Kaolinite/Polypropylene Nanocomposites. Part 1: Compounding

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Abstract - *Polypropylene (PP) is the most widely used semi-crystalline thermoplastic polymers due to its good thermal and mechanical properties, high dimensional stability and excellent process ability. In this research three types of different molecular weight PP named HP500 (HP), Atofina HG265FB (ATO) and Borealis PPH7089 (B) were used for production of nanocomposites at different kaolinite contents. Nanocomposites sample preparation starts by drying kaolinite and thorough mixing with PP and compounding at different formulations between 0 up to 30 wt% of kaolinite for two batches of processing; for spinning of fibers and filaments for 3D printing. Melt flow and crystallisation temperature of HP compounded formulations shows an increase with kaolinite content while Melt Flow Index for ATO PP decreased with increase of kaolinite content. Increase of crystallisation temperature implies kaolinite acts as a nucleating agent.*

Key words: **Polypropylene, Nanocomposites, Melt Flow Index (MFI), Kaolinte, Compounding, Crystallisation**

1. INTRODUCTION

In recent years, rapid growth and high consumption rates were predicted for various polyolefin composites, since they find applications in many areas such as automotive, home appliances, and construction. The introduction of particulate fillers into polypropylene matrix in the fabrication of thermoplastic composites is mainly governed by price-performance relationships. Apart from reducing the price of the final material, mineral fillers can also help to improve shrinkage on moulding, stiffness and flammability, which are the principal limitations of the bulk thermoplastics. The effects of fillers on the mechanical and other properties of the composites depend on their shape, particle and aggregate sizes, surface characteristics, and degree of dispersion [1–5].

Characteristic size of the inorganic filler particles in polymer composites are distinguished as micro-composites that contain micrometre-scale fillers or nanocomposites that contain nanometre-scale fillers, i.e. fillers that have at least a characteristic size less than 100 nm. As compared to neat polymer or microcomposites polymers, polymer nanocomposites exhibit markedly improved properties like modulus, strength, impact performance, and heat resistance at low concentration of the inorganic components. In this context, the nanocomposites are lighter in weight and easier to be processed [6].

Kaolinite is a 1:1 layered dioctahedralaluminosilicate composed of silicate sheets (Si_2O_5) bonded to aluminium hydroxide layers ($Al_2(OH)_4$) called gibbsite layers with the formula:$Al_2Si_2O_5(OH)_4$ or $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$. Kaolinites find use in a variety of applications, either because of specific technical properties and/or because of their very favourable cost/performance ratio. Fine kaolinites have semi-reinforcing properties and used as the primary filler in PP. In addition, the processing properties of kaolinite filler are often more advantageous, since they are easier to disperse efficiently. All kaolinite fillers share some common features because of the plate like shape of their particles, which results in some stiffening of the PP. This gives much better extrusion and drawing properties (exhibiting low die swell and good dimensional stability) than that can be obtained from most other mineral fillers.

In this work, it is decided to search and study different series of formulations with subsequent characterization; examine benefits obtained from polypropylene matrix by the inclusion of inorganic nanofiller, kaolinite, by direct mixing. Generally, agglomerations are highly dependent on dispersion of particles in a matrix, i.e. increase in the degree of particle dispersion results in decreasing particle agglomeration. On the other hand, distribution indicates how uniformly the primary particles or their agglomerates are distributed through the volume [7]. Composites prepared in this way showed improvement in mechanical, thermal and electrical properties. Recent study by Batistella et al [8] shows that the fire retardancy of an ultrafine kaolinite in EVA composites exhibited better performances in terms of peak of heat release rate.

