture of the nature of the properties of polymeric nanocomposites. Polymeric nanocomposites, by their nature, constitute an attractive family of hybrid materials through the incorporation of inorganic nanoparticles into organic matrices. Many studies have reported significant improvement in a number of desirable properties, such as mechanical strength [29], thermal stability, [30] barrier properties, [31] and corrosion resistance [32] by incorporating inorganic fillers into a polymer matrix. Recently, poly (methyl methacrylate)/silica nanocomposites have received great attention due to their unique advantages and multiple applications in fields such as scratch resistance [33], bioactivity [34], antimicrobials [35], electrical insulation [36], and optical applications [37].

Determining the final properties of nanocomposite materials depends significantly on both the nature and strength of the interactions between the filler material and the polymer, as both of these factors will determine the dispersion of the filler and the quality of distribution into the polymer matrix. Actually, an increase in the amount of filler agglomerates arises from weak interactions, while strong interactions will lead to the formation of either mixed aggregates or the dispersion of the primary particles individually [38]. Previous studies based on FT-IR analysis indicated the presence of hydrogen bonds between the silanol (SiOH) groups of the SiO<sub>2</sub> and the carbonyl (C=O) of the PMMA, and the results of these studies were explained by the fact that the transfer of the O=C peak (PMMA) toward low wave numbers (low energies) is concerning to the strength of the interaction of the H bond with the OH groups in SiO<sub>2</sub>. Several studies that have investigated the incorporation of SiO<sub>2</sub>NPs into PMMA have recommended using low

concentrations of SiO<sub>2</sub>NPs [11, 39-41]. A study conducted by both Cevik and Yildirim-Bicer [40], showed a decrease in the mechanical properties of the obtained nanocomposite by adding high concentrations (1 and 5 wt.%) of SiO<sub>2</sub>NPs to PMMA. Likewise, Sodagar et al. showed a decrease in flexural strength after incorporating 1.0 and 0.5wt.% SiO<sub>2</sub>NPs into PMMA and this decrease was directly related to the concentration of the added nanoparticles [39]. Also, it was observed that the highest increase in flexural strength was when using 0.25 wt.% compared to the higher concentrations, through two studies carried out by Gad et al and Abushowmi et al, where they added SiO<sub>2</sub>NPs in proportions (0.25, 0.5, and 0.75 wt.%) to the PMMA repair resin [40,11]. Just now, the literature lacks studies that include an evaluation of the tensile and shear bond strength between acrylic teeth and dental base materials reinforced with SiO<sub>2</sub>NPs, so this study investigated the effect of adding different concentrations of SiO<sub>2</sub>NPs (2.5, 5, and 7wt. %) to heat-treated PMMA on the tensile and shear bond strength between the artificial teeth and the reinforcement denture base resin.

The null hypothesis of this study was that the incorporation of SiO<sub>2</sub>NPs into PMMA at different concentrations (2.5, 5, and 7 wt. %) would not have a significant impact on both shear and tensile bond strength.

## MATERIALS AND METHODS

### Study Protocol

In this experimental study, a total number of 120 teeth (3-layer cross-linked synthetic resin teeth- Acryrock Ruthinium, badiapolesine - ITALY), 60 maxillary central incisors and 60 mandibular molars from the same mold in terms of shape and size were selected to be mounted on cylindrical-shaped specimens (5 mm in diameter and 20 mm in length) fabricated from heat-treated poly methyl methacrylate. (Lucitone 199, Dentsply) reinforced with SiO<sub>2</sub>NPs (SiO<sub>2</sub>, 95.9+wt.%, size 20-30 nm, coated with 3-4 wt. % KH570- Silane coupling agent) white color, specific surface area 130 to 600 m<sup>2</sup>/g, and intensity < 0.10 g/cm<sup>3</sup>) concentrations of (2.5, 5, 7 wt.%), were prepared for shear and tensile bond strength test.

In order to perform shear and tensile bond strength tests, 60 PMMA specimens were fabricated. For each test: the specimens in each test were assigned to four groups (n = 15)

according to the percentage of silica nanoparticles added: 0% (control); 2.5, 5, 7 wt. %.

60 metal dies (5 mm diameter and 20 mm length) were manufactured in accordance with ADA Specification No. 15 [42]. Using a digital caliper, the metal mold was measured, and standardization of the specimens was executed. After making the silicone mold for the metal mold (Vinyl polysiloxane putty impression material, 3M ESPE Express XT putty soft, USA), to facilitate the retrieval of the wax patterns from the silicone mold, the silicone mold was divided lengthwise into two halves. Then to obtain identical samples, modeling wax was decanted into a silicone mold, and to secure the accuracy of the dimensions of the samples, they were measured again with a digital caliper. After making wax patterns from the metal die and by using an acrylic trimming tool, the ridge surface consisting of 60 central incisors and 60 lower mandibular molars was trimmed flat. 60 mandibular molars were attached to the wax patterns from both sides (i.e. ridge lap surface and occlusal surface), and 60 central incisors were similarly attached to the wax patterns obtained from the silicone mold to facilitate use as a fixation device for tensile and shear testing.

*Fabrication of acrylic samples with nanoparticle additives*

For each test, an electronic analytical balance device was used to measure the SiO<sub>2</sub>NPs added to the acrylic powder at concentrations (2.5, 5, and 7% by weight). To obtain a more homogeneous and uniform distribution of nanoparticle powder, a sonicator device was used for 3 minutes to sonicate the PMMA resin powder and nanoparticle powder. Next, the modified and unmodified acrylic powder was mixed with MMA liquid according to the

manufacturer’s guidance and packed into dental stone molds (Hydrocal Dental Stone, Moldano, Bayer Lircuisen, Germany) at the dough stage. To make sure the dough flows equally all over the mold area, the two parts of the flask were tightly closed together and placed under a hydraulic press at 40,000 N, and pressure was slowly applied to the flask. Excess material was removed with a sharp lancet after releasing the pressure, opening the flask, and then the flask was closed in the second trail.

Ultimately, after closing both parts of the flask, left for 5 minutes under pressure (20 bar). Then, the flask is clamped and kept under reduced pressure for half an hour and then placed in a water bath at room temperature. Slowly the temperature was raised to 73 ± 1°C and then to the boiling point for 90 minutes and 30 minutes, respectively. The flask underwent cooling on the bench before opening. All the samples were finished and polished to get a sleek and shiny surface and then stored in water ( 37 °C) for a full day before testing them with a Universal Testing Machine (INSTRON 1195: England).

