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Retarding the Flammability of Polypropylene Based on the Synergistic Effect of Montmorillonite and Carbon Nanotubes

Nady A. Fathy[1], A. Amr[2], Alaa S. Abdelmoaty[3], A. Abou-okeil[2], Adli A. Hanna*[3]

[1]*Physical Chemistry Department, National Research Center, El Bohouth Street, P.O 12632, Giza, Egypt.*

[2]*Pretreatment and Finishing of Cellulosic Fabric, Textile Research Division, National Research Center, El Bohouth Street, P.O 12632, Giza, Egypt.*

[3]*Inorganic Chemistry Department, National Research Center, El Bohouth Street, P.O 12632, Giza, Egypt.*

THE aim of this work is to reduce the flammability of polypropylene (PP) materials which are plenty used in a variety of industrial applications. For the first time, the efficiency of eco-friendly and low-cost flame retardant of carbon nanotubes (CNTs) prepared from rice straw on the flammability degree of PP/MMt composite is reported in this study. Mixtures of polypropylene/ montmorillonite /carbon nanotubes (PP/MMt/CNTs) were prepared by using different percentages of MMt and CNTs. The used CNTs sample was prepared from rice straw waste and the MMt was collected from Wadi El Hamadia, Egypt. The prepared samples were characterized by using Fourier transform infrared (FTIR) spectroscopy and scanning electron microscope (SEM), while the flammability properties were measured by using Limiting Oxygen Index (LOI). The results of FTIR and the photos of SEM showed that the MMt/CNTs composition is incorporated into the PP matrix. The LOI test revealed that LOI value of PP increased from 18.8% to 21.5 % in presence of MMt particles while the addition of 0.05 and 0.1 % of CNTs to PP/MMt significantly improved the LOI value to become 22.4 and 23.5%, respectively. This finding indicates that the addition of CNTs as co-nanofiller can improve the flame retardancy of PP/MMt composites. Thus the obtained MMt/CNTs composites act as synergist fillers to offer admirable and fire safe PP products.

Keywords: Polypropylene, Flammability, Montmorillonite, Carbon nanotubes, Rice straw waste.

Introduction

The polymer materials are used in different technical applications such as electric, electronic, construction, painting, aircraft and etc., for their distinctive advantages such as excellent mechanical and electrical properties, low shrinkage, low cost and ease shaping [1]. The main drawback of these materials is its flammability which produces some toxic gases such as CO, CO_2 and halogen [2]. For this reason, improving the fire retardancy of polymer is a major challenge for extending its use to most applications. So that, several efforts were carried out to prepare halogen-free, recyclable and eco-friendly flame retardant materials. For instance, flame retardant materials including natural clays such as kaolin, montmorillonite (MMt) and other clays [3-12] were investigated as alternatives to common flame-retardants of halogenated additives [2]. This is due to that the Environmental Standards Units (ESU) restricted the use of halogenated additives as flame retardants due to their hazard effects on human health and ecological system [13]. Nevertheless, the Environmental Protection Agency (EPA) recently assessed the feasibility of replacing poly-brominated diphenyl ethers (PBDEs) by CNTs in upholstery textiles as potential flame retardants [4]. The importance of the CNTs (single-, double-and multi-walled tubes of graphene sheets rolled up in cylindrical shapes) is owing to their unique structure. This structure

*Corresponding author e-mail: a_hanna2006@yahoo.com
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causes a rise in the physical and chemical properties, the electrical and the mechanical properties. Also, CNTs provide effective flame retardancy even at very low concentration as compared to the other additives. This property may be ascribed to their tubular structure, high aspect ratio of surface area/volume, tunable morphology and large surface area, good thermal stability and chemical resistance against to the chemical environmental [13].

The integration of clay and CNTs as nanocomposites (nanofillers) into the matrix of polymeric materials has paid much attention of researchers because of its synergistic effect on the flame retardancy of polymer via the formation of a jammed network structure in the polymer matrix [14-19]. This network structure of layers shields the polymer units from the external radiation and also acts as excellent thermal insulation layers by producing a passive residual char on the surface of polymer when burned [14]. Additionally, the resulting combination of clay and CNTs leads to great enhancement in the physical and thermal properties of polymer matrix and hence reduces its flammability.

