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Research Article

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The Characteristics of Poly Propylene Oxide/Montmorillonite Nanocomposites

Lahouari Mrah*, Rachid Meghabar and Mohammed Belbachir

Laboratory of Polymer Chemistry, Department of Chemistry, Faculty of Science, Université D'Oran, Algeria

Abstract

The aim of our study is based to produce the Poly propylene oxyde /clay nanocomposites [3,5,7 and 10% (w/w) Maghnite - CTAB based on the propylene oxyde content] were synthesized by in situ polymerization. Maghnite-CTAB is montmorillonite-CTAB silicate sheet clay was prepared through a straight forward exchange process, polymer composites based on modified montmorillonite (montmorillonite-CTAB) and Poly Propylene Oxyde were prepared with different compositions by melt processing. The maghnite used was obtained with a cation exchange, using a green natural clay from Maghnia which is situated in the west of Algeria. This work is based also to demonstrate a morphology, which is obtained by combining AFM and MEB. The polymer composites were characterized using differenttechniques such as X-ray diffraction (XRD), differential scanning calorimetery (DSC), infrared spectrophotometery (IR),and Microscopic electronics with sweeping (MEB) and Atomic force microscopy (AFM). The results were showed that, the basal space of the silicate layer increased, as determined by XRD, from 12.97 A° to 32.60 A°. The addition of PPO shows distribution of platelets perparticules, and improve the interaction between clay and polymer matrix. The microstructure was detected by X-ray patterns and Microscopic electronics with sweeping (MEB) and Atomic force microscopy (AFM) at 5wt% MMt-CTAB, however, higher than 3 wt% MMt-CTAB reveals partial intercalation structure. The results confirm the presence of several intercalation of molecules salt in the clay layers, and it also shows a good interaction with the polymer.

Keywords: Montmorillonite; Nanocomposites; Modified Montmorillonite-CTAB; Cetyltrimethylammonium bromide; X-ray diffraction

Abbrevations: PO: Propylene Oxyde; PPO: Poly (Propylene Oxyde); NCPO: Poly (Propylene Oxyde)/Montmorillonite Nanocomposites; CTAB: Cetyltrimethylammonium Bromide; C: molar concentration Mol L^{-1}; T: Temperature °C; T: time S; Tg: The glass transition

Introduction

Polymer-organoclay nanocomposites are considered a new classof advanced organic-inorganic materials. The subject of nano composite is of great vitality and offers immense opportunity because a variety of compounds can be synthesised by combining a wide variety of host materials as well as polymers. These composites are formed through the union of two different materials with organic and inorganic pedigrees. The integration of a second component into a host system allows for the combination of physical or chemical properties that can not be achieved by modification of the matrix alone. The unmodified clays such as montmorillonite are hydrophilic and therefore incompatible with a wide range of hydrophobic polymers including epoxy resins. Polymer melt intercalation presents a great advantage in production of PPO over conventional polymer processing techniques. Compared to other synthesis methods, it is generally the most used, and environmentally friendly (no solvent is required) technique. Applicable to a variety of polymers. The silicate has been known for a long time, the field of polymer nanocomposites has grown to recently organically have important application in several fields [1-3]. Two morphologies of polymer/clay nanocomposites have been widely studied: intercalated and exfoliated structures. The different layers of a clay/exfoliated nanocomposites of 1nm thickness are dispersed in a continuous polymer matrix. Exfoliated structure consists of nanometer-thick silicate layers dispersed in a polymer matrix, which result from extensive penetration of thepolymer within the interlayer spacing initiator. Intercalated structure is in general obtained whenthe polymer is located between the silicate layers, even though the layer spacing increases. The nanocomposites polymer/clay were concentrated on mainly for the strengthening and the improvement of property of the barrier and the flammability of polymer systems [4-7]. Later studies on conductive polymers and liquid crystal polymers and polyester oxide [8-10]. based Montmorillonite is a naturally occurring clay which has the formula AL_2O_3, $SiO_2HOH + nH_2O$ [11]. The compatibility between the clay and non polar polymer hydrophilic clay surface which can be modified by ion-exchange reaction with a quaternary alkylamonuim salts to reduce its polarity and then an organophilic clay is obtained, such as organomodified montmorillonite to messersuith and giannelis [12-20]. We used a method to prepared PPO/montmorillonite nanocomposites. In the present work, Algerian montmorillonite was organophilized and tested to prepare nano-composite by melt blending. These results are really encouraging compared to other clays.

