



## THE EFFECT OF COLEMANITE ADDITION ON THE MICROSTRUCTURAL AND MECHANICAL CHARACTERISTICS OF IPP

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### ABSTRACT

The objective of this study was to investigate the effect of the addition of the colemanite having 45 $\mu$ m size on the significant characteristic features of the isotactic polypropylene (IPP). The microstructural properties (diffraction pattern,  $a, b$  and  $c$  unit cell parameters and grain size) and mechanical behaviors (tensile strength, Young's Modulus, impact strength and percent elongation of the samples relative to the colemanite content (5, 10, 15, 20 and 30 wt.%) were studied in details. The optimum amount of colemanite was determined in order to obtain IPP based composites having improved properties. The obtained samples were characterized by using XRD and conventional mechanical tests. The results showed that the content level of the colemanite considerably affected the fundamental properties of IPP. As for microstructural properties, it was observed from the XRD patterns that all composite samples mainly showed only  $\alpha$  form (monoclinic arrangement) in the crystalline domains without any  $\beta$  form (hexagonal arrangements). Moreover, the finding revealed that  $a$  and  $b$  the unit cell parameters of IPP based composites increased initially, reached the maximum values with the products containing 10% of colemanite, and then the consistent decrement trend was observed with the further increase in the colemanite content in the products. Conversely,  $c$  unit cell parameter almost remained relatively unchanged. Furthermore, the mechanical test measurements depicted that the reinforcements were achieved in the tensile, Modulus and impact strengths of the composite materials, while the percent elongation of the products decreased with the increasing of the colemanite content. 7.4%, 24.9% and 6.7% increases were recorded in the tensile strength, Modulus and impact strength at the product with 10% colemanite, respectively. The improvements were probably stemmed from that the presence of micro size colemanite particles gave rise to increment in the orientations and alignments of IPP chain in the matrix.

**Keywords:** Colemanite, Unit cell parameters, Mechanical properties, IPP based composites, Percent elongation

### 1. INTRODUCTION

The polymer composites (PCs) can be defined as the materials composed of the polymer (matrix) and the additive (filler) bonded together via the covalent bonds or the weak intermolecular forces [1]. Many fundamental characteristics features can be imparted into the polymeric materials by forming the polymer composites. Thus, they have been an indispensable part of many industrial applications such as advanced materials [2], automotive [3], medicine [4], engineering [5], optics [6], electrical products [7] and textile [8] etc. since they enables to the materials lot of advantages such as mechanical reinforcement, resistance to corrosive, advanced in the polymer processing conditions, thermal stability, microstructural improvements etc [9-12]. Especially, many researchers recently have been focused on the polymer composites containing the inorganic filler or natural fiber with their outstanding properties [13, 14]. Isotactic polypropylene (IPP), which is one of the significant member of the olefin family, have used widely in the world plastic industry with its excellent comprehensive properties such as low manufacturing cost, high heat resistance, low cost and high mechanical strength [15]. However, the usage of IPP is limited due to some undesired features like absence of reactive site, lower hydrophobic property, sticking challenge during processing, dye uptake and more vulnerable to photo-oxidation etc [16]. In order to deal with these drawbacks, many studies regarding IPP based composites were done so as to modify its mechanical [17], morphological [18], conductive [19], thermal [20] and crystallization properties [21].