In our previous studies [20-22], the MMt clay and the ammonium polyphosphate were used as flame retardants for polypropylene (PP). It was found that adding 10% of ammonium polyphosphate to PP could improve its thermal stability and the flame retardancy without considerable effects on the mechanical properties of the PP. Also, these studies extended to increase the efficiency of ammonium polyphosphate by using modified and unmodified clays. For example, a mixture of 6% ammonium polyphosphate and 4% clay was the highest flame retardant as compared to either clay or ammonium polyphosphate used individually with PP.

However, it is important to produce low-cost, eco-friendly and effective flame retardants of carbon nanotubes integrated with natural clay. As well-known, the commercial CNTs (SWCNTs and MWCNTs) products are very expensive. Recently, Fathy [23] and coworkers [24] studied the synthesis of CNTs from agricultural wastes produced in Egypt such as rice straw via hydrothermal treatment followed by direct pyrolysis in a closed vertical reactor.

As continuation of these works, the present work aims to study the effect of carbon nanotubes (CNTs) and the treated clay (montmorillonite) by sulfuric acid on the flame retardancy efficiency of the polypropylene (PP). The used CNTs sample was prepared from rice straw waste and the clay was supplied from Wadi El Hamadia region, Egypt. The prepared samples were characterized by using Fourier transform infrared (FTIR) spectroscopy and scanning electron microscope (SEM), while the flammability properties of this system measured by limiting Oxygen Index (LOI).

Experimental

Materials

The following materials were used in this study:

(i) Polypropylene (PP), [$C_{22}H_{42}O_3$, 354.57 g/mol] used in the preparation of the tested samples was purchased from LG Chem., Ltd. SEETEC.

(ii) Montmorillonite (MMt)

The MMt samples were supplied from Wadi El Hamadia region, Egypt. The start samples were exposed to grinding and chemical treatment with H_2SO_4 to modify the particle size and the composition

(iii) Carbon nanotubes (CNTs)

The sample of carbon nanotubes was prepared from rice straw waste as described previously [24].

Preparation of the samples

(i) Modification of MMt

Chemical modification of the MMt ore was carried out by adding 50 g of the ore to 100 mL of sulfuric acid solution (10 M) and refluxing at 110^{o}C under the atmospheric pressure in a ground bottomed equipped with a reflux condenser for 4 h. The resulting suspension was rapidly quenched by adding 500 mL ice cold water. The product was then filtered washed with distillated water and calcined at 500^{o}C for 1 h and grinded in mortar paste to form a homogeneous powder [25].

(ii) Preparation of CNTs

Rice straw was collected, washed, dried and grinded to powder without any pretreatment. After that a hydrochar of rice straw loaded with nanoparticles of Fe, Ni and Co catalysts was prepared hydrothermally at 180^{o}C for 3 h [24]. Taking the hydrochar into stainless steel reactor in presence of low amount of Al_2O_3 supported to Fe and Ni oxides, the temperature of reactor was raised slowly to 300^{o}C under pressing N_2 gas and then the temperature increased to 830^{o}C slowly at heating rate 10^{o}C/min without any gas flow and held for 1 h. After cooling the furnace, the black sample was withdrawn out and purified with 5M of HNO_3 and H_2SO_4 solutions at 80°C under refluxing and stirring for 4h. The final product was filtered and washed with hot water for several times till pH of the filtrate becomes about 6 and then dried at 80°C overnight.

Preparation of PP/MMt/CNTs nanocomposites

The tested nanocomposites were prepared with different concentrations of MMt and CNTs according to the recommended route by using the melt method [26]. Before melt spinning the PP pellets were compounded with the (MMt/CNTs) in twin screw co-rotator where RPM of the screw was 60-80 rpm. The temperatures of the five zones were adjusted through the extruder at 175, 185, 195, 210 and 220°C for the zones I, II, III, IV, and V respectively. The melt spinning of PP pellets blended with MMt/ CNTs amounts were carried out on laboratory melt spinning machine supplied by Fair Deal Association (FDA), New-Delhi, India. Then, the temperatures of this produced spinning extruder zones maintained were 190, 200 and 220°C for zone I, zone II and zone III respectively. Table 1 illustrates the percentage of PP, MMt and CNTs used in the study.