Materials and Methods

Materials

Propylene Oxyde'(po) (Reagent Plus, ≥ 99%) was supplied by BASF and cetyltrimethylammonium bromide (CTAB) were used as purchased from Aldrich Chemical, The sodium montmorillonite was provided by Maghnia plant, Algeria, and used without further

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***Corresponding author:** Lahouari Mrah, Laboratory of Polymer Chemistry, Department of Chemistry, Faculty of Science, Université D'Oran, BP 1524 31000 El'Menouer Oran, Algeria, Tel: Fax: 213-41510057; Fax: 213-41510057; E-mail: lmrah@yahoo.fr

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purificatio. Raw-Maghnite: Algerian montmorillonite clay which has been used as catalyst % (Table 1).

Preparation of montmorillonite-Na: After a grinding and a fine sieving, the benthonite is put in touch with the salt, NaCl (1M, to make an exchange of the present ions within the clay by the cations Na^+), then left under agitation during 12m, this operation is 03 times repeated, then the clay has sudden a wash several times in the water distiller to eliminate the excess of the salt, (haste of the ions Cl- by $AgNO_3$). The montmorillonitics suspensions are put in cylinders increased in sedimentation (test tube of 2 L) kept at room temperature during 48 hours. Then the suspension is siphoned by inhalation, by means of a pipette to get back the fraction montmorillonitics the size of particles of which is around 2 µm. After every tacking, we re- shake the remaining fraction and we begin again. The operation completes as often as possible, and then the collected montmorillonite is dried in 100°C and finally crushed with a mortar [21].

Organic modification of clay: We prepared our montmorillonite modified according to the protocol of Loïc Lepluart [22]. Introducing us into a phial gauged by a liter, 10 ml of hydrochloric acid 1N. The volume is completed in the line of capacity with some distilled water, the solution paid into an Erlenmeyer containing a magnetic baron.

This acid solution is carried in the temperature in which we wish to realize the cationic exchange (80°C for hi it proceeded optimized), on a heating magnetic agitator. When the temperature is stable, introducing us 10^{-2} moles of the cetyltrimethylammonium bromide. After three hours of agitation in 80°C, Amine is dissolved and ionized. Introducing us 5 g of seeds montmorillonite. After three hours of cationic exchange, we recover of the montmorillonite organophile. The latter is then six times successively by some water detailed in 80°C to eliminate the inorganic cations. The efficiency of rinsings is verified by the addition of some drop of silver nitrate in the residue of the rinsing. The alkylmmonium physisorbées ions were eliminated by a mixture water /ethanol (50/50) beforehand warmed in 60°C. The montmorillonite organophile obtained is dried in 100°C, then crushed. The latter is named in court of this study Montmorillonite -CTAB. In this paper, we call the cation-exchanged montmorillonites, where n is the carbon number of Montmorillonite [23].

Polymerization procedure: The polymerization of propylene oxyde was carried out in tubes. Each tube contained a mixture of 10.0 g of propylene oxyde and an amount of Maghnite-CTAB. Four types of composites of different compositions (3, 5, 7 and 10 wt% MMt-CTAB) were prepared by melt compounding. The mixtures were held at under ambient temperature and stirred with a magnetic stirrer, the reaction was terminated by precipitating the PPO/montmorillonite, and the monomer conversion was determined gravimetrically by weighing the precipitated materials. In this method, the liquid monomers or polymers are intercalated into clay layers and polymerizes within the clay layers polymerization can be initiated by heat or a suitable initiator.

Species	%% (w/w)
SiO_2	68.8
Al_2O_3	14.6
Fe_2O_3	1.3
MgO	1.14
CaO	0.295
Na_2O	0.5
K_2O	0.78
TiO_2	0.18
As	0.05

Table 1: Chemical composition of the Maghnite.

Characterization X-ray diffraction analysis (XRD): The X-ray diffraction (XRD) patterns were measured on a Rigaku D/MAX 2200 instrument with CuKα radiation (λ=1.5405 Å). Surface area measurement was performed on NOVA 2000e surface area and pore size analyzer . The samples were outgassed overnight (20 h) under nitrogen prior to the adsorption measurementThe spacing distance of basic silicate layers is obtained with Bragg's law d=ʎ/2SinØ$_{max}$ The spacing distance is (001) calculated of plane peak.

Atomic force microscopy (AFM): The topographic study of surfaces generally allows to extract information on the growth of thin films and oxyde layers in particular. The AFM images in tapping mode with a silicone type of spring constant K between 20 and 80 N / set a resonance frequency f_0 = 234 kHz. Recall that the atomic force microscopy (AFM) is often confronted with e resolution problems due to the size of the probe artifact resulting from an inhomogeneous scan in x and y or impurity and therefore little t always probing the surface accurately [24].

Balayage Electron Microscope (MEB): The study was conducted at the University of Aboubekr Belkaïd Tlemcen (Chetouane pole). The observations of the images show using a belief electron microscope (MEB) TM1000-HITACHI brand with a maximum of 10 000 times magnification can coupled to an X-ray detector.