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Boron minerals is one of today's natural compounds used in many different industrial areas such as filler, energy, textile, cleaning, agriculture, glass, ceramic and superconductor materials etc [22-26]. Therefore, there existed many studies in literature corresponding to the boron mineral which were utilized as a filler in the polymer composites in order to impart the advanced characteristics such as mechanical, flame retardancy, water absorption etc. to the polymers [27-30]. Furthermore, boron minerals make the scientists unable to fabricate halogen free polymer composite by means of their flame retardant characteristic feature [31, 32]. Among the boron minerals, colemanite,  $2\text{CaO}\cdot 3\text{B}_2\text{O}_3\cdot 5\text{H}_2\text{O}$  is the indispensable natural mineral due to the fact that not only it is cheap (\$0.215/kg) and eco-friendly but also it was used as filler in the thermoplastics thanks to the thermoplastic reinforcement, thermal stability, flame retardant and a radiation transmitter effects [32]. Isitman and Kaynak studied the improvement of the fire retardancy and mechanical properties of low-density polyethylene with decreasing the total filler presenting in the matrix with the use of aluminum hydroxide and colemanite minerals. They revealed that the limiting oxygen index was increased by 1%–4% depending on colemanite content due to its better protecting character of fire residues and being more effectual fuel trapping in the condensed phase. However, Higher ductility and lower elastic modulus were recorded in low density polyethylene due to decrement in the total filler loading in the matrix [33]. Besides, Atikler et. al. investigated the synergistic effect of colemanite in order to improve flame retardancy of polypropylene [34]. The hardness and thermal properties of the polypropylene (PP) composites containing varying amount (12.19, 22.6 and 31.76 wt.%) of colemanite mineral having 19  $\mu\text{m}$  particle size were investigated by Sahin [35]. The results showed that thermal expansion of PP decreased with the addition of colemanite to polymer matrix due to the fact that the colemanite particle had a lower expansion coefficient comparing to the polymer matrix. Moreover, the Shore D hardness of the IPP based composites increased with the colemanite content. In another foregone study, the colemanite and ulexite as the fillers were added individually to bisphenol A epoxy resins in order to observe the mechanical and thermal changes in the epoxy resin composites [27]. The results showed that maximum reinforcements of the epoxy resin were recorded with the 5% colemanite content, at further contents, the tensile strength started to decrease due to the exfoliation of colemanite. Furthermore, it was revealed that colemanite showed better synergetic effect on the thermal characteristics of virgin epoxy resin when compared to ulexite. Sen et. al. [36] studied the surface, mechanical and thermal properties of the thermoplastic polyurethane (TPU) by using colemanite mineral. They founded that the mechanical properties (especially tensile elongation and elongation at break) of the TPU composites were enhanced with the addition of colemanite. Moreover, the good thermal stability was observed in TPU composites with higher char yield formation as percentage of colemanite increased in the polymer matrix.

It was deduced that colemanite mineral was used as a filler in many thermoplastic matrix with the purpose of improving mechanical, thermal, flame retardant etc. However, only a few studies are focusing on microstructure and mechanical properties of isotactic polypropylene (IPP) with the additional of colemanite having certain particle size (45 $\mu\text{m}$ ). In this study we tried to analyze meticulously the variations of the microstructure and crucial mechanical characteristics features of IPP with 45  $\mu\text{m}$  colemanite loaded at five contents by the weight (5, 10, 15, 20, 30 wt.%) into the polymer matrix. The optimum colemanite content was determined by making serious discussions over the results by comparing the virgin IPP.

## **2. EXPERIMENTAL**

### **2.1. Materials**

Isotactic polypropylene (IPP) with MH418 code was purchased from the Turkish Petrochemical Industry (PETKIM) and its density was 0.905 g/cm<sup>3</sup>. The colemanite (di-calcium hexaborate pentahydrate) having 45  $\mu\text{m}$  particle size with the chemical formula of  $2\text{CaO}\cdot 3\text{B}_2\text{O}_3\cdot 5\text{H}_2\text{O}$  were supplied from the Eti Mining Operations General Directorate in Turkey as in 1 kg bag. The chemical

composition of used colemanite were  $B_2O_3$   $40.00\pm 0.50\%$ ,  $CaO$   $27.00\pm 1.00\%$ ,  $SiO_2$   $4.00\pm 6.50\%$ ,  $SO_4$   $0.60\%$  max., As 35 ppm max.,  $Fe_2O_3$   $0.08\%$  max.,  $Al_2O_3$   $0.40\%$  max.,  $MgO$   $3.00\%$  max., SrO  $1.50\%$  max., and  $Na_2O$   $0.50\%$  max. Moreover, L.O.I. (loss on ignition), moisture and bulk density values of the used colemanite were  $25.00\%$  max.,  $1.00\%$  max. and  $1.00\text{ ton/m}^3$  max., respectively.