*Characterization of Mechanical Properties*

Fixation devices were manufactured for the fabrication of the tensile and shear bond strength specimens and held secure to avoid any change of position, measuring tensile bond strength at cross speed 0.5 mm/min until failure [43] as shown in the failure load carried out on the universal testing machine. Horizontal forces were applied to test the shear bond strength. The reading on the digital monitor is attached to the machine a load of brake was recorded by (mode lwdw50 manufactured by Laryee) as seen in using a stainless-steel chisel-shaped rod, the shear bond strength was used

Table 1. Specimens grouping according to SiO<sub>2</sub>NP<sub>s</sub> concentration

Groups	Subgroups	Description	No. of Samples
Tensile bond strength (TSB)	Group 1	(PMMA) without SiO <sub>2</sub> NP <sub>s</sub>	15
	Group 2	(PMMA) with 2.5% SiO <sub>2</sub> NP <sub>s</sub>	15
	Group 3	(PMMA) with 5% SiO <sub>2</sub> NP <sub>s</sub>	15
	Group 4	(PMMA) with 7% SiO <sub>2</sub> NP <sub>s</sub>	15
Shear bond strength (SBS)	Group 1	(PMMA) without SiO <sub>2</sub> NP <sub>s</sub>	15
	Group 2	(PMMA) with 2.5% SiO <sub>2</sub> NP <sub>s</sub>	15
	Group 3	(PMMA) with 5% SiO <sub>2</sub> NP <sub>s</sub>	15
	Group 4	(PMMA) with 7% SiO <sub>2</sub> NP <sub>s</sub>	15



to deliver the shearing force with a crosshead speed of 0.5 mm/min (ISO TR 11405). The load cell was set at 100 Kg, was based on the force (F) in Newton (N) at fracture and adhesive surface area (S) in (mm<sup>2</sup>), and converted to MegaPascal (MPa) according to the following formula below:

$$B.S = F \times S^{-1}$$

B.S = Bond strength (N/mm<sup>2</sup>) or (MPa)  
 F= Force at failure (N)  
 S=  $(\pi / 4) \times D^2$ ;  $\pi = 22/7$  or 3.14, D (diameter) = 5 mm, S = 19.64 mm<sup>2</sup>

**FTIR, XRD, and SEM-EDX Characterization**

By field emission scanning electron microscopy (FESEM, ZEISS Co., Germany), the surfaces of the fractured samples were evaluated at an operating voltage of 15.0 kV and equipped with an energy dispersive X-ray (EDX) spectrometer (AURIGA, ZEISS), Finally, the specimens were coated with gold using the SCD-005 device (Bal-Tec/Leica, Wetzlar, Germany) before the SEM analysis.

To examine the interaction between PMMA and SiO<sub>2</sub>NPs, Fourier transform infrared (FTIR) spectra were measured (FTIR, Nicolet iS50, Madison, WI, USA). Spectra were taken in the range of wavenumbers from 400 to 4000 cm<sup>-1</sup> with a resolution of 4 cm<sup>-1</sup> for all samples at room temperature.

**Statistical Analyses**

To perform all statistical analyses, SPSS statistical software version 26.0 (SPSS Inc., Chicago, IL) was used. The mean and standard deviation values were used to present the data and to compare all

groups, a one-way analysis of variance (ANOVA) test was applied. P< 5% is set to be the significance level.

**RESULTS AND DISCUSSION**

**Characterization of PMMA and PMMA-SiO<sub>2</sub>NPs composites**

Before starting to discuss and explain the results obtained, it must be clarified that both the polymer and silica have an amorphous structure, therefore the peaks in the X-ray diffraction measurement will be in the form of broad, overlapping peaks. Moreover, increasing the percentage of silica will reduce the transmittance in the FTIR measurement, thus the spectra will be less clear. Therefore, the EDX measurement will be the most important among these measurements in determining Si presence, in addition to determining the size with SEM.

**FTIR**

FTIR measurements of the PMMA polymer and its composites show that the transmittance reached less than 5%, therefore this will give a spectrum with low clarity. FTIR measurement of the PMMA polymer, the structure of which is shown below (Fig. 1), showed the presence of all the bands characteristic of the structure of this polymer. The measurement showed bands at 2846 and 2715 cm<sup>-1</sup>, which are attributed to the stretching vibration of aliphatic C-H in -CH<sub>3</sub> and -CH<sub>2</sub>. Moreover, the spectrum showed a weak band of intensity at 1774 cm<sup>-1</sup>, which is due to the stretching of the vibration of the carbonyl group (C=O). Regarding the stretching vibration in the

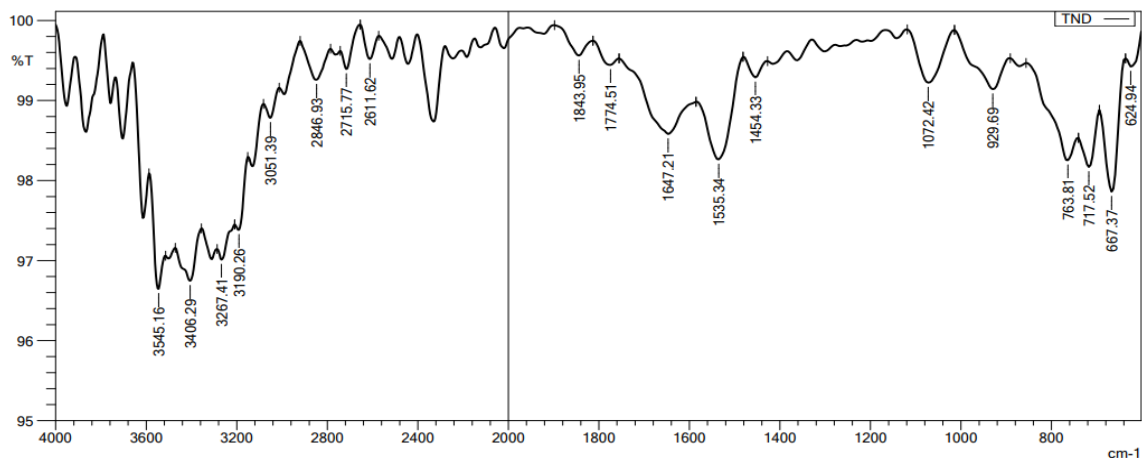


Fig. 1. FTIR spectrum of PMMA



C-O-C group, the measurement showed bands at 1072 and 929 cm<sup>-1</sup> in the form of medium-intensity bands. The band at 1535 cm<sup>-1</sup> is attributed to the deformation vibration of the CH<sub>2</sub> and CH<sub>3</sub> groups. Likewise, evidence of complete polymerization was the appearance of the band at 767 cm<sup>-1</sup>, which is attributed to the alpha-methyl group [44-46]. Finally, the bands at (3545, 3406), and 1647 cm<sup>-1</sup> are attributed to stretching and deformation vibrations of adsorbed water [46].

FTIR measurement of the PMMA polymer containing different percentages of nano-silica at 2.5, 5, and 7% (Fig. 2, Fig. 3, and Fig. 4, respectively),

the composition of which is shown below showed the presence of all the characteristic bands of the polymer structure. The measurement showed the stretching vibration band of aliphatic CH in CH<sub>3</sub> and CH<sub>2</sub> at the range 2843-2835 and 2704-2715 cm<sup>-1</sup>. Moreover, the spectra showed a weak band of intensity at 1654-1689 cm<sup>-1</sup>, which is due to the stretching vibration of the carbonyl group (C=O), which was observed to have a lower frequency compared to the free polymer (1774 cm<sup>-1</sup>), which confirms the coordination of the polymer via the oxygen of the carbonyl group with silicon, since silicon reduces the electronic density on the

