



Modification of Egyptian Clay by Different Organic Cations

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NOWADAYS, clay minerals are used extensively in a wide range of applications as nano additives for polymeric materials. Nevertheless hydrophilic characteristics of the nano additives reduce their degree of compatibility with polymeric chains. To overcome this problem it is necessary to modify the clay in order to render its surface more organophilic prior to the intercalation of the polymeric chains between its layers. In this work the Egyptian natural clay mineral montmorillonite (MMT) was modified with two cationic organic modifiers, namely hexadecyltrimethylammonium (HDTMA) and tributyl hexadecyl phosphonium using two different procedures. The first, using the magnetic stirrer while the second using a high speed mixer at different mixing times from 0.5 up to 2.5 hr. The samples with and without organic modifiers were characterized by X-ray diffraction (XRD), infrared spectroscopy (IR), thermogravimetric analysis (TGA), scanning electron microscope (SEM) and transmission electron microscope (TEM). XRD results revealed that the basal spacing of the organo-montmorillonite (OMMT) prepared using high speed mixer was larger than that using magnetic stirrer. The best result was obtained after 1.5 hr stirring in the high speed mixer. The modification of MMT was confirmed by FTIR as determined from $-CH_2$ stretching vibration of the organic modifiers. The TGA analysis revealed that the MMT modified with tributyl hexadecyl phosphonium cation had high thermal stability than that modified with hexadecyl trimethyl ammonium cation. The amount of surfactants within organoclays were found to be about 19 and 24%, respectively, for hexadecyl trimethyl ammonium bromide and tributyl hexadecyl phosphonium bromide. SEM demonstrated that the modified MMT exhibited a massive thin layered structure with some large flacks and some interlayer spaces. TEM images of the prepared organoclays showed exfoliated structure. This work proved to enhance the economical value of the Egyptian natural resources.

Keywords: Egyptian mineral montmorillonite, Organic modification, Basal spacing, High speed mixer.

Introduction

Egypt is rich with mineral resources which when treated in an appropriate manner are converted to materials with high economic value that can be used in several industrial applications. One of these natural resources is the smectite clay which covers most of the eastern and western deserts [1]. Montmorillonite (MMT) is one member of the smectite clays that is commonly used as nano

additives in small percentage in the synthesis of clay polymer nanocomposites [2-6]. These polymer nanocomposites have a wide range of applications in the field of material science and technology [7-11] due to the enhanced thermal [12,13], physical and mechanical properties [14]. Also the barrier and flame retardant properties of the produced nanocomposites are improved [15]. MMT has 2:1 layered structure as each particle consists of hundreds or thousands of

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layers stacked to each other with weak Van der Waals...forces; each layer consists of two tetrahedral sheets of silica sandwiching one octahedral sheet of alumina. These layers are separated from each other by interlayer spacing containing exchangeable earth cations [16, 17]. In order to obtain polymer clay nanocomposites with enhanced properties, the MMT layers must be well dispersed within the polymer matrix; this depends on the degree of compatibility between these layers and the polymer used. MMT itself is naturally hydrophilic; this makes it not compatible with most hydrophobic polymers [7]. For this reason pretreatment of MMT is necessary to render its surface more organophobic and hence more compatible with polymeric chains. The most common method of MMT modification is carried out via exchanging the interlayer cations with organic cations such as organic ammonium salts [18, 19] and organic phosphonium salts [20, 21]; this increases the interlayer spacing between MMT layers, increases its hydrophobic character [7] and thus increases its compatibility with polymer matrix. The MMT modified by this method is called organoclay (OMMT). Several procedures were followed for the organic modification of MMT [22]; that take several days to complete the modification process using the magnetic stirrer.

In the present work, the Egyptian mineral MMT was modified by two different methods; the first using magnetic stirrer and high speed mixer at different mixing times (0.5, 1, 1.5, 2, 2.5 hr). In both cases two different organic surfactants were used; hexadecyltrimethyl ammonium bromide and tributyl hexadecyl phosphonium bromide. The structure of the obtained organo-montmorillonites was characterized in terms of interlayer expansion (basal spacing), thermal stability and morphology.