The objective when producing filled and/or reinforced compounds is to balance the properties for optimum performance in the specific application. Several parameters need to be considered when working with mineral fillers, some of which can affect the compatibility of the material with the base polymer. The particle-size distribution, surface energy, and surface treatment must all be considered. The key property, however, is the aspect ratio, which is defined as the ratio of the length of the particle to its thickness. For materials with an aspect ratio between 1 and 10, the filler

acts only as an extender in the polyolefin, thus increasing only the stiffness of the material. When the aspect ratio is greater than 10, the filler also reinforces the polymer. This will increase the tensile strength, among other properties. Many different types of minerals are used as fillers with polyolefins; carbonates ($CaCO_3$), kaolinite (hydrated aluminium silicates or clay), magnesium hydroxide - $Mg(OH)_2$ and Talc are among the main fillers. Compounding composite where kaolinite particles are dispersed within the polymeric matrix possess significant processing challenges. Relatively there are few studies available in the literature regarding the development of kaolin-filled polymer composite [9].

To meet the specific challenges for compounding polyolefins with mineral fillers different approaches need to be considered. Among these approaches addition of fillers to the polymer could be via a variety of methods [10].

- Adding all the filler upstream (at an earlier point) with the unmelted polymer.
- Adding a portion of the filler upstream with the pellets and adding the remainder downstream (at a later point) of the melting section.
- Feeding the polymer upstream, then adding the filler downstream.

Extrusion is used in all methods of polymer processing as either the main method of forming or as the method of transporting and metering the molten plastic before it is formed. This process covers wide range of application from continuous articles, such as sheets, tubes, fibres, films, it can be also used to mix together different polymers and to introduce the additives. During the extrusion process, high shear stresses are generated and these forces allow the additive to penetrate and disperse homogeneously in the molten polymer. The components of the extrusion line are: polymer feeder, extruder, die, cooling system as shown Figure 1.5[11].

2. MATERIALS AND METHODS

2.1 Materials

1) 2.1.1 Polypropylene

There are three different types of polypropylene (PP) analyzed in this laboratory work: Moplen HP500 (HP) with melt flow rate1.8 g/10 min, Atofina PPH7089 (ATO) with MFI 12 g/10min and Borealis HF265FB (B) with MFI 26 g/10 min at 230°C and 2.16 kg. These polypropylenes and their mixture is used to prepare nanocomposites sample with different content of kaolinite (K) in powder form with the density 2.6 g/cm^3, surface area 12m^2/g and average diameter of 0.9μm.

2) 2.1.2 Kaolinite

Kaolinite up to 30 wt% and polypropylene were mixed in the molten state using a technique of melt compounding in Thermo-Haake Polylab Rheomix counter-rotating internal mixer at 200^{0}C. The compounded sample was grinded into small pellets of about 1-2 mm size and used for extrusion of fibers or filaments in Thermo Haake intermeshing co-rotating twin screw extruder. Polypropylene kaolinite nanocomposites fibers of K-HP, K-ATO and K-HP-B are extruded at different content of kaolinite and then drawn in a hot-plate drawing apparatusat145°C, in order to obtain highly extended fibers. HP-K up to 20 wt% of kaolinite filaments of about 1.7 mm diameter was extruded for 3D printing. 3D printed specimens of dumbbell shape and single filament of about 350 micron diameter was extruded by a Sharebot Next Generation desktop 3D printer feed with the extruded filaments of about 1.7mm diameter.

2.2 Methods

2.2.1 Composite preparation

Fibers and 3D printed Sample are produced through a series of production processes. At the first stage the kaolinite and polypropylene are dried, weighed and thoroughly mixed manually according to the required compositions and feed into compounding machine. Then compounded formulations are grinded into small pieces and used to feed extrusion machine in which filaments for fibers of about 500 micron or filaments for 3D printing of about 1.7 mm diameters will be extruded. Filaments for fibers will further extend in drawing machine at 145 ^{0}C and filaments for 3D printing are used to feed 3D printing machine. In 3D printing machine the extruded filaments will re-melt and re-orient through a heated nozzle to produce a filament of about 350 micron diameters.

3) 2.2.2 Compounding

Kaolinite and polypropylene were mixed in the molten state using a technique of melt compounding in Thermo-Haake Polylab Rheomix600 Pcounter-rotating internal mixer (Fig.1) operating at a temperature of 200 °C and rotational speed of 50 rpm. The residence time of the compounding varies from 10 to 15 minutes depending on how it develops a plateau of torque curve in the mixer. That means, when the curve reaches plateau and does not decrease anymore and stabilizes the compound is ready to be extracted. The maximum time limit of 15 minutes has been specially chosen to avoid degradation of the polymer matrix since the processing value of the PP in the table appears to be 230 °C.