Instruments of characterization

The PP/ MMt/CNTs) were characterized by using the Fourier transform infrared (FTIR) spectroscopy and the scanning electron microscopic (SEM).

FTIR

FTIR spectra of the samples were obtained using a KBr disk technique and FTIR 6500 spectrometer (JASCO, Japan) in the range of 400-4000 cm^{-1}.

SEM

The surface morphology of the samples was carried out using a JEOL JSM t20 scanning electron microscope (SEM, JEOL, Japan) at an accelerating voltage of 5 kV.

Flame retardant test

TABLE 1. Percentage of PP, MMt and CNTs in the resultant nanocomposites and their corresponding LOI values.

Sample No.	PP (%)	MMt (%)	CNTs (%)	LOI (%)
PP	100	0.00	0.00	18.8
PP/MMt	85	15.00	0.00	21.5
PP/MMt/0.05CNTs	85	14.95	0.05	22.4
PP/MMt/0.1CNTs	85	14.90	0.1	23.5

The flame retardant properties of the prepared samples as well as the pure PP was performed by using LOI apparatus ISO 4589-2 ASTM D 2863, Elevated- Temperature Oxygen Index ISO 4589-3, UK Naval Engineering standard NES 714 which measure the percentage of oxygen that has to be present the support burning of the samples.

Results and Discussion

The results of chemical compositions of MMt and CNTs in this investigation were showed in the next section.

Chemical analysis

The chemical analysis of the modified MMt by sulfuric acid revealed that it contains about 55. 3% of SiO_2 and about 28.9% of Al_2O_3 with some minor of other oxides such as MgO, CaO, K_2O, ZnO, TiO_2 and Fe_2O_3 as shown in Table 2.

Characteristics of CNTs

The produced CNTs sample was characterized elsewhere [24]. The results of TEM showed that the obtained CNTs are categorized as multi-walled nanotubes due to their outer diameter located in the range of 15 nm to 40 nm. CNTs exhibited a high surface area reached to 188 m^2/g. The chemical composition of the sample was found to be composed from C, O, Si, Al, Fe, Ni and Co elements.

FTIR measurement of the prepared samples

Figure 1 illustrates the FTIR spectra of all samples in order to indicate both the main surface functional groups and alterations in these groups. The IR spectrum of the pure PP (blank) showed that there are different absorption bands at 3070, 2985, 2922 and 890 cm^{-1}. These bands maybe corresponded to the C-H bond for the saturated and unsaturated of PP chain [26]. For PP/MMt and PP/MMt/CNTs nanocomposites, some new bands were appeared. The sharpness of the formed bands increased significantly with increasing the concentration of CNTs. It can be included that presence of MMt individually or MMt/CNTs can rearrange the structure of PP confirming that MMt and CNTs nanoparticles incorporated into the PP matrix. The FTIR spectrum of the CNTs only was recorded elsewhere [24]. It was found that the basic functional groups formed on the CNTs surface were oxygenated functionalities such as hydroxyls (3450 and 1450 cm^{-1}), carboxylic groups (1730cm^{-1}) and lactone groups (1060-1110 cm^{-1}) [24].

SEM of the prepared samples

Most of the previous works on the effects of retardant materials to the flammability of polymers was considered the morphology of the mixture has great effect. So that some works were focused on the relation between the flammability and the morphology of the system [27-31]. In this work the distribution of the MMt or MMt/CNTs in the PP matrix was examined. Figure 2 (a, b, c and d) represents the SEM images of the prepared samples. Figure 2a shows that the PP particles has smooth surface before modification. It can be seen clearly that there are substantial changes in the morphology of PP after modification with MMt and CNTs. For the PP/MMt sample, Fig. 2b illustrates that the MMt particles are distributed in layers-like structure and an individual clay layer are seen. By adding 0.05% of CNTs as presented in Fig. 2c, the distribution of the clay layers becomes clear and the surface is more amorphous and rough. As the CNTs concentration increases, the photograph shows that the more homogeneity with appearance of some of veins-like CNTs (the right of photo) as shown in Fig. 2d. It has been reported that the addition of CNTs improves the basal spacing and the dispersion of clay layers in nanocomposites accompanied by a

TABLE 2. Chemical composition of MMt sample.