Experimental

Materials

MMT was supplied from EGYPT NANO TECHNOLOGIES CO., Egypt, with cation exchange capacity (CEC) = 82 meq/100 g. This MMT was used as such without any further purification. The chemical analysis of MMT was performed on dried powder sample using XRF instrument and the results are given in Table 1. The two surfactants used; hexadecyltrimethyl ammonium bromide 98% purity, and tributyl hexadecyl phosphonium bromide 97% purity were purchased from SIGMA-ALDRICH and used as received, their molecular structure are shown in Fig. 1. Ethanol solvent was purchased from El Gomhoria Company of chemicals, Egypt.

TABLE 1. Chemical analysis of MMT using XRF instrument.

Analyte	Na ⁺	Mg ²⁺	Al ³⁺	Si ⁴⁺	P	S	K ⁺	Ca ²⁺	Ti ²⁺	Fe ³⁺	Cl ⁻
Concentration (%)	3.691	2.753	15.317	45.616	0.145	0.431	1.173	1.718	1.29	9.93	0.99

Hexadecyltrimethyl ammonium bromide

Tributyl hexadecyl phosphonium bromide



Fig. 1. Molecular structure of the used surfactants.

Organoclay Preparation

Modification of MMT was undertaken using two different organic surfactants applying two different procedures; the first using magnetic stirrer and the second procedure using high speed mixer.

First procedure

15 g of Egyptian sodium MMT was dispersed in 1500 mL of distilled water and stirred using a magnetic stirrer for 24 hr at room temperature (clay:water /1:100). After that molar equivalent of surfactants hexadecyltrimethyl ammonium bromide or tributyl hexadecyl phosphonium bromide surfactant dissolved in aqueous ethanol (70%) was slowly added to the clay suspension. The reaction mixtures were stirred for 14 h at room temperature. The obtained products were washed several times with distilled water until no halide ion was detected in the filtrate using 0.1M AgNO₃ solution. The product was dried at 80°C for 48 h, ground in an agate mortar and sieved to a size less than 100 µm particles.

Second procedure

15 g of Egyptian sodium MMT was dispersed in 1500 mL of distilled water and stirred in a laboratory high speed mixer for 1 hr at 3000 rpm. A solution containing the equivalent amount of surfactant dissolved in aqueous ethanol was then added to the MMT suspension. Five experiments were conducted at five different stirring times of the mixer 0.5, 1.0, 1.5, 2.0 and 2.5 hr. The products were then filtered, washed several times with distilled water until no halide traces were detected. The samples were then dried at 80°C for 48 hr. Finally, organically modified samples were grinded with ground in an agate mortar and sieved to a size less than 100 µm particle size.

The obtained organically modified samples are named here as OMMT1-A, OMMT1-B, for the MMT modified with hexadecyl trimethyl ammonium bromide surfactant by using a magnetic stirrer and a high speed mixer, respectively, and OMMT2-A and OMMT2-B representing the MMT modified with tributyl hexadecyl phosphonium bromide surfactant using the same procedures.

Characterization

Infrared Spectroscopy

MMT, OMMT1-B and OMMT2-B were characterized using Fourier transform infrared spectroscopy (FTIR). The experiment was conducted using IR Affinity-1 instrument manufactured by SHIMADZU CORPORATION (Japan) on KBr disc in the 400–4000 cm⁻¹ range.

X-ray Diffraction

X-ray diffraction patterns of the fine solid powder were obtained using a BRUKER x-ray diffractometer D8 Advance with Cu K_α radiation ($\lambda = 1.54 \text{ \AA}$) operated at 40 kV and 40 mA. The experiments were run at room temperature with an angle range (2θ) from 3° to 60° at 4° min and step size of 0.02°.

Thermogravimetric analysis

The weight loss of MMT, OMMT1-B and OMMT2-B which arises from the degradation of these samples under heating, was measured by SDT Q600 instrument. Samples of 6 - 8 mg were measured at a temperature range from ambient to 800°C at a rate of 10°C/min. The derivative weight loss percents of the three samples were studied versus temperature to show any changes occurring in the samples under heating.

Scanning electron microscope (SEM)

The Environmental Scanning Electron Microscope SEM- Quanta FEG-250 was used to show the difference in the morphology of MMT, OMMT1-B and OMMT2-B at a voltage of 20 kV and a magnification of 2×10^4 .