The total amount of material inserted in the compounder varies from 50 to 60 grams, these two limits are technical because below 50 grams it tends to have scarcity of volume of material obtained and 60 grams is a machine capacity

limit. For this reason during compounding of PP-K sample it was tried to keep the mass and volume in this range as shown in table 2.4 depending on the content of kaolinite, which has a higher density of about three times polypropylene density.The material doses were measured with the aid of the Mettler AM100 laboratory balance with sensitivity of 0.1 mg.

Three different molecular weight of polypropylene named HP500 (HP) with melt flow rate1.8g /10min, Atofina (ATO) with MFI 12g /10min and Borealis (B) with MFI 26 g/10 min at 230°C and 2.16 kg and various amount of kaolinite content used in compounding of different formulations are given in Table 1. In Table 1, HP and B with mass (gm), for example at 20 wt% of kaolinite HP & B with 33 & 11 gm means its a blend of HP500 and Borealis PP with ratio of 33 gm and 11 gm, respectively. Here the mass of kaolinite will be determined from the total weight i.e. 11 gm.
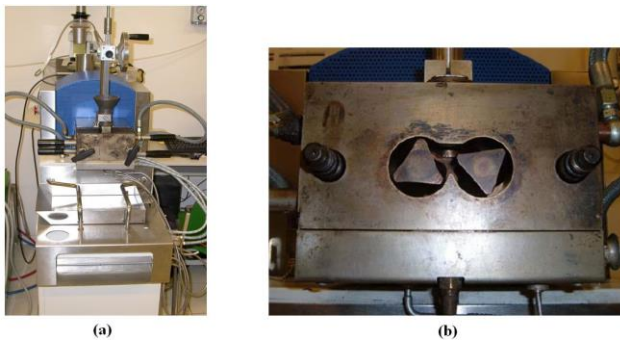

Fig.1 Photographs of (a) Thermo Haake® Rheomix 600P internal mixer (b) Mixing chamber.

Table 1 PP-K compounding formulations.

Kaolinite content (wt%)	PP type	Kaolinite mass (gm)	PP mass (gm)	Formulation number	Final Expenditure of compounded sample	
					Fibers	3D filaments
0	HP	0	50	1	√	
		0	50	2	√	
		0	50	20		√
		0	50	21		√
10	HP	5.5	49.5	3	√	
		5.5	49.5	4	√	
		5.5	49.5	5	√	
		5.5	49.5	22		√
		5.5	49.5	23		√
	ATO	5.5	49.5	6	√	
		5.5	49.5	7		
		5.5	49.5	8		
20	HP	12	48	9	√	
		12	48	10	√	
		12	48	11	√	
		12	48	24		√
		12	48	25		√
	ATO	12	48	12	√	
		12	48	13	√	
50	B	30	30	14		
		30	30	15		
20	HP&B	12	36&12	16	√	
		12	36&12	17	√	
30	HP&B	18	24&18	18	√	
		18	24&18	19	√	

4) 2.2.3 Grinding

Once compounded sample was collected it is necessary to grind these formulations by using a grinding machine, Piovan RN166 (Fig.2). Grinding operation for one formulation will take 5-8 minutes and the sample should be feed in small amount in step wise to avoid stacking and allow easy rotation of blades. Grinded material was stored on storage plate and it is followed by extraction of the sample which occurs in grain sizes of about 1-2 mm in length. The grinder must be cleaned thoroughly after each composition due to the presence of grains in everywhere inside the grinder. After extracted from grinding machine, granules sample can be processed in twin screw extruder through feeding unit (hopper) slowly to avoid clogging of units.

Fig.2 Grinding machine Piovan RN166.