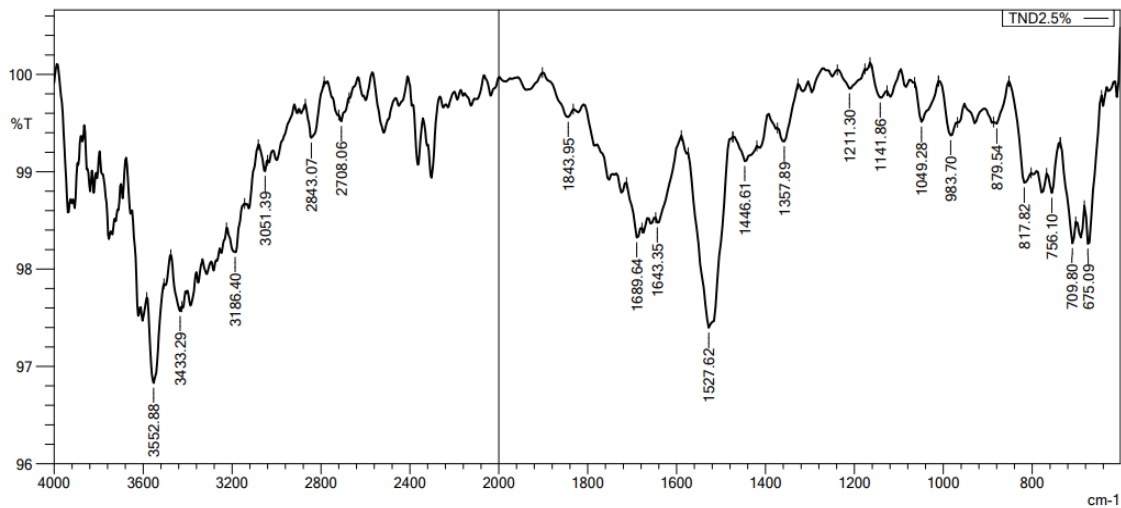


Fig. 2. FTIR spectrum of PMMA-SiO<sub>2</sub> NPs (2.5%)

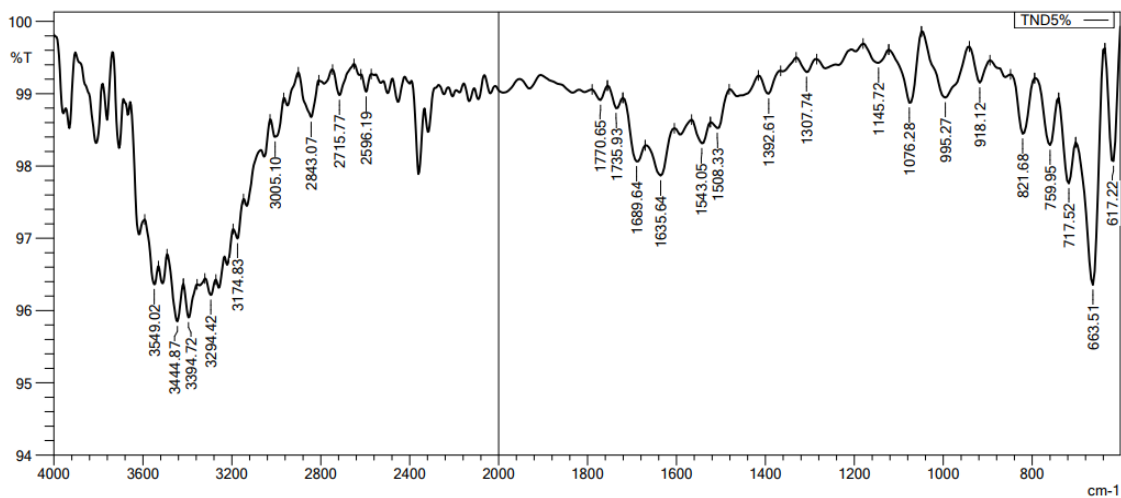


Fig. 3. FTIR spectrum of PMMA-SiO<sub>2</sub> NPs (5%)

carbonyl group, thus its frequency decreases, this is evidence of the success of the reaction in all proportions of silica used. Regarding the bands in the C-O-C group, bands at 1049-1087 and 906-983 cm<sup>-1</sup> appeared as medium intensity bands, while the band at the range 1527-1543 cm<sup>-1</sup> is attributed to the deformation vibration of the CH<sub>2</sub> and CH<sub>3</sub>

groups [44-46]. Furthermore, the spectrum showed the characteristic bands of silica, which is the stretching vibration bands of symmetric and asymmetric Si-O-Si at the range 1141-1149 cm<sup>-1</sup> and 817-821 cm<sup>-1</sup>, respectively, which is further evidence of the success of the reaction and the presence of silica within polymer mixture.

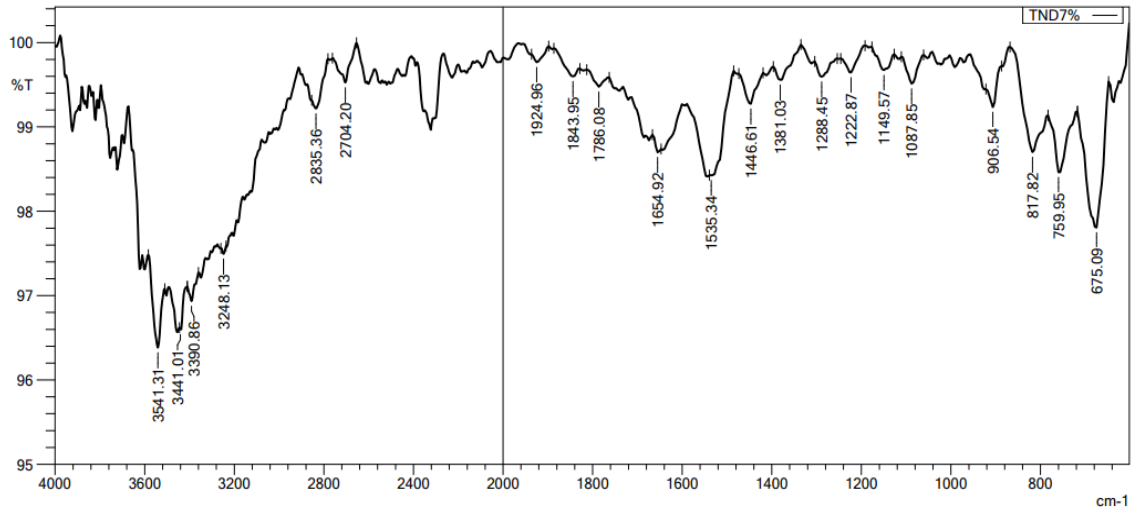


Fig. 4. FTIR spectrum of PMMA-SiO<sub>2</sub> NPs (7%)

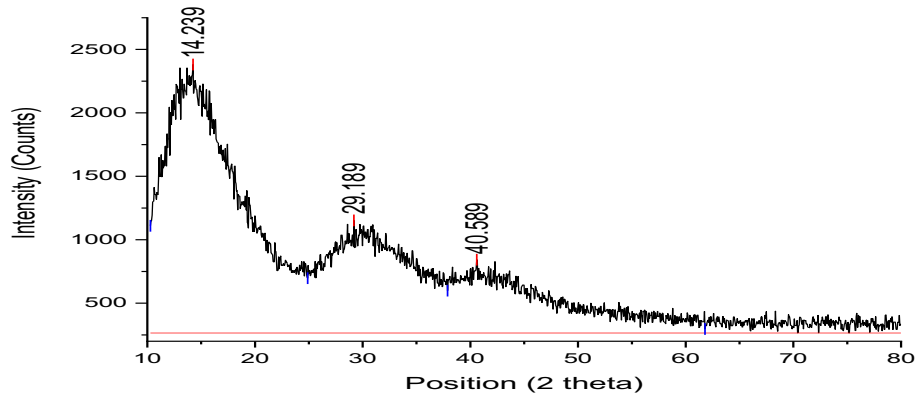


Fig. 5. XRD spectrum of PMMA

Table 2. XRD data and average particle size of PMMA

Position (degree)	Height (cts)	FWHM (degree)	Particle size (nm)	Average particle size (nm)
14.239	2116.8	7.19428	1.16	1.09
29.189	887.6	9.67401	0.89	
40.589	578.2	7.33836	1.21	