## **2.2. Preparation of IPP Based Composites**

IPP in the granule form firstly transformed into the powder form in order to obtain good homogeneity. For this purpose, granule IPP was dissolved in the xylene (50 g of IPP in 500 ml of xylene) at the boiling temperature of xylene and, refluxed for a day for completely solubility. Then, the dissolved IPP was precipitating with the addition of 250 ml of methanol. After the filtration, the powder IPP was dried under the vacuum at  $60^\circ\text{C}$  for 6 hours. In order to obtain colemanite-IPP composites, the prepared IPP in the powder form and the colemanite with the varying content levels (5, 10, 15, 20 and 30 wt.%) were mixed in the co-rotating twin screw extruder by using DSM Xplore 15 mL microcompounder machine. During extrusion processes, the hopper, mixing zone and die temperature and also the screw speed of the extruder were 100 rpm and  $210^\circ\text{C}$ . These parameters were kept constant for the production of all colemanite contents. The extruded samples composed of IPP and 5, 10, 15, 20 and 30 wt.% of colemanite were molded at  $220^\circ\text{C}$  for barrel and  $50^\circ\text{C}$  for mould by using DACA Micro Injection Molder. The minimum three samples both in the dogbone shape for the tensile tests (thickness of 2.1 mm, width of 7.6 mm and gauge length of 50 mm) and the rectangular solid shapes for the impact tests (thickness of 1 mm, width of 7 mm) were prepared with injection pressure of 8.6 bar.

## **2.3. Instruments**

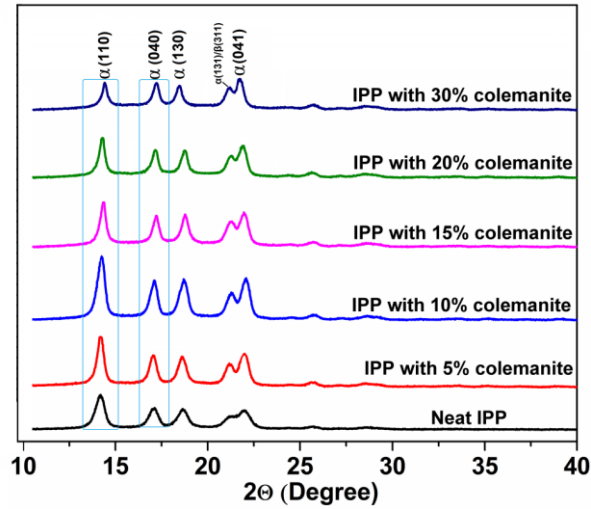
In order to investigate the microstructural properties of IPP based composites, XRD analyses of the molded samples were carried out with the help of Rigaku Multiflex powder X-ray diffractometer system with  $CuK\alpha$  target giving radiation ( $\lambda=1.54^\circ$ ) over the  $2\theta$  range of  $10-60^\circ$ . The XRD measurements were done at the room temperature via the scanning speed of  $5^\circ/\text{min}$  and step increment of  $0.02^\circ$ . The samples possessing the same weights were utilized in order to compare the intensities recorded from XRD patterns. Furthermore, the lattice constant parameters ( $a, b$  and  $c$ ) and grain sizes of the IPP domain were determined with the accuracy  $\pm 0.01\text{ \AA}$  by means of XRD patterns[37]. The tensile test of the samples in the dogbone shapes were conducted according to the standard of ASTM D-638 by using LLYOD LR30K Mechanical Tester. During analyses, the load cell and the crosshead speed were 5 kN and 5 cm/min, respectively. The stress-strain curves of the IPP based composite species were directly recorded by the instrument software. Moreover, the values of the tensile strengths, Young's Modulus and percent elongations of the samples were obtained from the software of the instrument. The Izod impact tests of the unnotched samples were conducted at room temperature according to ASTM standard of D256 with the use of Coesfeld Material Test Pendulum Impact Tester.