Nanocomposites were marked with the PP followed by kaolinite abbreviation like K-HP, where the first acronym shows filler content and while the latter indicate the PP types. For example, K10-HP indicates an anocomposites sample filled with10 wt% of kaolinite in HP500 polypropylene matrix.

2.2.4 Testing and characterization

Melt Flow Index measurements were performed by a Dynisco LMI 400 plastometer according to ASTM D1238-10. Scanning electron micrographs (SEM) were observed on a fractured surfaces of specimens immersed in liquid nitrogen through a Carl Zeiss AG Supra 40 field emission scanning electron microscope (FESEM), operating at an acceleration voltage of 3 kV. XRD analysis was performed by using Laue camera for transmission diffraction and over 2θ = 3–50° in the Bragg-Brentano configuration. Dynamic mechanical thermal analysis (DMTA) was performed on as-spun and drawn fibers with a DMA Q800 testing unit (TA Instruments) under tensile mode testing. The FTIR spectra were recorded by Spectrum-One equipped with an UATR (Universal Attenuated Total Reflectance) accessory for PP, kaolinite and its composite samples in a laboratory maintained at relative humidity of 48±2% and 23±1⁰C. Thermal Analysis was performed in a temperature range of 30 °C to 700 °C by a TGA Q5000-IR from TA Instruments on sample of about 14 mg at heating rate of 10°C/min tested in both air and nitrogen atmosphere at a flow rate of 100 ml/min.

Density measurements were performed by using a Micrometrics Accupyc 1330 helium pycnometer at 23.0°C in a testing chamber of 1 cm^3 and 100 measurements for each specimen. Differential scanning calorimetry (DSC) analysis was performed on samples of about 14 mg in 160 μL aluminium crucible by using a Mettler DSC30 calorimeter in the range of 0–220°C with a heating-cooling- heating cycle at ±10°C/min flushing nitrogen at 100 mL/min. Mechanical properties of fibers and 3D printed dumbbell were performed at room temperature by using an Instron 4502 dynamometer, equipped with load cells of 100 N and 1kN, respectively. Elastic modulus was determined as a secant value between deformation levels of 0.05 and 0.25% according to ISO 527 standard.

3. RESULTS AND DISCUSSIONS

3.1 Compounding

Compounding of three different types ofPP matrixes (HP, ATO and HP-B mixture) with kaolinite nanofiller was performed as shown in the compounding formulations Table 2.4. Accordingly F_{1-13} and F_{16-19} are used for extrusion of fibers while F_{20-25} are used for filaments of 3D printing process. Compounded formulations are grinded into small granules and its properties were analysed. Here the results of compounded neat PP and K-PP nanocomposites by melt flow analyser, DSC and FTIR would be discussed. After preliminary testing further characterization and discussion was focused only on some selected composition during laboratory work.

5) 3.2 Grinding

All compounded samples are ground into small pieces of granules with size of about 2-3 mm before testing and/or filament extrusion. The output from grinding process is about 95%, because some parts of samples are sticking to the machine, some remain between the blade and plate part and release only during cleaning while certain amount are flying out during start of grinding before closing the cover. Fig. 3 shows photos of compounded and ground samples.
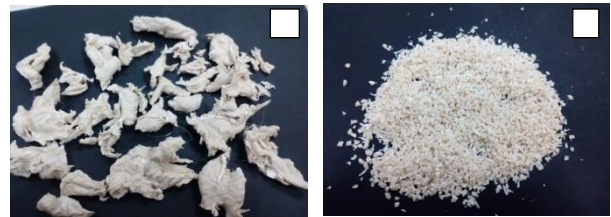

Fig. 3 Photos of compounded samples (a) and ground samples (b).

6) 3.3 Flow Properties

Melt flow index of neat HP, ATO and B polypropylene sample was tested and the results obtained was compared with the MFI from technical data sheet at temperature of 230°C and the weight of 2.16 Kg in Table 2

Table 2 Comparison of MFI of tested and technical data sheet of PP.

