

Effect of Kaolinite on Thermal Conductivity of Polymer based Nanocomposites

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ABSTRACT

In this study, kaolinite was used as nanofiller for the fabrication of High density polyethylene (HDPE) and polypropylene (PP) nanocomposites with various low clay loadings. These nanocomposites were synthesized by a by melt blend method, and polyethylene grafted maleic anhydride (PE-g-MA) was used as a compatibilizer. The morphology of nanocomposites was explored by X-ray diffraction (XRD) and the thermal conductivity were measured using Differential Scanning Calorimetry (DSC). Morphology results indicate that intercalated and partial exfoliation structures were formed. The thermal conductivity measurements showed an obvious improvement in the estimated thermal conductivity of HDPE/kaolinite nanocomposites within tested loadings and the optimum value ($0.93 \text{ W m}^{-1} \text{ }^{\circ}\text{C}^{-1}$) reached when clay content was 2 wt %. In the other hand, no enhancement detected in PP composites due to the poor adhesion which attributed to the immiscibility between PP and HDPE in PE-g-MA. Also, Mohaddespour and Lewis Nielsen(LN) models have been used to investigate their predictive capability for determining thermal conductivity of those nanocomposites. A deviation from the experimental data was obtained due to the missing information about the real molecular structure of the composite i.e dispersion of filler which is vital for the determination of related parameters used in the models.

Keywords: Thermal Conductivity, Polymer Nanocomposites, DSC , Kaolinite, PE-g-MA

1. Introduction

Polymer based composites are essentially important in thermal management field, due to their remarkable ability to add increased functionality that can help to meet today's requirements [1]. The unique combination of their potentially low production costs, light weight and key properties paves the way to a wide range of future applications rather than the existing ones.

Numerous studies investigated the feasibilities of applying various thermally conductive fillers such as carbon derivatives, metallic and ceramic fillers in polymer composites with different base materials; and usually high volume fraction of conductive filler is required to enhance the thermal conductivity of the polymer [2], [3] ; which leads to more or less comparable results. It should be noted that increasing loading level is not always preferred, as a high filler loading can cause the composite to be brittle, have poor processability and result in a high cost [1] . Among that, using the best heat conductive filler does not always lead to highest thermal conductivity in nanocomposite [4], [1] .

Polymer/clay nanocomposites (PCN's) is a very promising research area due to cost effective, unique structure, high specific surface area, and their ease of availability from natural resources [2]. However, engineers faced several challenges in the design of polymeric composites for thermal applications; one of the most challenging parts is the thermal resistance due to phonon scattering phenomena which caused by poor adhesion in the interface region [3]. Naturally, clay is hydrophilic whereas HDPE and PP are the most hydrophobic polymers. All clay types employed for PNC preparation are pre-modified, and modification starts with a purification step, which is a long and time consuming process [4]. Many surfactants were used to modifying clays but as of now, the number of molecular units that can directly intercalate in kaolinite clay is still very limited [5]; attributed to its unique structure.

While many studies on nanocomposites focus on the importance of chemical surface modification of the clay using surfactants without adding compatibilizer and the use of both surfactants and compatibilizer [6]; in order to increase the compatibility between clay and polymer matrix, the role of compatibilizer without the use of surfactant for the clay have not been extensively studied. In addition, thermal conductivity of PCN's has not been investigated as considerable as other nanocomposites.

Various clays have been used to improve the performance of polymers, such as spherical silica, layered silicates, sepiolite, etc. Amongst all the potential nanocomposite precursors, layered silicates have been extensively investigated because of the ease of intercalation of the polymer chains between the silicate layers [7]; the most popular used layered silicates, is montmorillonite (MMT). Kaolinite, is another layered silicate that different from MMT, which has the potential to be an ideal precursor for the preparation of nanocomposites since it is cost effective when compared to MMT [8]. It was observed in many studies that kaolinite has improved the properties of some polymer based composites [13]; and in particular gives better oxygen barrier and water transport property than polymer/MMT composites [8]. Also, kaolinite has a greater theoretical internal surface area per unit mass, and it exhibits a much greater crystallinity than the smectites [8]. Despite, kaolinite didn't get much attention as a filler because kaolinite layers are tightly bonded together due to 1:1 structure and that is the reason for exhibiting a low shrink swell capacity and a low cation exchange capacity which make those layers hard to delaminate [14]. Recently, polymer/kaolinite nanocomposites attracting considerable interest in polymer engineering research because they show dramatic improvement in properties at very low clay contents (< 5 wt. %) [7]; which leads to cost to property balance. Although, no research focused on the investigation of thermal conductivity of polymer/kaolinite nanocomposites and specially, no attention has been paid to use Libyan kaolinite as reinforcing filler for polymer nanocomposites [13]; where Libyan kaolinite can be considered as pure kaolin as reported by this study [9], [10]; their results showed that Libyan kaolinite is close to the value of pure kaolinite [9].

