

## Fabrication of polymethyl methacrylate composite films with silanized SiC nanoparticles

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### ABSTRACT

The properties of polymeric composites have often been altered with the incorporation of fillers. In this study, the poly(methyl methacrylate) (PMMA) was filled with silicon carbide nano-particles (SiC). The PMMA/SiC composite films were prepared through solution casting by using acetone as solvent. The different loading of SiC ranging from 0.25 wt% to 1.00 wt% were incorporated into the PMMA matrix. The effect of SiC loading and silane coupling agent on PMMA/SiC composite films in terms of mechanical, physical, and morphological properties was investigated. It was found that the increasing SiC loading and the silane treatment had increased the tensile strength and Young's modulus but reduced the elongation at the break of PMMA/SiC composite films. At 0.75 wt% of silanized SiC, the tensile strength of the composite films was found to increase by 25 % from 30 to 37.5 MPa as compared to the virgin PMMA. Besides, the hardness of composite films was also increased with SiC loading and silane treatment. The presence of 1.00 wt% silanized SiC had increased 21.7% the hardness of the virgin PMMA, resulting in the increase of Shore A value from 69 to 84. By using silane treatment, better filler-matrix interaction was established as smoother fracture surfaces were observed through SEM micrographs and higher d-spacing was found in X-ray diffraction (XRD) patterns. The PMMA/SiC composite films prepared were suitable to be used in sporting goods, additive manufacturing, and environmental and protective coatings.

**Keywords:** *Polymer composite, Poly (methyl methacrylate), Silicon carbide, Solution casting, Thin films*

### 1. INTRODUCTION

Polymer composites have been extensively employed and developed in recent years for numerous engineering applications, particularly in the aerospace and automotive industries due to their superior characteristics and benefits [1]. Poly(methyl methacrylate) often known as PMMA, is a type of thermoplastic material that belongs to the methacrylate esters family. PMMA is excellent for making displays and optical components, but its combination of hardness and brittleness prevents it from being used in applications where impact resistance is crucial. Past studies have demonstrated that adding filler to PMMA enhanced its characteristics [2].

Silicon carbide (SiC) is a broad bandgap semiconductor material with a few exceptional qualities, including good thermal conductivity, a high breakdown field, a high saturation drift velocity, and exceptional resistance to corrosion and oxidation [3]. The particles of SiC have been widely used as filler in ceramic composites, leading to superior mechanical properties. SiC nanoparticles also have applications in electron emitters, light emitters, and more as they are high in strength, elastic modulus, and thermal-chemical stability [4]. Here, SiC nanoparticles were chosen to be introduced into the PMMA matrix to improve

mechanical performance, such as increased strength and stiffness. Before that, authors had reported the use of SiC nanowhiskers in PMMA composite films [5], the results showed that SiC nanowhiskers were capable of reinforcing the PMMA composite films.

The incorporation of nano-sized fillers into a polymeric matrix may not achieve the optimum desired properties as a result of the incompatibility between fillers and matrix. Moreover, the nanoparticles with high surface energy are prone to form agglomeration when they are dispersed in a polymeric matrix. Therefore, chemical treatments such as silanization or chemical etching are commonly used to improve the interfacial bonding of matrix and fillers and improve the compatibility between the two materials. Silane was used as a coupling agent to treat the SiC nanoparticles prior to their addition into PMMA. Given the lack of prior research on PMMA composites filled with SiC nanoparticles, undertaking an in-depth investigation becomes essential for this study. The study aimed to determine the effect of various SiC nanoparticle weight percentages as well as the influence of silane coupling agents on the mechanical, morphological, and physical properties of the poly (methyl methacrylate) composite films filled with silanized SiC nanoparticles.

