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Poly Methyl Methacrylate (PMMA)/Polyether Ether Ketones (PEEKs)/ Miswak Fiber Particles (MFP) Ternary Composites Mechanical/ Morphological Assessment for Dental Application

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Abstract: Because of its exceptional biocompatibility as well as outstanding aesthetics, Poly methyl Methacrylate (PMMA) is widely recognized as a fundamental material for denture bases frequently used in dentistry and medical applications. Ternary composites made of PMMA/PEEK/ Miswak Fiber Particles (MFP) were fabricated and examined to examine the possibility improvements in contrast to pure PMMA. The chosen ratios of PEEK were fixed at 5%wt. and MFP were 5%wt and 8%wt (coarse and fine). The ternary composites specimens then tested for impact strength, bending strength, and tensile toughness, and characterized under SEM microscopic analysis. The findings revealed a notable enhancement in impact force for samples S5, S6, containing 5wt.% and 8wt.% fine MFP, respectively. These samples exhibited impact strength values of 56 kJ/m² and 56.1 kJ/m², representing a 47% improvement over the pure PMMA control sample. Even though a little decline in value of bending strength was observed, the highest value was obtained with S2 (PMMA/PEEK) at approximately 2451.12 kg/mm², which represents a 23.31% increase over the control PMMA (1987.8 kg/mm²). In contrast, the lowest value was recorded for S5 with coarse MFP at 1089.5 kg/mm² compared to the control PMMA. A gradual increase in tensile strength was observed for sample S2 reinforced with PEEK, reaching 83 MPa, which is 17% higher than the control PMMA sample (71 MPa). The SEM morphology characterization revealed a uniform and effective dispersion of both coarse and fine MFP particles within the PMMA/PEEK matrix, with no signs of particle agglomeration.

Keywords: SEM, Impact, Bending, Mechanical properties-poly methyl meth acrylic, Polymer blend, Tensile

Introduction

The most widely recognized polymer in the set of methacrylates, polymethyl methacrylate (PMMA), which is produced when methacrylate is subjected to in-chain polymerization. This stable, long-lasting, low-density Polymer offers outstanding transparency, hemocompatibility, and biocompatibility, completing it appropriate for numerous biomedical applications that call for long-term, structurally sound constructs, Constant oral environment when used in dentistry, such as bone tissue engineering and orthopedics (Al-Karam, 2019;Díez-Pascual, 2022; Hacker, 2019; Jessy , 2014). Because of its great transparency, PMMA is an ideal alternative to glass in situations where weight or impact is a significant concern (Abd Alwahab, 2020). Its appearance, price, ease of processing, and adaptability in terms are the ideal option due to its minimal density, simplicity of shaping, adjustable mechanical qualities, and ease of handling. Its uses in dentistry include occlusal splints,

temporary or permanent crowns, obturators, prosthetic teeth, foundation for dentures, dentures, and fixed restoration (Alla,2015; Díez-Pascual, 2022).

Interestingly, composite materials exist and employ polymer matrix that modified using fillers and additives to produce advanced polymer composites. Fibers, films, or particles (ranging from 5 to 100 nm) are found in polymer nanocomposites (Miao, 2013). In one study, two fiber types (polypropylene PP and polyacrylonitrile PAN) were added to the PMMA in order to create enhanced mechanical PMMA composite, as shown in Sharhan (2022) .

Synthetic polymers are also used like polyether ether ketones known as PEEK, PEEK can be defined as a linear, aromatic, semi-crystalline, polycyclic, thermoplastic polymer. Due to its high temperature and resistance to hydrolysis, PEEK exhibits exceptional electrical and mechanical properties. PEEKs with reinforced material and modifications have been used for dental applications as in Rahmitasari (2017). Also, Dentures have been made using a binary mixture of PMMA and PEEK, to noted that PEEKs chosen due to their superior mechanical, biological, and thermal properties, as stated in Majeed (2024). Poly ether ketone (PEEK) is a substance that may find application in dental restorations, according to this article. Restorations made without alloy are being used in dental operations more often now adays due to aesthetic concerns PEEKs is a substance that has high power, excellent resilience and toughness to chemical assault. PEEK can also be processed and is resistant to heat among different restorations without metal that could be applied in the field of dentistry. Due to its remarkable properties concerning prosthodontics and dental implant procedures, PEEK is getting more and more important (Parate, 2023; Nizam, 2014).