PP type	Tested MFI (g/10min)	MFI on data sheet (g/10min)
HP500H	1.85 ± 0.13	1.8
PPH7089, Atofina	12.47± 0.48	12
HG265FB,Borealis	26.16 ± 2.71	26

The increase in MFI of tested sample could be due to combined effect of heat and time while the material is in the melt state. Also the polymers used were stored for a long period of time in pellet form as observed on the package. Within this period of time PP suffer different chemical degradative reactions that may induce alterations in the polymeric chains by chain scission, crosslinking and formation of new functional groups. Furthermore, it may come in contact with a lot of impurities from the surrounding environment and their performances are considerably reduced and these degradative processes are responsible for the reduction of the PP long term stability.

In order to evaluate the effect of the incorporation of kaolinite on the processability of the material, MFI tests were performed on three different types of PP with nanofiller content up to 30 wt%. The result obtained shows that the addition of kaolinite increases the melt flow index values for HP500 (1.8 g/10min) and decreases for Atofina PPH7089 (12g/10min) and HP500-Borealis (26 g/10min)

mixture at a proportion of HP40-B30-K30 wt% was shown in Fig.4. The flow properties may be influenced by the interaction between particles and the interaction between particles with the polymer melt. The interaction between the microscopically rough surface of particles and polymer melt played a significant role and decreases or increase the flow ability, resulting in the increase or decrease of the viscosity, respectively.
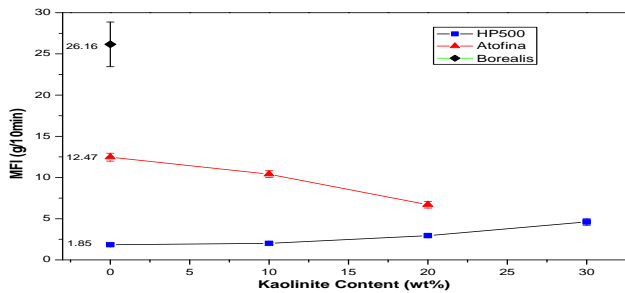
Fig.4 Melt Flow Index of PP-kaolinite composites containing different kaolinite contents HP500, Atofina and HP-Borealis mixture tested at temperature of 230°C and the weight of 2.16Kg.

For lower melt flow index PP i.e. HP500, as the filler content increases, the slippage between particles was the dominating effect, resulting in the decrease of the viscosity and vice versa for higher melt flow index PP, Atofina. Kaolinite particles have the ability to slide against each other during the application of shear force because of its platy shape, which allows an increase in the plastic flow. However, this ability only applies to fillers at certain parts of the composites that are near the die walls, and so, the effect of this ability on MFI is minimal [12]. Furthermore, the incorporation of very fine kaolinite could increase resistance to flow because of the increase of contact surfaces between particles and matrix.

7) 3.4 DSC Analysis

DSC analysis provides quantitative evaluation of the crystallization behaviour of neat PP and its composites. The crystallization behaviour of all samples was studied at 10 ^{0}C/min cooling rates. The DSC thermograms curves of neat HP and kaolinite filled HP compounded sample are shown in Fig. 5. Molecular chains of PP can crystallize by themselves (self-nucleation effect) or crystallize with the presence of inorganic filler, kaolinite leads to heterogeneous nucleation effect in filled PP system. When kaolinite fillers are well dispersed in the PP matrix, the heterogeneous nucleation effect dominates the crystallization process and one broad crystallization peak can be observed resulting from the co-existence of self-nucleation and heterogeneous nucleation effects.

The melting peak becomes broad when PP is filled with kaolinite ndicating that in comparison to PP there were more