## 2. MATERIALS AND METHODOLOGY

The PMMA pellets with a specific gravity of 1.18 g/cc used in this study were supplied by LG Chemical Ltd. (Seoul, South Korea). The MFI value was 2.50 g/10 min at 230 °C. Next, the SiC nanoparticles were purchased from Nanostructured and Amorphous Material Inc. (Texas, USA). Acetone was used as a solvent for PMMA. Both acetone and acetic acid were obtained from HmbG Chemicals (Hamburg, Germany). Silane coupling agent KH570 were provided by Acros Organics (Geel, Belgium). All the chemicals were used as received.

### 2.1. Chemical Modification of SiC Nanoparticles

Anhydrous ethanol was mixed with silane coupling agent KH570 (1 wt% from the weight of SiC) by a volume ratio of 1:10 and subjected to three hours of magnetic stirring for the hydrolysis process [6]. Prior to stirring, the pH of the silane solution was adjusted to 4-5 by adding acetic acid. It helps to catalyse the rate of both hydrolysis and condensation [7]. Next, the SiC nanoparticles were poured into the silane solution and the stirring process was continued for three hours. The SiC nanoparticles were rinsed with ethanol and deionized water a few times and then centrifuged to remove the unreacted silane molecules. Finally, the modified SiC nanoparticles were dried in an oven for 12 hours at 80 °C, to further promote the condensation of the silanol group on the SiC surface. The treated SiC nanoparticles were ready to be used for the fabrication of PMMA composite films.

### 2.2. Preparation of PMMA/SiC Composite Thin Films

The conventional melt mixing process may hinder the dispersion of SiC nanoparticles in PMMA, and further compromise the mechanical properties of the composite. Therefore, a solution casting technique with a nontoxic solvent, acetone, was used to prepare virgin PMMA and PMMA/SiC composite thin films. For the preparation of virgin PMMA film, 5 g of PMMA pellets was added into 30 ml of acetone. The mixture was subjected to a magnetic stirring process for three hours. After that, the solution was cast into a petri dish and covered with perforated aluminium foil. Then the solution was left for 24 hours drying in an open area.

The preparation of PMMA/SiC composite films started with the preparation of the PMMA solution. Similarly, 5 g of PMMA was dissolved in 30 ml acetone by using the ratio of 1 g: 6 ml and stirred for three hours. After the PMMA pellets were completely dissolved in acetone, the SiC was added slowly and the mixture was continuously stirred for 1 hour until homogeneous. In order to disperse the SiC evenly, the solution was then ultrasonically sonicated for an hour in a water bath at 50 °C. The solution was then cast into a petri dish and covered with perforated aluminium foil. Then it was left to dry for 24 hours. These steps were repeated with different weight percentages of SiC (0.25 wt%, 0.50 wt%, 0.75 wt%, 1.00 wt%). The PMMA/SiC composite films were then cut according to dimensions of 25 mm x 100 mm for tensile testing.

### 2.3. Tensile Test

The tensile test was conducted according to ASTM D882 at a cross-head speed of 1 mm/min by using a Universal Testing Machine (UTM) Instron 5569. For each formulation, 10 identical samples with a size of 25 mm x 100 mm were tested, and average values were taken. The tensile strength, elongation at break, and elastic modulus of the samples were measured.

**Table 1.** Formulation of untreated and treated PMMA/SiC composite films

Formulation	PMMA (wt%)	Untreated SiC (wt%)	Treated SiC (wt%)
PMMA/SiC_0.25	99.75	0.25	-
PMMA/SiC_0.50	99.50	0.50	-
PMMA/SiC_0.75	99.25	0.75	-
PMMA/SiC_1.00	99.00	1.00	-
PMMA/SiC_0.25S	99.75	-	0.25
PMMA/SiC_0.50S	99.50	-	0.50
PMMA/SiC_0.75S	99.25	-	0.75
PMMA/SiC_1.00S	99.00	-	1.00

### 2.4. Morphological Study

The morphological analysis of PMMA/SiC composite films was investigated by the means of Scanning Electron Microscope (SEM) model JEOL JSM-6010LV. By using scanning electron microscopy, a visual examination of the surface can help determine the cause of defects and interactions between different compositions of PMMA/SiC composite films. All the fracture surfaces composite films were sputter coated with a thin layer of platinum to prevent charging during testing.