According to reports, adding fiber reinforcement to acrylic resin increases the resin's flexural strength, impact resistance, and fatigue resistance. A variety of natural fibers have been used in numerous studies (Gad, 2017), including pistachio shells and crushed peanut shells (Hamad, 2019) and hemp fiber reinforcement (Ahmed, 2021), as well as two types of natural fibers like dates and pomegranate shells (Salih, 2018) coconut fibers (Boutaleb, 2023) bamboo powders and rice straw (Oleiwi, 2018) and eggshells for acrylic reinforcement (Alraziqi, 2020).

Moreover, natural fibers used in reinforcement are Miswak fibers that have been utilized in some studies as PMMA fillers material. In the field of dentistry, Miswak fibers have been used longitudinally with PMMA as in Salih (2018). Miswak fiber has also been used with polylactic acid where then it is cut into short fibers, the composite was prepared with varying weight composition ranging from 0 to 30 wt.% of MF content as in Nur Diyana 2(022). In Asia, the middle east, South America, and Africa, millions of people still use Miswak, one of the first known oral hygiene tools. Numerous theories have been put forth to explain Miswak's remarkable achievement. consisting of (i) the mechanical properties of its fibers, (ii) trimethyleamine, Vitamin C, resins, mustard oil, and salvadorine fluoride, Sterol, flavonoids, and/or a combination of (i) and (ii) may be necessary for its release in order to clean and preserve the health of teeth. Considering the cultural, social, religious, and historical ramifications of using miswak (Salvadora Perscia) for dental hygiene (Haque, 2015)

The purpose of the research was to create denture resin compositions that are advanced with greater biocompatibility and improved mechanical qualities, from PMMA/PEEK and a special addition of MFP at a selected ratio to presents ternary composites. In this work, the mechanical behavior of all hybrid/ composite was investigated (Impact strength, bending and tensile test) also particle size distribution (PSD), scanning electron microscope (SEM) additionally performed for more structural details.

Materials and Method

Control PMMA Making

In this research, PMMA as weight powder was supplied by the Turkish company SMD, and PEEKs is a white powder composed of polyether ether ketones. The amount of 46 g of liquid hardener and 93 g of PMMA were mixed together in a baker to prepare the control case samples (3:1 ratio monomer to polymers). The mixture then poured into a silicone mold in the selected required dimension for each test and left for a day. After that, samples were dried in about (50 – 60 ˚C).

PMMA\ PEEKs Preparation

The amount of a 54.2g PMMA reinforced with 16.66g of PEEKs were used for the other sample in this study. It is to be noted that PMMA was mixed with the weighted number of PEEKs then with the liquid hardener until dough stage, Then, the mixture was poured.to the silicon mold, allowed to dry for a day at the standard temperature.

PMMA\PEEKs\MFP Ternary Composites Preparation

For PMMA ternary composites, natural miswak fiber particles MFP were selected in various weight ratios (5 wt.% and 8wt.%). The selected amounts of MFP after several preparation techniques were used with PMMA/PEEK for ternary composites preparation. After adding the hardener, the liquid was placed in a silicone mold and allowed to cure for a day. The mixture which consisted of both coarse and fine particles fibers gradually blended.

Table 1. Composition of PMMA/PEEK binary blends

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

MFP Preparation

To verify the particle size of the miswak fibers used in the reinforcement, a particle distribution (PSD) test was conducted using MFP. As in form (1) which displays the outcomes of the PSD test. The measured size range (31112.3nm) for coarse MFP and (816.3) nm for fine MFP. For natural particles preparation, the miswak was crushed and ground very fine, after screening both the fine and coarse particles were chemically treated with alkali treatment.