Transmission electron microscope (TEM)

TEM images were used to visually evaluate the degree of intercalation of clay layers and the amount of aggregation of clay clusters in each of the MMT, OMMT1-B and OMMT2-B samples. The images were taken by JEOL, 2100HR instrument which is a high resolution instrument operating at 200 kV.

Results and Discussion

Infrared spectroscopy

Typical FT-IR spectra of neat MMT, OMMT1-B and OMMT2-B samples are shown in Fig. 2. In the spectrum of MMT shown in Fig. 2 (a), the Si–O and Al–O bonds are observed at 1031 cm⁻¹ and 911 cm⁻¹, respectively. The strong absorption peak at 1642 cm⁻¹ and the broad band at 3626 cm⁻¹ are assigned to the bending and stretching modes of absorbed water. Sharp peaks around 3402 cm⁻¹ are assigned to the hydroxyl group. For OMMT1-B, two new peaks were appeared at the absorption bands 2926 and 2852 cm⁻¹ and for OMMT2-B at 2941 and 2852 cm⁻¹; these peaks are due to the C–H asymmetric and symmetric stretching vibrations, respectively, of the -CH₂- in the aliphatic chains of the organic surfactants, indicating the organic modification of MMT [23,24].

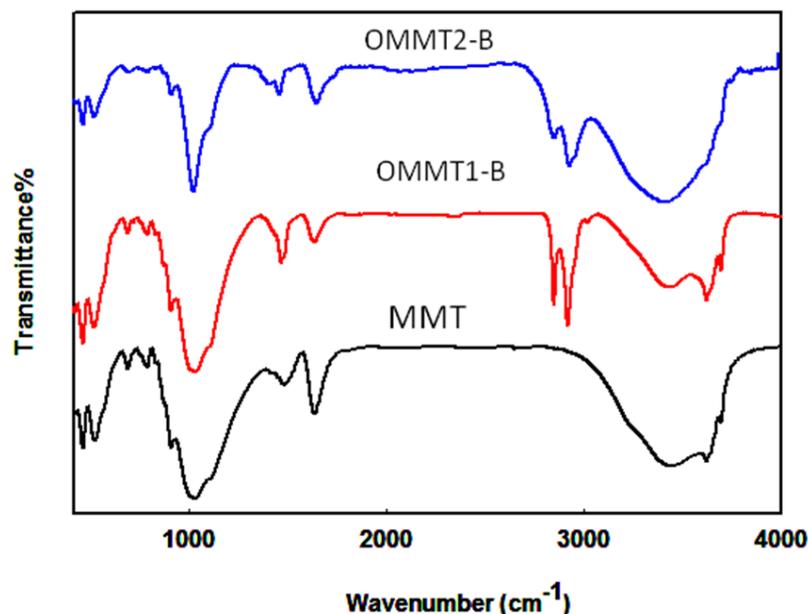


Fig. 2. FT-IR spectra of MMT, OMMT1-B and OMMT2-B.

XRD

The diffraction patterns of original MMT without any modification, OMMT1-A, OMMT1-B, OMMT2-A and OMMT2-B at 1.5 hr time reaction are shown in Fig. 3. The basal spacing of all these samples is given in Table 2. As can be seen from Table 2, the basal spacing of unmodified MMT was 13.3 Å ($2\theta = 6.62$), the basal spacing of OMMT1-B and OMMT2-B are 27.30 Å ($2\theta = 4.07$) and 27.79 Å ($2\theta = 3.18$), respectively, while the basal spacing of OMMT1-A and OMMT2-A are 19.20 Å ($2\theta = 4.50$) and 18.55 Å ($2\theta = 4.76$), respectively. These results indicate that the modification process with either the high speed mixer or the magnetic stirrer increases the value of the basal spacing of the unmodified MMT and shifts it to lower angles proving that the modifier chains have intercalated into the layers of MMT and change its structure. But in case of the modification using the high speed mixer the values of the basal spacing are greater than that in case of the modification using the magnetic stirrer and appears at lower angles, indicating that the clay layers are more separated from each other and hence this increases the ability of the polymeric chains to be intercalated or exfoliated between the clay layers in the polymer/clay nanocomposites synthesis process.