### 2.5. Hardness Test

Shore A Durometer test was used to determine the hardness of PMMA/SiC composite films. The test was carried out in accordance with ASTM D2240 A at a thickness of a minimum of 6 mm. For more accurate results, multiple measurements were performed at 3 different locations on the thin film sample and the average hardness value was calculated.

### 2.6. X-ray Diffraction (XRD)

Bruker D2 Phaser was used to analyse the crystalline states under normal atmospheric conditions. To conduct this test, an X-ray beam is pointed at a sample, and the intensity of the scattered radiation is measured with respect to the direction of the beam's output. After the beam has been separated, the scatter, known as a diffraction pattern, reveals the crystalline structure of the PMMA/SiC composite films. The scan range (2 $\theta$ ) for each sample was set ranging from 10° to 80° and the PDF card number used was cubic SiC (ICDD 01-073-1708).

### 3. RESULTS AND DISCUSSION

#### 3.1. Tensile Properties of PMMA/SiC composite films

Figure 1 demonstrates the tensile strength of PMMA/SiC composites at different loading of SiC. It was found that the tensile strength increased with the presence of SiC nanoparticles in the PMMA matrix. Compared to virgin PMMA, the tensile strength for untreated PMMA/SiC composites increased gradually with SiC loading up to 0.75 wt%. The PMMA/SiC<sub>0.75</sub> exhibited 25% higher tensile strength than virgin PMMA. This improvement was primarily contributed by the presence of SiC nanoparticles in the PMMA matrix. Due to their high strength and hardness, SiC nanoparticles provided additional structural integrity and load-bearing capacity to the composite. The SiC nanoparticles distribute and share the applied load, reducing stress concentration and enhancing the overall tensile strength of the PMMA/SiC composites. Since the SiC nanoparticles were untreated, the nanoparticles with high surface energy tend to form agglomeration. It may cause void formation and stress concentration that may compromise the mechanical properties of the composites. Therefore, the tensile strength for PMMA/SiC<sub>1.00</sub> was found to decrease slightly. On another hand, silanized SiC nanoparticles effectively form stronger adhesion with PMMA, ensuring that debonding at the interface is prevented [7]. Proper bonding between the SiC filler and the polymer matrix is crucial for efficient load transfer. As a result, the tensile strength of the treated PMMA/SiC composite films was higher than those of untreated counterparts.

Figure 2 shows the effect of SiC loading on the Young's modulus of PMMA/SiC composite films. The results indicated a gradual increase in both untreated SiC and treated SiC compositions compared to virgin PMMA. When SiC nanoparticles are incorporated into the PMMA matrix, they act as reinforcement, effectively increasing the overall stiffness of the composite films. The stiff SiC nanoparticles transferred stress more efficiently, resulting in enhanced stiffness of the composite material. Trivedi et. al. [8] also observed that the elastic modulus of graphene-filled polypropylene (PP) composites increased with increasing filler loading. The addition of filler increased the stiffness of

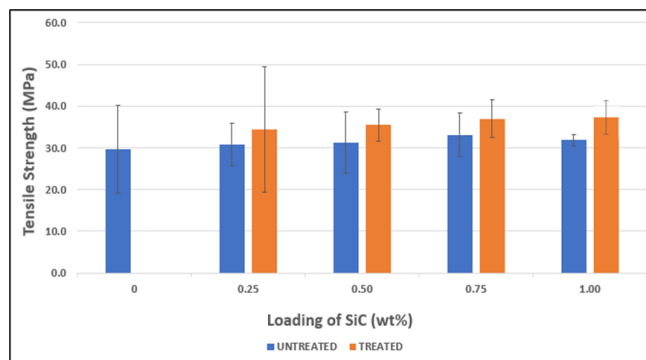


Figure 1. Tensile strength of PMMA/SiC composite films

the composites which in turn decreased the elongation at break. As for the silane-treated SiC composites, Young's modulus also increased steadily when the SiC filler content increased. The improved bonding leads to better stress transfer from the matrix to the filler, leading to a stiffer composite with a higher Young's modulus. The study of Rahman et. al. [9] also claimed that the benzene-treated abaca-PP composites are found to show higher elastic modulus compared to the untreated composites.

