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# Miswak fiber particles and polymer (MFP/PEEK) influence incorporation on properties, biocompatibility of PMMA composites.

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The PMMA (polymethyl methacrylate) composite is made using a particular percentage of polyether ketones (PEEK). In this study, 5% of PEEK is used, along with a chosen proportion of Miswak (MFP) natural fiber particles in two different particle sizes—coarse and fine, using a hand-layout technique. Using PSD the particle size dimension is a quantitative measure of the size of particles, typically expressed in terms of diameter, length, or other relevant dimensions, and is crucial in determining the physical and chemical properties of materials. of both kinds (31112.3 nm coarse and 816.3 nm fine) are assessed. Every manufactured PMMA composite underwent a number of tests, including ones for density, water absorption, and biocompatibility. The findings indicated for the density test revealed a reduction when sample S6 (1.042 g/cm3) is supplemented with 8wt.% of fine MFP. For water absorption rate, the addition of fine MFP as in in sample S6 attaining a maximum value of 38.42, whereas S2 had the lowest water absorption value, and its value is (1) which is reinforced with PMMA/PEEK blends, Miswak fiber nanoparticles (MFP), which exhibit strong antibacterial and cytotoxic properties and could be used as a source for medication delivery in health enhancement, show promise as a healing agent when added to PMMA composites. At 28 mm, sample S6 8% weight percentage of fine MFP had the greatest lethal impact on E. coli cells. Together with testing investigations, these PMMA tests demonstrated the formulation's effectiveness and bacterial resistances, which made it a highly successful combination in dentistry. In contrast, PMMA biocompatibility testing produced superior results and demonstrated resistance to microbes.

**Keywords:** PMMA composites; Miswak Fiber; Antibacterial activity; Water absorption.

## 1. INTRODUCTION

Polymethyl methacrylate (PMMA), the most well-known polymer in the methacrylate family, considered excellent transparency, blood compatibility, and biocompatibility are provided by this stable, long-lasting, low-density polymer, which makes it appropriate for a range of biomedical applications that call for durable structures, including orthopedic surgery and bone tissue engineering [1_3]. Polymeric composites materials are employed recently to modifying the performance of PMMA as polymer matrix with special additives and fillers. Polymeric Nano composites contain fibers, sheets, or particles with sizes ranging from 5 to 100 nanometers. The production of polymeric Nano composites (PNC) and polymeric composites (PCM) uses Polymers, both synthetic and natural, are employed as binders [4]. PEEKs, or polyether ether ketones, are another type of synthetic polymer that is use as advance material as reinforcement polymers as in [5]. Polyetherether ketone (PEEK) has also been used as a material for dental prostheses as in [6]. Additionally, two poly(aryl)ether-ketones (PAEKs) used with acrylic denture base resin are examined for bonding performance and heat cycling [7]. PEEKs have been modified and utilized with strengthening elements for dental implant applications. Hence PEEKs possess excellent mechanical, biological, and thermal qualities, and dentures have been created utilizing a binary combination of PMMA and PEEK as in [8]. Numerous research has employed a range of natural fiber [9]. Incorporating hemp fiber reinforcing [10]. And pistachio shells [11]. The addition of such eggshells to reinforce acrylic used in dentistry [12]. Coconut fibers [13]. Also, two kinds of natural fibers added as fillers, the peels of dates and pomegranates [14].

Miswak fiber reinforcing is one of the natural fibers utilized in this study, in few investigations, PMMA material has been reinforced with natural fibers. For instance, miswak fibers and PMMA have been employed longitudinally in dentistry [15]. Particles have also been used in several research projects for dental applications. In one study, two fiber types (polypropylene PP and polyacrylonitrile PAN) are added to the matrix polymer PMMA to create a composite material with enhanced mechanical capabilities, as shown in [16]. In addition, a study of HA with dental hydroxyapatite crystals has been conducted. These nanoparticles have been incorporated into various dental formulations for various applications to ensure comprehensive oral health care [17]. PMMA/$TiO_2$ NPs are utilized in this investigation since the inclusion of nanoparticles is done as a filler [18]. One of the oldest known oral hygiene instruments, miswak, is still used by millions of people in Asia, the Middle East, South America, and Africa. The exceptional performance of miswak has been explained by a number of theories. These include (1) the mechanical characteristics of its fibers, (2) substances such as trimethylamine, salvadorine, mustard oil, vitamin C, resins, fluoride, flavonoids, saponins, and sterols, and/or a combination of (1) and (2) that may be necessary for tooth cleaning and maintenance [19,20]. The objective of this research is to fabricate denture resin materials with enhanced qualities and increased the biocompatibility using natural fibers particles MFP. In addition, performing surface hardness, density and water absorption evaluation.