The ultimate goal of this study is to investigate the thermal conductivity of polymer/ Libyan kaolinite nanocomposites through: (1) investigating the influence of surface modification approach (using PE-g-MA) on the morphology of the polymer/kaolinite nanocomposites and the impact that will cause on their thermal conductivity, (2) determining the optimum loading that kaolinite can show maximum change in the thermal conductivity of HDPE and PP nanocomposites.

2. Materials and Methods

2.1 Materials

The materials were provided by (Polymer research center, Tripoli city in Libya). HDPE and PP were used as matrices. PE-g-MA was prepared in Lab. according to [11]; and used as a compatibilizer. Raw kaolinite (collected from Jarmah Member, Sabha city in Libya) were used as nanofiller consisted of particles (< 75) μm in diameter. Libyan kaolinite had perfect crystallization, high purity ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) with chemical composition 46.54% SiO_2 , 39.50% Al_2O_3 , as reported by [10]; a work that studied Libyan kaolinite chemical composition and microstructure. P-Xylene (Alfa Aesar 99%) was used to melt the polymer before compounded processes. The xylene serves to smooth the melting of the polymer and mixing of the clay.

2.2 modification process using compatibilizer

The modification process was carried out by melting 2 wt% of compatibilizer (PE-g-MA) in a small amount of xylene in a hot plate at 180 °C and then mixed with different wt% amounts of raw kaolin in a separate bowl, each sample individually. The pre-intercalate of "kaolinite ~ PE-g-MA" (precursor) will then dried in an air circulating oven at 80 °C ~ 85 °C over night.

2.3 Composite preparation

The manufacturing process was carried out using twin screw extruder (Brabender Plastic Corder) with (L/D ratio 48) with screw speed of (30 r.p.m.). The host polymer was fed into the twin screw extruder at the feed zone and the precursor were introduced subsequently at the melting zone. The barrel, die temperatures and screw speed were monitored and controlled, as shown in table 1.

Table 1: Temperature and screw speed profiles of the extruder barrel

	Zone 1	Zone 2	Zone 3	Zone 4	Zone 5	Zone 6
Temperature (°C)	140	160	170	180	190	190
Screw Speed (r.p.m.)	30					

Different composites were prepared with different compositions described as following:

- I. a desired amount of HDPE were mixed with 2 , 4, 6 wt% of precursor .
- II. PP of desired amount were mixed with 2, 4, 6 wt% of precursor.

The extruded materials were cooled in air and then granules to small pieces. Details of the composites and codes are reported in table 2.

Table 2: Composites composition and codes

Composite Code	Polymer wt%	Clay wt%	Compatibilizer wt. %
HDPE	100	---	---
HDPEC1	96	2	2
HDPEC2	94	4	2
HDPEC3	92	6	2
PP	100	---	---
PPC1	96	2	2
PPC2	94	4	2
PPC3	92	6	2

2.4 XRD analysis

XRD tests were carried out on prepared samples to evaluate the dispersion of nanoclay at the scan rate of $0.5^{\circ} \text{ min}^{-1}$ at room temperature using an X-ray diffractometer (Philips model X'Pert) in 2θ between 3 and 34° . The X-ray beam was a Co K α radiation ($\lambda = 1.7889\text{\AA}$) using a 40 kV voltage generator and a 30 mA current. The basal spacing of nanoclay layers were calculated using the Bragg's law from the position of the plane peak in the XRD intensity profile, $d = \lambda / (2\sin\theta \text{ max})$. Samples for XRD were prepared by compression- moulded sheets of 2 mm in thickness.

