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Preliminary Study: Characterization of UHMWPE and PP Polymer for Extrudability of PP/UHMWPE Composite to FFF Process

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Abstract

Ultra-high molecular weight polyethylene, or UHMWPE, is a thermoplastic semicrystalline polymer with outstanding wear resistance, low friction coefficient, and mechanical qualities. It also can exhibits powerful resistance to corrosive substances. Even Ultra-high molecular weight polyethylene (UHMWPE) has unique characteristics, but it features an extremely low melt flow rate (MFR) of near zero, which renders it unsuitable for processing using traditional polymer processes. Thus, PP is one of the most potential plasticizers for increasing the extrudability of UHMWPE-based composites. PP, commonly known as Polypropylene, is a thermoplastic polymer that is widely used in many industry sector. It is made from the monomer propylene using chain-growth polymerization. However, there is a problem where the chemical incompatibility of the PP-UHMWPE composites makes the composite is difficult to manufacture. As a result, the intent of this research is to examine the characteristics of both materials in order to acquire a better knowledge of the requirements and to present a deeper insight into these two materials in order to increase the extrudability of PP-UHMWPE composite for Fused Filament Fabrication (FFF). The characterization procedure comprises the morphological and flow properties, thermal behavior and stability, and crystallinity of UHMWPE and PP in powder and granular form, respectively. The research illustrates the feature for both polymer behavior that is influenced by particle shape. This paper's findings were utilized to manufacture the extrudability of PP-UHMWPE composites using FFF as a bone repair implant.

1. Introduction

Ultra-high molecular weight polyethylene (UHMWPE) possesses distinctive properties such as non-toxicity, simple chemical composition and structure, low water absorption, as well as chemical and radiation resistance [1-

2]. Due to its high molecular weight, UHMWPE has an extremely low melt flow rate (MFR) of about zero, which makes it unsuitable for processing by standard methods for polymers [3-6]. Recently, in order to increase the viscosity of the UHMWPE, several materials have been added up with plasticizing components such as polypropylene (PP) [7], polyethylene glycol (PEG) [8], high pressure polyethylene (HDPE), alpha-tocopherol (vitamin E), hydroxyapatite (HA) [9-10].

However, recently study show that the plasticizers partially destroy the structure themselves. It is also decreased the mechanical and tribological characteristics of two- or three-component UHMWPE-based composites. PP, commonly known as Polypropylene, is a thermoplastic polymer that is widely used in many industry sector [11-13]. It is made from the monomer propylene using chain-growth polymerization [14-15]. High PP heat resistance enables to asepticism the products at temperatures up to 130℃. Furthermore, PP is characterized by a high MFR of about 0.5-2.0 g/10 min [16-18]. For these reasons, PP is one of the most promising plasticizers in terms of improving extrudability of the UHMWPE-based composites [19-20]. Since, by using PP-UHMWPE composites allows for the simultaneous resolution of two problems: increasing the wear resistance of polymer and producing complexly shaped products that can be manufactured using Fused Filament Fabrication via 3D printing techniques [21-22].

However, hand mixing (manual shaking) proved incapable to integrate the distinct polymer forms—powder and granular—in a homogenous manner [23-26]. As result, homogeneous blends were formed utilizing melt blending in line with the weight percentages specified [27]. To accomplish the mixing process, the properties of both polymers must be analysed and examined. Hence, the objective of this study is to investigate the properties of both materials in order to gain a better understanding of the requirements and to present a deeper understanding of these two materials in order to enhance the probability of successful blending and extrudability of PP-UHMWPE composite for Fused Filament Fabrication (FFF).

2. Methodology

A comprehensive approach to investigate the fundamental properties of raw polypropylene (PP) and ultra-high-molecular-weight polyethylene (UHMWPE). This section details the materials used and the methods employed for analysis. The study seeks to provide detailed insights into the particle size, morphology, and structural characteristics of these polymers using a variety of analytical techniques.

2.1 Material

The PP employed in this study satisfies the standards medical grade standard, and is available in granular form. Emory provided the content. Meanwhile, the UHMWPE employed in this study comprised Medical Grade GUR1020 for surgical implants. Emory provided the substance in powdered form [9], [28]. Both material is distinguished by their particular specs, as indicated in Table 1.