Fourier transform infrared spectroscopy: The infrared analysis was conducted on a Shimadzu FTIR - 8300 spectrometer N°. 9501165, 0.5 mg of the sample was added to 300 mg of KBr and mixed in a vibratory mill prior to pressing of a disc.

Differential scanning calorimetry: Thermal analysis is carried out by differential scanning calorimetry (DSC). Using a DSC. 204F_1 instrument.

Microscope optique polarisant: The microscope is equipped with an image acquisition in real time camera with lenses: x100, x50, x20, x10, x5. Photos taken by FijiFilm camera F820 EXR.

Results and Discussion

Figure 1 shows the mechanism which three chain cyclic monomer ion tertiary oxonium were activated in the distribution which consists to add an active monomer of the group hydroxylate terminal of the initiator.

The structural characterization of nanocomposites was performed using XRD patterns, which could allow direct evidence of the intercalation of the polymer chains into the silicate galleries. The results indicate that the dominant peak of the silicate layer shifts toward the lower diffraction angle with CTAB intercalated into its silicate galleries, as the interlayer distance increases from 12.97 A° to 32.60 A°. Figures 2a and 2b shows the XRD patterns of montmorillonite -Na and montmorillonite -CTAB.

For montmorillonite -CTAB, the d_{001} value is 32.60 A° which is larger than montmorillonite -Na (12.97 A°). For the montmorillonite -Na, the peak of the montmorillonite is found at 2Ɵ = 6.95°, corresponding to the interlayer distance (d_{001}-spacing) of 12.79 A°. The d_{001} peak for the montmorillonite -CTAB was shifted to a lower region (2Ɵ = 2.46°), That corresponds to an increase in basal spacing from 12.97 A° to 32.603 A° when montmorillonite is organophilized, its mean that

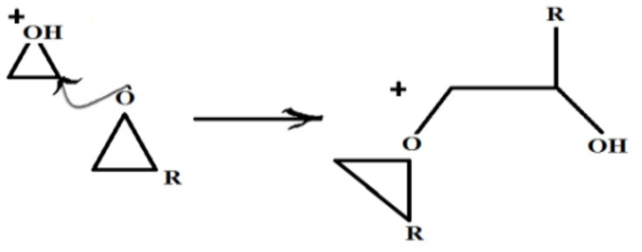

Figure 1: The mechanism in which three-membered cyclic monomer tertiary oxonium Program: Chemdraw [24].

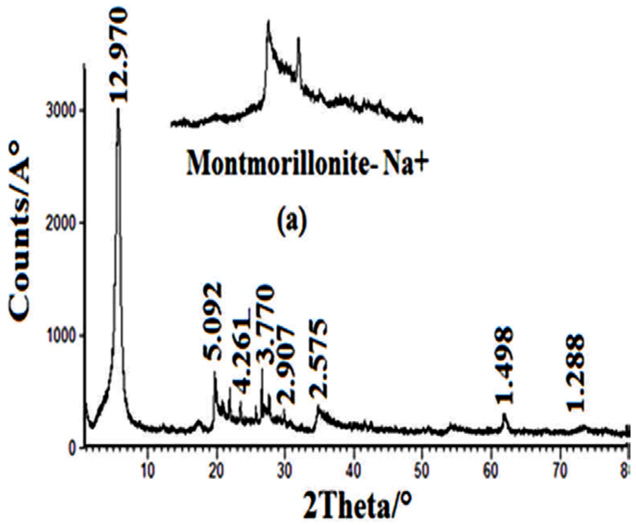

Figure 2a: XRD patterns of Montmorillonite –Na+ (d_{001}=12.97 A°). Program: XPERT PLUS01.

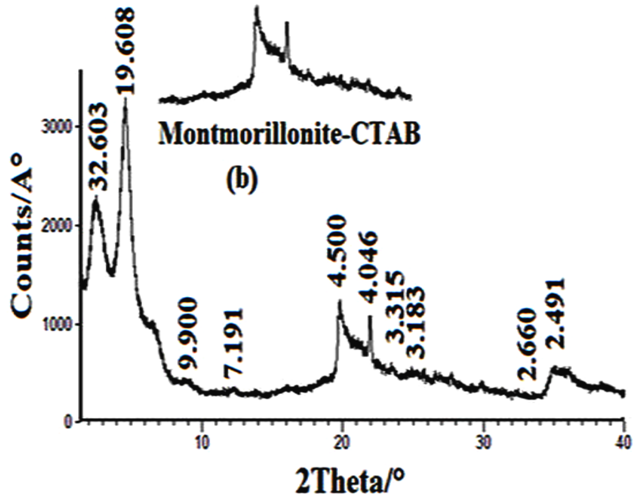

Figure 2b: XRD patterns of Montmorillonite- CTAB (d_{001}= 32.603 A°, d_{002}= 19.680 A°) Program: XPERT PLUS01.

organic moiety replaced the intra-gallery Na^+ of montmorillonite -Na, resulting montmorillonite -CTAB. This result is due to the replacement of smaller Na ions by bulky molecules of montmorillonite -CTAB inside the clay galleries during the ion - exchange reaction.