2.5 Thermal conductivity measurements

In this work thermal conductivity were measured using DSC device according to Flynn and Levin method [12] ; were the thermal contact resistance between the sample and heater considered. DSC 204F1 Phonic (NETZSCH) was used at a heating rate of $10^{\circ}\text{C}/\text{min}$. in atmospheric conditions. Phenantren and benzoic acid materials were used as a sensors. The melting points for the sensors measured with the DSC were 95.3°C and 122°C , respectively.

2.6 Modeling techniques

In this study two different models were used to test their prediction capability of thermal conductivity of PCN's. A new model [2]; and Lewis -Nielsen model [13] ; were used to analyze the results obtained from the experimental work.

3. Theory and Calculation

3.1 Flynn and Levin method

This method based on the measurement of rate of heat flow into a sensor material during its first order transition to obtain the thermal resistance of a material placed between the sensor material and the calorimeter in the DSC as illustrated in fig.1. According to this method; sensor materials, which have sharply melting peak in various temperature ranges, is selected and melting points of the sensor materials was used to calculate the thermal conductivity values.

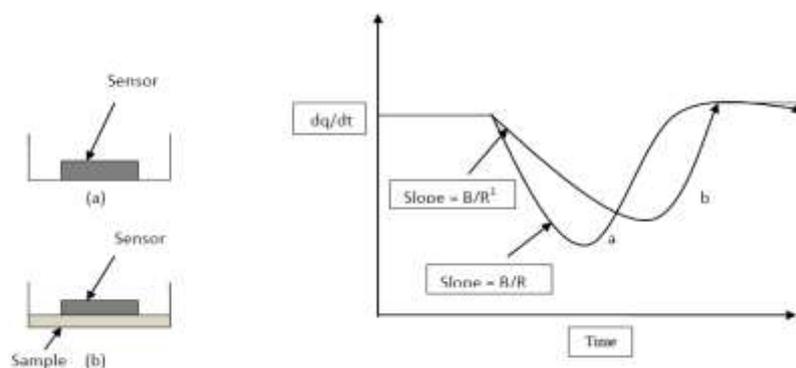


Fig. 1 (a) Sensor (b) sensor + sample and DSC curves sensor and sensor + sample (reproduced from Tavman et al., 2011)

3.2 Models theory

Mohaddespour and his co-workers developed Effective medium approximation (EMA) formula for PCN's media derived from Maxwell Garnet (MG) model, this model presented excellent results for low range of clay content into PP and HDPE nanocomposites by using OMMT as nanofiller [2] . By considering these factors; (1) random dispersion of clay particles into the matrix, (2) effective interface between matrix and clay particles in nano dimension for the energy transportation across the bulk of the samples, (3) taking the matrix phase as a homogeneous medium, (4) the thermal conductivity coefficient of clay particles is the same in both x and z directions as they are randomly dispersed into the polymer matrix, the model can be simply given by:

$$\frac{K_n}{K_p} = \frac{3 + f(2K' + K'')}{3 - 2fK'} \quad (1)$$

Where
$$K' = \frac{K_c - K_p}{K_c + K_p}, \quad K'' = K_c - K_p$$

K_p is the thermal conductivity coefficient of pure polymer at room temperature;
 K_c is the thermal conductivity of clay particles;
 K_n is the thermal conductivity coefficient of the composite;
 f is the volume fraction of clay particles.

Lewis - Nielsen model (LN) is a modification of Halpin-Tsai model (H-T) which considered the maximum volumetric packing fraction of the filler as an additional parameters in order to improve the prediction ability of the classical HT model. In LN the effect of packing of the dispersed particles and particle shape are considered important factors when dealing with the prediction of thermal/electrical conductivity [13].

The effective thermal conductivity of a composite according to the LN model is given as:

$$\frac{K}{K_1} = \frac{1 + AB\omega^2}{1 - A\psi\omega^2} \quad (2)$$

$$A = K_E - 1, \quad B = \frac{\frac{K_2}{K_1} - 1}{\frac{K_2}{K_1} + A}, \quad \psi = \frac{1 + [(1 - \omega_m)]}{\omega_m^2} \omega^2$$

In these equations, K is the thermal conductivity of the composite, K_1 and K_2 are the thermal conductivities of the polymer and filler, respectively, A is the shape factor that related to the generalized Einstein coefficient K_E , B is a constant related to the relative conductivity of the components, ψ is a function related to the maximum packing fraction ω_m of the filler, and ω_2 is the volume fraction of the filler.