Table 1 *Specification of specified polymer material*

Specification	Polypropylene (PP)	UHMWPE
Density (g/cm^3)	0.9	0.93
Molecular Weight (g/mol)	620,000	5,000,000
Yield Tensile Strength (Mpa)	27.5	21
Flexural Modulus (Mpa)	1320	-
Notched Izod Impact (kJ/m^2)	-	210

2.2 Method

The methodology employed in this study encompasses a multi-faceted approach, incorporating several analytical techniques to comprehensively characterize Polypropylene (PP) and Ultra-High Molecular Weight Polyethylene (UHMWPE) polymers.

a) Particle Size and Morphology

A scanning electron microscope (SEM) is an advanced imaging instrument widely utilized in various scientific fields to examine specimen surfaces with exceptional resolution. By focusing a beam of electrons onto the sample, SEMs capture emitted signals such as secondary electrons, backscattered electrons, and characteristic X-rays [29]. This process generates detailed images that reveal the surface morphology and composition of the sample. SEMs are invaluable in materials science, biology, geology, and nanotechnology, allowing researchers to investigate materials at micro and nanoscale levels. [30].

Surface morphology of the samples was examined utilizing COXEM EM-30AX scanning electron microscopy (SEM). Prior to SEM analysis, the material underwent a 24-hour drying process in the oven. Small sample quantities were mounted onto carbon tapes attached to upright stubs, with gold pre-coating applied to mitigate excessive charge reflection. Observations were carried out under vacuum conditions, employing a magnification of 1.0k and an accelerating voltage of 20.0 kV. Both materials were magnified from 200x to 2.0k magnification, focusing on particle size, shape, and potential contamination.

b) Fourier transform infrared (FTIR)

FTIR spectroscopy, utilizing Fourier transform technology, is a well-established analytical method utilized for studying polymeric materials. Over the past two decades, it has been extensively employed to scrutinize microscopic regions within polymers, facilitated by the integration of an infrared interferometer with a microscope outfitted with specialized detectors. With the advent of instrumentation utilizing focal plane array (FPA) detectors, the scope of FTIR spectroscopy applications has expanded further [31-33].

FTIR micro spectroscopy has recently undergone a renaissance, enhancing its instrumental capabilities and visualization techniques for studying multicomponent polymer systems. The functional groups of both materials were identified using FTIR (Perkin Elmer Spectrum One) with a universal diamond attenuated total reflection (ATR), recorded across a wavenumber range of 400 cm^{-1} to 4000 cm^{-1}. Small amounts of powdered UHMWPE and small granules of PP were positioned in the testing area. The experiment aimed to gather information about the polymers, including bonding, chemical class, and molecular structure.

c) X-Ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) analysis of the polymer provides details regarding crystallinity, size, and orientation of crystallite and phase composition in semi-crystalline polymers [34].

A fully crystalline material has a series of sharp peaks in its XRD pattern, each of which represents a reflection from a different crystallographic plane. An XRD pattern with a large peak that represents the average distance between and along the polymer chains can be seen in a totally amorphous material. As a result, the crystalline reflections superposed over an amorphous halo constitute the XRD pattern of a semi-crystalline polymer. An index of crystallinity can be obtained from the ratio of the crystalline peaks integrated intensity to the total area under the XRD curve [34-35].

The X-ray diffraction (XRD) analysis was performed using a Bruker D2 PHASER 2nd generation instrument. Both material samples were mounted on the stage and scanned from 10° to 70° with a step size of 0.05° and a time per step of 10 seconds. The diffraction angle range for XRD analysis was $2\theta = 10\text{-}80°$, using Cu Kα radiation ($\lambda = 1.5406$ Å) with a step size of 0.01°. The collected data was processed using Origin software, and the crystalline phases in the materials were identified by matching the significant XRD peaks with standard PDF files using Eva Diffrac Plus (Version 4). Since the XRD experiment requires samples in powder form, the granular PP was crushed into powder. Both materials then underwent a 24-hour drying process in the oven. Subsequently, a small amount of powder from each material was mounted on a provided plate before being inserted into the machine. The data was processed to identify the group class by analysing the peaks in the obtained graph and to assess the level of contamination in the materials.

d) Degree of Crystallinity

Many chemical and mechanical characteristics of polymers (such as Young modulus, yield stress, strength, fatigue, and shrinkage) are known to be impacted by crystallinity. Thermal examination utilizing, volumetric analysis, infrared, Raman spectra, and X-ray diffraction may all be used to measure the degree of crystallinity in polymers. Each of these approaches yields different findings since the testing measure various physical characteristics and morphological features [36].