Figure 3 illustrates the elongation at the break of PMMA/SiC composite films as a function of SiC loading. For virgin PMMA, the sample recorded average elongation at break at 8.6% before fracture. For untreated PMMA/SiC composite films, the data showed a decreasing pattern when the SiC filler loading increased. The reduction in elongation at break can be attributed to the SiC nanoparticles effect. When incorporated into the PMMA/SiC composite, these SiC nanoparticles can restrict the movement of the polymer chains and hinder their ability to undergo plastic deformation. Ramli et. al. [10] also reported a decrease in elongation at break with the presence of filler in eggshell powder-filled recycled high-density polyethylene/ethylene vinyl acetate composites. The rigid fillers reduced the mobility of the polymer chains. Besides, the results showed that the elongation at break for treated PMMA/SiC composites decreased gradually with higher SiC nanoparticle loading as compared to untreated counterparts. Surface treatment of SiC nanoparticles enhanced the interfacial bonding and dispersion within the PMMA matrix, leading to a stiffer composite.

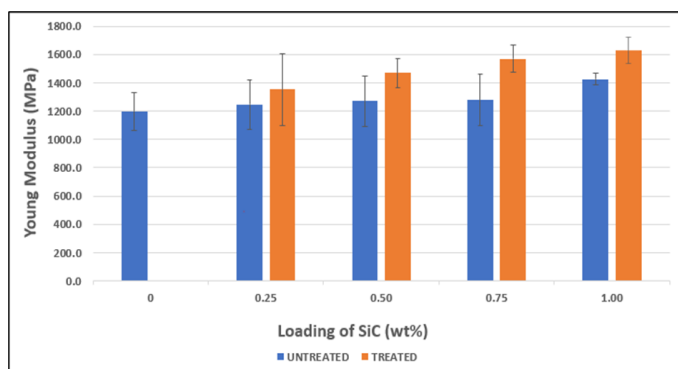


Figure 2. Young's Modulus of PMMA/SiC composite films

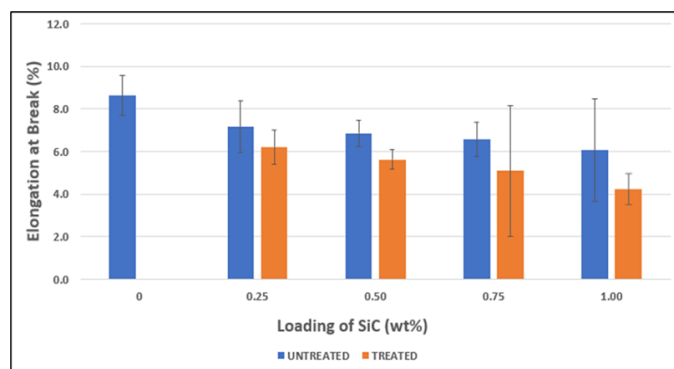
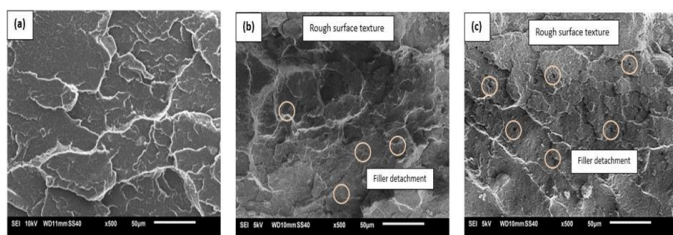