## 2. EXPERIMENTAL

### *2.1 Synthesis of MFP preparation*

The miswak fibers is crushed and ground extremely fine for the purpose of creating natural particles as in the Figure1. Following screening, the both coarse and fine particles are chemically treated with alkali (chemical treatment). One liter of distilled water and (50g) of sodium hydroxide are progressively combined to create the base solution used in the chemical treatment. To dissolve, the liquid is vigorously swirled. After the basic solution is left overnight, the prepared particles to be treated are added. For a whole day, the mixture is to stand at room temperature. After the allotted amount of time, the treated item is repeatedly rinsed with distilled water to get rid of any remaining base solution and reach neutralization (pH 7). After that, the substance is allowed to cure at room temperature for five days. To verify the drying process, they are lastly dried for 45 minutes at 50 to 60 degrees Celsius in a dedicated oven [ 21]. A particle size distribution (PSD) test is performed for MFP to confirm the particle size of the natural addition utilized with PMMA composites. as seen in Figure1, which displays the PSD test results. For coarse MFP, the measured size range is 31112.3 nm, whereas for fine MFP, it is 816.3 nm.

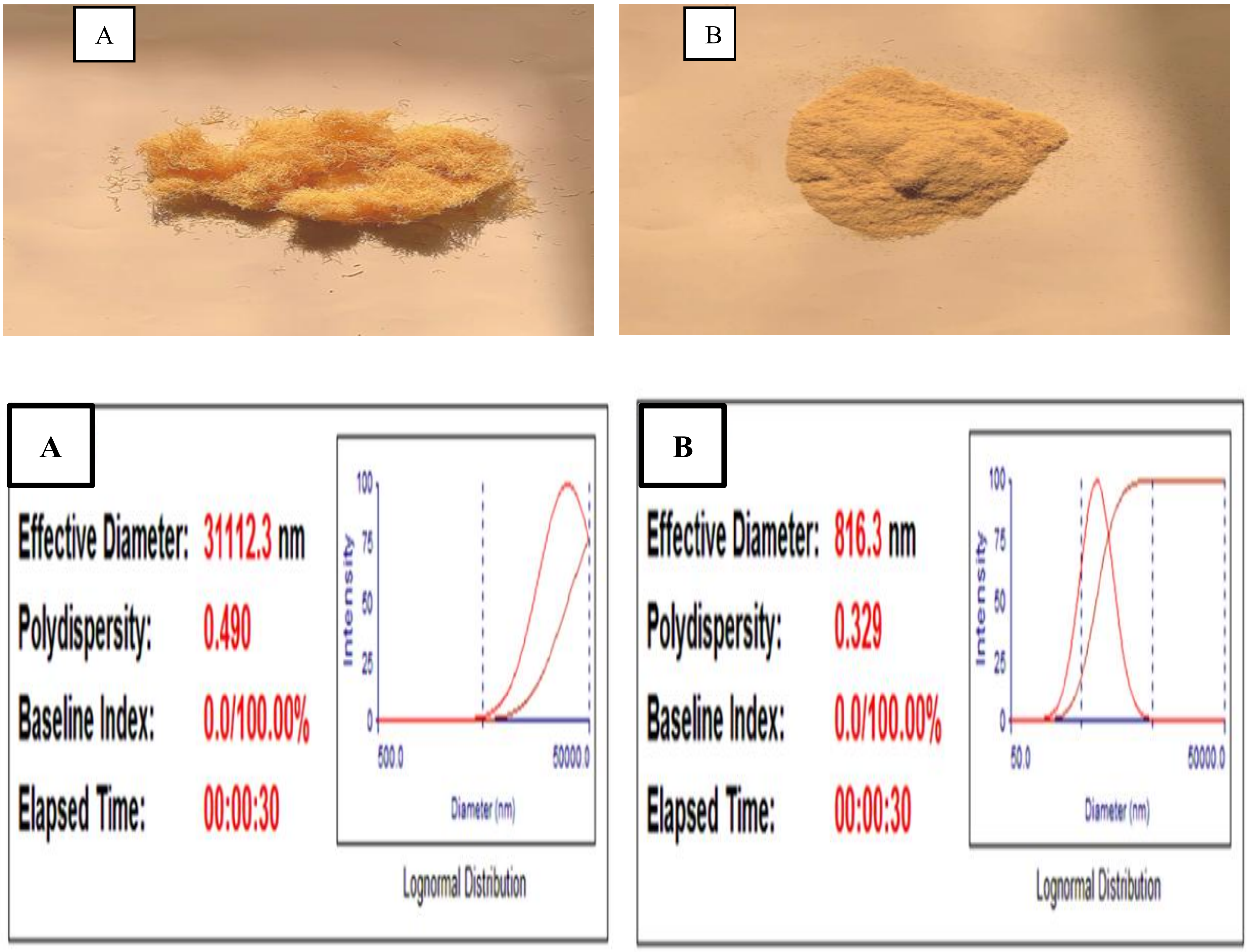


**Figure 1** Particle size analysis results and particles shape (A) MFP coarse, (B) MFP fine.

*2.2 Synthesis of PMMA/PEEK/MFP composite preparation*

A self-curing PMMA mixture and PEEK are used as polymer blends with addition of natural Miswak fibers, particles (MFP) in this research. The PMMA manufactured in the form of self-polymerizing acrylic. Using the Hand lay-up technique, samples of PMMA and PMMA/PEEK and PMMA/PEEK/MFP composites are prepared as base materials for artificial denture. The control