Sample	SiO_2	AL_2O_3	MgO	CaO	K_2O	ZnO	TiO_2	Fe_2O_3
MMt	55.3	28.9	0.02	0.013	0.011	0.006	0.25	0.004

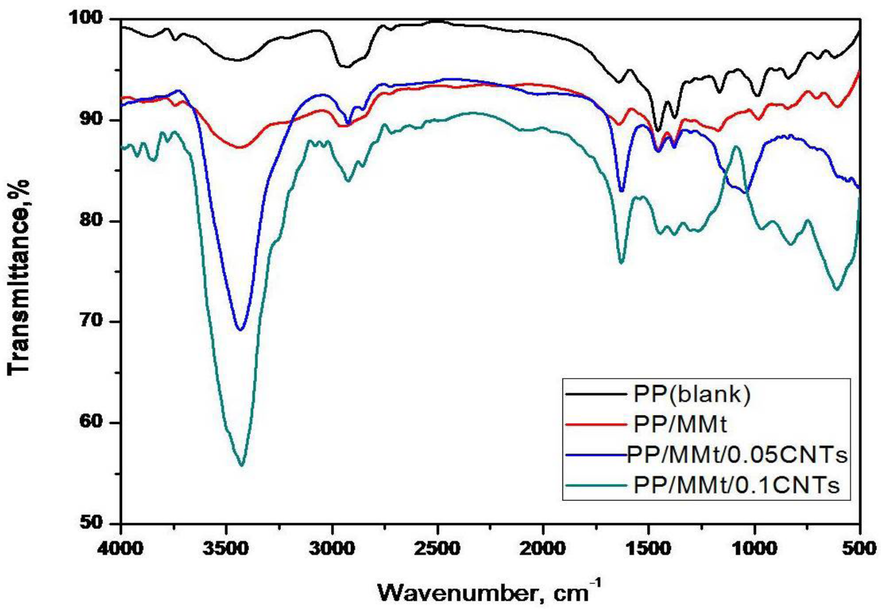

Fig. 1. FTIR spectra of the prepared samples.

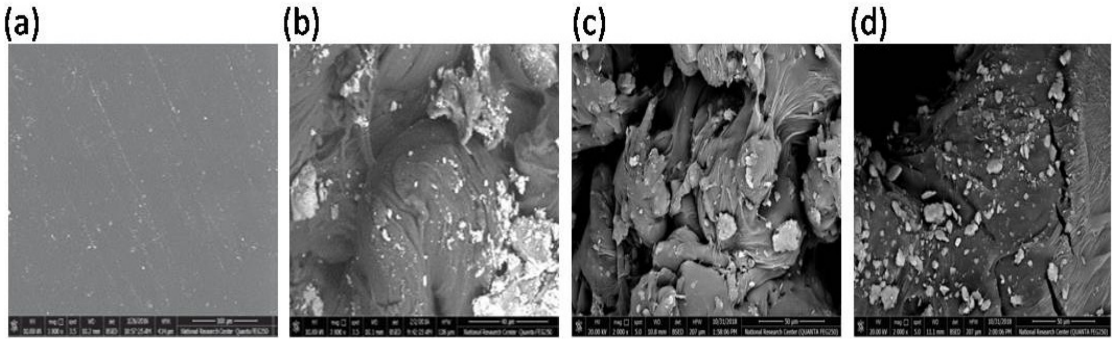

Fig. 2. SEM Photographs of the prepared samples (a) PP-blank, (b) PP/MMt, (c) PP/MMt/0.05CNTand (d) PP/MMt/0.1CNT.

strong interaction between clay and CNTs [18]. Thus the morphology studies here showed also that the adding CNTs with MMt clay inside the PP matrix would improve the physical, chemical and mechanical properties of PP polymer. Hence, the flame retardant performance of PP is considerably enhanced after combined with CNTs as shown in the next section.