4. Results and Discussion

4.1 Morphology characterization

Fig. 2 and fig. 3 exhibit the XRD patterns of raw kaolinite corresponding to the d-spacing of 6.80757 Å at $2\theta = 13.0051$ and the HDPE and PP/ nanocomposites with various clay loadings, respectively. In general, the characteristic peak of the raw kaolinite observed in the figures is not detectable for some composites.

In fig. 2, we notice an absence of kaolinite peak in 2% loading sample and a wide shifting in the other two samples which could interpreted as a partial exfoliation of kaolinite layers in 2% sample, and intercalation in 4% and 6% samples. This might happens for the following two reasons: (1) the low content of kaolinite in the composites, and (2) the molecular chains of HDPE are intercalated into kaolinite layers, and this results in the collapse of the orderly structure of clay through the entropy increase caused by the disorganization of the layers in the solvent [14]. We should mentioned that exfoliation is important in producing a PCN's with unique properties because it is only with such separation of individual clay layers that high aspect ratios are obtained [15]. However, drawing a final conclusion about nanocomposite structure based on XRD results can only be tentative and often an electron microscope observations are necessary specially for exfoliation microstructure [4].

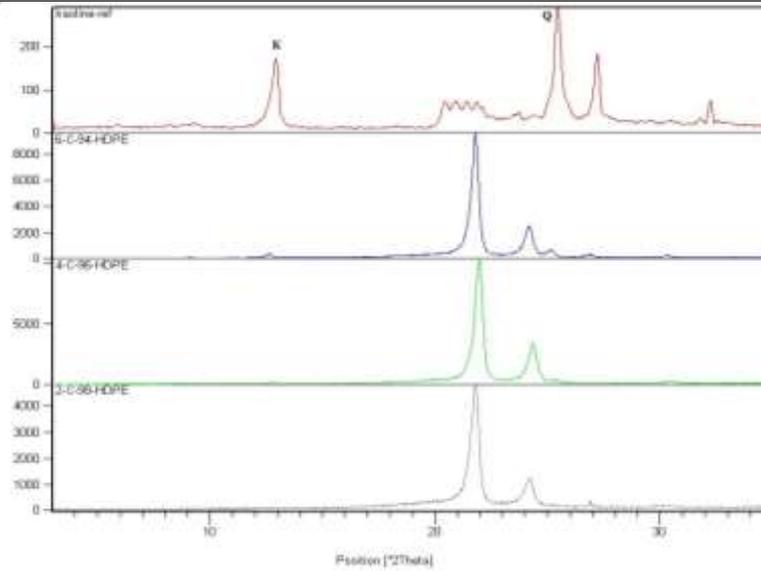


Fig. 2 XRD patterns of natural kaolinite and HDPE/ nanocomposites with clay loadings of 2, 4 and 6 wt% (K: Kaolinite , Q: Quartz)

In the other hand, no detectable peak of kaolinite in PP nanocomposites in fig. 3 and the layers seems to retain stacked to each other, this could be attributed to the poor adhesion between PP and precursor.