## **3. RESULTS AND DISCUSSION**

### **3.1. Microstructural Properties of IPP Based Composites**

The effect of different content level of colemanite addition on the microstructural characteristics of IPP based composites was investigated in details using XRD measurements carried out with the usage of the same amount of the products at room temperature. X-ray powder diffraction patterns belonging to neat IPP and IPP based composites were depicted in Figure 1. XRD measurements revealed that the only  $\alpha$  form (monoclinic arrangement) was observed in the crystal domains of neat IPP and IPP based composites without any detection of  $\beta$  form (hexagonal arrangements). The X-ray diffraction peaks

recorded at  $2\theta = 14.1^\circ, 16.8^\circ, 18.6^\circ, 21.1^\circ$  and  $21.8^\circ$  were attributed to  $\alpha(110)$ ,  $\alpha(040)$ ,  $\alpha(130)$ ,  $\alpha(131)/\beta(311)$ , and  $\alpha(041)$  reflections, as seen in Figure 1 [38, 39]. The obtained results showed that the relatively intensity of the products depended highly on the additional of the colemanite contents. Especially, when examining the diffraction peaks corresponding to  $\alpha(110)$  and  $\alpha(040)$ , it was seen that the relative intensity of the products increased with the initial addition levels of colemanite, but decreased with the further colemanite addition. It may be attributed that the presence of the higher amount of colemanite in the IPP matrix brought about the lattice distortion of the crystal structure of the IPP domains. Moreover, the broadening of the diffraction peaks and decrement in the sharpness of the peaks might be seen the experimental evidence for this disorder and defects formed in the crystal structure of the IPP matrices [40].



**Figure 1.** X-ray diffraction patterns belonging to neat IPP and IPP based composites containing 5, 10, 15, 20 ad 30% colemanite.

The unit cell parameters ( $a$ ,  $b$  and  $c$ ) regarding to the monoclinic crystalline arrangements of the neat IPP and the composites were calculated by means of XRD patterns. The calculations to determine were carried out depending on the basis the basis of a least square method with the usage of both  $d$  values and  $h, k, l$  planes (040, 110, 041 for  $a$ ,  $b$ ,  $c$  unit cell parameters, respectively.  $d$  values of the neat IPP and IPP based composites were obtained directly from the software of the instrument. In addition to that, the crystal sizes (grain sizes) of the neat IPP and IPP based composites were calculated using the following equations (1 and 2) based on the  $\alpha(110)$  main diffraction peak.

$$B^2 = B_m^2 - B_s^2 \quad (1)$$

where  $B_s$  and  $B_m$  designated the half width of the standard material and the difference between the angles at full width at half maximum (FWHM) of the corresponding peak, respectively.

$$d = 0.941\lambda / B \cos\theta_B \quad (2)$$

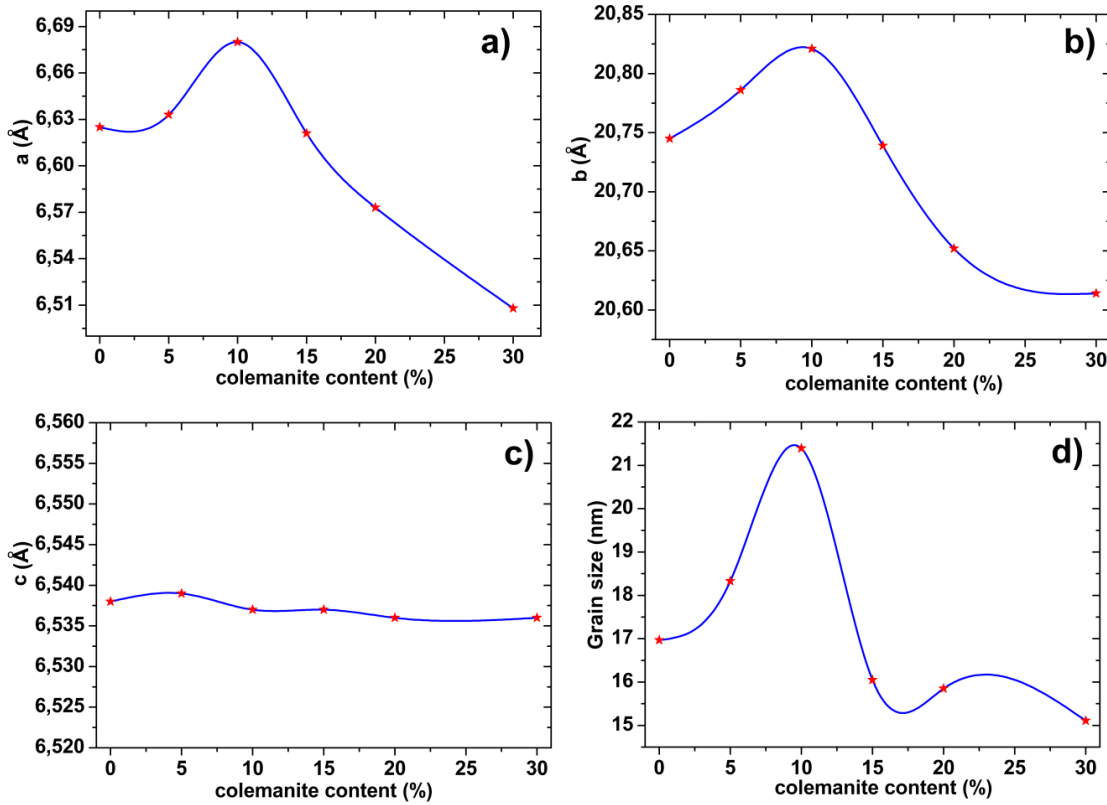
where  $d$  showed the thickness of the crystal,  $\lambda$  designated the wavelength of the used XRD source,  $B$  exhibited sign of FWHM corresponding Bragg peak and also  $\theta_B$  implied the Bragg angle.