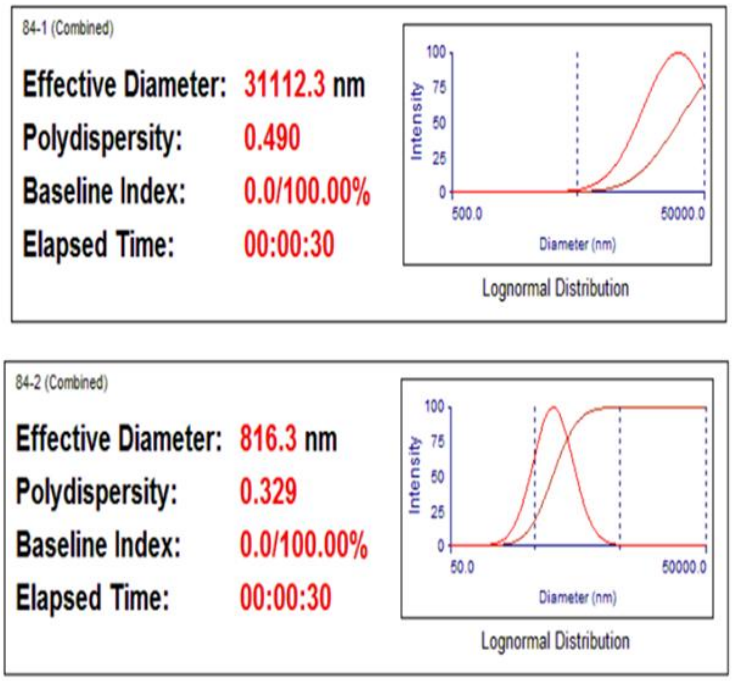

Figure 1.PSD of (A) MFP course, (B) MFP fine.

The chemical treatment contains a base solution which was made by gradually combining 50g of (sodium hydroxide) with one liter of distilled water. The liquid was thoroughly stirred to dissolve. The prepared particles to be treated were introduced after the basic solution had been left overnight. The mixture was left to stand at room temperature for a whole day. To remove any leftover base solution and achieve neutralization (PH 7), the treated item was repeatedly rinsed with distilled water after the designated period of time. The material was then left to cure for five days at room temperature. Finally, they were dried in a special oven for 45 minutes at 50- 60 Celsius, to confirm drying process (Benyahia, 2014).

Mechanical Properties and Morphology

Impact Test

Charpy impact test apparatus was utilized for this test. The sample was manufactured in compliance with the international standard (ASTM D 6110-04) (Adnan, 2023), as illustrated in figure 2. Where Equation 1 is utilized to compute the impact strength (I.S.).).

$$(I.S) = F.E(KJ)/A(m^2) \tag{1}$$

Where: I.S = impact force (KJ/m2), F.E = The energy of fracture (KJ), A = the area (m2

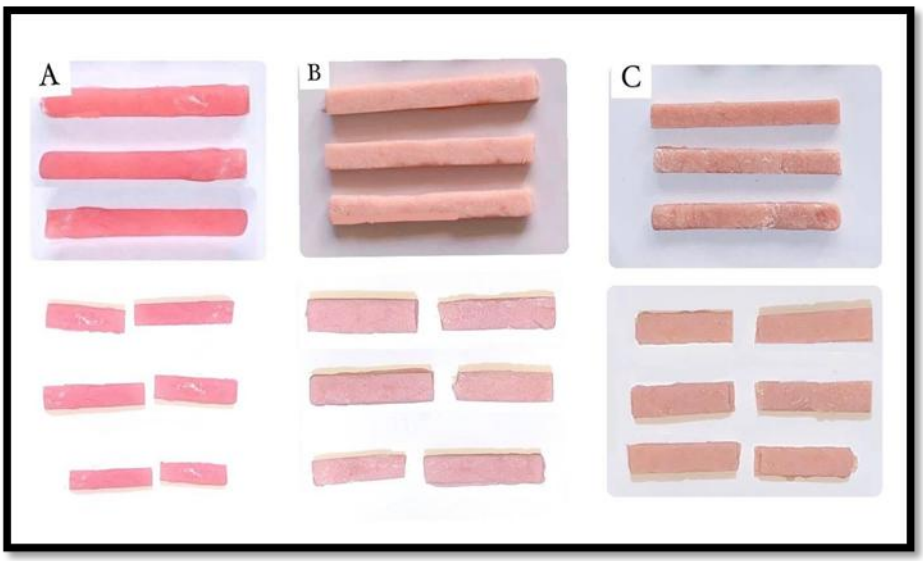

Figure 2. Sample before and after the impact test

Bending Test

The bending modulus It was tested. conducted in compliance with the international typical (Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials. , 2016). There was a three-point bending test conducted on sample and the figure 3 showing the shape sample used. The pointed head in the middle of the sample was progressively fastened to both ends of the ports until failure was reached in order to determine the material's maximum resistance. Equations.2and 3 were used to calculate the bending coefficient.

