Mechanical Properties and Morphology Studies of Nanocomposites Based on RSF/Nanoclay Modified /HDPE Nanocomposites

Abdel- Hakim A, A.[1]; Abdel -Salam Sabbah I.[2]; Metwally M., S.[2]; El Begawy S.[3] and Elshafie E., S[3].

[1]Polymer and pigments Department, National Research Center, Dokki, Cairo, Egypt.
[2]Department of Chemistry, Faculty of Science, Al-Azhar, Cairo, Egypt
[3]Science &Technology Center of Excellence, Ministry of Military Production, Cairo, Egypt
emadsobhy2007@gmail.com

Abstract: The use of natural fibers and nanoclay as reinforcement materials in composites has proved the ability of providing several environmental and economical advantages. In this work, two groups of rice straw fibers (RSF) reinforced high density polyethylene (HDPE) nanocomposites were prepared in the presence of 3wt% maleated polyethylene (MAPE) as coupling agent. To the former group, nanomontmorillonite clay (n-MMT) was added after being treated with cetyltrimethyl ammonium bromide (CTAB), in order to enhance dispersiability, compatibility and interfacial bonding with the polymer. Calcium oxide (CaO) was added as a filler to the second group in fine powder form .These groups have been examined with regard to their mechanical properties and thermal stability. The interfacial adhesion has been also investigated by scanning electron microscopy(SEM).Results revealed that, significant improvement was attained for mechanical properties as the modified nanoclay %increases to~ 2.5% .Maximum loaded amount of ca.10% CaO has found to be sufficiently required to enhance tensile strength with reasonable flexural strength.

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1. Introduction:

Natural fibers have been increasingly used as reinforcement materials in commercial thermoplastics and thermoset matrix composites.The use of these material has proved the ability of providing several advantages such as low densities, low cost nonabrasive nature, high specific properties and renewable nature [1-4].Some types such as rice straw fibers and saw dust are considered as important potential reinforcing filler for thermoplastic composite because of their lignocellulosic characteristics.[5].Composite materials based on cellulosic fibers, namely wood-plastic composites (WPC) [6], nanoscience and nanotechnology have opened up a completely new way to develop wood-plastic composites (WPCs).Nanoparticles are obtained from available natural resources and they generally need to be treated because the physical mixture of a polymer and layered silicate may not form a nanocomposites; in this case a separation into discrete phases takes place. The poor physical interaction between the organic and the inorganic components leads to poor mechanical and thermal properties. In contrast, strong interactions between the polymer and the layered silicate nanocomposites lead to the organic and inorganic phases being dispersed at the nanometer level. As a result, nanocomposites exhibit unique higher properties than conventional composites [7-10]. Modification of clay due to Hydrophilic clays and hydrophobic polymers are not compatible in their virgin states and a modification of either the polymer or clay is necessary for dispersion, intercalation and/or exfoliation of clay tactoids in the polymer matrix. Surface modification of clay has commonly been used to achieve a greater compatibility of the clay and polymer. Ion exchange of Na+ or Ca2+ gallery cations in the mineral by alkyl ammonium ions is frequently chosen to modify the clay [11]. Recently, the use of inorganic nanoparticles as additives to enhance the polymer's performance has been studied extensively. The incorporation of organically modified layered silicate (OMLS) into polymers has been of particular interest because of their demonstrated significant enhancement, relative to an unmodified polymer resin of a large number of physical properties, including barrier, flammability resistance, thermal and environmental stability, solvent uptake, and rate of biodegradability of biodegradable polymers [12]. Montmorillonite (MMT) Of the many type of nano-reinforcements available for polymers, layered silicates clay has attracted the greatest interest in recent years, both in industry and in academia, because of the ability of silicate particles to disperse into individual layers and also the ability to tune their surface chemistry through ion exchange reactions with of organic inorganic cations [13]. The layered silicates commonly used in nanocomposites belong to structural family known as 2:1 phyllosilicates and the most commonly used layered silicates are MMT. It was discovered in 1847 in Montmorillion France . Among the potential nanomaterials used in polymer nanocomposites, nanoclay (layered silicate) which has been widely investigated primarily because of its remarkable improvement in properties. Furthermore, clay materials are easily available, environmentally friendly, and their intercalation chemistry has been investigated since a long time These make nanoclay one of the most widely accepted and effective nano-reinforcements [14-16]. Surface modified nanoclay are widely used in, technological applications because of their improved strength[17-19].