The findings obtained from the XRD analyses were tabulated in Table 1. It was revealed that the colemanite addition into IPP matrix was considerably influential in the microstructural properties of the IPP. Namely, the initial addition of the colemanite gave rise to the increasing of  $a$  and  $b$  unit cell parameters, Figure 2.a and b. The maxima,  $6.680\text{\AA}$  and  $20.821\text{\AA}$  for  $a$  and  $b$  (0.83% and 0.36% of expansions, respectively) were achieved with the composite sample with 10% colemanite. This

expansion was probably caused from that the colemanite having the chemically different character from IPP presumably forced IPP chains, which results in the formation of larger free volumes in the matrix. Thus, IPP chains may form larger monoclinic units during crystallization. However, as the colemanite content reached up to 10% colemanite content level, these parameters (*a* and *b*) depicted the decreasing trend as seen in Figure 2a and b. Even, *a* and *b* unit cell parameters reached the minimum values, 6.508 Å and 20.614 Å, which was lower than that neat IPP had. These decrements were attributed to the presence of the larger amount of colemanite in the certain area in IPP matrix. In other words, since the unit cell parameter were highly dependence of the filler composition, the further addition of colemanite surrounding IPP molecules in the matrix brought about the inhibiting or preventing effect to form larger IPP monoclinic unit. This resulted in the formation of smaller monoclinic units compared to neat IPP, Figure 2a and b. On the other hand, XRD results depicted that almost no effect of colemanite addition on the *c* unit cell parameters of IPP based composites were observed, Figure 2c. This parameter remained almost constant although the colemanite content increased in IPP matrices. This was believed to account for that the chain axis of the IPP molecular segments presenting in the monoclinic arrangements was the parallel with the axis of *c* unit cell parameter. Thus, any changes were not recorded in *c* unit cell parameters. Furthermore, as well as unit cell parameters, the founded results as for the crystal sizes of the neat IPP and IPP composites containing varying content of colemanite particles were tabulated in Table 1 and drawn in Figure 2d. It is to be emphasized that the crystal sizes of the composite sample illustrated the good correlation with the *a* and *b* unit cell parameters. That is, the crystal sizes of the composite increased with the initial addition of the colemanite, after maximum, the drastic decrements were observed, Figure 2d. At low colemanite content, the colemanite particle may assist the ordered nucleation of the IPP crystals, causing the formation of larger crystals. However, the high presence of colemanite particles in the matrix might prevent the development of the IPP crystals, which results in the smaller IPP crystals in the matrices.

**Table 1.** The dependence of the unit cell parameters a, b and c, and crystal size of IPP samples on the colemanite content in the matrices.