$$\boldsymbol{E} = \frac{MGL3}{48IS} \tag{2}$$

$$I = bd^3/12 \tag{3}$$

where M/S is the slope of the curve derived from the relationship between each sample's mass (M) and deflection (S), g is constant (⁄), and L is the separation in millimeters between the instrument's two backs. I, the

geometrical moment of inertia, can be found using the following formula. The breadth and thickness of the sample are denoted by b and d, in turn. The result of (I) is measured in (4mm), but the units of (E) are megapascals (MPa) (Adnan, 2023).

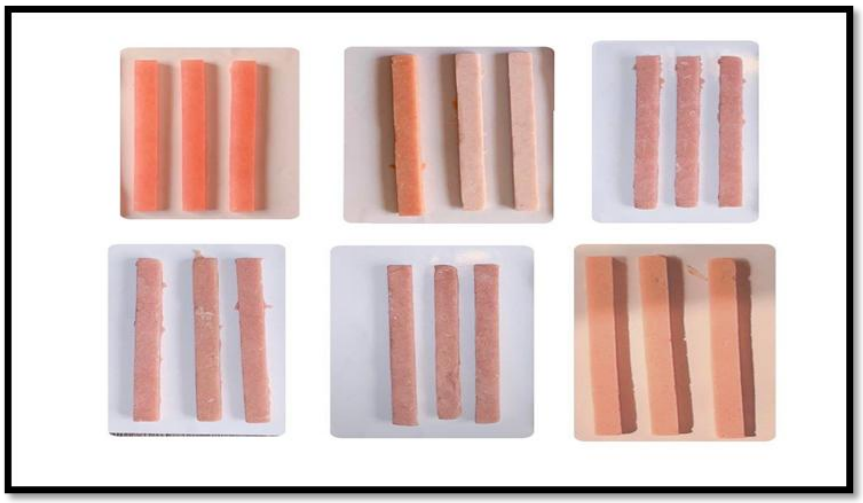

Figure 3. Bending test samples

Tensile Test

One important test is the tensile test. It measures the extent of a sample's development before breaking and the substance's resistance to deformation stresses (Monem, 2021). The Instron model (1195) tensile test apparatus was employed in this investigation. All of the samples as in the figure 4 were prepared in accordance with ASTM-D638, a standard specification (Materials, 2016). All PMMA composite samples' cross sections are frequently rectangular.

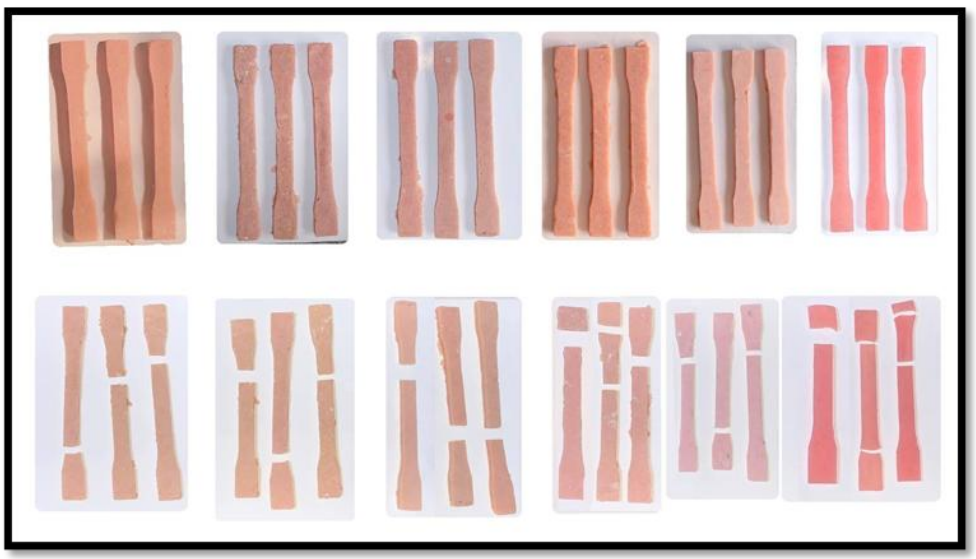

Figure 4. Shape the sample before and after the tensile test

SEM Test

The morphological characteristics of the selected samples surface were evaluated using FEI's INSPECT S50 scanning electron microscope. This test was beneficial. since it explained the diffusion of the filler inside the polymer matrix and the quality of the bonding by examining the sample surface as well as the internal structure of the composite.