2. Materials

High Density Polyethylene (HDPE) was purchased from Exxon Mobil Chemical with Density 0.964 gm/cm^3, melt index 8.0g/10min and melting temperature 134^{o}C.Rice straw from Egypt agriculture fields. Montmorillonite clays were used as supplied (Cloisite Na$^+$ (Na$^+$-MM), Southern Clay Products Inc. Calcium Oxide (CaO): was from Sigma-Aldrich Company; grade, Assay 96-100.5 % Dicumyl Peroxide (DCP): was from Akzo Chemi, Netherlands,density: 0.98- 0.99 gm/cm³, processing temperature was 120^{o}C, suitable cross linking temperature: 150-170^{o}C. Maleic anhydride: was from Fluke. Cetyltrimethylammonium bromide(CTAB) was purchased from Sigma–Aldrich and used as received.

2.1. Modification of montmorillonite with CTAB.

The CTAB-modified MMT was prepared as follows; 5.0 g of MMT was mixed with 3.4 g CTAB in 200 ml of water. The mixtures was subjected to mechanical stirring for 8 hours at 70°C and finally, the modified montmorillonite was collected by filtration and washed thoroughly using distilled water and dried at 80°C.

2.2 Melt Intercalation

In this technique, no solvent is required and the layered silicate is mixed within the polymer matrix in the molten state. The thermoplastic polymer is mechanically mixed by conventional methods such as extrusion and injection molding with organophillic clay at an elevated temperature. The polymer chains are then intercalated or exfoliated to form nanocomposites.[20, 21] . this work rice straw was dried at 105^{o}C for 24 h, then blended with HDPE and modified nanomontmorillonite in Brabender twin screw for 10 minutes at 180 ^{o}C with rotational speed of 60 rpm, to. The rice straw /HDPE composites were then removed from the Brabender twin screw and pressed to sheet-like sample using the hydraulic press at 180^{o}C for 5 minutes.

2.3 Testing Methods

The flexural and notched izod impact strength properties of the prepared composites were measured according to ASTM standard D790 and D256 respectively.The morphology of the prepared composites was observed by scanning electron microscopy (SEM) type a Joel JXA-840 A.

2.4 Water absorption:

Water absorption of the composites was measured according the ASTM D570 specification. The dried specimens were immersed in distilled water to maintained at 25 ^{0}C. for different times All values were calculated as the mean of three samples. Using the following formula:

Water uptake % = $(Ws - W_1)/W_1 \times 100$

Where Ws is the weight of water saturated specimen and W_1 is the weight of oven dried speciment.

3. Results and Discussion

3.1 Preparation of Maleated Polyethylene (MAPE):

Maleated polyethylene was prepared by grafting of maleic anhydride at high density polyethylene in the presence of the initiator; Dicumyl peroxide (DCP) as initator[22].This was carried out by melt mixing of HDPE with maleic anhydride in a Barabender twin screw at 180 °C and 60 rpm. Fourier transforms infrared spectroscopy (FTIR) was used to measure the relative maleic anhydride (MA) grafting in polyethylene. Fig. (1) and Fig. (2). Shows the FTIR spectra of HDPE and HDPE-g-MA. New peaks appeared at 1786 cm^{-1} is due to asymmetric stretching modes of carbonyl (C=O) of saturated maleic anhydride[23-25]. The band at 1859 cm^{-1} and 1786 cm^{-1}, which are characteristic of cyclic anhydride. The backbone molecule, polyethylene, had a strong peak of (-CH) at about 2917 cm^{-1} and 2848 cm^{-1}, and the attributed band to the rocking vibrations of $-CH_2$ bonds in HDPE was observed at 719 cm^{-1}.

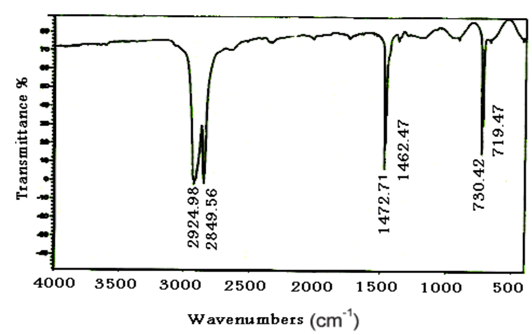


Fig.(1) FTIR for virgin high density polyethylene (HDPE).

