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# Unmodified and Organo-Modified clay content effect on Mechanical and Thermal Properties of the Waste Low Density Polyethylene

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### ABSTRACT

The Organo modified and unmodified sodium montmorillonite clay effect on thermal and mechanical properties of the waste low density polyethylene (wLDPE) were studied. Commercialize unmodified (MMT) and Organo-modified clay (OMMT) were added to the wLDPE to prepare wLDPE-clay noncomposites by melt intercalation method. OMMT and MMT were added in a range of 1-5 wt %. Fourier transform infrared spectroscopy (FTIR) used to evaluate polymer structure before and after the fabrication. Thermogravimetric analysis (TGA) and Differential Scanning Calorimetry (DSC) were used to analyse the thermal stability and thermal properties for the wLDPE and fabricated nanocomposites. Tensile mechanical characteristics of the waste specimens before and after nanocompsite fabrication were evaluated. The FTIR exhibited no change in the chemical structure of the wLDPE used after clay addition. Melting temperature and crystallization percentage were increased up to 1 wt% loaded and decreased in with clay content increasing when compared to the original waste matrix. The thermal steadiness of the wLDPE /clay nanocomposites were found enhanced in the case of loading 3 wt% of OMMT. The elastic modulus has improved in the 3% OMMT loaded.

### Keywords: Unmodified clay, Organo-clay, Nanocomposites, wLDPE.

### INTRODUCTION

Polymers are currently distinguished as expanded and advanced applications , which are used widely in different fields in our social life such as electronic, agriculture, sporting industries and other various fields. Polymer is a significant category of material science and maintains as a development area in the coming decennary [1]–[3]. The recent times have witness the increased number of population. Accordingly, the demands made by the public as to enjoy their respective living conditions have led to a dramatically increasing number of polymer consumption (mostly plastics). [4], [5].

The manufactured quantities of plastic solid wastes escalate in no time, despite the growing efforts to minimize, reprocess, recycle and recover such great amount. This is primarily because of their extensive applications in the packaging production for the foodstuff manufacture in addition to other daily, everyday products simply because they do not possess any side effects that can be detrimental to human living beings. As well as, the period of life for the plastic scraps is very short (estimatedly, 40% have life duration lesser than thirty days). Nevertheless, because of the environment of considerably toxic plastic scraps, just part of them can be recycled into new products. Hence, the plastic wastes is slowly increased creating a serious environmental each year problem.[6]

Montmorillonite is regarded as one of the most frequent smectites, that is increasingly applied in various applications due to its powerful cation interchange capacity, swelling capacity, high surface areas, and resulting powerful adsorption[7].

Polymer–clay nanocomposites have been a remarkable field of study consideration. Different kinds of polymer matrixes (thermoset, thermoplastics, and rubbers) packed with clays have been investigated . In many of these studies, they concluded that the clays, (MMT)can be used as a nano-filler in the polymer matrixes and exhibiting enhancement of the properties (thermal, mechanical, and optical) even at very low concentrations of about 3–5 wt % [8]–[10].

Hasegawa et al. [11] and Kawasumi et al. [12] have reviewed polyolefin and polypropylene /clay hybrids based on altered polyolefin and organophilic clay.

Olewnik et al. [13], studied the formation of polyolefin / altered montmorillonite nanocomposites with 1.5–5 wt% clay improved with 4, 4'-methylenebisaniline to observe the thermal and structural properties of generated nanocomposites. It was reported that the

thermal stability of the nanocomposites can be enhanced furter, with a loading higher then 5 wt%.

Commercial and laboratory prepared linear low density Polyethylene (LLDPE), PE-g-MA these two grade were used by Wang et al. [14], melt mixing method was used for prepare of silicate nanocomposites, by laboratory prepared LLDPE-g-MA and LLDPE reacted with PE-g-MA. The application of LLDPE-g-MA as a compatibilizer for HDPE/OMMT system was studied by [15]. LDPE, a widely used packaging material, looks to be particularly splindid for nanocomposite preparation, commonly because of the anticipated enhancement of barrier features. Nevertheless, the details concerning such nanocomposites, with PE-g-MA, as a compatibilizer, is uncommon [16]. The activity of clay peeling in LDPE based nanocomposite in [16] was extermely worse than in HDPE based system due to a very complicated dispersion of branched LDPE macromolecules into clay galleries.