Results and Discussion

Mechanical Testes

Impact Test

Impact Tests were carried out to evaluate the materials conduct under a fast force; this test is one of the most significant dynamic mechanical tests in material science. The results illustrated advance behavior to fast fracture with PMMA ternary composites compared to control one. The first of two primary elements influencing the force of impact of composite the materials are the particles' capacity of absorb vitality and stop cracks from spreading. Second, small gaps between the particles and matrix caused by inadequate interfacial bonding make it easier for cracks to spread. Salih (2018), as indicated in figure 5 a decrease in determined value from (38kJ/m2 to 24kJ/m2) Whereas the S1 value of pure PMMA is 38 kJ/m2 and the S2 value is 24 kJ/m2 after adding 5wt.% PEEKs. This could be due to Increased macromolecular chain immobilization results from the addition and while PMMA considered as brittle this property may increase further when mixed with PEEKs. So, during this test, the prepared polymer blend absorbs less energy through fractures and deformation. with less energy (Mohanty, 2010; Kumar, 2015). In addition, for coarse MFP /PEEK/PMMA as in S3 the results were higher than S4 for the fine MFP /PEEK/PMMA. In S4 was about (54kJ/m2), while in S3 was approximately (40kJ/m2). This could be due to the weak interconnection between coarse particles with PMMA. In S3, tiny spaces are formed between the coarse MFP and matrix, which facilitates the spread of cracks (j., 2014). Also, the difference in value are related to the strong cross-links that are developed between fine particles and the PMMA. Which permits energy transfer from the matrix to the particles by stopping fractures from spreading throughout (Sharhan, 2022; Rafiq, 2012). Additionally, it observed the strength value increase when the reinforcement ratio is increased (8%) in both cases. For S5 the determined value was 56kJ/m2, whereas S6 value was 56.1kJ/m^2.