PMMA 46wt% of hardener and 93wt.% of PMMA These ratios are set through the following relationship:

$$Weight\ of\ component = (Ratio\ /\ Total\ ratio) \times Total\ weight \quad (1)$$

are mixed together in a beaker, the pure liquid is then poured into a silicone mold in various shapes as Figure2 and left there 24 hours.After using the mixture, which contained a 3:1 ratio of monomers to polymers. (3 powder: 1 MMA liquid), the pure samples are taken out of the mold and post cured 50 to 60 C$^{o}$and fig2 shows the samples after mixing. On the other hand, to prepare PMMA/PEEK polymer blends Common solvents for PEEK include,Sulfuric acid, NMP (N-Methyl-2-pyrrolidone), DMSO (Dimethyl sulfoxide)These solvents can dissolve PEEK under specific conditions. a specific and constant amount of PEEK 5% is added to PMMA powder. Furthermore, 5wt.% of PEEK is added with all other samples and mixed to obtain the PMMA/PEEK/MFP composite. The addition of coarse and fine MFP is (5and 8) wt.%, respectively. Then, the prepared mixture is cast and poured into the silicon mold. Finally, the mixture should be left at (25°C) for 24 hours for curing purposes. The post-curing process is carried out by using oven at (55°C) for (45 minute) as shown in Table 1.

**Table 1** shows the sample sets used in this research.

| Sample No. | Material proportion %(w/w) PMMA | Material proportion %(w/w) PEEKs | Material proportion %(w/w) Fiber | Weight of PMMA (g) | Weight of PEEK (g) | weight of fiber (g) |
|---|---|---|---|---|---|---|
| S1 | 100% | - | - | 92.52 | _ | - |
| S2 | 80% | 20% | - | 54.2 | 16.66 | _ |
| S3 | 90% | 5% | 5%(coarse) | 71.2 | 6 | 6 |
| S4 | 90% | 5% | 5%(fine) | 88.2 | 7.5 | 7.5 |
| S5 | 87% | 5% | 8%(coarse) | 68.8 | 6 | 9.4 |
| S6 | 87% | 5% | 8%(fine) | 68.8 | 6 | 9.4 |

*2.3 Experimental Test*

*2.3.1 Density Test*

Three materials made up the composite samples used in this investigation, and their density is determined using the formula [22]

$$Specific\ Gravity\ (SP.Gr) = Md/(Md + Mi)\_M \quad (2)$$

$$\text{Density (D)} = \text{(SP.Gr)} * (0.997) \quad (3)$$

Where: (Md) The specimen's mass in the atmosphere (gm). (Mi) The specimen's mass (gm) in distilled water,(M) The partially submerged wire's mass, approximately 0.02 grams.

*2.3.2 Water absorption test*

In compliance with ASTM D 570, the test sample is weighed in air before being immersed in water [23]. Once the duration of the immersion has been determined, the sample is removed after a 24-hour immersion in distilled water at room temperature (27±3) C. Once any remaining water has been wiped off the sample's surface with a dry cloth, the sample is weighed, and the difference between the two weights is calculated [24].

*2.3.3 Biological test/ antibacterial activity*

The antibacterial activity of the generated samples (Control, S2, S3, S6) is tested against strains of both Gram-positive and Gram-negative bacteria using the agar well diffusion assay [25, 26]. Muller-Hinton (MH) agar (20 milliliters) is aseptically placed into sterile Petri dishes. The bacterial species are isolated from their stock cultures using a sterile wire loop [27]. After the organisms are cultured, wells of 6 mm in diameter are drilled into the agar plates using sterile needles. The samples are used in the bored wells at different concentrations. Before determining and recording the average diameter of the zones of inhibition, The test organisms and samples are placed on cultured plates and incubated for a whole night at 37°C [28,29].

*2.3.3.1 Prepare of mueller hinton agar*

38 g of the powder is heated in 1 L of distilled water while stirring to create Muller-Hinton (M-H).M-H must be autoclaved for 15 minutes at 121°C to disinfect it. It is cooled to 50 °C, then poured onto a petri dish, allowed to harden for about 15 minutes, then inverted and chilled at 4 °C.