In the case of NCPO7, a sharp peak is observed at about 2ϴ = 2.21° (37.32 A°), corresponding to the (001) plane of the stacked and intercalated silicate layers dispersed into the PPO matrix, accompanied by the appearance of a small peak at 2ϴ = 25.02°, which was attributed to the (002) plane (d_{002}) of the silicate layers dispersed into the Poly propylene oxyde matrix

Figures 3a-3c, shows the XRD analysis of different nanocomposites prepared in (3,5 and 7 wt %). The clay content increases for NCPO3, NCPO5 and NCPO7, an interlayer distance of 33.8933 A°, 34.9047 A°, 37.321A° respectively. This indicates the incorporation of a considered quantity of organic molecules pop. The clay layers are intercalated by the polymer chains by the increase of the d_{001} value. The position of the characteristic 3 peak of clay shifts to a lower angle value corresponding to well intercalated polymerization chains compared the position of

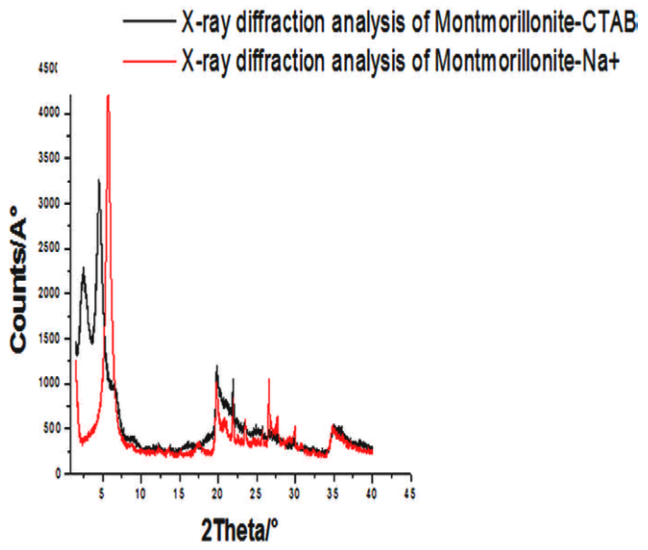

Figure 2c: X-ray diffraction analysis of Montmorillonite -Na$^+$of Montmorillonite -CTAB Program: Origine6.0.

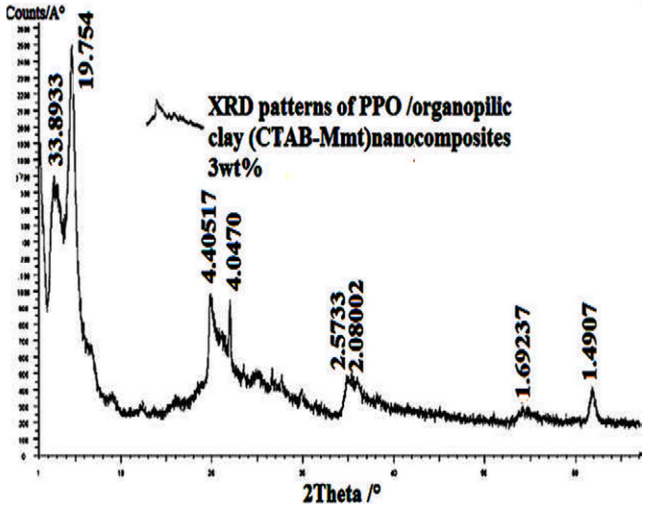

Figure 3a: XRD patterns of Poly propylene oxyde / organophilic clay (CTAB-MMt) Nanocomposites with various 7wt% (NCPO7) (d_{001}= 37.321A°, d_{002}= 19.0015A°) Program: XPERT PLUS01.