**XRD**

The measurement showed the presence of only three main peaks indicating the carbon structure of the PMMA polymer chains, which appeared in the form of broad peaks, which indicates that the

polymer structure is an amorphous structure. The highest intensity peak was at 14.23°, followed by the peak at 29.18°, then the third peak, which was less intense, at 40.58°, as shown in Fig. 5, which are attributed to the crystal planes 111, 112, and

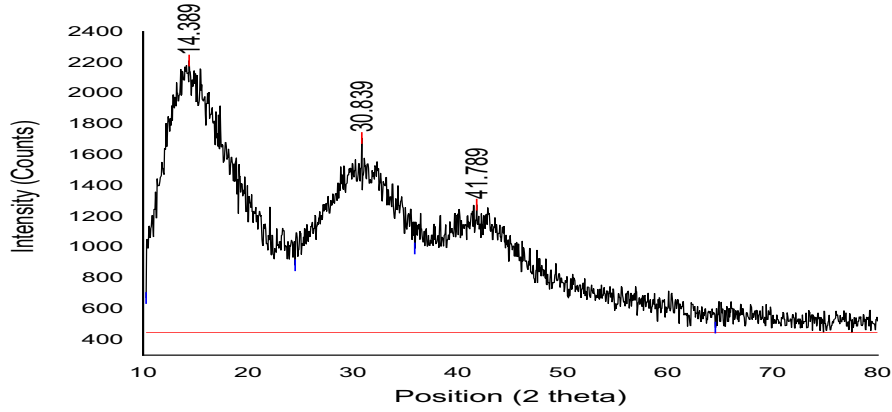


Fig. 6. XRD spectrum of PMMA-SiO<sub>2</sub>NPs (2.5%)

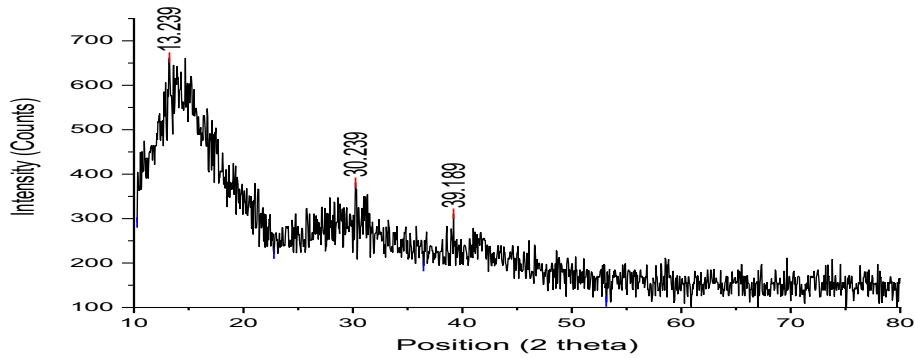


Fig. 7. XRD spectrum of PMMA-SiO<sub>2</sub>NPs (5%)

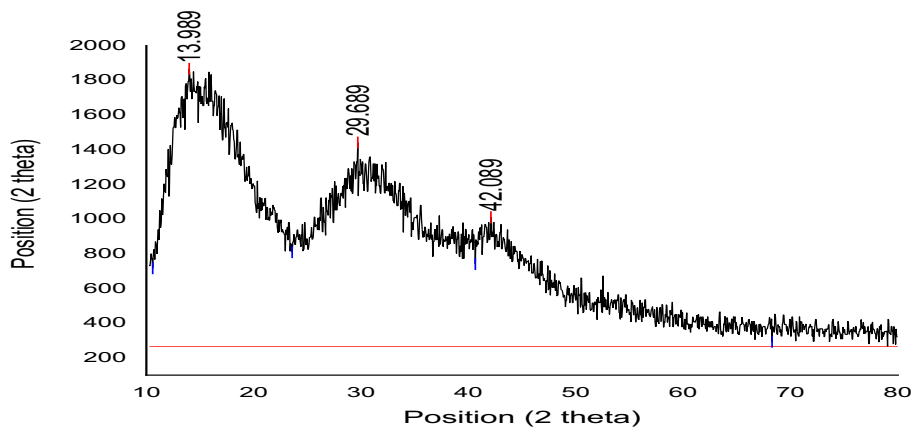


Fig. 8. XRD spectrum of PMMA-SiO<sub>2</sub>NPs (7%)



211, respectively [47, 48]. Based on the Scherrer equation, the size of the particles reached 1.09 nm, but here the material does not express the true size of the polymer particles because the structure is not spherical, and the material is also amorphous, therefore the width of the peaks (FWHM) results from the amorphous property and not from the complete small size of the particles. Therefore, the Scherrer equation fails to give a real picture of the average particle size [49, 50] as

shown in Table 2.

This measurement was also used to determine whether the interaction between the polymer and silica occurred or not, as the measurement clearly demonstrated that there was an interaction between the polymer and silica, despite the fact that nano-silica and the polymer share the property of amorphous, which causes overlap peaks in this measurement. However, the measurement showed the peaks for the polymer within the

Table 3. XRD data and average particle size of PMMA-SiO<sub>2</sub>NPs (x %)

Position (degree)	Height (cts)	FWHM (degree)	Particle size (nm)	Average particle size (nm)
PMMA-SiO <sub>2</sub> NPs (2.5%)				
14.389	1764	7.74737	1.08	0.97
30.839	1261.4	9.12054	0.94	
41.789	828.8	10.13857	0.88	
PMMA-SiO <sub>2</sub> NPs (5%)				
13.239	567	7.49278	1.12	13.95
30.239	285.6	0.38229	22.50	
39.189	215.6	0.48348	18.23	
PMMA-SiO <sub>2</sub> NPs (7%)				
13.989	1601.6	8.64912	0.97	13.32
29.689	1176	0.4111	20.90	
42.089	746.2	0.49192	18.09	