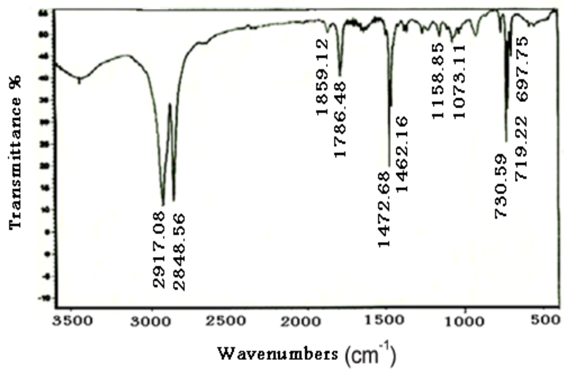
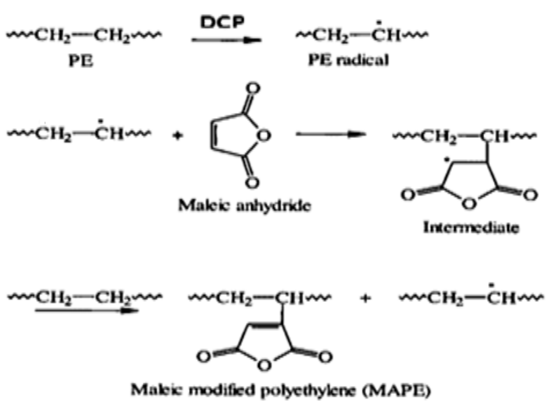


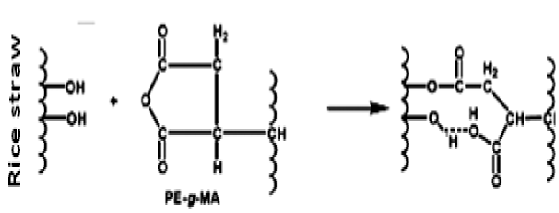
Fig (2): FTIR for Maleated high density polyethylene (MAPE).

3.2 Effect of MAPE% as Coupling agent of Composites.

Maleated polyethylene plays a vital role in the improvement of the mechanical properties such as tensile strength, flexural strength and hardness of the prepared composites due to the increase of different ratios of coupling agent compatibility and interfacial bonding between rice straw and HDPE [26].Different ratios of rice straw /HDPE contents were used in the presence of different ratios of coupling agent coupling agent (1, 3 and 5%). The obtained results are shown in Fig. (3).



Scheme1: Reaction mechanism of maleic anhydride modification of PE. [27].



Scheme 2: Reaction mechanism interaction between rice straw and PE-g-MA.

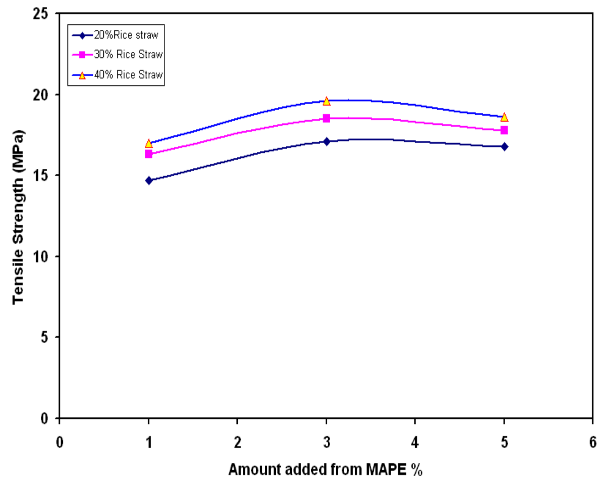


Fig. (3) The change in tensile strength with amounts of coupling agent (MAPE) at different feed ratios

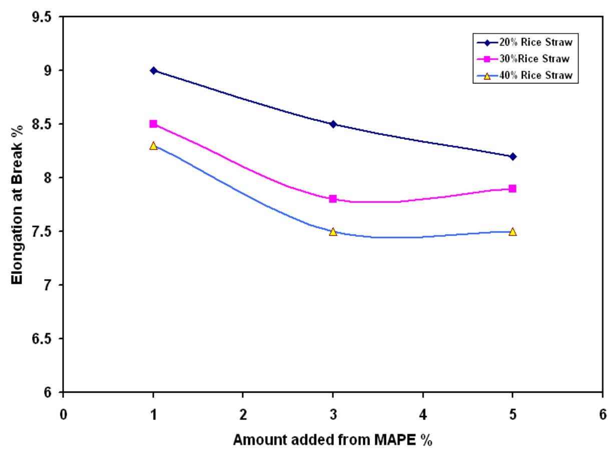


Fig (4): The change in elongation % with the amounts of coupling agent (MAPE) at different feed ratios.