The diffraction patterns of different OMMT1-B samples modified using high speed mixer at different mixing times are shown in Fig. 4. The basal spacing of these samples is given in Table 3. It can be seen from Table 3 that the basal spacing increases with increasing mixing time reached a maximum of 27.30 Å after 1.5 h mixing time. This value decreased with increasing mixing time up to 2.5 h. From these results we conclude that the best mixing time for the organic modification of MMT using the high speed mixer is 1.5 hr at which the highest value of basal spacing is obtained.

Thermogravimetric analysis

TGA gives information about the difference in the thermal stability between the original clay MMT and the organically modified samples OMM1-B and OMMT2-B. Typical TGA weight loss curves and their corresponding DTG curves of the unmodified MMT and the organically modified clays OMMT1-B and OMMT2-B are shown in Fig. 5. The mass loss of the MMT occurs via two steps. The first mass loss takes place around 67°C is attributed to the desorption of adsorbed water molecules from the clay and the second mass loss step around 465°C is due to the loss of the structural hydroxyl groups from the clay structure [22]. However in case of organically modified samples (OMMT1-B,

OMMT2-B) the decomposition takes place in four steps due to the presence of the organic cations. For OMMT1-B, the first decomposition step takes place between ambient temperature and 85°C, which is due to water desorption, the second step is from 269°C up to 340°C due to the dehydration of the hydrated cations in the interlayer spacing, the third step is at 421°C due to the removal of the surfactant while the fourth step is at 634°C which is assigned to dehydroxylation of the structural hydroxyl groups in the original clay structure. In case of OMMT2-B the first decomposition step is from ambient to 63°C, the second at 348°C, the third

at 475°C and the fourth step is at 990°C these steps are due to water desorption, dehydration of the interlayer cations, loss of the surfactant and the dehydroxylation of the clay structure, respectively [25]. The OMMT2-B exhibited a higher onset decomposition temperature of 255°C compared to the OMMT1-B with an onset decomposition temperature of 210°C. Thus, the thermal stability of phosphonium organoclay is higher than that alkyl ammonium organoclay. The organic content of the OMMT1-B and OMMT2-B was found to be about 19 and 24 %, respectively.

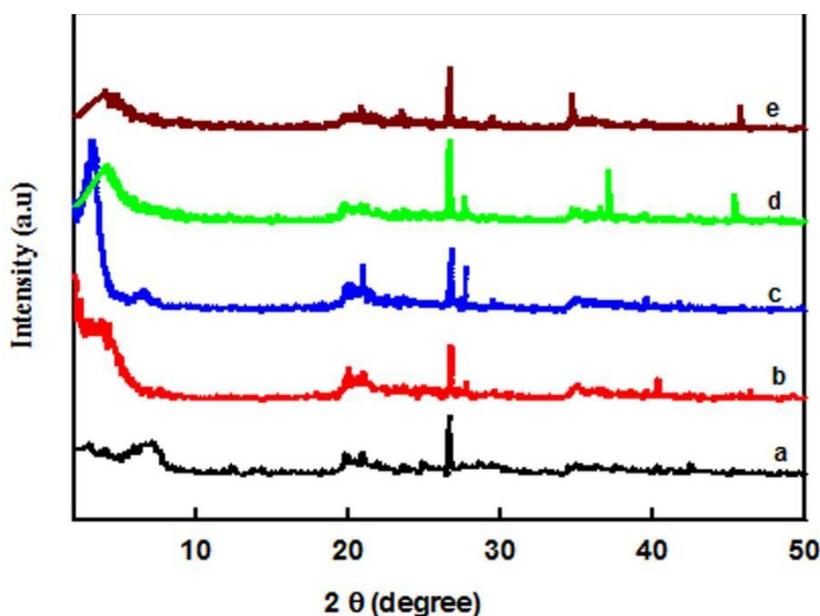


Fig. 3. XRD comparison between (a) MMT, (b) OMMT1-B, (c) OMMT2-B (d) OMMT1-A and (e) OMMT2-A.

TABLE 2. Comparison between the basal spacing of MMT, OMMT1-B and OMMT2-B and OMMT1-A& OMMT2-A.