Figure 3. Elongation at break PMMA/SiC composite films



**Figure 4.** SEM micrograph of the tensile fracture surface of (a) virgin PMMA, (b) PMMA/SiC<sub>0.50</sub> and (c) PMMA/SiC<sub>1.00</sub>

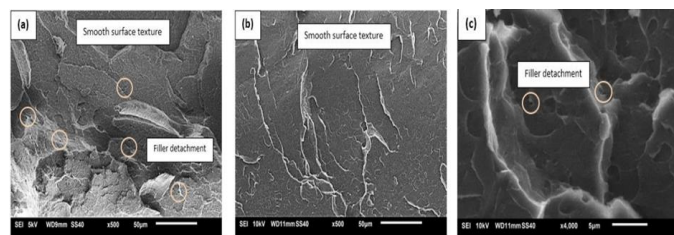
### 3.2. Morphological Study of PMMA/SiC Composite Films

SEM micrograph of tensile fracture surface for virgin PMMA thin film is shown in Figure 4 (a). It can be seen that virgin PMMA thin film exhibited brittle properties because virgin PMMA thin films are often deposited as solid layers with a relatively low molecular weight. Due to their rigid and amorphous nature, they are lacking in flexibility, making them prone to cracking and fracturing under mechanical stress. The presence of sharp edges resulted from the propagation of cracks through the material, which does not undergo extensive plastic deformation.

In Figure 4 (b) and (c), the rough fracture surfaces were observed. This can be attributed to the amount of filler loading that had been incorporated in the PMMA matrix. The fracture surface of PMMA/SiC<sub>1.00</sub> was rougher than that of PMMA/SiC<sub>0.50</sub>. At higher filler loadings, there was a higher probability of filler particles clustering or agglomerating within the PMMA matrix. These agglomerates can create uneven distribution and protrusions on the surface of the composite, leading to increased roughness. The tensile strength of PMMA/SiC<sub>1.00</sub> deteriorated due to the detachment of SiC as shown in Figure 4 (c). Jajam et. al. [11] reported the effect of particle size scale and filler loading can be clearly seen on the surface roughness of silica nanocomposites. The fracture surface of silica nanocomposites showed very rough and highly textured surfaces.

Besides, plenty of filler detachments also can be observed in PMMA/SiC composite films for untreated SiC in Figure 4 (b) and (c). Untreated SiC nanoparticles were more susceptible to this phenomenon due to the weaker bonding and interface strength with the PMMA matrix. There were more filler detachments in PMMA/SiC<sub>1.00</sub> compared to PMMA/SiC<sub>0.50</sub>. Similar results were also shown by the research of Xu and Mellor [12] where a brittle filler was found to have a higher wear rate compared to unfilled polymer because the brittle fillers were fractured and detached from the matrix easily.

Figure 5 (a) and (b) shows the SEM micrograph of the tensile fracture surface of silane-treated PMMA/SiC composite films. As compared to the SEM micrograph between Figure 4 and Figure 5, smoother fracture surfaces can be observed in Figure 5. This was due to the silanized SiC nanoparticles promoting better adhesion or bonding with the PMMA matrix. This improved bonding led to a stronger interface between the filler and matrix, reducing

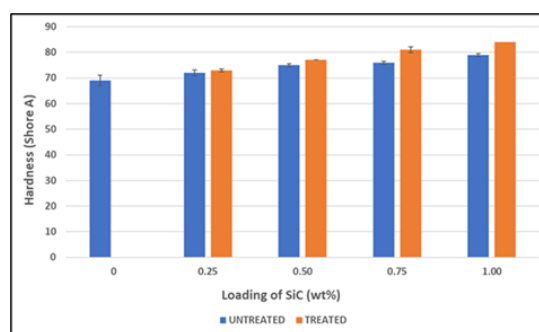


**Figure 5.** SEM micrograph of the tensile fracture surface of (a) PMMA/SiC<sub>0.50s</sub> at 500X, (b) PMMA/SiC<sub>1.00</sub> at 500X and (c) PMMA/SiC<sub>1.00</sub> at 4000X magnification