XRD techniques were applied in this investigation to measure the degree of crystallinity of both UHMWPE and PP based on their own form approach. One method that is frequently used to assess the level of crystallinity in polymers is X-ray diffraction. Sharp peaks in the diffraction profile indicate the diffraction of crystallites, while wide peaks indicate the scattering of the amorphous phase. The intensity of the scattering may be measured and used to quantify the degree of crystallinity based on the utilizing Equation (1) [37]:

$$X_C(\%) = \frac{I_C}{I_C + I_A} \times 100 \tag{1}$$

where IC and IA are the scattered intensities for the crystalline and the amorphous phases, respectively The degree of crystallinity was calculated using Equation (2) [37-38]. The theory posits that the areas under the peaks are proportional to the scattering intensities of both the crystalline and amorphous phases.

$$X_C(\%) = \frac{A_C}{A_C + A_A} \times 100 \tag{2}$$

where AC represents the area of crystalline phase (i.e., A110 +A200) and AA is the area of amorphous phase.

3. Result and Discussion

The analysis of polypropylene (PP) and ultra-high-molecular-weight polyethylene (UHMWPE) offers valuable insights into their fundamental properties. By integrating data from these techniques, the understanding of the properties of PP and UHMWPE, facilitating their effective use in combining both material. The results and discussion presented here elucidate the distinctive differences and similarities between these two polymers, highlighting their respective advantages and limitations.

3.1 Particle Size and Morphology

UHMWPE particles range approximately 10 μm in size and display elongated shapes with high aspect ratios, contributing to exceptional toughness and wear resistance. In contrast, PP particles vary from micrometer to sub-micrometer sizes, featuring a finer grain structure due to shorter polymer chains, which renders them flexible and suitable for diverse applications. This result shows that the average particle size of PP is larger than UHMWPE, suggesting that PP will serve as the primary material and UHMWPE as the filler to enhance the PP-UHMWPE composite.

The coarser nature of UHMWPE aligns with its robust mechanical properties, while the smaller size of PP enhances surface finish and facilitates processing. These findings underscore the importance of particle size in determining material characteristics and suitability for specific applications, offering insights into optimizing material selection and processing techniques for desired outcomes. According to SEM analysis, the UHMWPE powder particles have a highly agglomerated structure of smaller particles and are non-spherical in form, as seen in Figure 1. UHMWPE particle microstructure is highly complex and from the SEM seems porous [3]. At 200x magnification as per Figure 1(a), the powder particles exhibit a generally uniform shape, with some variation in size and morphology apparent.

However, finer details such as the presence of fibrils are not clearly visible at this level. As the magnification increases to 500x based Figure 1(b), the individual particle shapes become more defined, and the existence of fibrils, which are elongated and thread-like structures within the UHMWPE matrix, starts to become discernible. These fibrils may appear as thin strands or fibres protruding from the surface of the powder particles.

Based on Figure 1(c) at 1000x magnification, the morphology of the particles becomes more pronounced, with distinct boundaries and surface features becoming evident. The presence of fibrils becomes more apparent, with their intricate network becoming visible, particularly on the surfaces and edges of the powder particles. Finally, at 2000x magnification according to Figure 1(d), the nanoscale features of the fibrils and their arrangement within the UHMWPE matrix become clearly observable. Detailed examination reveals the presence of individual fibrils and their alignment, providing valuable insights into the material's structure and properties. Therefore, the surface structure reduces the ability of the material to pack into regular structures.