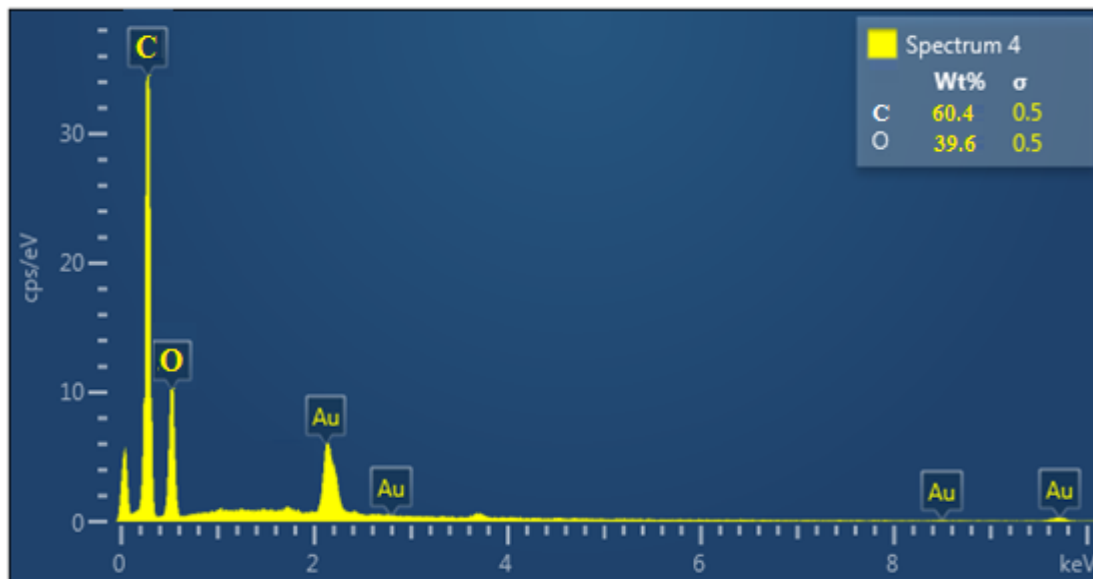


Fig. 9. EDX of PMMA

same range specified in the characteristic of nano-silica appear at approximately 24°, which is attributed to the crystal plane 101 [51], thus there is an increase in the intensity of this peak only with a greater width of it due to this overlap. Based on this, it was observed that the intensity of the peak at 30° increased approximately directly with the increase in the percentage of added silica, as in Figs. 6, 7, and 8. Through the Scherer

equation, the average particle size was calculated, which amounted to 0.97, 13.95, and 13.32 nm for the added percentages of silica, respectively. We notice that with an increase in the percentage of silica, the calculated particle size increases and thus approaches the possibility of applying the equation, due to the increase in particles to which the equation applies compared to the polymer, as shown in Table 3.

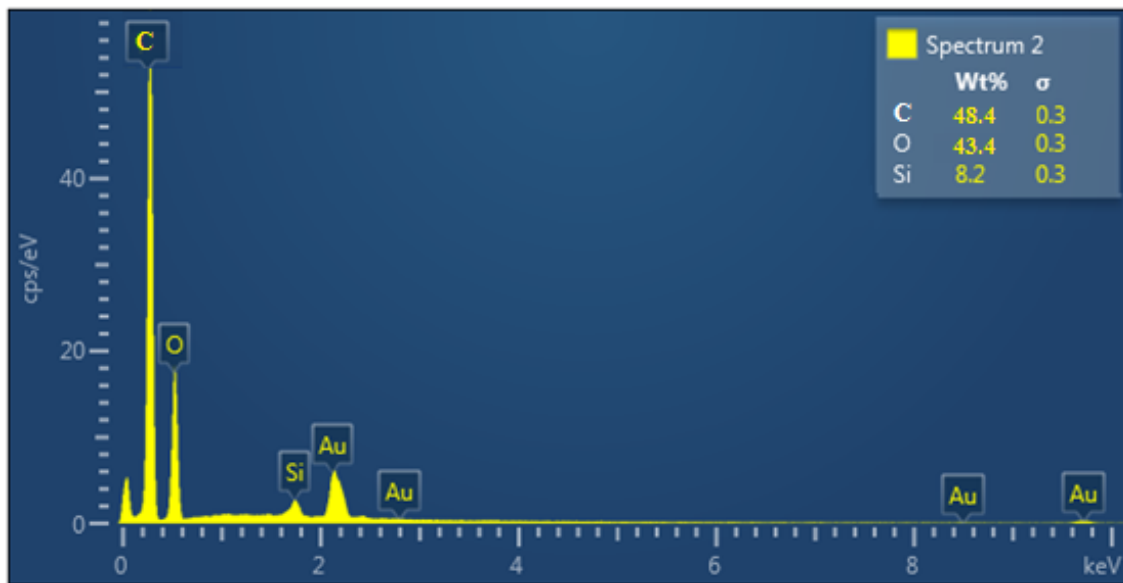


Fig. 10. EDX of PMMA-SiO<sub>2</sub>NPs (2.5%)

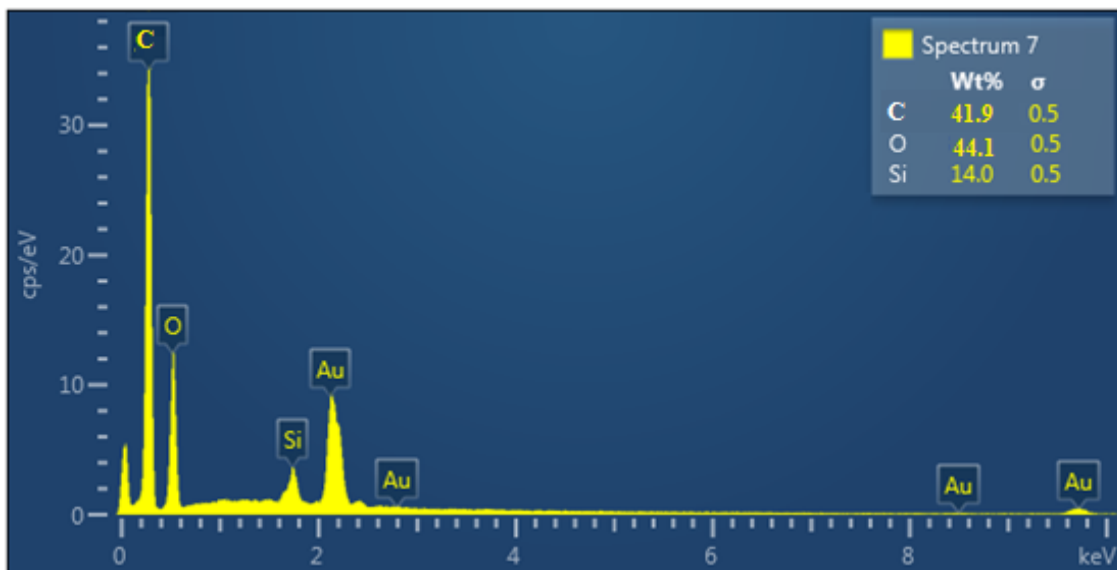


Fig. 11. EDX of PMMA-SiO<sub>2</sub>NPs (5%)

SEM-EDX

EDX measurement was used to determine the elements involved in the composition of the prepared composites and determine the percentage of silica in them. The measurement proved that the polymer consists of oxygen and carbon, as it gave two peaks, one at 0.277 and the other at 0.525 KeV, which is attributed to carbon and oxygen, respectively, which are considered the basic elements of this polymer, in ratios of 60.4 and 39.6%, which agree with the theoretical ratios of this polymer. The measurement did not show peaks specific to hydrogen, as it is an element with a low atomic number that this technique

cannot estimate. We also note the presence of peaks specific to gold that is attributed to its use in showing the peaks as a coating material, as shown in Fig. 9.