the likelihood of interfacial debonding during fracture. As a result, the fractured surface appeared smoother. In addition, the better interfacial bonding between the SiC and PMMA contributed to the enhanced mechanical properties as discussed in Section 3.1. The study of Sahraeian et. al. [13] discovered that the nanoparticle that was treated with a silane coupling agent, resulting a smoother fracture surface in polypropylene/nanoperlite nanocomposite films. Moreover, lesser filler detachments were found in PMMA/SiC<sub>1.00S</sub> at the magnification of 4000X as shown in Figure 5 (c). The good dispersion of the filler reducing stress concentrations and minimizing the occurrences of filler detachment [14].

### 3.3. Hardness Properties of PMMA/SiC Composite Films

Figure 6 shows the results of Shore A hardness testing and demonstrated that the hardness of the PMMA/SiC composite films increased as the SiC nanoparticles increased from 0.25 wt% to 1.00 wt%. With an increased filler content, more SiC nanoparticles can be found in the PMMA matrix. The SiC particles were able to share the applied load more effectively. Similar results were also obtained by Kumar and Arun [15] where the hardness of PMMA nanocomposites increased with the incorporation of nano clay and compatibilizer. All the nanocomposites exhibit higher hardness than virgin PMMA. The effect of the coupling agent on the hardness of PMMA/SiC composite films can also be seen in Figure 6. The hardness values for treated PMMA/SiC composite films were higher than untreated counterparts. The hardness value of PMMA/SiC<sub>1.00S</sub> was 21.7 % higher than that of the virgin PMMA. Surface treatment enhanced the dispersion and



**Figure 6.** Shore A Durometer hardness testing of PMMA/SiC composite films



distribution of SiC nanoparticles within the PMMA matrix. Improved dispersion helps to prevent agglomeration and ensures a more uniform distribution of fillers, leading to a more homogeneous composite structure and increased hardness. In addition, the better interfacial adhesion of SiC and PMMA can prevent local deformation and lead to higher Shore A readings. Alhotan et. al. [16] also observed that the hardness of the PMMA nanocomposites linearly increased with the increasing concentrations of  $\gamma$ -MPS silane-treated titanium dioxide (TiO<sub>2</sub>) nano-fillers.

### 3.4. X-ray Diffraction of PMMA/SiC Composite Films

Figure 7 shows the XRD pattern of virgin PMMA and its untreated SiC nanoparticle-filled composites. The diffraction peaks located at 34.19°, 46.97° and 54.54° was corresponding to the cubic SiC (ICDD 01-073-1708) in the (h k l) plane of (111), (200) and (220) orientation, respectively. The high intensity of SiC fillers reveals the high crystallinity of SiC in nature. A similar result was observed by Tony et. al. [17] where XRD peaks corresponding to  $\beta$ -SiC were observed at (111), (200), and (220) planes associated with 2 $\theta$  values of 36.5°, 41.2°, and 60.5°.

The d-spacing was calculated according to Bragg's Law for peak with high intensity which is 33.91° for PMMA/SiC\_1.00 and 33.83° for PMMA/SiC\_1.00S as shown in Figure 8. A smaller d-spacing suggests a closer packing of lattice planes, which can be an indication of increased particle agglomeration. Conversely, a larger d-spacing suggests a more spaced-out arrangement of lattice planes, indicating better particle dispersion in the matrix. By comparing the d-spacing values between the untreated and the treated PMMA/SiC composites, there was a small difference of 0.08 nm. This indicated that the treatment applied to the SiC nanoparticles may have a subtle influence on the dispersion of SiC nanoparticles. The improved dispersion of SiC nanoparticles within the PMMA matrix can lead to enhanced interfacial bonding, which can subsequently improve the tensile strength of the composite material [18].