A comparative study on the physicochemical properties of polymer/clay composite and reported were performed Drozdov et al. [17], "There are many experimental studies morphology and mechanical properties of intercalated and exfoliated nanocomposites with HDPE matrices. The reinforcement of HDPE with MMT clay platelets results in an increase in the Young's modulus, and a decrease in elongation at break, respectively."

Continuous with our previous work recycling of the polyolefin using dissolution/reprecipitation technique [18]–[22]. In this study we explain the synthesis of waste waste low density polyethylene / Organ-modified and unmodified sodium montmorillonite nanocomposites by melt interaction method and explore the effect of clay content on the waste LDPE structure, thermal and mechanical properties. The characterization of the resulting polyolefin/clay nanocomposites will be evaluated by mean of FTIR. The mechanical of new nanocomposite sample using tensile testing, nanoindentation and the thermal properties will be analyses using TGA, DSC. The properties of the new wLDPE/clay nanocomposites will compare with the original waste polymer.

#### 2. EXPERIMENTAL PART

### 2.1 Materials

The waste low density polyethylene material was collected from houses trash, waste LDPE was general career used bags name in this study for easy treatment (bag), density 0.927 g/cm<sup>-3</sup>, melt flow index 0.841 g/ 10 min. The unmodified sodium montmorillonite and organo-modefied clay (Cloisite 20A) were commercial products are purchased from Southern Clay Products Inc., USA.

#### 2.2 Preparation of the nanocomposite

Polymer samples were prepared by cleaning using tab water and cutting into randomly small pieces using plastic crusher machine to 2-4 cm. the clay will add as 1, 2, 3 and 5 wt % to the waste polymer. Clay and waste polyolefin sample are blend in a Kitchen Aid type mixer about 15-30 minute after the waste polymer and clay are dried in oven at 60 °C separately at overnight. The mixture then is feed to an extruder for 20 minute as blending time. Twin-screw extruder (EuroLab 16 XL), co-rotating, screw diameter 16 mm, L/D=25 was used for the nanocomposites preparation, temperature for the extruder will set as 150 °C at the hopper to 190 °C in the die and the screw speed was 80 rpm.

#### 2.3 Characterization

FTIR study was performed by a Nicolet (Avatar 370 DTGS) FT-IR spectrophotometer with a resolution of 4 cm<sup>-1</sup>. The recorded wave number range was from 500 to 4000 cm<sup>-1</sup>. Small pieces of 1mm thickness were prepared for testing using a hand press, as specified for FTIR testing in ASTM D5477.

Melting temperature, heat of fusion and crystallinity of samples was measured by Differential Scanning Calorimetry DSC Q1000 (V9.6, Build 290) from TA instrument. The testing are performed according to ASTM D3418-03, with temperature interval 20-300 °C and a scan rate of 10 °C/min, in nitrogen atmosphere. Thermal stability for the samples before and after fabrication were determined thermogravimetric analyser (TGA) during heating at the rate of 20 °C/min from 20 to 800 °C, in dry Nitrogen. 25-30 mg samples were prepared for the testing purpose. The testing was carried out using a Universal V4.5A, TA Instruments.

The tensile mechanical properties were measured using Universal Testing Machine Shimadzu AG-X, according to the standard ASTM D638-03 (type I), with the speed of 5 mm/min. Samples were prepared by using a hydraulic thermal press under the following conditions: temperature, 190 °C, pressure, 10 MPa; time of heating, 15 min. The testing was done after 48-72 hours at room temperature with 55-60% humidity.

#### **3. RESULTS AND DISCUSSION**

#### 3.1 FTIR analysis

**Fig.1** shows FTIR testing, for the original wLDPE compared with the fabricated nanocomposites. No significant change was observed in the chemical structure or the characteristic bands for all the wLDPE samples used. Slight differences were recorded in wavenumbers, which is due to the additives present in small amounts in the waste products. However, the distinguishing peaks of the wLDPE/clay nanocomposites were found identical to the original waste LDPE peaks in all the cases.



Figure 1. FTIR spectra of waste wLDPE (bag) and wLDPE/clay nanocomposites.