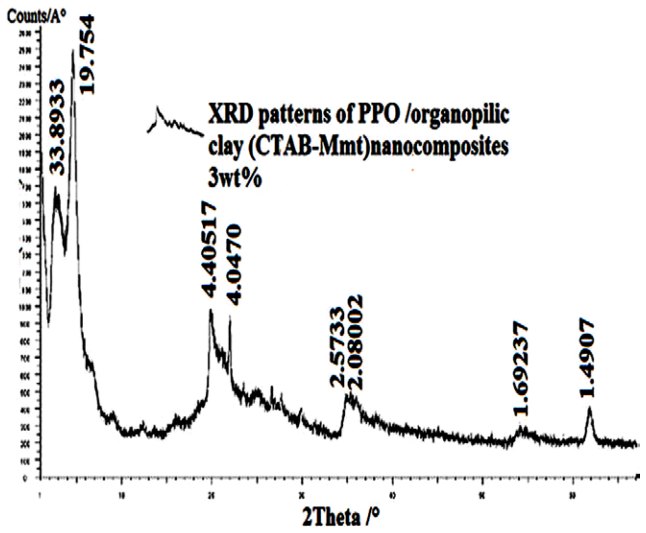

Figure 3b: XRD patterns of Poly propylene oxyde / organophilic clay (CTAB-MMt) Nanocomposites with a various 3wt % (NCPO3) (d_{001}= 33.8933 A°, d_{002}= 19.754 A°) Program: XPERT PLUS01.

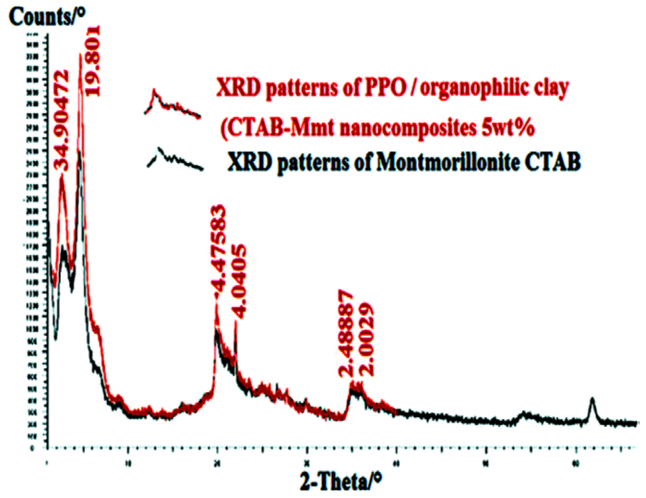

Figure 3c: XRD patterns of Poly propylene oxyde / organophilic clay (CTAB-MMt) Nanocomposites with various 5wt% (NCPO5) (d_{001}= 34.9047 A°, d_{002}= 19.801 A°) Program: XPERT PLUS01.

basal reflection. All of PPO/organoclay nanocomposites are increased position which that clay plates in the PPO matrix are more dispersing exclusively after addition of propylene oxyde.

Differential scanning calorimetric

Figure 4a shows the DSC scans for The glass transition (Tg) temperature of compound PPO/ montmorillonite -CTAB at (3 wt%) was observed at -53.9°C while compound PPO / montmorillonite -CTAB (5,7 wt%), exhibited Tgs ~ -52.6°C, -50.2°C respectively. These peak shifts can be explained on the basis of restricted mobility of the Poly Oxyde Propylene chains within the montmorillonite layers, there are due to the intercalated/exfoliated nature of the Poly Oxyde Propylene/CTAB modified montmorillonite clay nanocomposites.

It is interesting to analyze the crystallinity of PPO in the various samples as detected by differential scanning calorimetry (DSC) (Figure 4b).

Fourier transforms infrared spectroscopy

Figure 5a shows the absorption bands for montmorillonite (-OH stretching, vibration and Si-O stretching, vibration) which we note the following values 3614 .08 cm^{-1}, 977.16 cm^{-1} and (Al-O stretching, Si-O is bending vibrations of MMt) are 524, 432 cm^{-1}, respectively. Figure 5b shows a band at 2850.79 and 2920.66 cm^{-1}. The band shifts to 2924 cm^{-1}, may be due to the increase of the available space in the interlayer space in the intercalated structure. Figure 5c shows two bands at 2930.31 and 2861.84 cm^{-1}. They are assigned to the CH stretching of the ethylene sequence. In the region of 1300-900 cm^{-1}, we note peaks getting broader with the addition of organic clay, particularly the 1116.36 cm^{-1} peak. The characteristic peaks of Poly propylene oxyde which appear shows a band of asymmetric -CH_2 stretching 3020.94 cm^{-1}. For montmorillonite-CTAB complexes. It can explain by the