## 3. RESULTS AND DISCUSSION

*3.1 The density*

As shown in Figure2, the density of PMMA/PEEK/MFP Composite with all different addition ratio. First, there is a clear decrease in density value between S1 and S2 which displays that PEEK density lower than PMMA polymer, the density of PMMA 1.63 g/cm$^3$while 1.27 for PEEK [30]. Moreover, As the weight percentage of reinforcing fibers increase, the density values decrease. For S3 the weight fraction of PMMA/PEEKs/MFP coarse is (1.319) g/cm$^3$) whereas S5 the weight fraction of PMMA/PEEKs/MFP coarse is (1.167 g/cm$^3$ ), the densities of added fibers are significantly lower than those of PMMA. the density of Miswak fiber about (1.319 g/cm$^3$) [31] PEEKs/MFP coarse/PMMA density as in S3 and S5 compared to PEEKs/MFP fine/PMMA as in s4 and S6 is significantly higher, (1.074g/cm$^3$ ) and (1.042 g/cm$^3$) respectively. As the fiber size decreases, the lower the density affect PMMA's, this can be attributed to the fact that larger particles increase the void ratio in the matrix, which in turn affects the relative density of the material, resulting in a higher density [32] This implies that fine particles decrease the void ratio in the composite, resulting in a lower density. Also, these results could be as outcome from better dispersion inside PMMA/PEEK mixture of the fine compared to coarse MF during preparation. So, Maximum enhancement (lower density) is observed clearly overall in S2, S6 as in Figure 2.

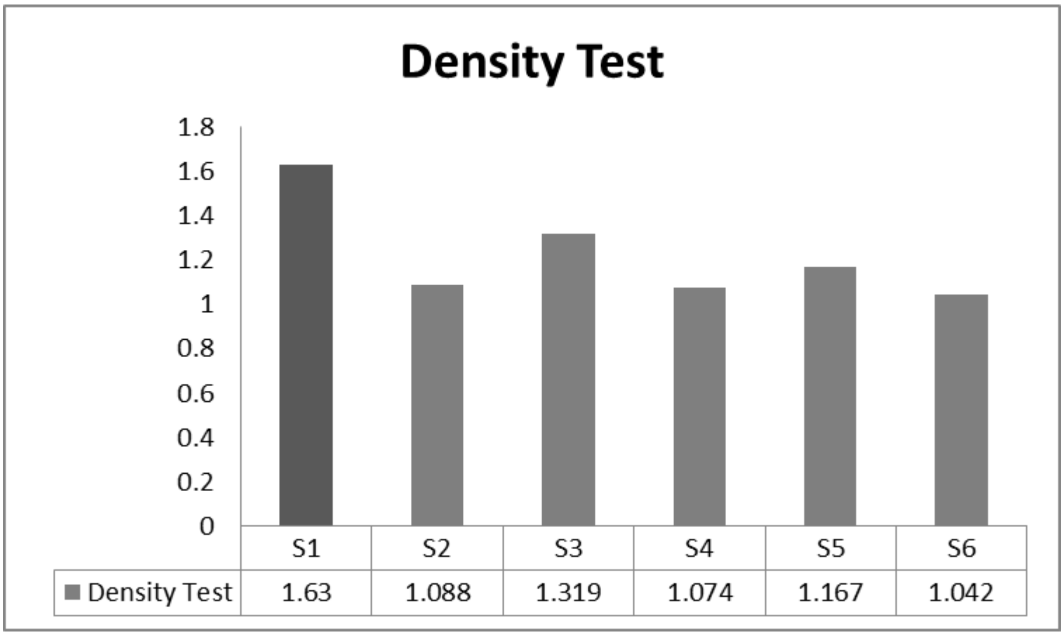


**Figure 2** density test results Vs composite addition ratio.

*3.2 Water absorption*

Figure (3) shows the relationship between the water absorption determined and addition ratio of the plain PMMA, PMMA/PEEKs, and PMMA/PEEK/MFP samples. This value showed a considerable decrease after mixing with PEEK in 5wt.% ratio. From (20.71% ) to (1%) the water absorption for S1 and S2 respectively. This phenomenon may be attributed to the reduced water absorbency resulting from mixing, which is consistent with PEEK's inherent low water absorbency properties, particularly its stability in water-bound environments [33]. Furthermore, water absorption is directly proportional to the fiber ratio, likely due to the natural fibers' inherent capacity for water absorption and their susceptibility to moisture exposure., and fibers' high cellulose content, which encourages capillary action and water penetration [34-37]. PMMA/PEEK/MFP composite with both fiber type absorbs more water compare to S1 and S2. Moreover, fine MFP in composite sample as in S4 and S6 exhibits a greater water absorption rate than the coarse MFP composite as in S3 and S5.