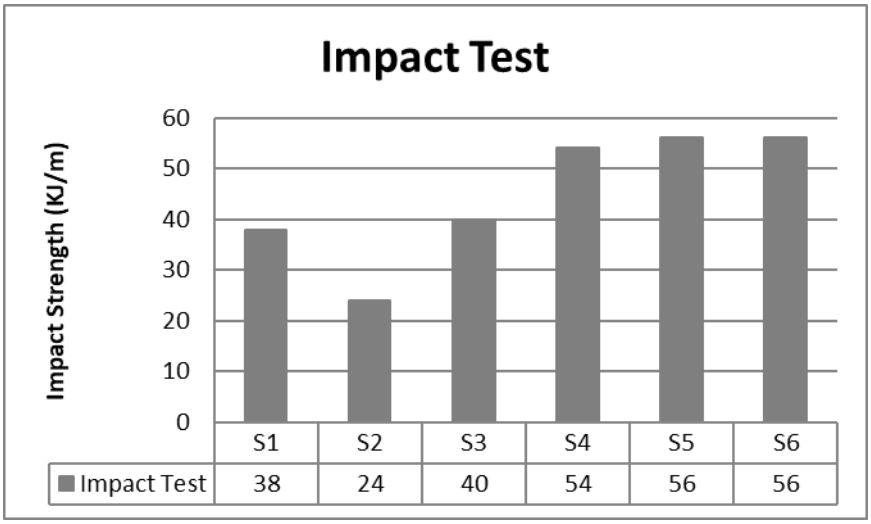

Figure 5. Impact strength value Vs PMMA/PEEK/MFP at different ratio

Bending Test

Figure 6 illustrates the determined bending values of all PMMA ternary composites at different ratios. The increase in value from (1987.8 to 2451.1 kg/mm^2) was clear from S1 to S2, the increase in this case when reinforced with PEEKs (5%). This behavior. may be explained by the reality that the mechanical features to PEEK the primary fillers effects. PEEK particles may absorb the imparted forces following the bending test, supporting one another at the matrix deformation site. This is predicated on the filler and polymer matrix having adequate bonding; if not, force would cause the filler and matrix to glide over one another (Schwitalla, 2015). While there was a decrease in values after the addition of MFP both types, after adding fine particles as in S4 and S6 at rates of 5wt.% and 8wt.%, respectively, most likely due to the aggregation effect. for S4 (1835.29 kg/mm^2) and S6 (1464.14kg/mm^2) were as in Figure (6). However, the decrease was greater in the coarse particles as S3 and S5, S3 (1420.6 kg/mm^2) and S5 (1089.5kg/mm^2) respectively. Notably both types coarse and fine PMMA/PEEK/MFP sample showed a drop in bending value as the reinforcement ratio increased. This is

elucidated by the reality that load transfer from A matrix to particles is lessened when the particle-matrix link is weakened due to aggregation (j., 2014).

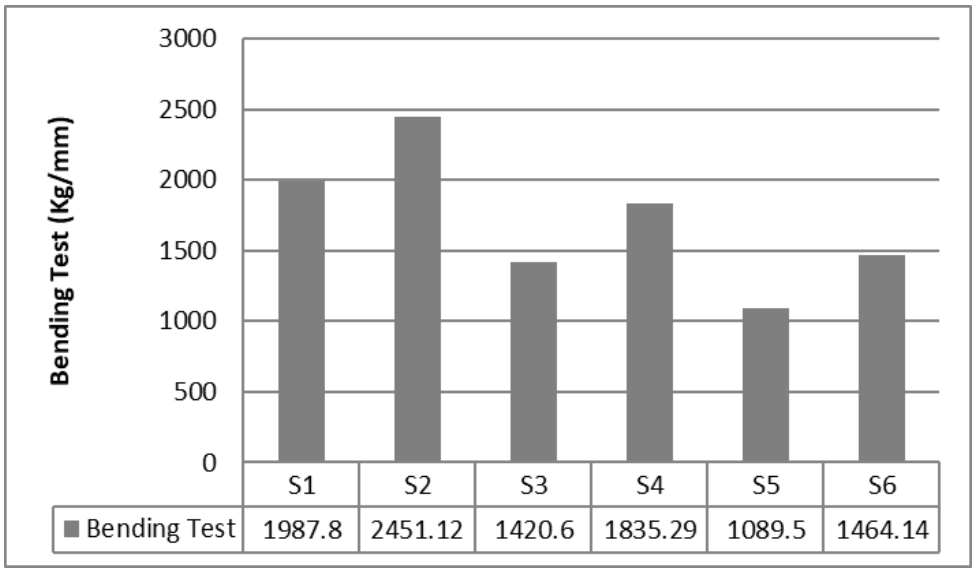

Figure 6. The bending value Vs PMMA/PEEK/MFP at different ratio

Tensile Test

Tensile were undertaken to analyze the behavior of materials under the influence of tensile strength of PMMA as control case and compare with others ternary composites samples, the figure 7 shows the result. TS value of S1 was (71 MPa), when reinforced with 5 % PEEKs, the value determined (83 MPa), S2 has known contained (95wt.%PMMA and wt.5%PEEK). So, due to TS value for PEEK (90-100 MPa), the increase in TS is related to presence of PEEK (Sampaio, 2015; Friedrich, 2011). Moreover, after the addition of coarse and fine particles as in S3 and S4 at 5wt.%, and compared with the control one, Between S3 and S1, there was a minor drop (68.5 MPa) the TS value was close to the control value. However, for S4 the decrease was bigger reaching (65.5 MPa). This variation could be the result of loosely bound cluster formations caused by the agglomeration of the fine particles, which could lead to fracture propagation and reduce the TS (Gad, 2016). S5 and S6 for more coarse and fine particles at selected ratios of 8wt.%, a clear decrease in TS was found. Which attributed to the low adhesion and poor bonding at the interface between the two phases that considered the reason of the fall in tensile strength values as the reinforcing phase increases (Nur Diyana, 2022) .