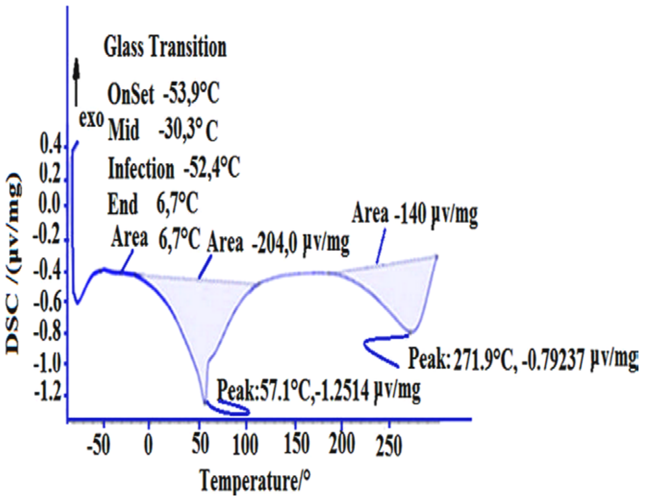

Figure 4a: DSC for Poly propylene oxyde /Montmorillonite-CTAB (3 wt%). Program:NETZSCH DSC 204F1PHOENIX 240-12-0110-L DSC for Poly propylene oxyde /Montmorillonite-CTAB(3 wt%).

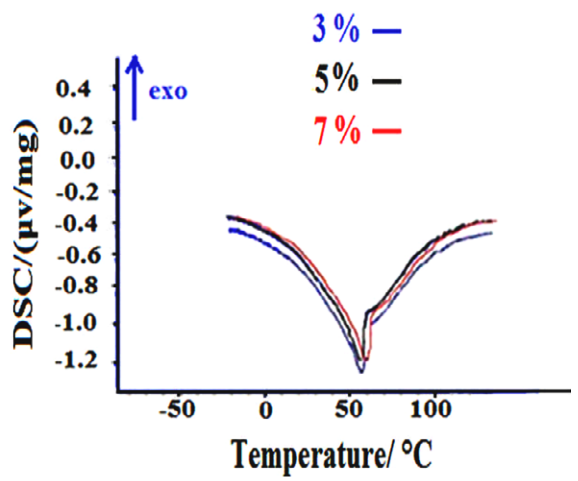

Figure 4b: DSC Scan of poly propylene oxyde /clay nanocomposites with varying concentration of clay Program:NETZSCH DSC 204F1PHOENIX 240-12-0110-L.

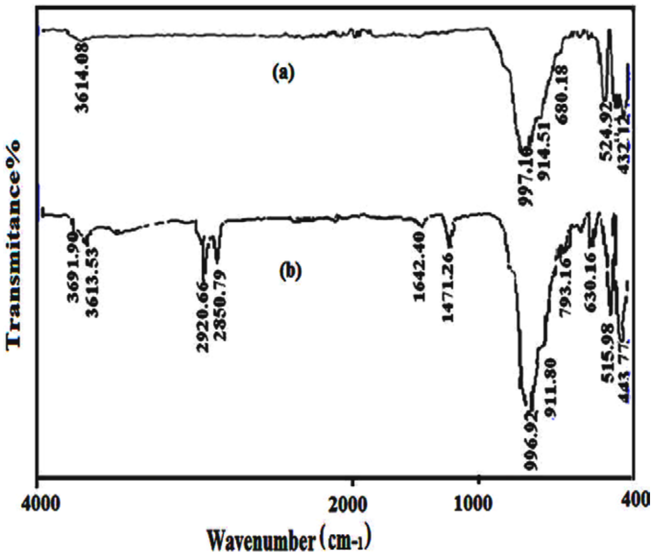

Figure 5a and 5b: (a) FT-IR spectra of montmorillonite –Na$^+$ & (b) FT-IR spectra of montmorillonite -CTAB Program:Files/ OPUS_65/M EAS / COCH4.0.

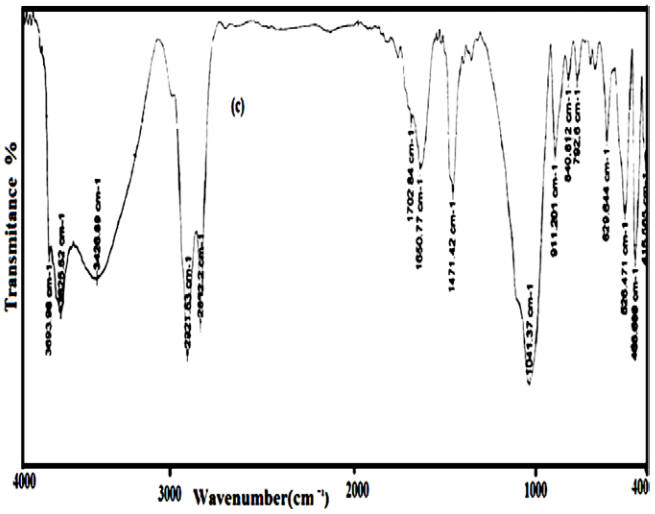

Figure 5c: FT-IR spectra of PPO/ Montmorillonite-CTAB. Program:Files/ OPUS_65/M EAS /COCH4.0.

intensities of the vibrations strongly depend on the increase of loaded surfactant [25].