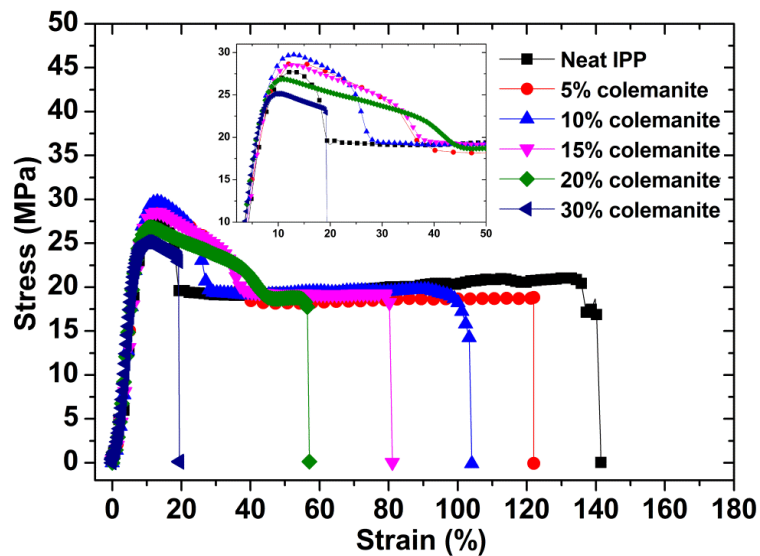
Species	<i>a</i> (Å)	<i>b</i> (Å)	<i>c</i> (Å)	Crystal size (nm)
Neat IPP	6.625	20.745	6.538	16.97
5%	6.633	20.786	6.539	18.33
10%	6.680	20.821	6.537	21.39
15%	6.621	20.739	6.537	16.05
20%	6.573	20.652	6.536	15.85
30%	6.508	20.614	6.536	15.11



**Figure 2.** The variation of  $a$ ,  $b$ ,  $c$  and crystal size with colemanite content

### 3.2. Mechanical Characteristics of IPP Based Composites

In order to unfold the effect of colemanite content on the mechanical characteristics of IPP, the mechanical behavior of the IPP based composites were investigated in details. Prior to the discussion of the results, it was to be stated that the micro injection molding temperature and injection pressure during the preparation of the test samples were determined as 220°C and 8.6 bar to cope with the difficulties in the flow and viscosity. Thus, at this condition, both the ideal dispersion and good melt flow were achieved in the products. The representative stress-strain curves of the neat IPP and IPP based composites with 5, 10, 15, 20 and 30% colemanite were depicted in Figure 3, which determined by using 5 cm/min<sup>-1</sup> elongation speed during the tests. The finding revealed that neat IPP showed the great extent of cold drawing and the maximum percent elongation, 141% was achieved with neat IPP. Moreover, it can be deduced from the figure that all IPP based composites showed ductile failure with neck formations. Only, the product with 30% colemanite failed during the strain softening region. Moreover, the maximum ultimate tensile strength was obtained from the product containing the 10% colemanite content as shown in Figure 3. On the other hand, the figure showed that the percent strain of the products consistently decreased with the increasing of the colemanite content in the IPP matrix. This was the experimental evidence for that the increment in the colemanite content in IPP matrix gave rise to increment in the brittle characteristics of the products.