Sample code	Modification method	Basal spacing (\AA)	2θ ($^\circ$)
MMT, a	•	13.30	6.62
OMMT1-B, b	High speed mixer	27.30	4.07
OMMT2-B, c	High speed mixer	27.79	3.18
OMMT1-A, d	Magnetic stirrer	19.20	4.50
OMMT2-A, e	Magnetic stirrer	18.55	4.76

TABLE 3. Basal spacing of OMMT1-B at different mixing times in the high speed mixer.

Time of mixing (hr)	d-spacing value (Angstrom)	2 θ (degree)
0.5	25.84	4.290
1.0	22.34	4.960
1.5	27.295	4.065
2.0	24.39	4.549
2.5	15.08	7.357

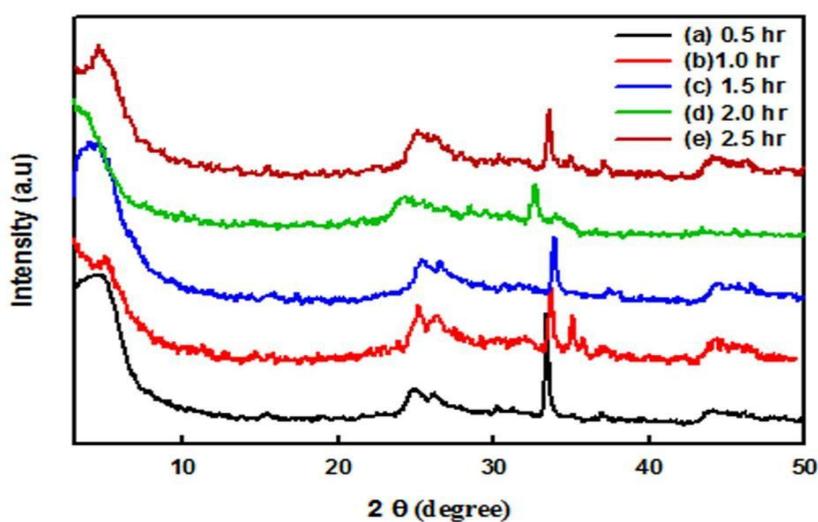


Fig. 4. XRD patterns of OMMT1-B at different mixing times of the mixer (a) 0.5 hr, (b) 1 hr, (c) 1.5hr, (d) 2hr and (e) 2.5 hr.

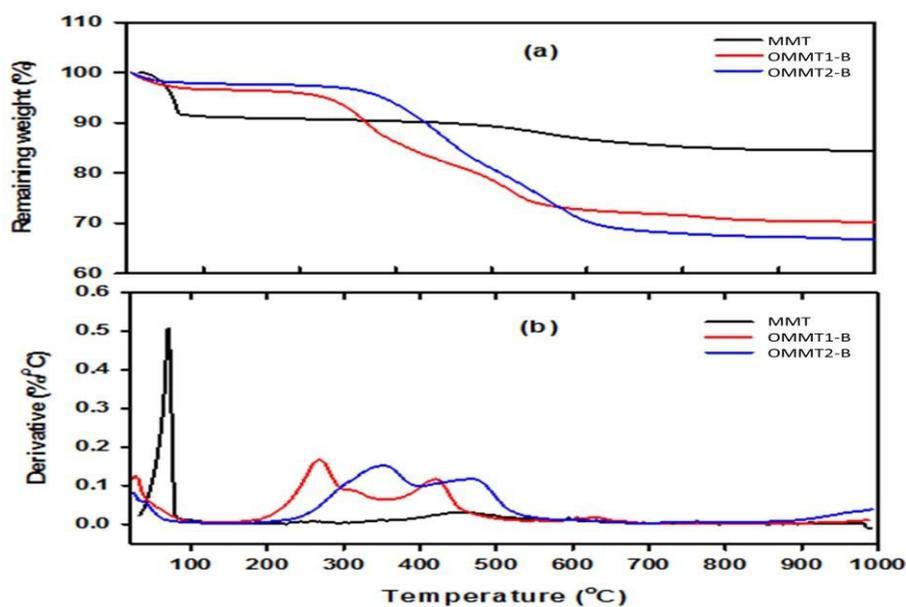


Fig. 5. TGA and DTG curves of MMT, OMMT1-B and OMMT2-B.