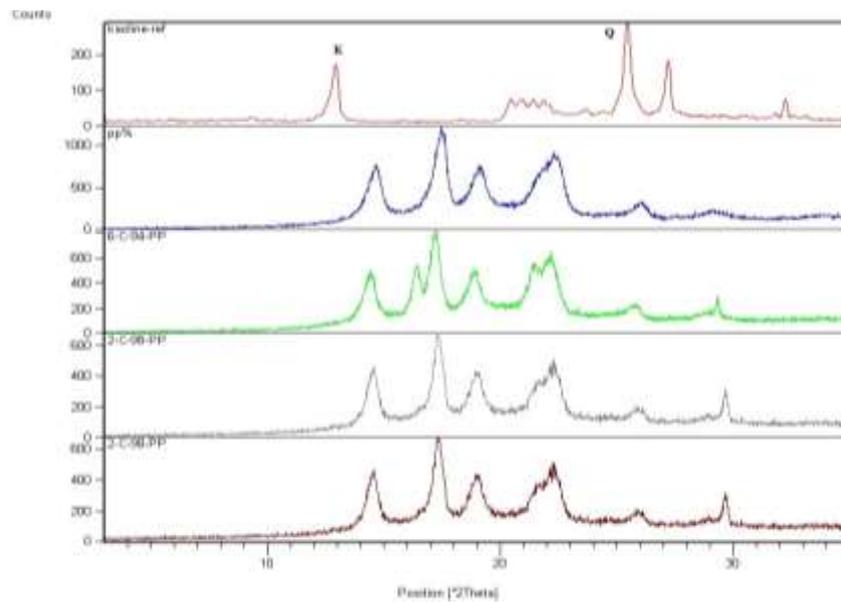


Fig.3 XRD patterns of natural kaolinite and PP/ nanocomposites with clay loadings of 2, 4 and 6 wt% (K: Kaolinite , Q:Quartz)

4.2 DSC measurement results

Thermal conductivity of pure HDPE and PP were measured using Flynn and Levin method and to examine the accuracy of the results obtained from this method, the obtained results compared to literature are shown in table 3 and in fig. 4.

The results shown in table 3 and fig. 4 are in good agreement with literature values; thermal conductivity decreases with increasing temperature.

Table 3 : Thermal conductivity of pure HDPE and pure PP results compared to the literature values

Polymer Type	Experimental Value	Literature Value*
	Thermal Conductivity (W/m. °C)	
HDPE	0.48	0.42 ~ 0.51
PP	0.15	0.1 ~ 0.22

*Source: (https://www.engineeringtoolbox.com/thermal-conductivity-d_429.html), Accessed August 2018

Thermal conductivity of HDPE and PP nanocomposites containing 2, 4, and 6 wt.% of clay were measured according to [12], the measurements were done at 122 °C . An increase in thermal conductivity was observed with the addition of the precursor to HDPE. The thermal conductivity increased from 0.48 W/m. °C for pure HDPE to 0.93 W/m. °C for the sample that contain 2% by weight of precursor. This increasing in HDPE it may be due to two reasons: (1) based on XRD results the partial exfoliated structure of 2% sample and intercalation in 4% and 6% nanocomposites (2) the compatibility between HDPE samples and the precursor which caused by forming strong bond and making thermal conduction pathways connecting between the clay layers and the HDPE samples.

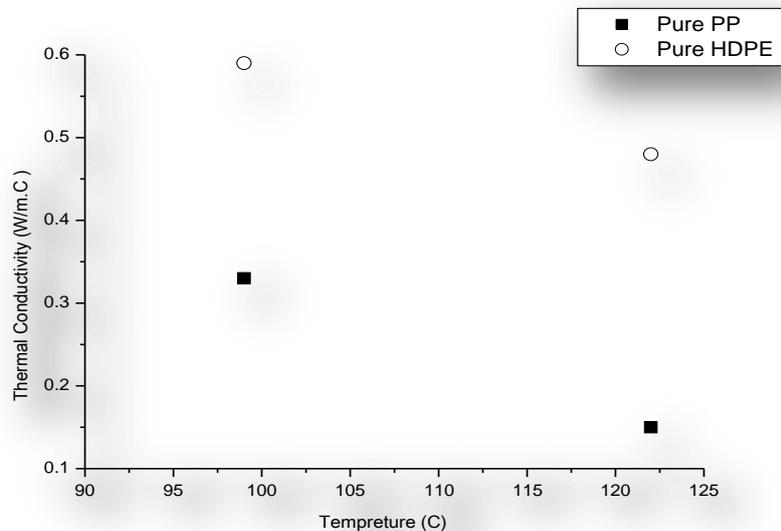


Fig. 4 The variation on thermal conductivity of pure HDPE and pure PP as a function of temperature

It worth to mention that all DSC measurements are based on the assumption of uniform temperature distribution within the sample and the reference. This is true for the high thermally conductive reference but may not be true for the low thermal conductivity sample as polymers. In this case, the measured temperature does not represent the true sample temperature and there will be a time lag in the measured temperature. Therefore, the estimated substantial value in the nanocomposite containing 2% of precursor could be attributed to char forming which composed of graphite, and the thermal conductivity of graphite is much greater than the thermal conductivity of polymers. Thus, large changes in thermal conductivity may be expected for these char forming polymers as they degrade. According to Flynn and Levin [12] ; very large number of polymeric materials form chars as a result of thermal or oxidative degradation.