crystallites with lower perfection formed in PP-kaolinite composites. It is believed that this may result from some impediment in the melting process of filled PP because of combination of small particle size and uniform distribution of kaolinite in PP matrix. At this phase, the growth of crystallites was delayed due to restricted alignment of PP molecules network by kaolinite fillers. It leads to low mobility of polymer segments in the regions of lamellae surfaces during polymer melting process. These analyses showed that the incorporation of kaolinite fillers enhanced the PP nucleation mechanism but also hindered the crystallite growth which corresponds to the broader melting peak of PP/kaolinite composites in DSC curves.
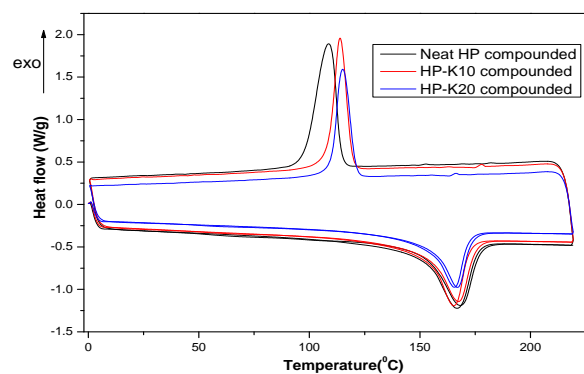
Fig. 5 DSC thermograms of neat HP, K10-HP and K20-HP compounded formulations.

From these curves, Fig.5, some useful parameters for crystallization analysis, such as the melting temperature (Tm), the crystallization temperatures, e.g. the exothermic peak maxima and the end temperature of crystallization can be obtained. The degree of crystallinity ($\mathcal{X}_c$) of PP nanocomposites was calculated using Equation given below.

$$\mathcal{X}_{pp}(\%) = 100 \ \frac{\Delta H_i}{\Delta H_{PP}.(1-f)} .... \text{Eq. 1.}$$

Where ΔH_i = melting enthalpy, ΔH_{PP} = reference enthalpy of a fully crystalline PP taken as 207 J/g [18], f = weight fraction of nanofillers. The crystallization enthalpy (ΔH_i) was determined from the area under the melting peak.

Table 3 summarizes the most important DSC results of compounded sample of neat HP and HP-kaolinite composites. Introductions of nanoparticles increase crystallization temperatures, from 108.6 ^{0}C for neat HP to 116.6 ^{0}C for K20-HPand have almost the same melting temperature in comparison to the neat PP. The crystallinity content $\mathcal{X}_c$ of compounded samples after cooling from first melting displayed higher value. This earlier crystallization phenomenon can be interpreted as the result of an increase in nucleation activities. Melting process induces the molecular chains mobility of PP, and reduces the free energy

barrier for crystallites formation which is able to accelerate the rate of crystallization.

Table 3 DSC analysis: melting temperature (Tm), crystallinity content (X_c) and crystallization temperature (Tc) for neat and K-PP nanocomposites compounded samples.

Sample Composition	1st melting temp.		Crystallization temp.		2nd melting temp.		Crystallinity Content (%)	
	peak (°C)	integral (J/g)	peak (°C)	integral (J/g)	peak (°C)	integral (J/g)	1st Melting	After Cooling
HP500	168	82	108	96	167	97	40	47
K10-HP500	167	84	116	85	165	85	44	45
K20-HP500	167	67	117	70	166	70	39	40

The crystallinity content after cooling for compounded sample is almost the same for kaolinite content up to 10 wt%. Nonetheless, degree of crystallinity (X_c) tend to decrease when the kaolinite content is 20 wt%. At a given cooling rate, crystallization temperature of kaolin-filled HP composites was higher than that of unfilled HP polypropylene. This clearly implies that incorporation of kaolinite results in the heterogeneous nucleation to the PP matrix. At an early stage of crystallization, the molecular chains of PP might be in contact with the filler surface and limit the movement of polymer chains. The movement of polymer chains which is in a form of distorted network can be quickly relaxed and re-established. This scenario helped to increase the structural stability of polymer matrix [13] giving an increase in the crystallization peak temperature. This finding confirms that kaolinite behaves as a nucleating agent in polypropylene.