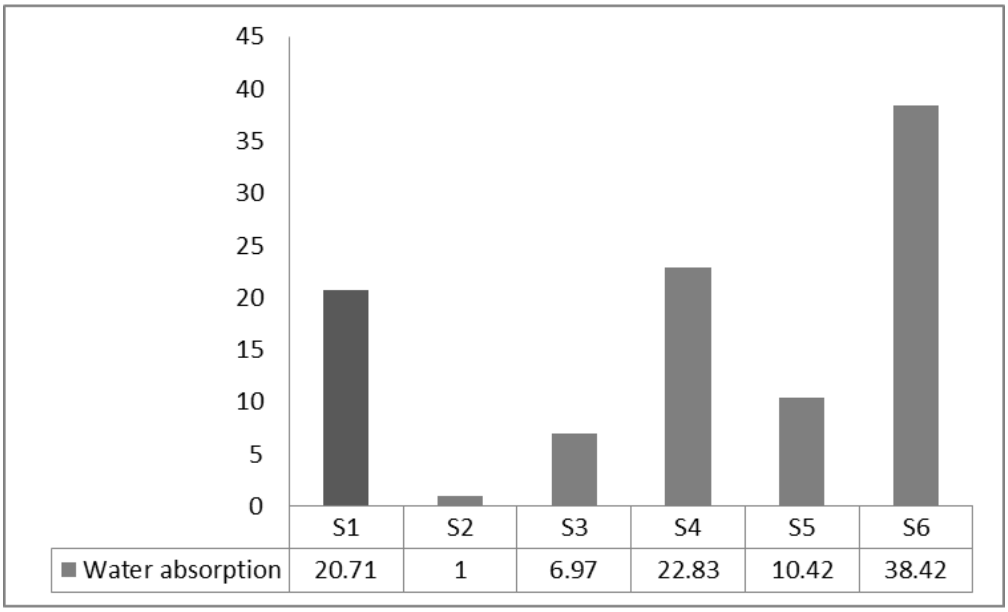


**Figure 3** water absorption test Vs composite addition ratio.

*3.4 Antibacterial activity*

Antibacterial activity properties of S2, S3, and S6 in contradiction of isolated pathogens—gram-positive Streptococcus mutans (S. mutans) and gram-negative Escherichia coli (E. coli), are listed below. The results shown as in Table (2).

**Table 2** showed that every sample is effective against every pathogen assessed. The range of their inhibition zones is 6 ±0.8 mm to 28 ±0.8 mm.

| Antibacterial analysis (Zone of inhibition (mm)) | | | | | | |
|---|---|---|---|---|---|---|
| Sample | | A | B | C | D | E |
| *E.coli* | S2 | 8 | 11 | 15 | 16 | 22 |
| *S.mutans* | | 6 | 13 | 14 | 18 | 20 |
| *E.coli* | S3 | 13 | 17 | 18 | 22 | 24 |
| *S.mutans* | | 6 | 16 | 17 | 22 | 25 |
| *E.coli* | S6 | 6 | 18 | 19 | 25 | 28 |
| *S.mutans* | | 6 | 20 | 22 | 24 | 27 |

E. coli is the most active, after S. mutans. The largest zone of inhibition against E. coli is achieved by (S3) (28 ± 0.8 mm). As shown in Figures (4) and (5), the sample had the smallest zone of inhibition against S. mutans, measuring only 12 ± 0.5 mm. These results suggest that while E. coli is significantly affected by the tested compounds, S. mutans exhibits a higher level of resistance, indicating a need for further investigation into alternative treatments, measuring 20 ± 0. 5 mm. This disparity highlights the importance of understanding the specific interactions between antimicrobial agents and different bacterial strains, which could lead to more effective therapeutic strategies in combating resistant infections [38-40]. Further research could explore the underlying mechanisms contributing to this resistance in S. mutans. 8 mm.

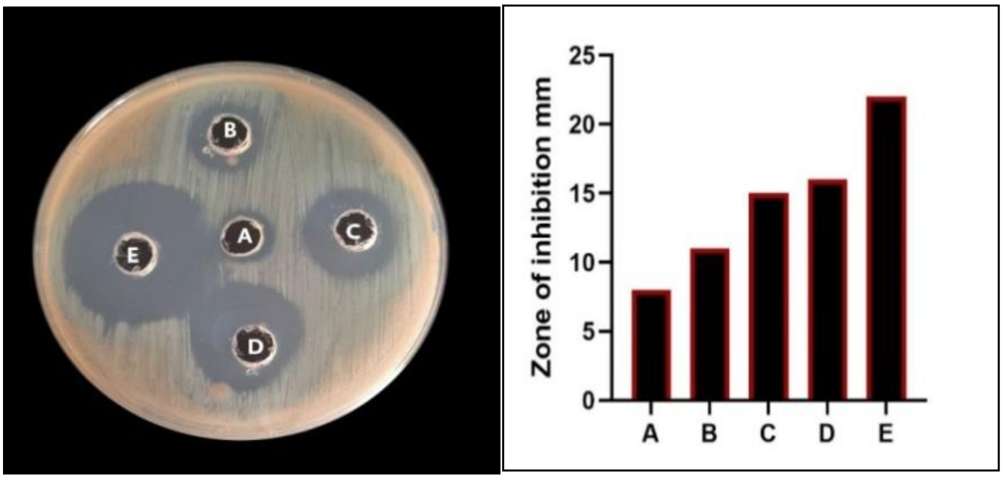


**Figure 4** Antibacterial activity of against *E.coli.* A, control (250 µg/mL). B, (S2 (31.2 µg/mL)). C, (S2(62.5 µg/mL)). D, (S2 (125 µg/mL)). E, (S2 (250 µg/mL).