For PP nanocomposites, the results showed a decrease in the values of thermal conductivity for all the samples. This phenomenon might be explained by: (1) forming filler clusters i.e filler/filler interactions, in those samples where clay agglomerate to fit into the small packing volume which increased the interfacial thermal resistance. As reported in reference (Huang et al., 2017) when the fillers aggregate, the contact area between the filler and the polymer matrix decreases dramatically, and the thermal conductivity also decreases. This phenomenon takes place in molecular level but the effect is macroscopically observed in the form of thermal conductivity reduction; (2) it is assumed also that the decrease in thermal conductivity is basically caused by the poor interaction between the precursor and PP matrix attributed to the immiscibility between the PP and PE in the compatibilizer (PE-g-MA) which leads to agglomeration. As reported, PP and HDPE are generally considered immiscible in the whole composition range and shows a remarkable phase separation during cooling/crystallization [16], [17] . In contrary, in the work of Lee et al. [18]; were they examines the feasibility of melt intercalation for preparing better exfoliated PE/OMMT nanocomposites with various compatibilizers (PP-g-MA and PE-g-MA) were MMT organically modified before being melt blended with the compatibilizer. They found that the clay in the nanocomposite synthesized with PP-g- MA were better exfoliated and resulting a better properties. The cause of this could interpret to either the treatment with the surfactant or the preparation condition including the fabrication method. Other research group [2] ; used MMT modified by quaternary ammonium salt before using PE-g-MA for preparing HDPE nanocomposites and PP-g-MA for preparing PP nanocomposites where they got intercalated and exfoliated structures. In fact, the majority of studies on the PP/clay nanocomposites have been carried out using (PP-g-MA) because it provides the best compatibility among all modified PP investigated so far [19] .

4.3 Model Predictions

The experimental data obtained within this study of HDPE and PP nanocomposites were used to investigate predictive capability of Mohaddespour and LN models for determining thermal conductivity of those nanocomposites.

For Mohaddespour model, the calculations were straight forward in the contrary to LN model, which require the consideration of maximum volumetric packing fraction ϕ_m and the shape factor A . A values are published in reference [20]; for many geometric shapes and orientation but platelet shape not included . Since we got no information about the dispersion state or the characteristic of the nanoparticles and no published papers mentioned a specific value for ϕ_m and A for kaolinite filler. A number of assumptions are inherent to this approach based on previous related works [21], [22] .

The shape factor A depends upon the shape of dispersed particles and how they oriented with respect to the direction of flow of the thermal currents [13]; and largely influenced by the Einstein coefficient of the filler K_E , which is related to the aspect ratio of the filler particle. Clay platelet was considered like a flake in the exfoliated structure and the thickness of the single clay platelet is only 1 nm, while the length dimension varies from 20 to 200 nm depending on the type of clay [23] ; which exhibit a high aspect ratio. Hence, in this study a flake shape was suggested for kaolinite and the current direction is parallel to the plane of flake orientation in one axis. By considering high aspect ratio ($l \gg t$) the value of A will be 1.5 .

The predicted results of the thermal conductivity obtained from Mohaddespour model and from LN model were compared with the experimental data for HDPE and PP nanocomposites as shown in the fig. 5.

The models results for HDPE nanocomposites didn't fit the estimated experimental data as shown in the figures below due to many factors. In general, for both models the major factor were the estimated values and the absences experimental value of kaolinite; where the kaolinite value for calculations was taken as 1.3 from literature [24]. Also, it can be seen that at low filler loadings, the dependence of composite thermal conductivity on filler content is nearly linear for both HDPE and PP samples in both models.

Mohaddespour model based on the assumptions of effective interface and homogenous dispersion of the filler which showing the limitation of this model to some very specific situation (e.g. monodispersed fillers in homogenous matrix), contrary to author's work itself [2]; which exhibits a reasonable agreement with their experimental work and no other literature used this model yet to make a comparison with.