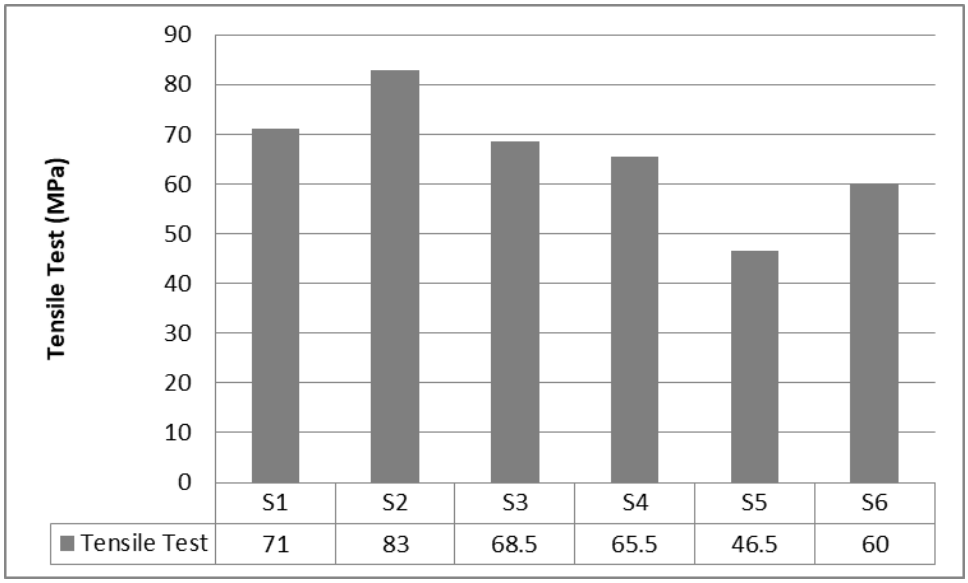

Figure 7. The tensile values PMMA/PEEK/MFP at different ratio

SEM Analysis

Surface morphology of all prepared PMMA ternary composites was illustrated in figures 8 a, b, c, d, e. which represents PMMA/PEEK, PMMA/PEEK/MFP 5wt.% coarse, PMMA/PEEK/MFP 5wt.% fine, PMMA/PEEK/MFP 8wt.%, coarse and PMMA/PEEK/MFP 8wt.% fine respectively, using SEM microscopic techniques. As it is clear from figures 8a for S2, image clearly shows the synthesized polymer blend of PMMA/ PEEK polymers, which was monodispersed in nature with a very limited degree of agglomeration. Figures 8b &c illustrated S3 and S4 (MFP at 5wt.% for both types) from the figures that functionalized natural particles were effectively and uniformly dispersed into the PMMA/PEEK which exhibited the absence of particle agglomeration. Figures. (8d &e) illustrated S5 and S6 (MFP at 8wt.% for both types) for this case the natural particles were monodispersed with a limited degree of aggregation in PMMA/PEEK. The increased addition of MFP closed the gaps between PMMA/PEEK and adhered them to one another, avoiding phase separation and promoting force transmission. from matrix to added fillers. So, a greater quantity of network structures supposedly created.