Figure 5c shows that the montmorillonite-CTAB displays two bands at 2921 cm^{-1} for asymmetric CH_2 stretching mode and 2851 cm^{-1} for symmetric CH_2 stretching mode and similar to those of the organoclay [26,27].

The band shifts 2921.63 cm^{-1} is due to the increase of the space available in the interlayer space. It can be explained that PPO has been successfully intercalated into the silicate gallery inside the nanocomposite.

Microscopic electronics with sweeping (MEB)

We observe coexistence of geometrical shape. The first particle size varies between (25 and 40 µm) and the second, which are bigger, their size varies between (100 and 45 µm). Let us presume that the fillers were mostly none homogeneously dispersed in the PPO matrix (Figures 6a and 6b).

Polarizing optical microscope

According to Figure 7, We note that the existence of crystals initially increases with the incorporation of clay. The platelets react as a nucleation center and decreases with further addition of clay.

Atomic force microscopy (AFM)

This was shown at Figure 8a and 8b.

Topography AFM 3D

This was shown at Figure 8c.

Profiletopography autocorrelation

This was shown at Figure 8d.

The analysis reveals that various images of the morphometry can be obtained as a function of the augmented training time, the spherical shape of the grains is indicated by in Figures 8a and 8b. Moreover, it is clear that the grain size increases in height along the Z axis and also in X and Y directions mentioned side .the depth of the autocorrelation

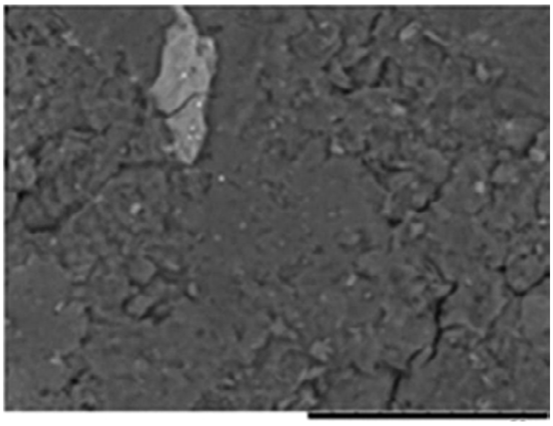

Figure 6a: MEB Image for montmorillonite –CTAB. Program:PHOTO Filtre7.

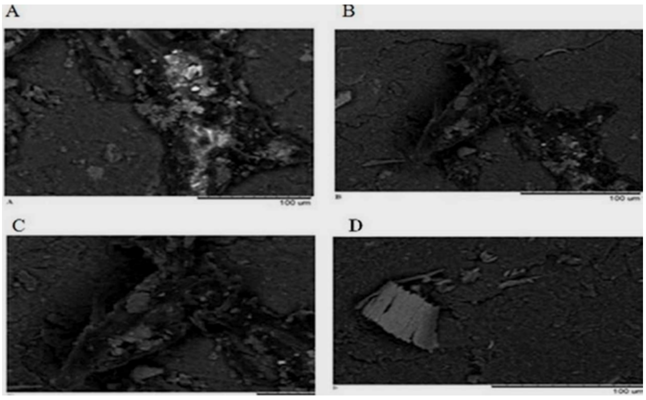

Figure 6b: MEB Image for PPO / montmorillonite -CTAB nanocomposites with various wt.% for (A)3Wt%; (B) 5Wt%; (C) 7Wt%; (D) 10Wt% Program:PHOTO Filtre7.

function shows us benefit layer by using the software image processing WSxM by application of the autocorrelation function of the AFM topography image, we get an image with a central spot. This central spot gives the average grain of the scanned surface (5×5 μm^2) and after expansion of this spot we draw a line profile in the direction 'x' in the direction and 'y', the width at half height was measured at each time. The values obtained from the grain size are regarded as the average values for the function. Using the image processing software WSxM, by applying the function autocorrélation in the AFM topography image. This central spot gives the average grain of the scanned surface (5×5 μm^2) and after expansion of this spot we draw a line profile in the direction 'x' in the direction and 'y'. Height, the half width was measured each time. The values obtained from the grain size are regarded as average values.

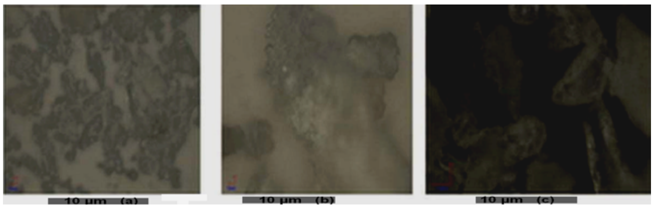

Figure 7: Optical microscope images for PPO/ montmorillonite -CTAB nanocomposites with various wt.% for (A) 3Wt%, (B) 5Wt%, (C) 7Wt%, (D) 10Wt% Program: PHOTO Filtre7.