Investigation of LOI test

In this study the LOI was used to evaluate the CNTs/MMt as flame retardant for reducing the flammability of PP. The LOI is measurement for the percentage of oxygen (vol. %) that will support the burning (i.e., combustion) of the polymer. The higher of the LOI is the lower flammability, while the lower of LOI is the higher flammable materials. So, this test gives an indication about the efficiency of the fire retardant materials and not any information about the mechanism of the different samples. Four samples of PP (blank), PP/MMt, PP/MMt/0.05CNTs, PP/MMt/0.01CNTs were tested and the values of LOI are listed in Table 1. According to the obtained results, the LOI value of the PP (blank) is equal to 18.8%; this means that the percentage is lower than 21.0 % which is the percentage of oxygen in the atmosphere. This result confirms that the PP is a flammable material. For sample PP/MMt which contains 15% of treated MMT by sulfuric acid, the value of the LOI is increased to 21.5% affirming that the ability of burning is decreased. For PP/MMt/0.05CNTs nanocomposite which contains 0.05 % of CNTs inside the composition of PP/MMt sample, the value of LOI is increased to 22.4%. While with increasing the percentage of CNTs to 0.1% as presented in sample PP/MMt/0.1CNTs, the value of LOI is increased to 23.4%. These findings mean that the MMt causes an increase in the LOI value and inhibits the spread of the flame while the addition of a little amount of CNTs improves the retardant efficiency of MMt/PP composite. This leads to state that a synergistic effect has been occurred. These results are evident by the following photographs (Fig. 3) which demonstrates the residues of prepared samples after exposure to ignition process.

In Fig. 3, it can be seen that the pure PP sample melted and accompanied by vigorous bubbling and weight loss. While PP/MMt sample behaved

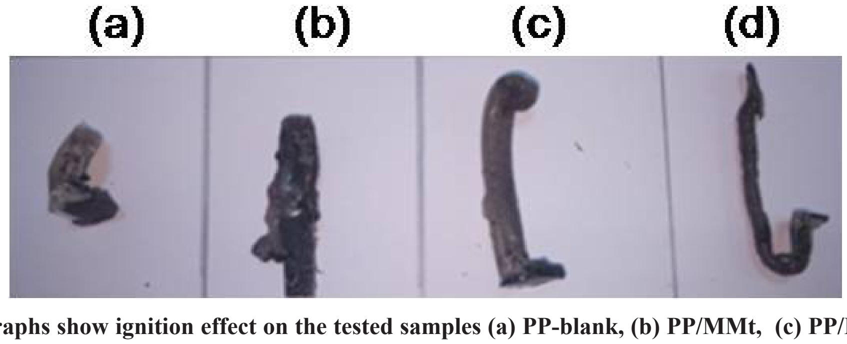

Fig. 3. Photographs show ignition effect on the tested samples (a) PP-blank, (b) PP/MMt, (c) PP/MMt/0.05CNTs and (d) PP/MMt/0.1CNTs.

like a solid without obvious melting beside black discrete islands observed and thin char grew with a tendency for cracking between them during combustion. On the other hand, PP/MMt/CNTs samples showed a relatively intact char layer without melting and cracking. The similar results were documented by others [32].

According to the previous studies [14-19, 29, 30], the mechanism based on the synergistic effect between MMt clay and CNTs on the flammability properties of PP can be explained by the role of the two additives. There are three mechanisms occur in the presence of the flame retardants; a) burning of this nanocomposite would swell or promote char forming a barrier layer to protect the underlying material or to smother the flame, b) by-products (solid or gas) can be generated that slow down the heat released during combustion, effectively reduce the burning rate and the rate the fire spreads and finally c) short lived molecules called free radicals maybe released and reacted with the material and other combustion by-products in order to slow down or stop the flammability of polymer. Probably the mechanism of flame retardancy by the tested samples takes place through two or the three steps. It has been reported earlier that the clay may enhance the graphitization of CNTs and graphitic structure was obtained which tends to give better protection of materials from thermal oxidation.