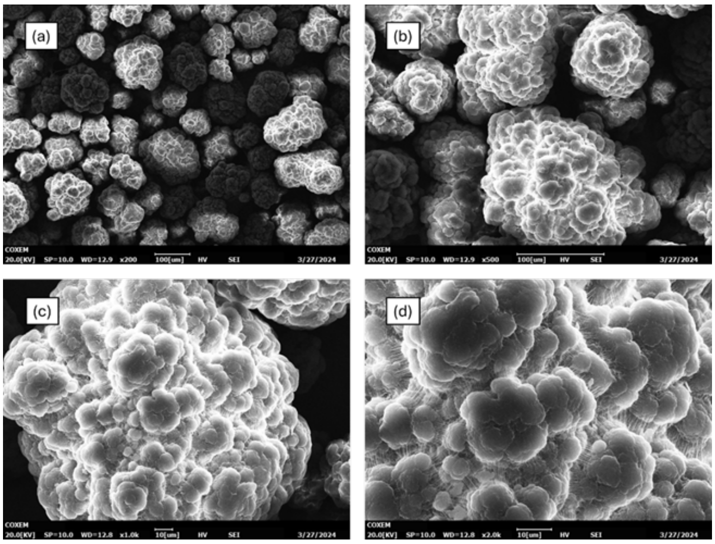


Fig. 1 *Scanning electron microscopy (SEM) images of virgin UHMWPE powder. The image shows the irregular structure and non-spherical shape. Magnifications: (a) 200×; (b) 500×; (c) 1000× and (d) 2000×*

Under SEM image obtained in the Figure 2 below, the surface morphology of the PP granules appears smooth and featureless at low magnification 500x as Figure 1(a). However, upon increasing the magnification to 1000x according Figure 1(b), fine details become visible. The surface of the pellets exhibits a moderate level of roughness with sporadic shallow depressions and small protuberances. These surface irregularities are likely attributed to the manufacturing process and the cooling history of the PP. At even higher magnifications 2000x based on Figure 1(c), the microstructure of the polypropylene becomes discernible. Crystalline regions appear as distinct domains with a granular texture, interspersed within the amorphous matrix. The crystalline domains exhibit varying sizes and shapes, indicating non-uniform nucleation and growth during polymer solidification.

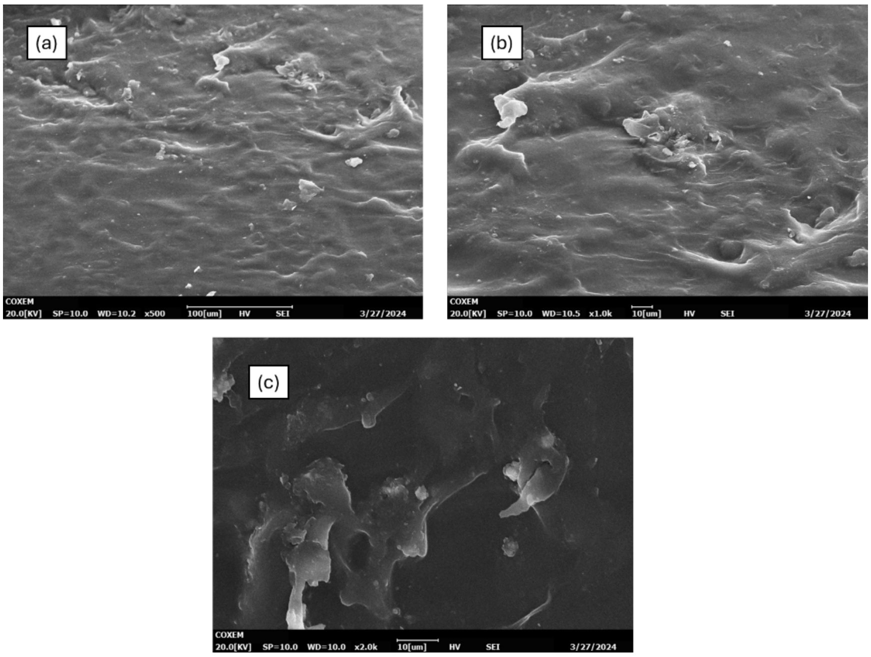


Fig. 2 *Scanning electron microscopy (SEM) images of virgin PP granular form. The image shows smooth and featureless. Magnifications: (a) 500×; (b) 1000×; and (c) 2000×*

3.2 Fourier Transform Infrared (FTIR)

Based on Figure 3, The FTIR analysis on UHMWPE revealed distinct spectral features indicative of its chemical composition and molecular structure. Strong absorption bands observed at 2917 cm^-1 and 2844 cm^-1 correspond to the asymmetric and symmetric stretching vibrations of methylene (-CH2-) groups, respectively, suggesting the presence of aliphatic chains in the polymer backbone.