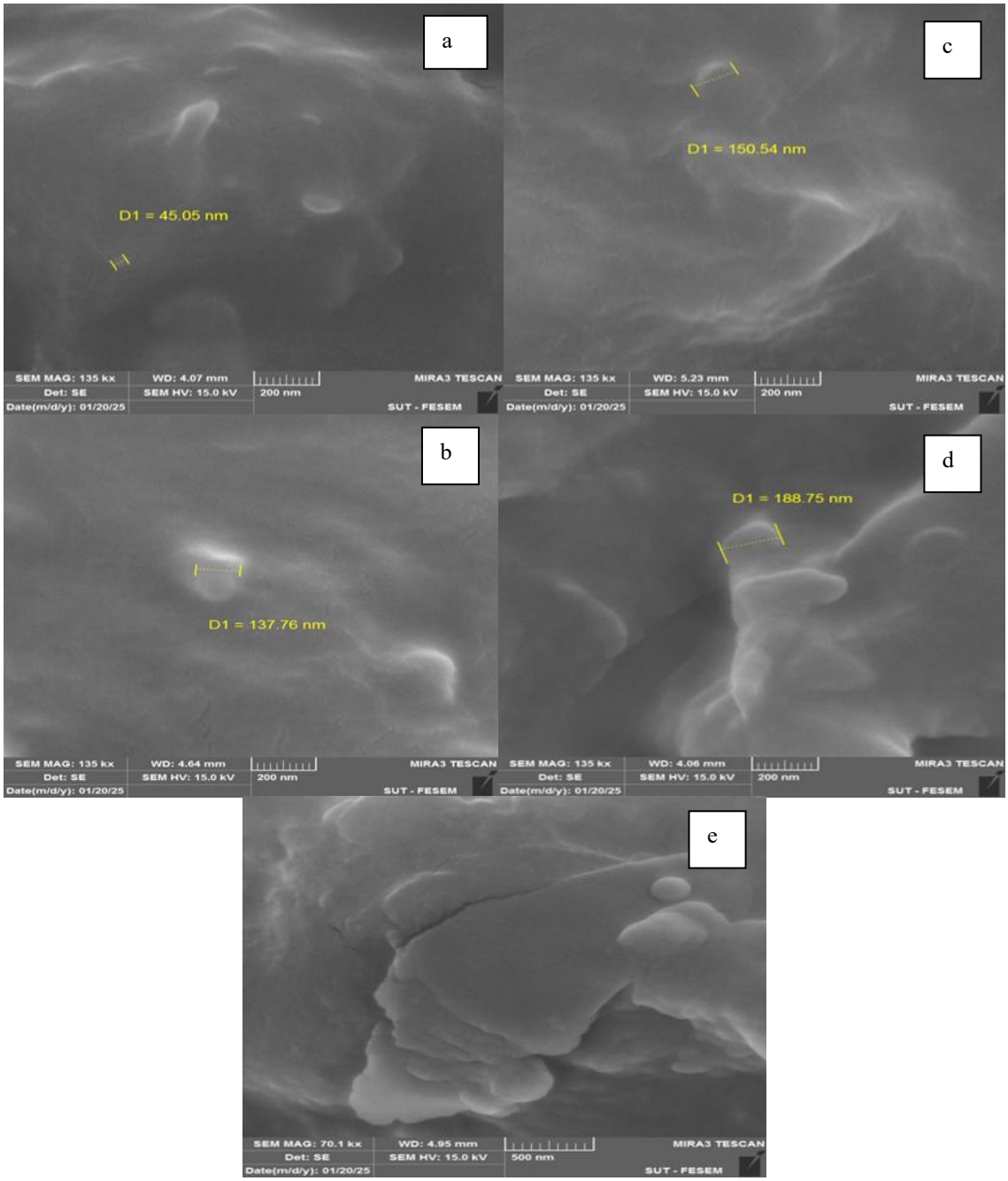

Figure 8. SEM Morphology: A) PMMA/PEEKs/, B) PMMA/PEEK/MFP coarse at 5%, C) PMMA/PEEK/MFP fine at 5%, D) PMMA/PEEK/MFP coarse at 8%, and E) PMMA/PEEK/MFP fine at 8%

Conclusion

In this current study, PMMA/PEEK successfully fabricated which reinforced with varying amounts of MFP to prepare PMMA ternary composites with advanced characteristics. The following results are inferred from the experiments. For impact tests compared to the control PMMA, samples S5 and S6 had the highest value (5wt, 8wt.% fine) with IS about (56KJ/m^2, 56.1 kJ/m^2) respectively, with (47% and 47%) higher over control PMMA. As for bending test, the maximum value obtained with S2 was PMMA/PEEK about (2451.12 kg/mm^2) which presents (23.31%) over the control PMMA, while the lowest value was obtained from S5 with coarse MFP, about (1089.5kg/mm^2). The best tensile strength value was obtained with S2 PMMA/PEEK about (83 Mpa) which presents (17%) over the control PMMA. For other prepared samples a decrease trend clearly found with the lowest value obtained for S5 when reinforcing with 8wt.% coarse MFP about 46MPa. SEM morphology characterization for both coarse and fine PMMA/PEEK/MFP samples, which display effectively and uniformly dispersed of both types of MFP into the PMMA/PEEK and exhibited the absence of particle agglomeration.

Recommendations

Based on the experience gained from this study, the following topics were proposed: evaluating more prepared mechanical properties such as tensile and bending behavior for both PMMA/PEEKs and PMMA/PEEK/MFP composites, compared to the results obtained from the pure polymer with a new composite.

Scientific Ethics Declaration

* The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM journal belongs to the authors.

Conflict of Interest

* The authors declare that they have no conflicts of interest

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