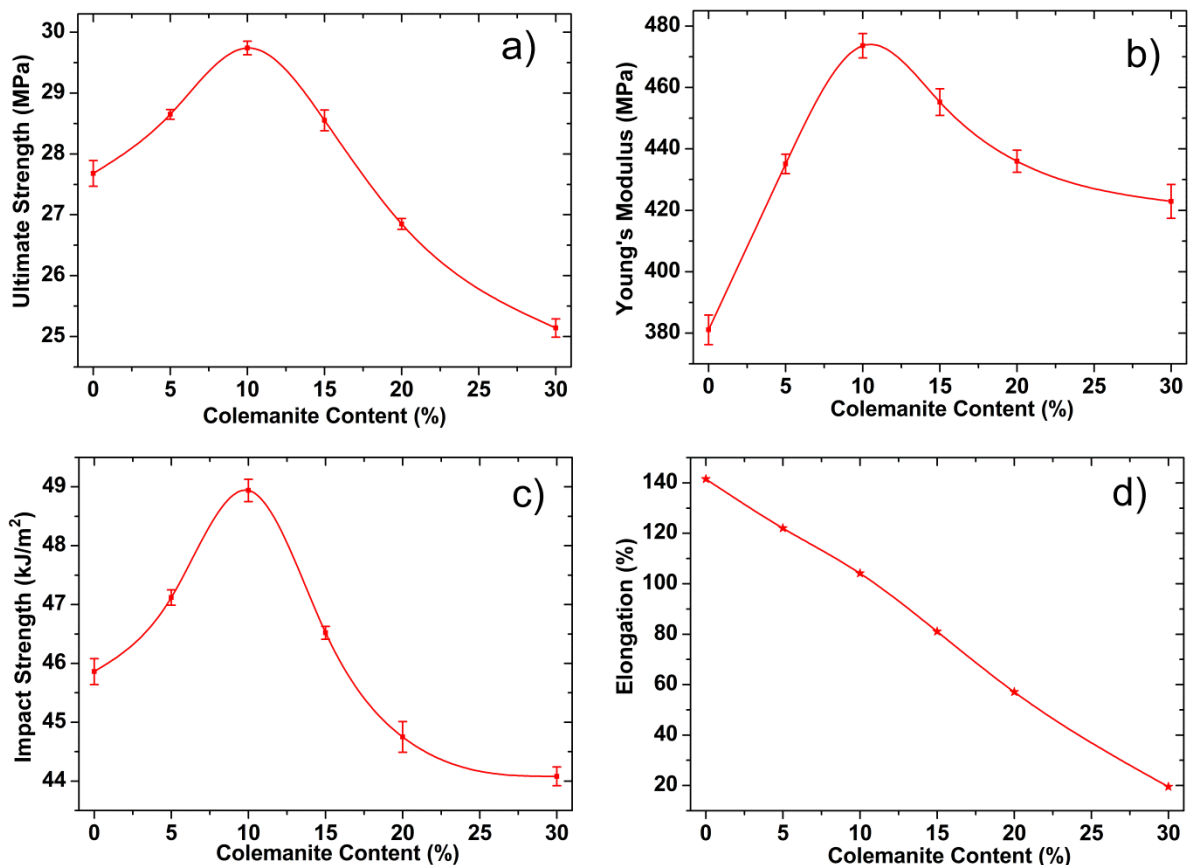
**Figure 3.** Stress-strain curves of neat IPP and IPP based composites including varying percentage of colemanite.

The significant mechanical properties (ultimate strength, Modulus, impact strength and percent elongation) of the neat IPP and IPP based composites were also studied by discussing the effect of colemanite content in the matrix. The numerically mechanical test findings belonging to the products were tabulated in Table 1 and were drawn in Figure 4. The obtained results depicted that the varying amount of colemanite addition into the IPP matrix affected slightly the all mechanical properties of the products, except for percent elongation. The tensile strength of the products illustrated the increment at the initial addition of the colemanite and reached the maximum strength, 29.74 MPa with the IPP based composite including 10% colemanite. This value was 3.5% greater compared to the strength of neat IPP. This increment probably caused by the strengthening effect of the colemanite incorporated into the IPP polymer matrix [41]. After maxima, the consistently decreasing trend was observed in the strength of the products and, at higher content, the tensile strength was found to be 25.14 MPa, which was 9.18% lower than the value that neat IPP had in Figure 4.a. The initiatory increasing trends with colemanite content were also observed in Modulus of the products. The maximum values, 473.63 MPa (24.3% greater than that neat IPP had) were noted at the content of 10% colemanite for the modulus. After maxima, the dramatic decrements were also recorded in modulus of the products as illustrated in Figure 4.b. However, even at the highest colemanite content (422.92 MPa), Young's Modulus of the products was found to be greater than that neat IPP had. This was evident that the colemanite addition into IPP matrix caused clearly that the products gained the resistance against to deformation. This was believed to result from the enhancement in the alignment and orientation of IPP polymer chain due to the colemanite assisting to IPP chain to orient. On the other hand, the impact strength of the products was also investigated in detail by means of Izod impact tests. It was seen that the change in the colemanite content did not affect considerably the impact strengths of the products. The impact strength findings were presented as the absorbed energy by the products during the test. The results depicted that the impact energy absorption capabilities of the products increased initially with the content, and the maximum value was recorded as 48.94  $\text{kJ/m}^2$  with the composites containing 10% colemanite, which was 6.7% greater compared to neat IPP. After maximum, the decreasing trend was also observed in the impact strength of the IPP based composites, in Figure 4.c. The minimum value, 44.08  $\text{kJ/m}^2$  related to impact strength was obtained from the product with %30 colemanite content. The improvement in the impact strength with the initial additions of colemanite presumably caused from that the colemanite particles presenting in the matrix gained to the composites extra energy absorption ability by sharing the stress applied suddenly in the certain regions. However, at further colemanite content, this positive effect may turn into the negative effects. Namely, the presence of larger amount of colemanite microparticles in the IPP matrix probably brought about the formation of