From Fig. (3).one can observe that, the addition of 3% of the coupling agent is more effective than 1% and 5% which gives a slight decrease in tensile strength which increases from 14.7 to 17.1 MPa in case of 20% addition of rice straw and there is a slight decrease in case of 5% of the coupling agent to 16.8 MPa. This indicates that 3% better to improve interfacial bonding in the prepared composites. From Fig(4) indicates that Elongation at break decreases as the amount of rice straw loaded increases due to increasing rigidity of the composites .

Table(1) :Composition of the studied formulations.

No.	HDPE (wt %)	Coupling agent (Wt %)	Rice straw flour (wt %)	MMT-CTAB (wt %)	CaO (wt %)
S_1	37	3	59.5	0.5	-
S_2	37	3	59	1	-
S_3	37	3	58.5	1.5	-
S_4	37	3	57.5	2.5	-
S_5	37	3	55	-	5
S_6	37	3	50	-	10
S_7	37	3	40	-	20

3.3Tensile Test:

The effect of nanomontmorillonite loading on the mechanical properties of the composites shows that, proper amount of nanomontmorillonite can improve the tensile, flexural and impact properties of the composites, when nano montmorillonite is organized with cetyltrimethylammonium bromide [28].From Fig(5) which represents the relation between the tensile strength and the added amount of nanoclay modified with CTAB to RSF/HDPE composites in the presence of MAPE as coupling agent .From the figure. It was found that the tensile strength increases as amount of MMT-CTAB increases. Modification of montmorillonite and presence of maleated polyethylene have important role in improving the mechanical properties of the prepared composites, Koo et al [29],reported that the final morphology and the anisotropic phase formation of MAPE/layered silicate nanocomposites depend on the clay content. With the aid of MAPE. But the elongation % of the prepared composites was found to decrease as the amount loaded with MMT-CTAB increases as seen in Fig.(6).

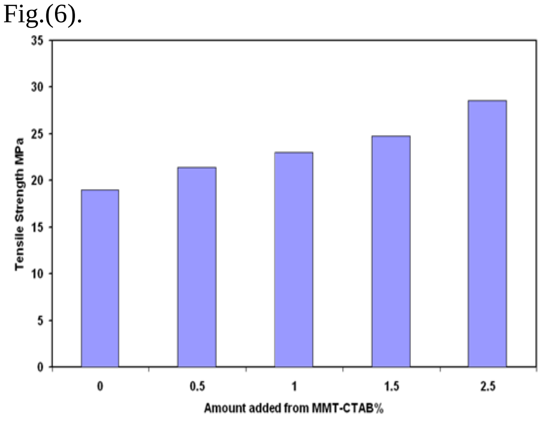


Fig.(5) The relation between Tensile strength (MPa) with different amount of MMT-CTAB.

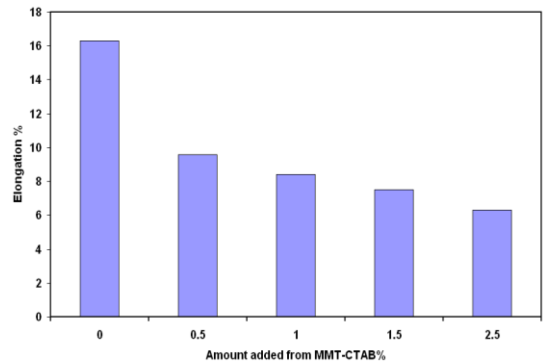


Fig.(6) Represents the relation between the Elongation% (MPa) with different amount of MMT-CTAB.

Fig. (7) Represents the tensile strength of composites in the presence of CaO. Addition of CaO significantly improved the mechanical properties [30]. Good effect was noticed at 10%loaded CaO .It was found that elongation decreases as the amount of CaO increases as clearly shown in Fig.(8).

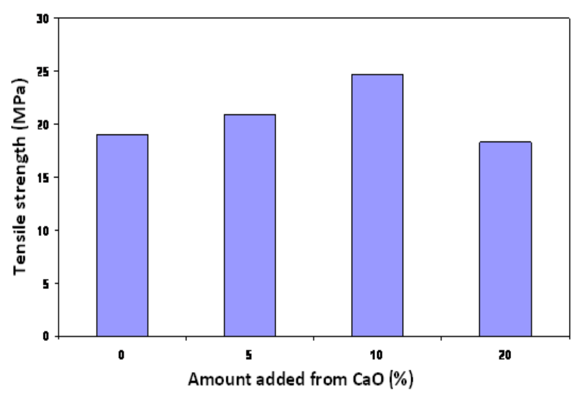


Fig. (7) The relation between Tensile strength (MPa) with different amount of CaO.