SEM

The SEM micrographs of the unmodified MMT, OMMT1-B and OMMT2-B are given in Fig. 6. From this figure it can be seen that the structure of modified MMT exhibited a massive thin layered structure with some large flacks and some interlayer spaces; this is probably due to the chemical modification by large alkyl ammonium or alkyl phosphonium moieties in the clay galleries.

TEM

Transmission electron microscopy analysis is crucial in order to support the X-ray diffraction analysis. The TEM micrographs of all MMT, OMMT1-B and OMMT2-B are shown in Fig. 7. The presence of the interlayer silicates is observable with particles of hexagonal shape and dimensions of the order of 100 nm. The image of prepared organoclays shows the exfoliated layer, which provides an efficient interface with the polymer chains when melted with it to prepare polymer clay nanocomposites.

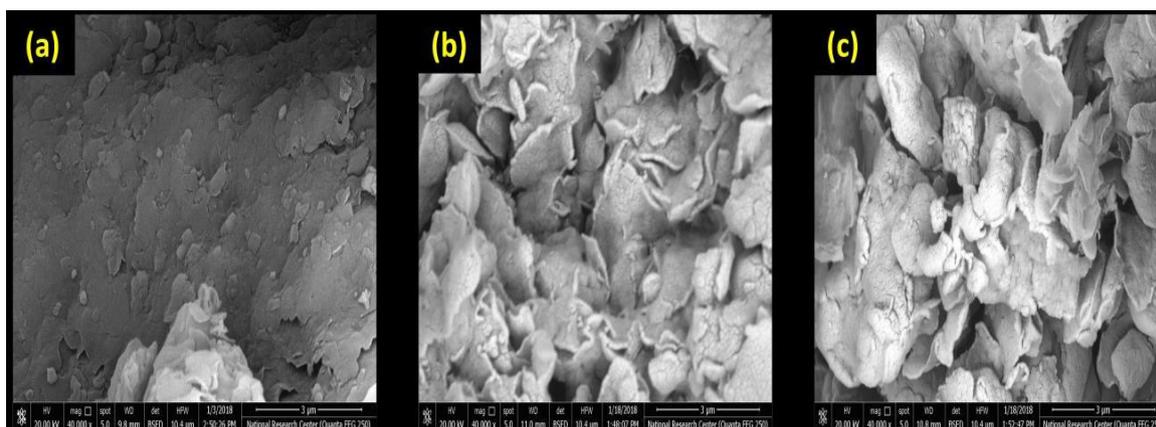


Fig. 6. SEM micrographs of (a) MMT, (b) OMMT1-B and (c) OMMT2-B.

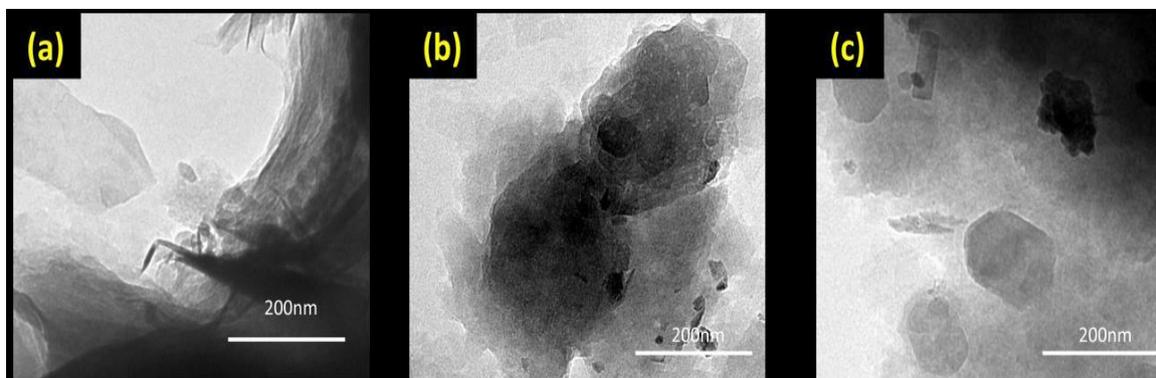


Fig. 7. TEM micrographs of (a) MMT, (b) OMMT1-B, (c) OMMT2-B all at magnification of 200nm.