the agglomeration, which resulted in the appearing the cracks rather than absorbing the energy when the force applied suddenly to the composite materials[42]. Furthermore, Figure 4.d. illustrated that the percent elongation of the composites decreased with the increasing of the colemanite content in the IPP matrix. This may be attributed to that the stiffness of the polymer matrix was smaller than the colemanite inorganic filler. Namely, since the increment in colemanite content gave rise to the increasing of the rigid characteristic of IPP matrices, the free movements of IPP chains in the matrix were probably hindered by colemanite [43].

**Table 2.** The variation of the mechanical characteristics of neat IPP and the composites containing different content of colemanite.

Species	Tensile Strength (MPa)	Young's Modulus (MPa)	Impact Strength (kJ/m <sup>2</sup> )	Maximum Strain (%)
Neat IPP	27.68±0.21	381.06±4.82	45.86±0.22	141.46
5%	28.65±0.08	435.10±3.16	47.12±0.13	122.03
10%	29.74±0.11	473.63±3.97	48.94±0.19	104.11
15%	28.55±0.17	455.25±4.36	46.52±0.11	81.05
20%	26.85±0.09	436.00±3.59	44.75±0.26	57.08
30%	25.14±0.15	422.92±5.51	44.08±0.16	19.48



**Figure 4.** The variation of a) ultimate strength b) Young's modulus) impact strength and d) percent elongation of the neat IPP and the composites with colemanite content.



#### 4. CONCLUSIONS

In this present work, the effect of varying percentage of colemanite addition on the significant microstructural (XRD pattern, unit cell parameter, crystal size) and mechanical (strain vs. stress behavior, tensile strength, Young's Modulus, impact strength, percent elongation) properties of IPP was studied meticulously. The experimental findings revealed that these properties were depended considerably on the percentage of colemanite presenting in IPP matrices. Moreover, it was revealed that the optimum colemanite content was found to be 10% in order to obtained IPP based composite having advanced characteristics in terms of microstructural and mechanical aspects. Only  $\alpha$  form (monoclinic arrangement) in the crystalline domains of IPP was observed from X-ray powder diffraction patterns belonging to neat IPP and IPP based composites without any formation of  $\beta$  form (hexagonal arrangements). The increment in the colemanite content in IPP matrices gave rise to both decreasing of the sharpness and broadening of the diffraction peaks [especially  $\alpha(110)$  and  $\alpha(040)$ ]. This may be seen as the experimental evidence for this disorderly and defects formed in the crystal structure of the IPP. Moreover, it was founded that the colemanite addition into IPP matrix was considerably influential in the microstructural properties of the IPP. Namely, the expansions and contractions in  $a$  and  $b$  unit cell parameters were recorded depending on the addition of colemanite into IPP matrices, however,  $c$  unit cell parameter remained almost unchanged. Furthermore, the obtained results showed that the initial addition of colemanite brought about the reinforcement in tensile strength, Modulus and impact strength of IPP, which probably caused from the strengthening effect of the colemanite incorporated into the IPP polymer matrix. But, at further colemanite content, the mechanical properties get worse. Moreover, it was founded that percent elongation of the products consistently decreased with the increasing of the colemanite content.

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