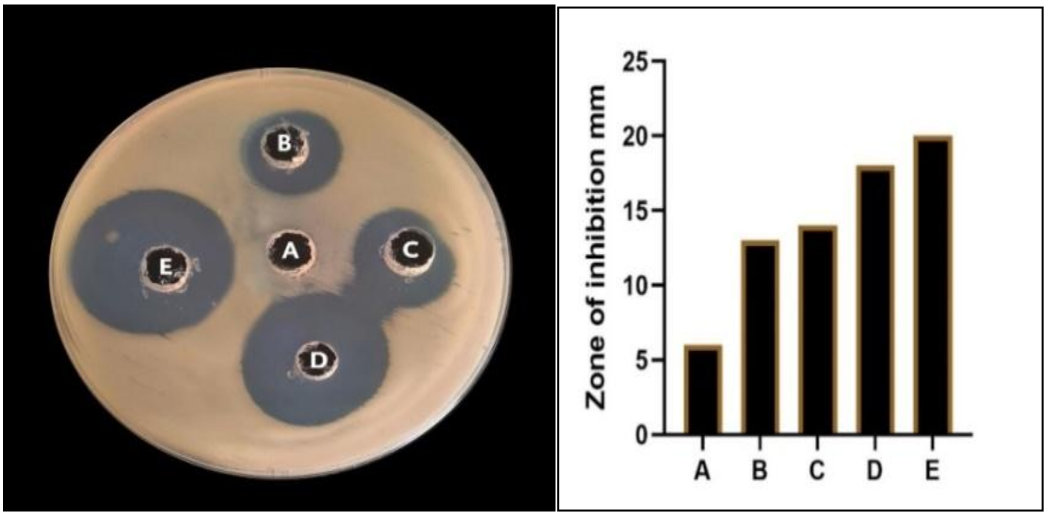


**Figure 5** Antibacterial activity of against *S.mutans.* A, control (250 µg/mL). B, (S2 (31.2 µg/mL)). C, (S2 (62.5 µg/mL)). D, (S2 (125 µg/mL)). E, (S2 (250 µg/mL).

PEEK is a hydrophobic polymer at sample (S2) (PMMA-PEEK), as Figures (4and 5) demonstrate. The water contact angle of PEEK is therefore increased when it is combined with other hydrophobic materials. PMMA can increase the hydrophobicity and water contact angle of PEEK-PMMA composites, depending on its concentration. Its surface roughness changed as the structure's PEEK conten, improving its hydrophobicity [41-43]. The surface's hydrophobicity stops hydrophilic germs from adhering to it. A hydrophobic substance can be added to create hydrophobicity, or the surface texture can be altered [35, 36]. Can also clarify another factor contributing to composites' antibacterial properties: Generation of reactive oxygen species (ROS) The integrity of the bacterial cell membrane is weakened by electrostatic interactions with antibiotic ions. Antimicrobial particles break through the cell membrane and generate reactive oxygen species (ROS), which harm mitochondria, proteins, or

DNA [37]. In Figure (6),(7), in the composite (B) and Figure 8&9 the composite (C) exhibited antibacterial activity against S. aureus and E. coli upon the addition of Miswak [44-47].