#### **3.2** Differential Scanning Calorimetry

Table1 present the measured thermal features (melting point, fusion behaviour and crystallinity) of the wLDPE and nanocomposites samples with different clay loading. The percentage crystallinity C can be derived as the experimental heat of fusion of the sample tested divided by the heat of fusion of the 100% crystalline polymer. Experimental heat of fusion is calculated by measuring the area under the thermogram peak. Heat of fusion of the 100% crystalline were taken as 293 J/g LDPE [23], [24].

MMT and OMMT addition has minimal effects on the melting temperature of wLDPE samples. The melting temperature of pristine wLDPE was 112.8 °C which increased vey slightly to 113.9 °C in all clay percentage added. This phenomenon may be because of the functional nucleating effects of the silicate layers/ tactoids [25], [26].

**Table 1.** Melting temperature Tm, Heat of melting Hm, and degree of crystallinity C, of nanocomposites as a function of MMT and OMMT content.

Sample	Tm (c )		Hm/ Jg <sup>-1</sup>		C (%)	
	MMT	OMMT	MMT	OMMT	MMT	OMMT
bag	112.8		121		0.41	
bag-1%	113.9	113.9	104.8	129	0.36	0.44
bag-2%	113.67	113.3	96.1	120	0.33	0.41
bag-3%	113.78	113.3	92.3	104.5	0.32	0.36
bag-5%	113.64	113.7	88.9	95.2	0.30	0.32

The heat of fusion Hm for nanocomposites increases slightly, particularly at a minimum, 1 wt% OMMT loaded and decreases with further increase in clay addition compared with the value of pure wLDPE. In MMT addition Hm value decreased less than the pure wLDPE with increase in clay concentration. This phenomenon may be attributed to the "blockage of crystalline growth front caused by the clay platelets, dispersed in an irregular array in the nanocomposite, as it is evident in view of hot-stage microscopy studies"[27].

#### 3.3 Thermogravimetric analysis

The thermal degradation of the materials was studied using TGA. Onset temperature was estimated as the point in which 5% of the specimens is lost, the degradation at 10% of the sample lost also are taken in respect, and the final calculation of thermal degradation is the part of material which is non-volatile at 873 K, known as char [28]. **Fig.2**, present the TGA curves for neat wLDPE with fabricated nanocomposites containing 1, 2,3, 5 % wt of OMMT and 1,5 % wt of MMT, respectively. The temperature at weight loss 5 and 10 %, while the residue at 600 °C are presented at Table 2.



wLDPE /OMMT nanocomposites at 1-2 % wt clay exhibit a very high thermal stability as compared to neat wLDPE. The wLDPE /OMMT nanocomposites exhibit significantly enhanced the first thermal stability at 5, 10 % weight loss compared with pure wastes. The wLDPE /OMMT nanocomposite with 2 % wt clay, exhibits the highest initial thermal stability. When the OMMT content is rised more than 2 % wt, the first thermal stability reduction, but even in 3 and 5 % wt is still higher than pristine wLDPE, but it is less than that with sample loaded with 2 % wt content. The results in wLDPE/MMT nanocomposites were different, the temperatures at 5 and 10 % weight loss did not change as compared with pure wLDPE. This may be because of the "aggregates of clay, which are less effective in blocking heat than the MMT layers/tactoids, increasing with clay content" [26].

Sample	Temperatur	Residue at	
Sample	5%	10%	600 °C %
bag	413	428	0.62
bag 1% m	426	438	1.30
bag 2% m	430	440	1.73
bag 3% m	429	440	1.70
bag 5% m	428	440	2.17
bag 1%	417	432	1.83
bag 5 %	414	429	3.91

**Table 2.** TGA data for wLDPE and nanocomposites under nitrogen flow.

#### **3.4** Mechanical properties

wLDPE/clay nanocomposites mechanical properties were measured and compared with those of the pure wLPE. The elastic modulus, tensile tension at highest load strain at break and tensile stress at yield are show in **Fig.3**.

The measured modulus of pristine wLDPE was 150 Mpa, which increased significantly when modified clay (OMMT) was added from 1-3 wt % and were decreased at 5 wt % OMMT, in constract the unmodified clay (MMT) addition decreased the modulus. The maximum modulus were found as 207 MPa in wLDPE/ OMMT nanocomposites increase of 32 % relative to wLDPE. It has been reported that the elastic modulus increases when organo-clay is added in low content 1-3 wt %.