8)   3.5 Fourier Transform Infra-red (FTIR)

FTIR spectra of Compounded neat HP, dried kaolinite and nanocomposites of HP-kaolinite are shown in Fig.6(a). The spectra of dried kaolinite show the appearance in the hydroxyl zone of the bands at the wave numbers of 3696, 3672, 3646, and 3621 cm^{-1} where the three first bands characterize the outer hydroxyl groups vibrations, respectively. Also, observation of the bands at 3672 and 3646 cm^{-1} confirms that the used kaolinite is a highly ordered mineral. The band at 3621 cm^{-1} corresponds to the inner hydroxyl groups which do not participate to the establishment of the hydrogen bonds responsible of the

cohesive energy of kaolinite. The Si-O- and Si-O-Si vibrations are illustrated by the bands at 1118, 1029, and 998 cm^{-1} whereas the Al–OH bond vibrations are visibly characterized by the bands appearing at the wave numbers 911, 795, and 749 cm^{-1}.
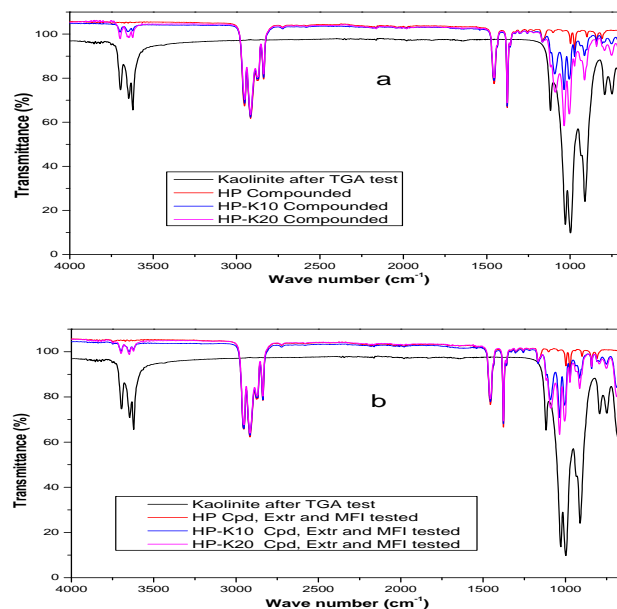
Fig.6 FTIR spectra of neat PP, kaolinite and PP nanocomposites with 10 and 20 wt% of kaolinite compounded only (a) and compounded, extruded and after melt flow tested (b).

Similarly FTIR spectra of compounded neat PP indicates the following main assignments and peaks for specific functional group: ν_{as} (methyl group -CH$_3$) – 2962 cm^{-1}, ν_s(-CH$_3$) – 2914 cm^{-1}, ν_{as} (methylene group - CH$_2$ -) – 2876 cm^{-1}, ν_s (- CH$_2$ -) – 2838 cm^{-1}, δ_{as}(-CH$_2$) – 1454 cm^{-1}, (m), δ_s (-CH$_3$) - 1378 cm^{-1}. The FTIR spectra of PP-kaolinite nanocomposites show spectra at the same wave number as that of neat PP and dried kaolinite and its intensity depends on the composition of the nanofillers.

FTIR spectroscopy is also used to illustrate the degradation mechanisms [14] of polymers by identifying and quantifying the degradation products. Significant degradation mechanisms for polymers in this case will be thermo-oxidation which results in the formation of hydroxylated compounds, which may be identified by examination of the 3800–3100 cm^{-1} regions in the infrared spectra. Fig.6(b) shows FTIR spectra for compounded, extruded and melt flow tested samples to investigate about the degradative effects at a molecular scale to evaluate the stabilization effectiveness of the additives [15]. By analysing the changes in chemical functional groups it can be determined that both Figure 3.4 (a) and (b) shows almost similar spectra which implies that there is no degradation effects i.e. no functional groups transformation on the polymeric structure due to heat

treatment during the extrusion and melt flow testing process.

Fourier Transform Infrared (FTIR) spectroscopy can be also described as a means of quantifying the filler loading of polypropylene composites. Though filler content of polymer composite is commonly determined by using the thermogravimetric analysis, the infrared spectroscopy technique may also be used as a method for the quantitative analysis of those materials. This technique has the advantage of being more specific and is suited to the analysis of mixtures. Although numerous absorption bands of various compounds may appear, and it is possible to select the bands specific to the components of interest in the mixture. The choice of correct absorption bands is of great importance for quantitative analysis work. Among criteria of suitable band are: strong or large absorption coefficient, isolated from other bands to avoid interferences and outside regions of compensated absorption other than CO_2 and H_2O.