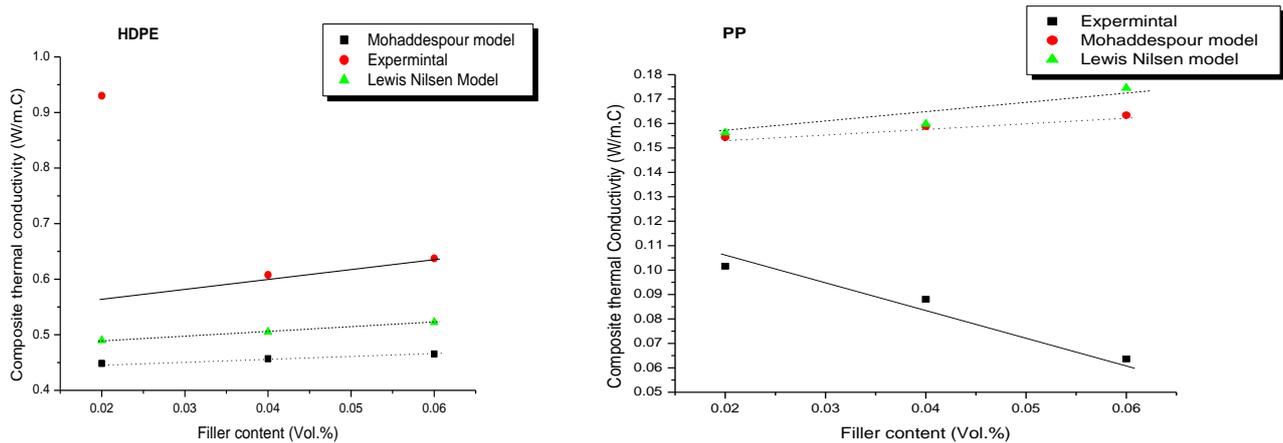


Fig. 5 Experimental and theoretical value of the thermal conductivity of HDPE and PP nanocomposites

The factor that influence the results obtained from *LN* beside the absence of the exact value of the thermal conductivity of kaolinite is the assumptions of *A* and ω_m . In conclusion, the deviation between the experimental and models results attributed to the missing information about the real molecular structure of the composite which is vital for the determination of related parameters used in the models. In PP composites case, the poor adhesion obtained experimentally will never fit those models because they are developed in order to predict a good adhesion between matrix and the filler.

2. Conclusions

The effect of clay content and fabrication route on thermal conductivity of HDPE and PP/ kaolinite nanocomposites are studied. Intercalated and partial exfoliation of HDPE nanocomposites were successfully fabricated using the melt blend method and PE-g-MA as compatibilizer as XRD analysis depicted. While no detectable change observed in PP composites structure. Thermal conductivity of pure HDPE and pure PP with their nanocomposites were measured using DSC. The inclusion of kaolinite improves the thermal conductivity of the HDPE nanocomposites but not for PP composites. An increase in thermal conductivity was observed with the addition of kaolinite to HDPE where precursor can act as a molecular bridge between the polar polymer (PE-g-MA) and polar clay layers, enhancing filler matrix adhesion and the dispersion of clay particles in polymer matrix. The optimum thermal conductivity 0.93 W/m. °C is obtained at 2 wt% clay content. However, the results obtained for PP showed a linear reduction in the thermal conductivity for PP composites attributed to the poor interaction between the filler and matrix caused by the immiscibility between PP and PE in the compatibilizer.

Approximations of Mohaddespour and LN models for HDPE nanocomposites showed a deviation from experimental values which could attributed to the missing information about the real molecular structure of the composite i.e dispersion of filler, which is vital for the determination of related parameters used in LN model and the absence of the exact value of the thermal conductivity of kaolinite. In PP nanocomposites case, the poor adhesion obtained experimentally will never fit those models because they were developed in order to predict a effective adhesion between matrix and a filler.

In addition the experimental values are not exact values. It must be concluded that the improvement in the thermal conductivity of HDPE nanocomposites can be related to a good dispersion of clay particles in the host polymer and this can be achieved through the optimizations of clay loading, modification approach and fabrication process.

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