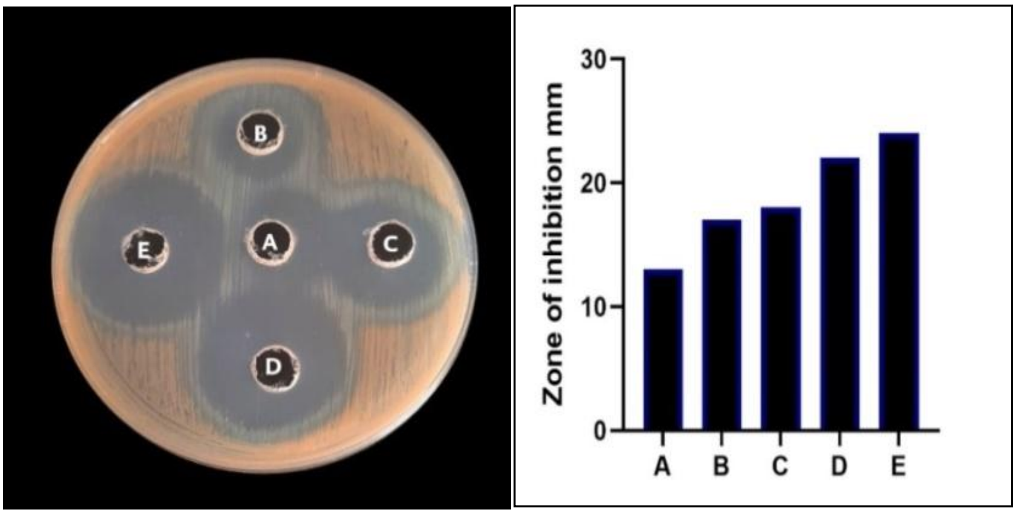


**Figure 6** Antibacterial activity of against *E.coli.* A, Control (250 µg/mL). B, (S3 (31.2 µg/mL)). C, (Sample S3 (62.5 µg/mL)). D, (Sample S3 (125 µg/mL)). E, (Sample S3 (250 µg/mL).

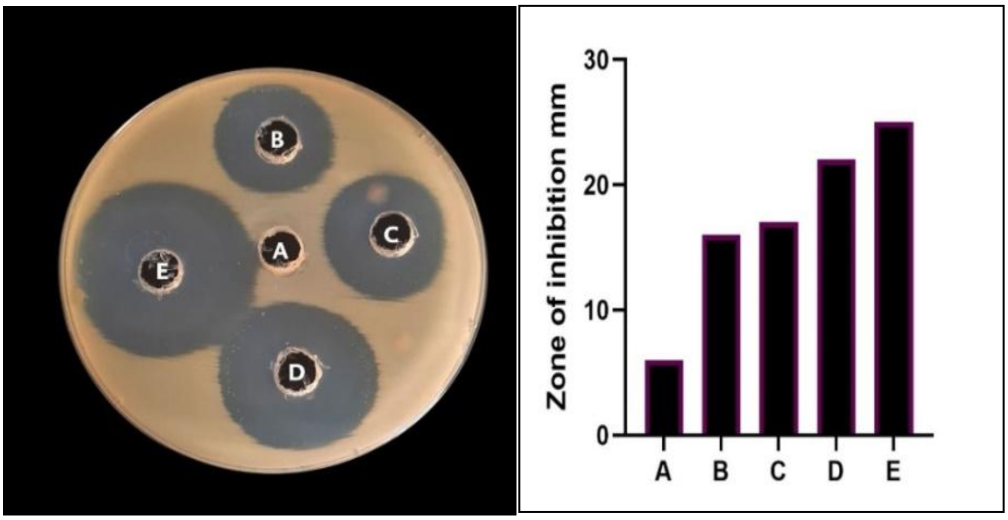


**Figure 7** Antibacterial activity of against *S.mutans.* A, Control (250 µg/mL). B, (Sample S3 (31.2 µg/mL)). C, (Sample S3 (62.5 µg/mL)). D, (Sample S3 (125 µg/mL)). E, (Sample S3 (250 µg/mL).

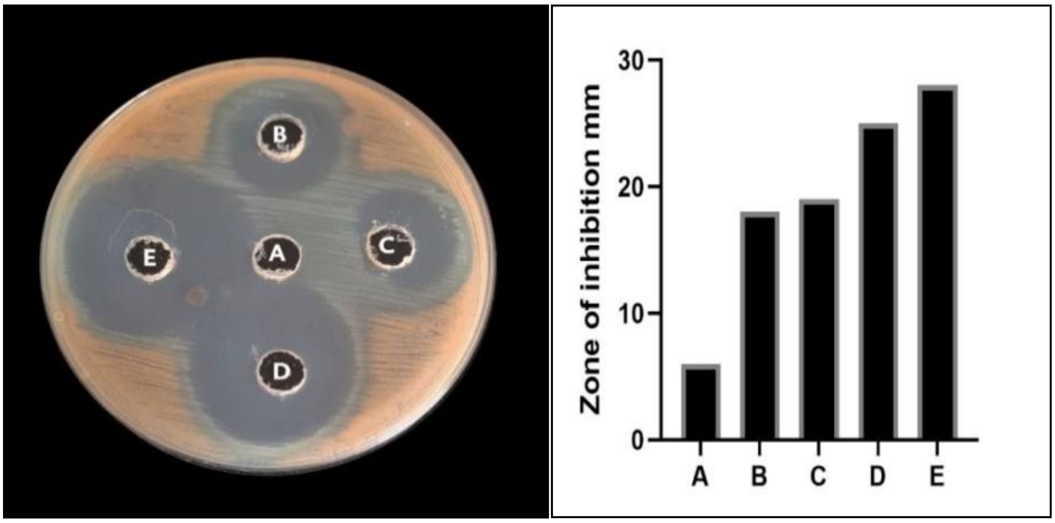


**Figure 8** Antibacterial activity of against *E.coli.* A, Control (250 µg/mL). B, (Sample S6 (31.2 µg/mL). C, (Sample S6 (62.5 µg/mL). D, (Sample S6 (125 µg/mL). E, (Sample S6 (250 µg/mL).