9) 4. CONCLUSION

In summary of compounding process, this paragraph will describe and compare melt flow properties, calorimetric analysis and FTIR analysis of neat PP and K-PP nanocomposites samples. The MFI values have decreased with increasing content of kaolinite for Atofina and increasing with kaolinite content for HP500. These results show that the flow properties of kaolinite filled PP composites are significantly influenced by the natural properties of the PP. Although fillers particle shape, the aspect ratio, particles size and particles size distribution are properties believed to influence the flow properties as well as the MFI values of the PP kaolinite nanocomposites. It was observed that above 30 wt% of kaolinite it is difficult to obtain a continuous chain of HP composite.

An investigation of crystallization behaviour of compounded neat HP PP and its composites was performed through DSC analysis in the heating-cooling-heating cycles of neat HP and HP-kaolinite nanocomposites were compared. The crystallinity content of compounded samples after cooling displayed the highest χ_c than first melting due to increase in nucleation activities. Melting temperature and particularly the level of crystallinity of compounded samples after cooling were almost similar for neat HP and K-HP nanocomposites for kaolinite content with 10 wt%. Nonetheless, crystallinity content (χ_c) of the composites tends to decrease in 20 wt% of kaolinite content. Crystallization temperature of kaolin-filled HP composites was higher than that of unfilled PP. From the DSC cooling stage, it is noticeable that crystallization temperature of K-HP nanocomposites of compounded sample is higher than neat PP, i.e. up to 116.6 °C versus 108.6°C, suggesting a moderate effect of kaolinite as nucleating agent. The increase in the crystallization temperature after introduction of the filler means that lower under cooling of the melt is necessary

for the crystallization to occur [16]. The reason for this behaviour is believed to be the strong influence of the kaolinite on the chain dynamics. Fillers usually act as nucleation centers [17].

FTIR spectroscopy is used to identify functional group in matrix and fillers, quality and quantity of fillers and also illustrate the degradation mechanisms of polymers by identifying and quantifying the degradation products. Significant degradation mechanisms for polymers in this case will be thermo-oxidation which results in the formation of hydroxylated compounds, which may be identified by examination of the 3800–3100 cm^{-1} regions in the infrared spectra. FTIR spectra for sample passed through compounding, extruding and melt flow testing steps to investigate about the degradative effects at a molecular scale to evaluate the stabilization effectiveness of the additives. By analysing the changes in chemical functional groups it can be determined that both compounded only and compounded, extruded and melt flow tested sample shows similar spectra which implies that there is no degradation effects i.e. no functional groups transformation on the polymeric structure due to heat treatment during the compounding, extruding and melt flow testing process.

According to these experimental results compounded samples of all the formulations mentioned under compounding process can be used for filaments extrusion other than formulations 14 and 15, composition that contains Borealis at 50wt% of kaolinite content. Even if MFI of PP nanocomposites increase or decrease with increasing kaolinite content, they have the processability for filaments by varying the screw rotation and the collection rate to obtain the required diameter. Kaolinite will act as a heterogeneous nucleating agent in PP-kaolinite nanocomposites and expected to enhance the filament crystallization temperature further.

Compounding of 25 formulations using different molecular weight PP and their blend at different kaolinite content was performed. From analysis of compounded sample melt flow test result shows an increase in melt flow index for HP (with original MFI of 1.8 g/10min) and decrease in MFI for ATO (original MFI of 12 g/10min) polypropylene as kaolinite content increases in compositions. The presence of a kaolinite in the PP nanocomposites results in the enhancement of the PP crystallization temperature through heterogeneous nucleation. The melting behavior and crystallinity content of a material was not affected by the nanofiller content. FTIR analysis shows kaolinite used has good quality and different intensity of spectra was observed depending on kaolinite quantity. It also illustrates absence of degradation of polymers during processing compared to compounded, extruded and melt flow tested samples.

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