The measurement of polymer composites containing silica in different proportions demonstrated the presence of oxygen, carbon, and silicon, as it gave three peaks, one at 0.277 and another at 0.525 KeV, which is attributed to carbon and oxygen, respectively. The peak at 1.739 KeV is attributed to silicon, which was found to be 8.2%, 14%, and 21.5% for the PMMA-SiO<sub>2</sub>NPs (2.5%), PMMA-SiO<sub>2</sub>NPs (5%), and PMMA-SiO<sub>2</sub>NPs (7%) composites, respectively which do

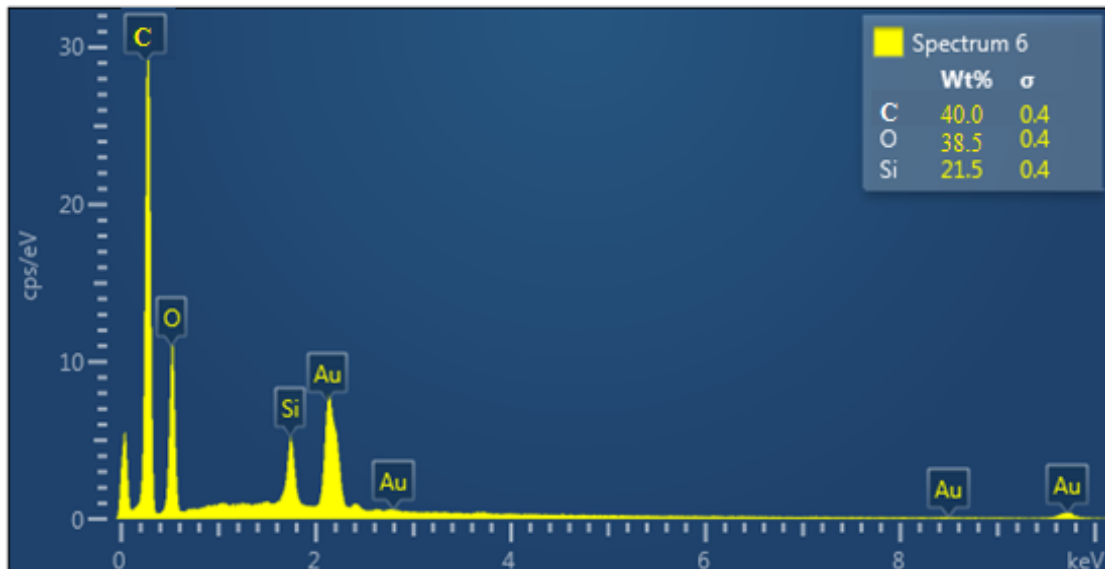


Fig. 12. EDX of PMMA-SiO<sub>2</sub>NPs (7%)

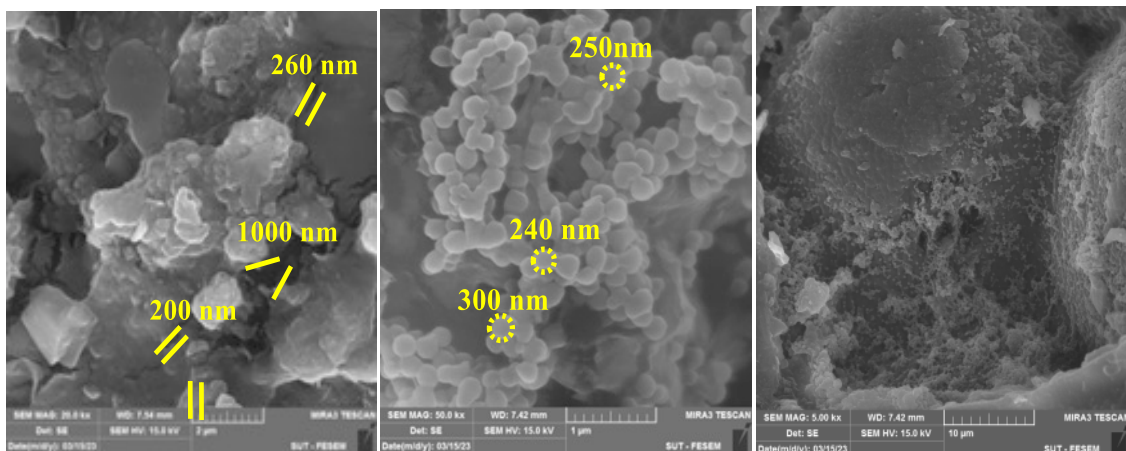


Fig. 13. SEM of PMMA

not agree with the percentages added from silica to the polymer. This defect is due again to the fact that this technique excludes many elements from the estimation due to the insensitivity of this technique to some elements. However, the technique recorded evidence of an increase in the percentage of silica in a manner consistent with the added percentages, as shown in Fig. 10, Fig. 11, and Fig. 12.

In order to verify the shape and size of the prepared materials, SEM was measured, and the polymer showed that it consisted of irregular particles resulting from the fusion of the polymer, which were formed in the form of microspheres spread on the surface [52], whose size reached 240-300 nanometers, as shown in Fig. 13. The measurement was confirmed. Moreover, the cracks were extended with a diameter of 200-1000 nanometers, as shown in Fig. 13. The

measurement proves the presence of surface roughness resulting from the presence of these spherical structures of the polymer itself.

The surface roughness, the amount of cracks, and the surface composition of the polymer were studied after adding different percentages of silica. SEM measurement of the mixture containing 2.5% silica (Fig. 14) showed that there was a higher roughness than that recorded for the pure polymer, in addition to a change in the shape of the surface with the complete disappearance of the polymer microsphere. In addition to this, the measurement showed the presence of particles reaching 41.96 nm and smaller, in the form of irregular spheres which is attributed to the added nano-silica. However, it was not possible to determine the presence of silica in abundance in the measurement area because the percentage is small in addition to its presence within the

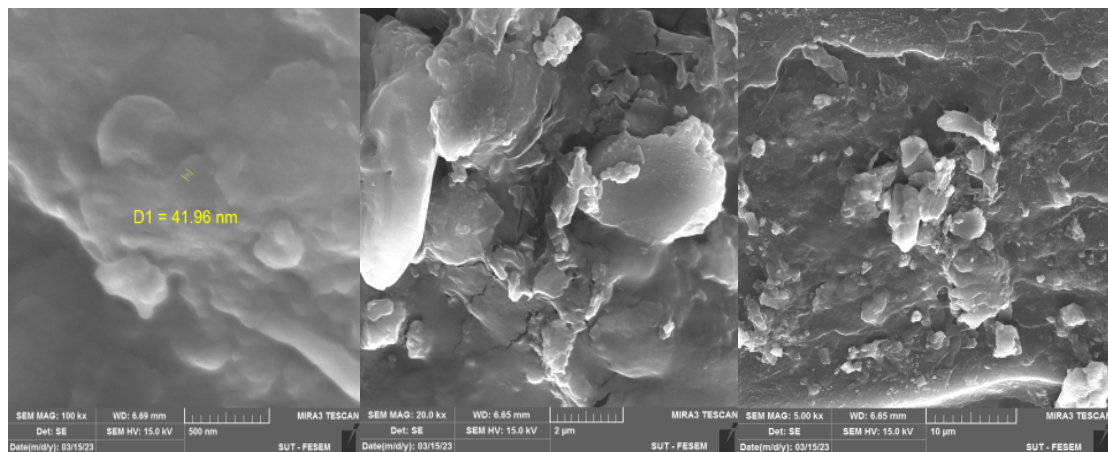


Fig. 14. SEM of PMMA-SiO<sub>2</sub>NPs (2.5%)