Conclusions

The Egyptian clay MMT has been successfully modified by two different organic surfactants; hexadecyl trimethyl ammonium bromide and tributyl hexadecyl phosphonium bromide using two different procedures via magnetic stirrer and high speed mixer. XRD results showed that the values of the basal spacing in case of modification using high speed mixer are greater

than those obtained using magnetic stirrer and increased with the time of mixing up to 1.5 h. The TGA analysis showed that the prepared organoclays exhibited three steps of weight loss; the first one is due to desorption of the adsorbed water, the second is assigned to dehydration of the bound water and the third degradation step is related to the decomposition of the organic modifier. In addition, the MMT modified with tributyl hexadecyl phosphonium cation was found

to be more thermally stable than that modified with hexadecyl trimethyl ammonium cation. The organic content of the prepared organoclays using hexadecyl trimethyl ammonium bromide and tributyl hexadecyl phosphonium bromide surfactants were found to be about 19 and 24%, respectively, using efficient speed mixer method. Both SEM and TEM confirmed that the organic modification of clay causes an increase of the basal spacing between its layers and hence increase the ease of intercalation of polymer chains between these layers in the polymer clay nanocomposites synthesis process.

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تحسين الطفلة المصرية باستخدام الكاتيونات العضوية المختلفة

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في الوقت الحاضر، يتم استخدام المعادن الطينية على نطاق واسع في التطبيقات كمواد مضافة في حجم النانو للمواد البوليمرية. ومع ذلك، فإن الخصائص المحبة للماء للمواد المضافة بحجم النانو تقلل من درجة توافقها مع السلاسل البوليمرية. وللتغلب على هذه المشكلة كان من الضروري تعديل الطفلة لجعل سطحها اليف عضويًا قبل دخول السلاسل البوليمرية بين طبقاتها. في هذا العمل تم تعديل الطفلة المصرية الطبيعية مونتومورونيوت (MMT) باستخدام اثنين من المعدلات العضوية وهما سداسي الديسيل ثلاثي الميثيل امونيوم و ثلاثي البيوتيل سداسي الديسيل فوسفونيوم باستخدام طريقتين مختلفتين. الأولى، باستخدام القلاب المغناطيسي والثانية باستخدام خلط عالي السرعة في أوقات خلط مختلفة من ٠.٥ إلى ٢.٥ ساعة. وقد تم تحليل كلا من العينات التي تحتوي والتي لا تحتوي على محسنات عضوية بواسطة تحليل حيود الأشعة السينية، التحليل الطيفي باستخدام الأشعة تحت الحمراء (IR)، التحليل الوزني الحراري (TGA)، ماسح المجهر الإلكتروني (SEM) و مساح انتقال المجهر الإلكتروني (TEM). و أظهرت نتائج حيود الأشعة السينية أن التباعد بين طبقات المونومورونيوت العضوي المحضر باستخدام الخلط عالي السرعة كان أكبر من المحضر باستخدام القلاب المغناطيسي وكانت أفضل نتيجة بعد مضي ١,٥ ساعة في الخلط عالي السرعة. تم تأكيد تعديل MMT بواسطة FTIR كما هو محدد من تمدد الاهتزاز الخاص بـ CH₂ من المعدلات العضوية. أظهر تحليل الوزن الحراري أن MMT المعدلة مع الكاتيون ثلاثي بوتيل الفوسفونيوم ثلاثي البيوتيل لديه ثبات حراري أعلى من ذلك المعدل مع سداسي ميثيل إيثيل الأمونيوم الموجب. وقد وجد ان كمية المادة العضوية داخل المعدل الطيني المعدل تساوى حوالى ١٩ و ٢٤٪، على التوالي، لبروميد سداسي ميثيل الأمونيوم وبروميد سداسيكل فوسفونيوم ثلاثي بوتيل. أثبت تحاليل ماسح المجهر الإلكتروني أن MMT المعدلة اكتسبت تراكيب رقيقة ضخمة فشرية وبعض المساحات البينية. أظهرت الصور TEM للمواد المحضرة organoclays بنية تقشرية. وقد أثبت هذا العمل تعزيز القيمة الاقتصادية للموارد الطبيعية المصرية.