The OMMT is dispersed in the LDPE matrix at nanometre scale and part of the OMMT is intercalated by LDPE chains. This may have narrowed the segmental movement of LDPE macromolecules. [26], [29].On the other hand, the other three mechanical properties, tensile tension at highest load, strain at break and tensile tension at yield show in **Fig.3**, decreased with clay addition. In MMT content, it was observed that the decrease in values is higher than in the OMMT content. The significantly decreased mechanical properties at clay loading may be due to the uniformly dispersed MMT tactoid with intercalated structures [26].

### **4. CONCLUSION**

The melt intercalation method was used to fabricate waste LDPE clay (unmodified and organo-modified) nanocomposites. Modified and unmodified clay effected on the structure and characteristics were investigated perfectly. FTIR testing shows there is no significant change in the chemical structure of the polymer after clay adding. OMMT considerably affect the exfoliation and interaction of OMMT with in polymer matrix and there is no effect and intercalation by using MMT without any modification.

The DSC results show that in almost of the samples the melting temperature is remain as it is without observably change. The enthalpy of fusion was increased of nanocomposites at low OMMT content 1wt % in comparison with the pure wLDPE and decreased with clay content increasing. Same results were observed in the thermal behaviour of the nanocomposites there are enhancement at low OMMT content. The greatest elastic modulus in the LDPE nancomposites increased 34% relative to pure waste LDPE. Tensile tension at highest load, strain at break and tensile stress at yield were slightly decreased in the low OMMT clay content. Further increasing the content of clay would result in decreasing the mechanical properties of resultant composites.

It was concluded that by increasing the content of clay above 2% by weight a decline in the mechanical properties of the resultant composites. However, we can conclude from this study that the recycling of the waste polymer using dissolution/reprecipitation method was better than reinforcement of the waste LDPE by nanocomposites fabrication in melt interaction method.

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تأثير مكونات الطين غير المدعم والمدعم عضويا على الخصائص الميكانيكية والحرارية لمخلفات البولي اثبلين الواطئ الكثافة

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### الخلاصة

في هذا البحث تم در اسة تأثير الطين المدعم عضويا وغير المدعم على الخصائص الحرارية والميكانيكية لمخلفات البولي اثيلين الواطئ الكثافة (wLDPE). حيث تم اضافة الطين غير المدعم المستخدم تجاريا (MMT). حيث تم اضافة الطين غير المدعم المستخدم تجاريا (MMT) clay والمدعم عضويا (OMMT) والمدعم عضويا (Organo-modified clay (OMMT) الى مخلفات البولى اثيلين الواطئ الكثافة (wLDPE) لغرض تحضير المركبات النانوية الطين – مخلفات البولى اثيلين الواطئ الكثافة (wLDPE-Clay nanocomposite)باستخدام تقنية الاذابة بالأقحام Melt Intercalation Method كما تم اضافة OMMT و MMT بنسب وزنية تتراوح بين (%5wt).

استخدمت تقنية تحليل طيف الاشعة تحت الحمراء (FTIR) لاثبات تركيب البوليمر قبل وبعد التصنيع. استخدمت تقنية التحليل الحراري الوزني (TGA) و Differential Scanning Culorimetry (DSC) لتحليل الثبات الحراري والخصائص الحرارية لمخلفات البولي اثيلين والمركبات المحضرة في هذه الدراسة. تم قياس الخصائص الميكانيكية لنماذج wLDPE قبل وبعد تحضير .wLDPE- Clay nanocomposite

أظهر طيف FTIR عدم وجود اي تغير في التركيب الكيمياوي ل wLDPE المستخدمة بعد اضافة الطين. الا ان حرارة الانصهار (Hm) ونسبة التبلور (C%) ارتفعت عند اضافة الطين بنسبة 1wt%. بينما انخفضت عند زيادة نسبة مكونات الطين المضافة عند مقارنتها بمخلفات البولى اثيلين الواطئ الكثافة قبل اضافة الطين وقد اظهرت الدراسة ايضاً بأن الثبات الحراري ومعامل المرونة لمركبات wLDPE-clay nanocomposite قد تحسنت بعد اضافة wt% of OMMT المرونة لمركبات wLDPE.

الكلمات الرئيسية: الطين غير المدعم، الطين المدعم عضوياً، المركبات المتناهية في الصغر (النانوية)، مخلفات البولي اثيلين الواطئ الكثافة.