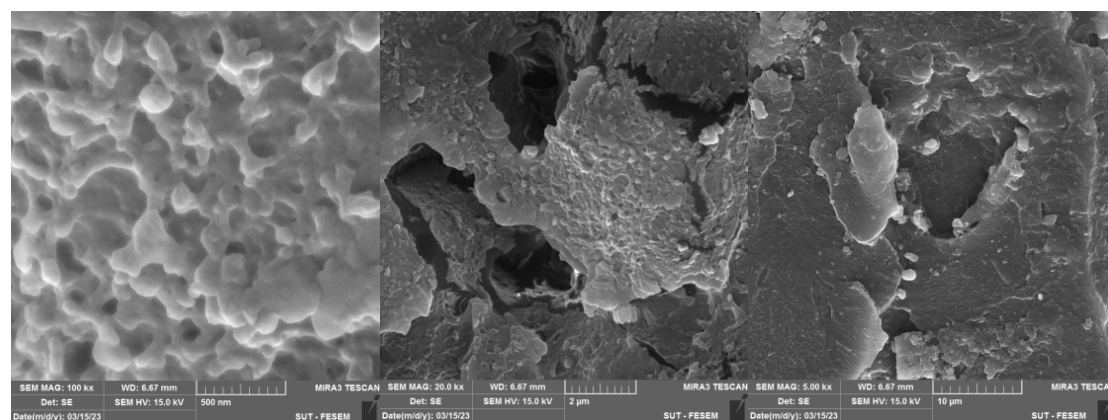


Fig. 15. SEM of PMMA-SiO<sub>2</sub>NPs (5%)

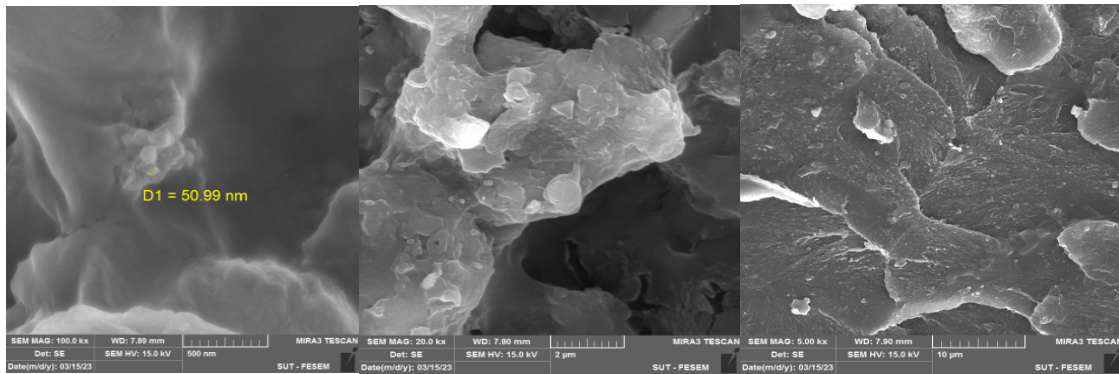


Fig. 16. SEM of PMMA-SiO<sub>2</sub>NPs (7%)

polymeric structure and between its chains. Moreover, the measurement showed cracks not exceeding 120 nm, which is much smaller than the cracks recorded for the pure polymer.

SEM measurement of the polymer containing 5% silica showed an increase in surface roughness more than the first mixture and the pure polymer. This is due to the small size of the added silica particles, which causes an increase in roughness [53]. It was noted that the size of the particles on the surface reached 140-150 nm, which does not represent the added silica, but rather a polymeric mixture containing silica. Furthermore, the cracks in this mixture were in an irregular circular shape not vertical cracks as above. However, the cracks were larger and reached 1000-3000 nanometers, as in Fig. 15.

SEM measurement of the mixture containing 7% silica (Fig. 16) showed that there is a higher roughness than that recorded for the pure polymer and similar to the roughness of the mixture containing 5% silica, in addition to a change in the shape of the surface with the complete disappearance of the polymer microsphere. In addition to this, the measurement showed the presence of particles. It reached 50.99 nm and is smaller and in the form of irregular spheres, which is attributed to the added nano-silica, which is within the polymeric structure of the polymer and incorporated in polymer chains. Moreover, the measurement showed cracks with a range of 200-400 nanometers and in the form of local cracks, therefore this ratio represents the optimal percentage in terms of lack of cracking.

#### Mechanical Properties

ANOVA results showed statistically significant

differences among groups ( $P < 0.001$ ). The mean values of tensile and shear bond strength in MPa are illustrated in Table 4. A highly significant statistical difference was noticed between the SBS and TSB of the different groups ( $P < 0.001$ ). Among the reinforced groups, the tensile bond strength increased significantly ( $P < 0.001$ ) at 2.5% followed by 5.0 %, 7.0 %, and least in the control group. Moreover, the shear bond strength values of the 2.5 wt. % and 5 wt. % modified samples were significantly higher than those of the control ( $P < 0.001$ ), while the 7wt. % group was lower compared to the control group.

The durability of dentures depends on the strong adhesion between denture base resin and the acrylic teeth [54], nevertheless, failure of bonds in acrylic teeth and the denture base resin interface remains a common clinical concern in prosthodontics that faces both the patient and the dentist [55]. As mentioned above, various methods have been adopted to promote the bonding of denture bases to acrylic teeth, for instance, it was found that adding SiO<sub>2</sub>NPs to PMMA enhances some of its biomechanical properties, due to the distinctiveness of these nanoparticles represented by their strong interfacial interaction with organic polymers, high surface activity, and large specific surface areas [16, 56, 39].

Due to the unique biomechanical and optical properties of SiO<sub>2</sub>NPs [57–63], it was chosen for investigation in the current study. Previous research dealt with concentrations of these particles that ranged between 5% and 10% by weight, which in itself is a large variation that has generated controversy about the effect of SiO<sub>2</sub>NPs on the mechanical properties of PMMA. In general, the positive effects were limited to low

Table 4. One-way analysis of variance and pairwise comparison tests between different subgroups of PMMA for all properties tested.

Groups	SBS (MPa)	TBS (MPa)
V0 control	10.33± 0.52	19.59 ±4.54
SiO <sub>2</sub> (2.5 %)	18.61± 0.78 <sup>A</sup>	24.17 ± 5.56 <sup>A</sup>
SiO <sub>2</sub> (5 %)	13.19 ± 0.67 <sup>A,B</sup>	28.44 ±3.4 <sup>A, B</sup>
SiO <sub>2</sub> (7 %)	7.73 ± 0.45 <sup>A,B,C</sup>	33.81± 2.23 <sup>A, B, C</sup>
P-value	0.000 (<0.001)	0.000 (<0.001)

SBS= Shear bond strength  
 TBS=Tensile bond strength  
 A= comparing with control group  
 B= comparing with SiO<sub>2</sub> (2.5%) group  
 C= comparing with SiO<sub>2</sub> (5%) group

concentrations, in contrast to high concentrations, which showed negative effects [58, 62–64], this is in addition to what has been reported in more than one study, that there is a significant change in the color of the nanocomposite as a result of using concentrations exceeding 7 wt. % of these particles [57, 64]. Therefore, concentrations of (2.5, 5, and 7) wt. % were selected in the present study to describe relatively low and high concentrations [8, 58, 61]. Depending on the results of the present study, the shear and tensile bond strength of the PMMA nanocomposites changed significantly after the addition of SiO<sub>2</sub>NPs; therefore, the null hypothesis adopted by this study was rejected. The aim of this study was to fabricate polymethyl methacrylate (PMMA) nanocomposites with better mechanical properties by combining them with different concentrations of silicon dioxide nanoparticles (SiO<sub>2</sub>NPs) (2.5, 5, 7 wt. %) and to investigate the effect of loadings these particles affect both the tensile and shear bond strength and what effect does this have on the strength of the attachment of the artificial teeth to the base of the acrylic denture.