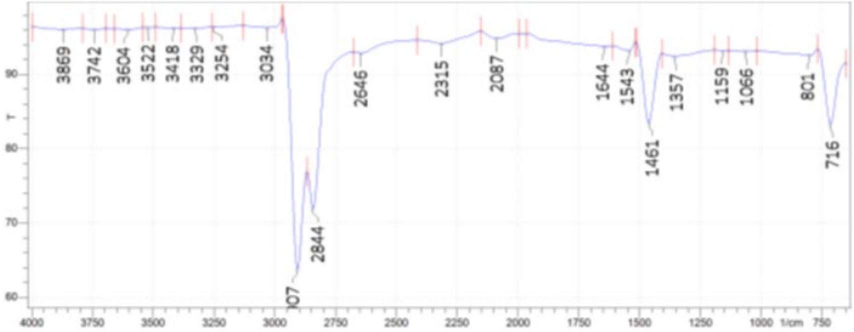


Fig. 3 *FTIR spectra of raw ultra-high molecular polyethylene*

Additionally, sharp peaks at 1461 cm^-1 and 1357 cm^-1 signify the bending vibrations of methyl (-CH3) groups, further confirming the presence of alkyl groups within the polymer structure. Weak absorption bands detected in the region of 716 cm^-1 correspond to the wagging vibrations of carbon-hydrogen (C-H) bonds in the polymer chains, indicating the presence of saturated hydrocarbon moieties. Moreover, the absence of characteristic peaks in the carbonyl (C=O) stretching region (2140-2085 cm^-1) and the hydroxyl (OH) stretching region (3600-3200 cm^-1) suggests minimal presence or absence of carbonyl or hydroxyl functional groups in this sample. Overall, the FTIR analysis confirms that this material is predominantly composed of hydrocarbon molecules with aliphatic chains and minimal functionalization.

As seen in Figure 4, the FTIR analysis of raw PP unveiled distinct absorption bands indicative of its molecular structure. There is a few corresponding chemical classification related to the peak of the analysis. Firstly, in the aliphatic CH stretching region (2800-3000 cm^{-1}), prominent peaks signalled the presence of aliphatic methyl (-CH_3) and methylene (-CH_2-) groups within the polypropylene chain. Specifically, a robust absorption at approximately 2970 cm^{-1} was attributed to symmetric stretching of CH_2 groups, while another peak around 2845 cm^{-1} corresponded to asymmetric stretching of CH_3 groups [39].