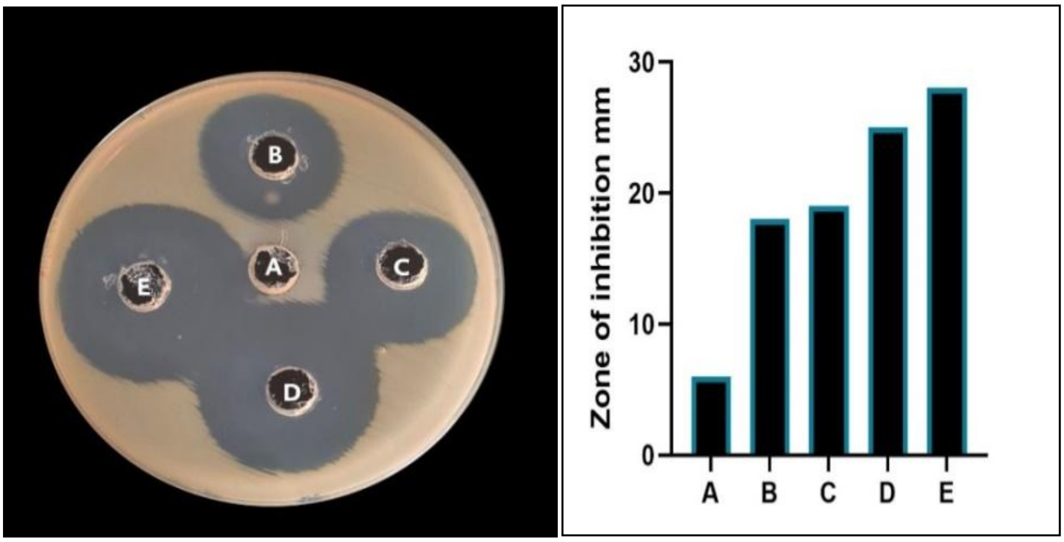


**Figure 9** Antibacterial activity of against *S.mutans.* A, Control (250 µg/mL). B, (S6 (31.2 µg/mL). C, (Sample S6 (62.5 µg/mL). D, (Sample S6 (125 µg/mL)). E, (Sample S6(250 µg/mL).

The contact reaction mechanism used to explain the negatively charged bacterial membrane and the positively charged substance ions interact electrostatically. When 8 % weight percent Miswak is introduced to the PEEK-PMMA composite, the ion deactivates the protease, an enzyme that breaks down proteins into peptides, which results in a decrease in the physiological activity of bacterial cells. By binding hydrophobic imidazole amino and carboxyl groups, the ions in a composite system integrate into the bacterial membrane, rupturing the cell membrane and letting the cell content leak out [38]. The results demonstrated that samples C possess antibacterial qualities against E. coli and S. aureus. Furthermore, our results demonstrate that the Miswak-reinforced composites improved oral health, due to the presence of compounds (chemical compositions) having anticariogenic qualities. *S.*

*persica* Miswak had a cute effect on oral microorganisms, according to Sofrata et al. [39,40]. Furthermore, in a study using S. persica extracts against microorganisms, Almas et al. [41, 42] found that E. faecalis is the most sensitive pathogen [48, 49]. Furthermore, there is no appreciable difference in the effects of fresh and old Miswak cuttings in this investigation [50, 51]. This work includes techniques to modify surface topography, make composites, evaluate with antimicrobial material, and improve the mechanical properties of functional materials [52, 53]. Miswak chopped fiber is used to boost PMMA-PEEK's antibacterial properties [43].

## 4. CONCLUSIONS

In this current study, PMMA/PEEK/MFP trinary composites with modified characteristics. The following results inferences from the experimental: For the Regarding the density test, it showed a decrease when reinforced with (8wt.%) fine MFP miswak fiber particles (1.042 g/cm³) in sample S6.The water absorption rate of fine PMMA/PEEK/MFP increases with the weight fraction, reaching a maximum value of 38.42 at 8% weight fraction in sample S6, whereas the lowest value of 1 was recorded for the PMMA/PEEK sample S2. Biologically, the addition of Miswak fiber nanoparticles (MFP) to the PMMA matrix holds promise as a healing agent, demonstrating significant antimicrobial effects and cytotoxicity, and serving as a potential source for drug delivery in health enhancement. The best killing effect against E.coli cells was observed at (28mm) with an 8% weight percentage of fine MFP in sample S6.

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