## 4. CONCLUSION

The preparation of poly(methyl methacrylate) composite films filled with silicon carbide nanoparticles was conducted. The tensile strength and Young's Modulus of PMMA/SiC composite films increased with increasing SiC weight percent (wt%). However, more than 0.75 wt% of untreated SiC had decreased the tensile strength of the composites due to the formation of SiC agglomeration. The silane treatment improved the interfacial bonding of SiC and PMMA, thereby enhancing the mechanical properties of the composites as compared to untreated counterparts. The findings were evidenced by the smoother fracture surface and reduced filler detachment shown in SEM micrographs. The hardness of PMMA/SiC composite films was increased with SiC loading and with the application of silane treatment. On the contrary, the elongation at the break of the composite films decreased gradually with the addition of higher SiC loading. In sum, this study proposes a novel method of preparing PMMA/SiC composites using solution

casting. This study highlights the importance of applying silane treatment to enhance the potential of using SiC nanoparticles to improve the mechanical properties of PMMA composites while addressing the challenges of nanoparticle dispersion.

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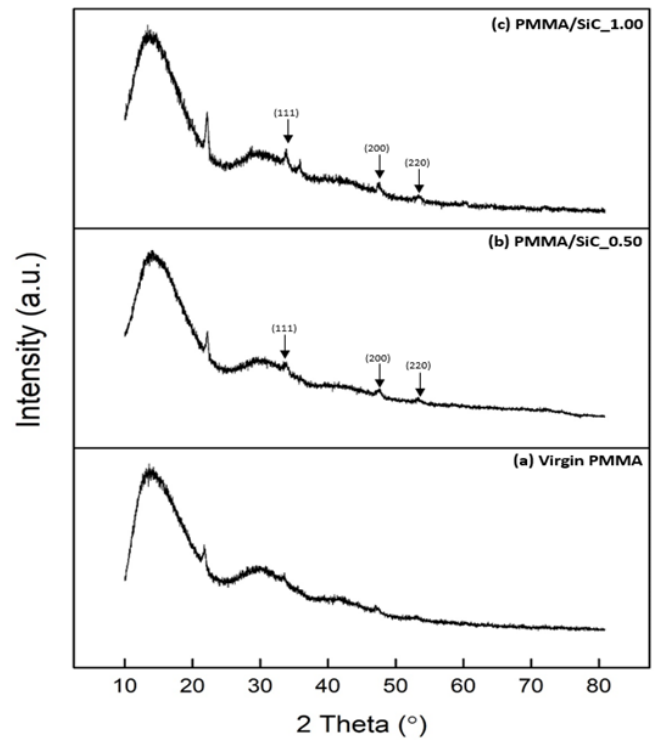


Figure 7. XRD pattern of SiC from PMMA/SiC composite films

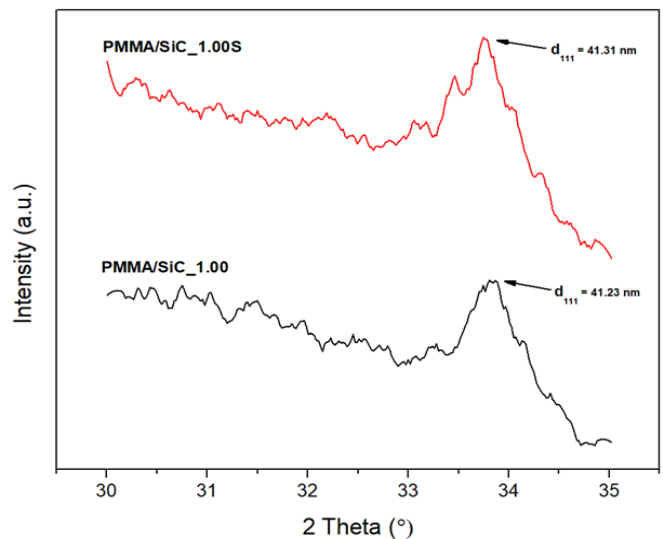


Figure 8. D-spacing between 1.00 wt% untreated and treated PMMA/SiC composite

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