The role of the CNTs when used as flame retardant additives concerned with forming a relevant structured floccules layer which re-emits much of the incident radiation back into the gas phase. This formed layer acts as a protective barrier layer of char. While MMt particles which formed of layered structure could traps some molecules water and when burned; results in a char of SiO_2 and Al_2O_3 which acts a protective layer between the flame and the burned polymer. In addition, MMt has low heat capacity, this it can retard the heat transfer during combustion and it works as isolated barrier to reduce the diffuse of gases combatable [33]. Therefore, the combination of MMt and CNTs generated layered network structure which re-emitted oxygen radicals from the surface of the layer to outside and decreased the flammability rate of PP polymer.

Conclusions

This work investigates the efficiency of ecofriendly and low-cost flame retardant of CNTs prepared from rice straw on the flammability degree of PP/MMt composite. From analysis of the obtained results, it may conclude that the modified MMt improved the flammability of PP. The adding small amount of CNTs increased the efficiency of PP/MMt composite as a flame retardant. The limiting oxygen index (LOI) improved from 21.5% to 23.4% when the PP/MMt contains 0.1% of CNT. However, a synergistic effect is established from the combination between MMt clay and CNTs prepared from agricultural waste leading to an enhancement in the flame retardancy of PP material and hence it worthy necessarily to further study.

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تثبيط القابلية للاشتعال من مادة البولي بروبيلين بواسطة التأثير التآزري لخام المونتموريللونيت وأنابيب الكربون النانومترية

نادي عطية فتحي[١]، احمد السيد عمرو[٢]، علاء صلاح عبدالمعطي[٣]، اشرف ابوعقيل[٢]، عدلي عبدالله حنا[٣]

[١]قسم الكيمياء الفيزيقية - المركز القومي للبحوث – الجيزة - مصر.

[٢]قسم التحضيرات والتجهيزات للالياف السليلوزية - المركز القومي للبحوث – الجيزة - مصر.

[٣]قسم الكيمياء غير العضوية - المركز القومي للبحوث – الجيزة - مصر.

الهدف من هذا العمل هو تقليل القابلية للاشتعال لمواد البولي بروبيلين (PP) والتي تستخدم كثيرًا في مجموعة متنوعة من التطبيقات الصناعية. للمرة الاولي، في هذه الدراسة اجري تعيين كفاءة مثبطات اللهب صديقة للبيئة ومنخفضة التكلفة من انابيب الكربون النانومترية (CNTs) المحضرة من قش الأرز على درجة القابلية للاشتعال لمتراكب من البولي بروبيلين/مونتموريللونيت (PP / MMt). تم تحضير خليط من البولي بروبيلين/مونتموريللونيت/انابيب الكربون النانوية (PP / MMt/ CNTs) باستخدام نسب مختلفة من MMt و CNTs. تم تحضير عينة الـ CNTs المستخدمة من نفايات قش الأرز وتجميع خام الـ MMt من وادي الحمادية، مصر. تم توصيف العينات المحضرة باستخدام التحليل الطيفي للأشعة تحت الحمراء (FTIR) والميكروسكوب الإلكتروني الماسح (SEM) ، في حين تم قياس خصائص القابلية للاشتعال باستخدام معامل الأكسجين المحدد (LOI). أظهرت نتائج FTIR وصور SEM أن تركيبة MMt / CNTs مدمجة في مصفوفة الـ PP . كشف اختبار LOI أن قيمة LOI لـ PP زادت من ١٨,٨ ٪ إلى ٢١,٥ ٪ في وجود جزيئات MMt في حين أن إضافة ٠,٠٥ و ٠,١ ٪ من الـ CNTs إلى PP / MMt فإن قيمة LOI تحسنت بشكل ملحوظ لتصبح ٢٢,٤ و ٢٣,٥ ٪ ، على التوالي. توضح هذه النتيجة إلى أن إضافة CNTs كحشو مساعد متناهي الصغر يمكن أن يحسن مثبطات اللهب لمتراكبات .PP / MMt وبالتالي ، فإن متراكبات MMt / CNTs التي تم الحصول عليها تعمل كمواد حشو متآزرة لتقديم منتجات رائعة وآمنة للحريق من البولي بروبيلين.