The distribution in the polymer matrix, the shape, and size of the filler particles, as well as the contact with the matrix, function important roles in boosting the mechanical properties of polymer composites. It is necessary that the size of metal oxides be low enough for homogeneous mixtures [65]. In this study, almost 20–30 nm of SiO<sub>2</sub>NPs were mixed in about 121.2 μm of acrylic powder. This in turn prevents the formation of a heterogeneous mixture by filling the cracks between the polymer

molecules with nanoparticles, thus preventing the movement of the polymer chain. On the other hand, to ensure the incorporation of nanoparticles into the resin, low percentages of filler must be maintained [66]. Based on preceding studies, it is possible that different percentages of nanoparticles can lead to positive and/or negative effects on the flexural strength of acrylic resins, where Akkus et al. reported that adding 1% and 3% of SiO<sub>2</sub>NPs with a size of 15 nm to the heat cured acrylic resin, did not increase the flexural strength values, but rather it was low compared to the control group [67].

The outcomes of the present study showed a significant difference in the shear and tensile bond strength compared to the unmodified group. We noticed an increase in the tensile bond strength in proportion to the concentration of the added silicon nanoparticles. The rationale for this increase can be ascribed to the uniform distribution of small-sized SiO<sub>2</sub>NPs which enables them to fill the spaces between the linear chains of the polymer matrix and thus result in limiting the segmental movement of the macromolecular chains [68, 69]. While the shear bond strength increased at low concentrations (2.5 wt.% of SiO<sub>2</sub>NPs), which can be attributed to the homogeneous allocation of nanoparticles and their capability to penetrate spaces in the polymer chain, in addition to controlling their movement [70, 11]. It is worth noting that treating silicon nanoparticles with silane coupling agents formed a durable bond with the polymer matrix and reinforced the surface shear strength between the

inorganic nanoparticles and the resin matrix which is believed to have resulted from cross-linking or supramolecular cross-linking that prohibited crack expansion and consequently enhanced mechanical interlocking [72, 71]. Unfortunately, the shear bond strength decreased and showed its lowest value among the groups modified with nanoparticles when the concentrations of added SiO<sub>2</sub>NPs increased from 2.5 wt.% to 7 wt.%, this can be attributed to the aggregation of SiO<sub>2</sub>NPs and cluster formation, which in turn led to weak bonding and stress concentration [73, 70].

Since previous research did not investigate the effect of adding silicone to acrylic resin on both shear and tensile bond strength, this study is considered the first to evaluate this effect. In comparison with studies that evaluated the effect of adding silicon to acrylic on many other mechanical features, the results of the current study can be relatively compared with the results of previous studies that investigated the effect of adding silicon to acrylic on some other mechanical properties such as flexural strength, hardness, impact strength, etc. Azmy et al revealed that the reinforcement with low concentrations (3 wt. %) of SiO<sub>2</sub>NPs leads to an increase in the flexural and impact strength, and wear resistance, while the hardness increased at high concentrations of SiO<sub>2</sub>NPs (7 wt. %) [74]. These results, in turn, were similar to the findings by Alnami who reported that adding 3 wt. % and 5 wt. % of SiO<sub>2</sub>NPs powder to acrylic resin, significantly better the impact and transverse strength of modified PMMA and repaired dentures, but the surface hardness increases with increasing concentration (7 wt. %) of SiO<sub>2</sub>NPs, resulting in bestead mechanical properties compared to its higher content [75]. Also, Abushowmi showed a significant decrease with increasing concentration of the additive (SiO<sub>2</sub>NPs) for both flexural strength and impact strength; 0.25%-SiO<sub>2</sub>NPs showed the highest value of impact strength and flexural strength and their lowest value with 0.75%- SiO<sub>2</sub>NPs.

Balos et al found that the maximum values of microhardness and fracture toughness were obtained for PMMA reinforced with the lowest nano-silica content (0.023%), and when the concentration of SiO<sub>2</sub>NPs increased to 0.91% by volume, the microhardness and fracture toughness decreased to a minimum among SiO<sub>2</sub>NPs-strengthened groups [73].

A study conducted by da Silva et al. [76]

indicated that surface treatment of microwave-activated acrylic resins with 0.5 or 1.0% SiO<sub>2</sub>NPs improved flexural strength, but similar results were not achieved in terms of hardness. It was found that using 0.5% and 1.0% SiO<sub>2</sub>NPs concentrations in acrylic resin showed significantly higher flexural strength than the control group. While the addition of 5.0% SiO<sub>2</sub>NPs reduced the flexural strength. Regarding hardness, the control group (without silica) presented the highest mean value. The addition of silica from 0.1 to 5.0% led to gradually decreasing hardness values. Several previous studies reported that there was a significant improvement in surface hardness when acrylic resin was reinforced with SiO<sub>2</sub>NPs [73, 75, 77]. In contrast, Cevik et al. [40] reported that the hardness of the denture base did not improve significantly. This was supported by the results of da Silva et al. [76], who reported that surface treatment with concentrations of 0.1–5 wt.% of SiO<sub>2</sub>NPs caused a decrease in the hardness of PMMA/SiO<sub>2</sub>NPs nanocomposites. Sodogar et al. [39], found that the incorporation of SiO<sub>2</sub>NPs into PMMA can negatively influence the flexural strength of the nanocomposite, and this reduction was directly related to the content of SiO<sub>2</sub>NPs. Also, Gad et al revealed that the incorporation of SiO<sub>2</sub>NPs significantly enhanced the flexural strength of the repaired acrylic resin compared with the unadjusted group. This improvement was greater at lower concentrations of SiO<sub>2</sub>NPs than at higher concentrations [41].

The present study achieved an evident improvement in the tensile bond strength with all percentages of SiO<sub>2</sub>NPs compared to the control group. This improvement was related to the concentration of the nanoparticles added, as 7 wt.% showed the highest tensile strength values, followed by 5 wt.% and then 2.5 wt.%. This increase can be explained by the existence of the nanoparticles on the sample surface and perfect bonding between the resin matrix and nanofillers, which makes breaking this bond require more energy [71]. Clinically, modification of PMMA with inorganic SiO<sub>2</sub>NPs to improve its mechanical properties (tensile and shear bond strength), resulted in strengthening the bond between artificial teeth and acrylic denture base materials. By adhering to the rules of scientific research, we had to mention the limitations correlated to this study, including imperfect prediction of the clinical achievement of the tested substance, as realistic

clinical conditions impose some further factors, for example, the existence of saliva, feeding habits, neuromuscular strength, semi-functional habits, and various disinfectant techniques can impact the results, and this leads us to say that the results presented by this study are only a promising starting point for further investigations. In addition, we must mention that only one type of acrylic resin denture material was tested, in addition to the lack of simulation between the shape of the simple cylindrical sample on which the tests were conducted and the shape of the actual denture.

## CONCLUSION

Within the limitations of this study, we deduce that the consolidation of SiO<sub>2</sub>NPs into PMMA denture base resin significantly increased the tensile bond strength for all groups tested (2.5%, 5%, and 7%) compared with the control group, and this increase was concentration-dependent. Low SiO<sub>2</sub>NPs (2.5%) is the preferred concentration which may increase the shear bond strength of modified denture base resins. Therefore, this fusion process will reduce the possibility of separation of the artificial teeth from the denture base.

## CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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