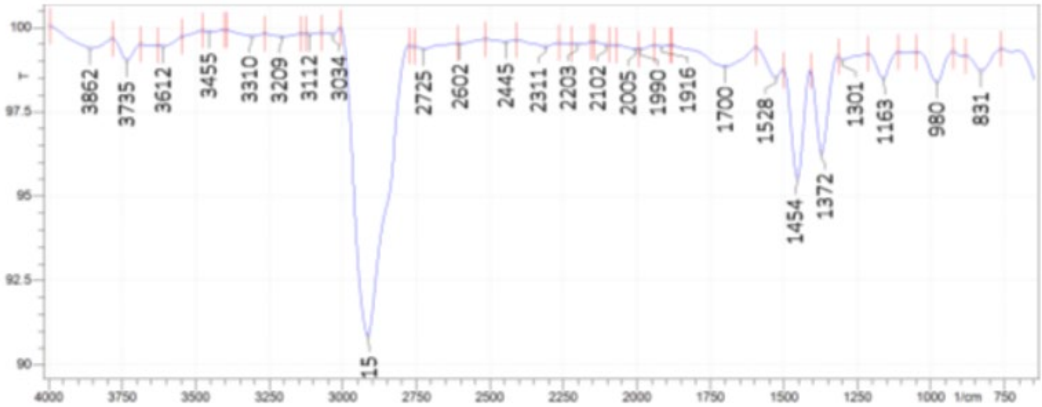


Fig. 4 *FTIR Spectra of raw polypropylene*

Moving to the C-H bending region (1460-1375 cm^{-1}), observable peaks were associated with the bending vibrations of methyl (-CH_3) and methylene (-CH_2-) groups. Notably, the peak at approximately 1454 cm^{-1} was linked to symmetric bending of CH_3 groups, while the peak near 1372 cm^{-1} was ascribed to asymmetric bending of CH_3 groups. These findings offer valuable insights into the chemical composition and molecular arrangement of raw polypropylene, crucial for comprehending its properties and optimizing its myriad applications. Table 2 show the corresponding chemical classification and the priority of the vibration bonding.

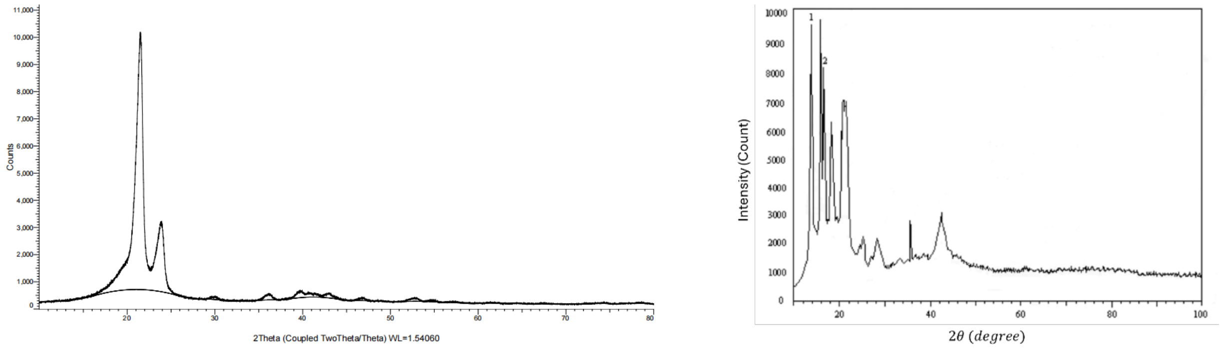
Table 2 *Corresponding chemical classification rules*

Vibration	Start WN	End WN	Threshold	Priority
C-H Stretch	2970	2845	Strong	Mandatory
C-H Bend, CH2/CH3	1480	1450	Variable	Mandatory
C-H Bend, CH3	1390	1365	Variable	Mandatory
No Band Region	3700	3050	Medium	Excluded
No Band Region	2000	1650	Medium	Excluded
No Band Region	1280	1000	Medium	Excluded
No Band Region	1000	650	Very Strong	Excluded

3.3 X-Ray Diffraction Analysis (XRD)

Figure 5(a) displays the UHMWPE powder's XRD spectrum. According to the results, UHMWPE is a semi-crystalline polymer made up of two phases: an amorphous phase and the typical orthorhombic crystalline phase of polyethylene, which has two distinct, sharp peaks at 22.5◦ (110) and 24◦ (200). Meanwhile, Figure 5(b) showed the granular form of PP's XRD spectrum.

According the finding obtained, PP is also a semi-crystalline polymer made up of two phases: an amorphous phase and the typical orthorhombic crystalline phase of polypropylene that typically reveals a series of sharp diffraction peaks corresponding to its crystalline structure, indicative of the presence of ordered regions within the polymer chain [40-41]. These peaks, which appear at specific angles, approximately 14°, 17° reflect to the orthorhombic crystal structure characteristic of polypropylene. The intensity and sharpness of these peaks suggest the presence of well-defined crystalline regions within the polymer chain. The relative intensities of the peaks provide insights into the degree of crystallinity of the sample.



(a) **(b)**

Fig. 5 *X-ray diffraction pattern of: (a) UMWPE powder; (b) PP in granular form*

3.4 Degree of Crystallinity

Using XRD, the degree of crystallinity of UHMWPE powder and granule PP was assessed; the results are displayed in Table 3.