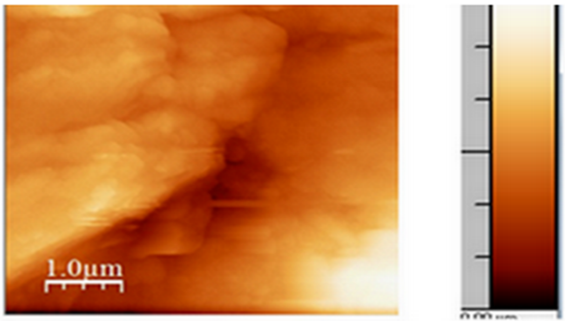

Figure 8a: AFM Montmorillonite-CTAB. Program:PHOTO Filtre7.

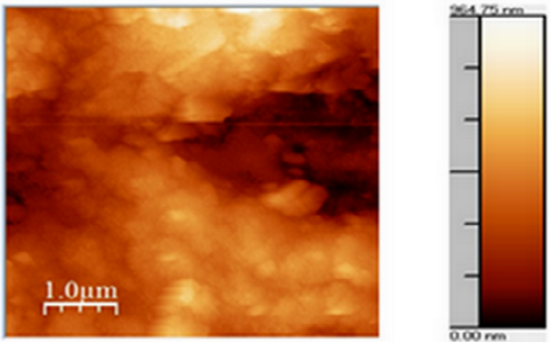

Figure 8b: AFM PPO/montmorillonite-CTAB nanocomposites. Program: PHOTO Filtre7.

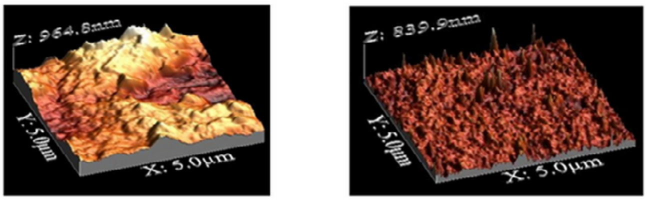

Figure 8c: Poly propylene oxyde / montmorillonite-CTAB nanocomposites. Program: PHOTO Filtre7.

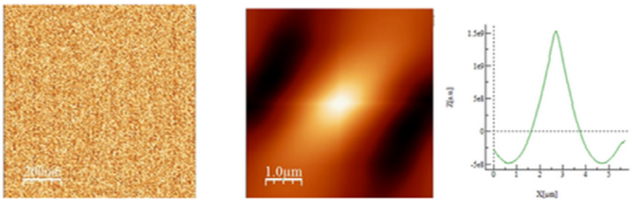

Figure 8d: Application of the autocorrelation function on an AFM picture. Poly propylene oxyde / montmorillonite-CTAB nanocomposites. Program: PHOTO Filtre7.

Conclusions

After this work, we synthesized the poly propylene oxyde / clay Nanocomposites with Organ of clay in polymerizing in situ propylene oxyde catalyzed with montmorillonite clay. The chain transfer agent intercalated by ions is quaternary ammonium was verified by spectroscopy XRD. The modified clay with the ions ammonium can be considered a model practice for biological membranes. These absorbents may be used for treatment flow contaminated waste. The alkyl ammonium ions in the modified clay are inserted in the spaces between the plates of silicate crystallites. In such an embodiment of the CH_2 ion channels adopt different positions in the galleries. We got a very marked improvement in the mechanical properties of the composite nanocomposites to poly propylene oxyde / montmorillonite CTAB used. The Montmorillonite modified in the matrix led to a significant reduction of the viscosity to the molten state and the improvement of the resistance so the effect of by the polarity of compatibility and the dispersal of the clay on the thermal, mechanical properties of the nanocomposites were evaluated. We are able to identify only propylene oxyde created a strong dihedral interaction between the layers of the clay and the phase polymer. The analysis by XRD showed that a better dispersion of the clay within the matrix was obtained with various 3Wt%. The samples show the presence of crystallization phenomena cold. The glass transition (Tg) température of compound PPO/ montmorillonite -CTAB is less than that of pure poly propylene oxyde. According to the Balayage Electron Microscope, the resulting structure is represented by a complete intercalation of the silicate sheets in the poly propylene oxyde /Clay Nanocomposites with a low quantity of montmorillonite -CTAB (3, 5 and 7 wt%). The study by autocorrelation allowed us to determine the morphometry of grain and changes depending on the duration of training. This is attributed to the interactions between the set of polymer chains and the organic compounds of the modified class. The reinforcing effect of the clay in the polymer is confirmed by the increase of rigidity in the system. In the end. This Algerian clay can be used as nanoparticle nanocomposites in the propylene oxyde.

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