Table 3 *Degree of crystallinity (Xc) of UHMWPE powder and granule PP*

Material	Crystallinity (%)
UHMWPE Powder	63.63 ± 0.50
PP Granular	71.43 ± 0.50

According to Dong's studies in 2021, UHMWPE exhibits an XC value within a relatively broad range of 29% ≤ X_C ≤ 65% [42]. The analysis of X-ray diffraction (XRD) data reveals important information about the crystalline structure of the sample. Peaks observed at 22.5° and 24° correspond to the (110) and (200) crystallographic planes, respectively, indicating the presence of ordered arrangements within the material. Integration ranges of ±0.5° around each peak position are defined to capture the entirety of the peak signals accurately.

Through integration, the intensity values within these ranges are summed, and the average background or amorphous contribution is subtracted to account for non-crystalline components. The resulting adjusted intensities are used to calculate the total area under the crystalline peaks (Ac) while considering the background or amorphous contribution to determine the total background or amorphous area (A_A)Using these values, the calculation is shown in the equation below:

$$X_C(\%) = \frac{700}{700 + 400} \times 100 \tag{3}$$

$$X_C(\%) = 63.63\% \tag{4}$$

The degree of crystallinity X_C is calculated, revealing a value of 63.63%. This indicates that approximately 63.63% of the sample's composition consists of ordered, crystalline regions, underscoring the material's structural integrity and providing valuable insights for further characterization and application considerations.

The degree of crystallinity X_C of Polypropylene (PP) granules, calculated based on X-ray diffraction (XRD) peaks at 14° and 17°, considering the background or amorphous contribution, is approximately 71.43%. This value indicates the proportion of crystalline regions within the PP granules relative to the total crystalline and amorphous phases present in the material. The integration of intensity values within specific ranges around the peaks at 14° and 17° allows for the determination of the total area under the crystalline peaks, while accounting for the background or amorphous contribution ensures the accuracy of the calculation. The calculation is made using the equation below:

$$X_C(\%) = \frac{1000}{1000 + 400} \times 100 \tag{5}$$

$$X_C(\%) = 71.43\% \tag{6}$$

More crystallinity implies a greater organization of polymer chains into crystalline structures, which typically results in enhanced mechanical properties such as stiffness and strength. However, it's essential to consider that the degree of crystallinity can vary depending on factors such as processing conditions, molecular weight, and additives present in both material forms, highlighting the importance of precise characterization techniques like XRD for understanding the material's properties and performance.

4. Conclusion

The UHMWPE powder's particles have a non-spherical shape and a highly agglomerated structure made up of smaller particles. The powders' microstructure also contains fibrils. However, for PP's particles have a smooth surface and a highly agglomerated structure made up of bigger particles. These conclude that the PP will be the main material and the UHMWPE will be the filler to completion the PP-UHMWPE composite since the capability of the powder to free flowing through the bigger particle of PP. Many chemical and mechanical characteristics of polymers, such as young modulus, yield stress, fatigue, and shrinkage, are known to be impacted by crystallinity. Based on the testing, X-ray diffraction spectroscopy was utilized to validate the degree of crystallinity for PP and UHMWPE both in their own form. PP and UHMWPE measurements revealed that the degree of crystallinity was 71.43 ± 0.50 and 63.63 ± 0.50, respectively. The results demonstrate that the crystallinity value obtained by PP is greater than UHMWPE since the form of both material is different. In summary, the study's goal was effectively accomplished because of the in-depth analysis and investigation of the two materials as well as the outcome, which demonstrates that PP-UHMWPE can go through the mixing process to create PP-UHMWPE composite, which can then be extruded and used in the FFF process for 3D filament printing. The study can be further by find out the characteristics of the composite after the mixing process occur.

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Conflict of Interest

Authors declare that there is no conflict of interests regarding the publication of the paper.

Author Contribution

Fakrul Nur Aiman: ***conceptualization, methodology, writing****. Kamarudin. K:* ***supervision, review****. Shufeng Sun:* ***collaboration, technical input****. M.F. Shaari, Mohd Nazrul Roslan, A. Ahmad, Norrizal Mustaffa, Mohd Khairuddin M. S.:* ***data collection, analysis****.*

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