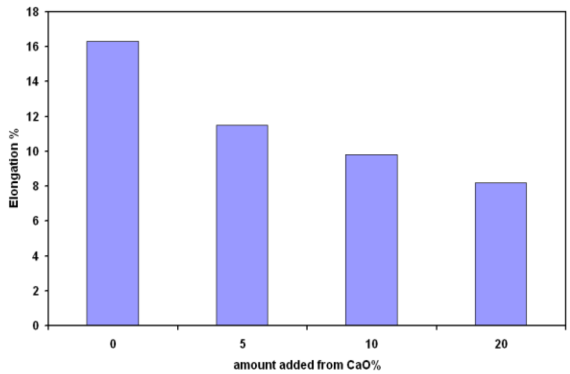


Fig.(8) The relation between Elongation % of the RSF/HDPE composites with different amount of CaO added.

3.4 Flexural Test

The flexural strength of the composites measured using 3-point bending tests are shown in Fig. (9) The flexural strength increases with increase of modified nanoclay up to2.5 wt% .This increase was expected due to the improved adhesion between components in the composites. These results imply that the flexural properties of HDPE/rice straw fiber composites could be enhanced significantly by tailoring the Coupling agent content and the method of nanoclay addition in the composites. [31].

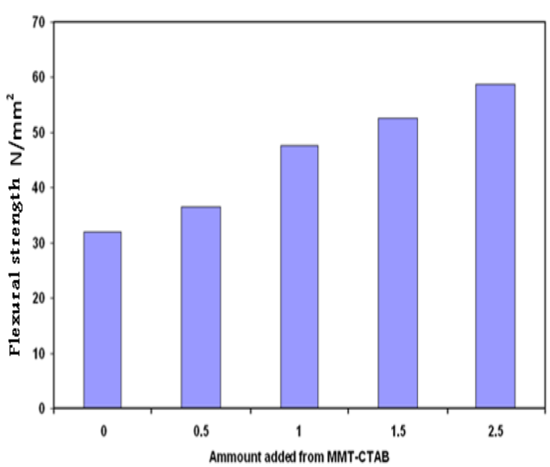


Fig.(9) Effect of added amount of MMT-CTAB on the flexural strength (N/mm^2)of the prepared nanocomposites.

The effect of adding CaO amount on the flexural strength of the prepared polymer composites was studied .Results are shown in fig (11) which reveals flexural strength increases as calcium oxide increases .

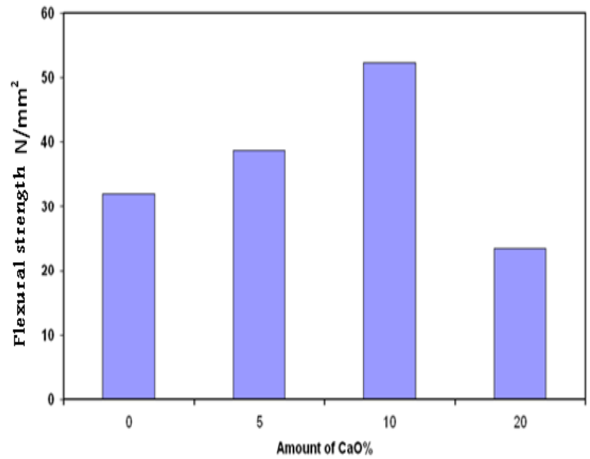


Fig.(10) The relation between the tensile strength (MPa) with different amounts of CaO.

3.5 Charpy Notched Izod Impact Test

Effect of (Na-MMT) treated with cetyltrimethylammonium bromide (CTAB) content on rice straw fibers and high density polyethylene composites on the notched izod impact test are shown in Fig.(11). Represents the effect of MMT-CTAB amount loaded on the impact strength of RSF/HDPE composites. The impact strength decreases at small amounts loaded of Nanoparticles MMT-CTAB, followed with increasing the impact strength sharply with increasing the loaded Nanoparticles. Fig (12) it was observed that, the impact strength decreases as the calcium oxide increases,

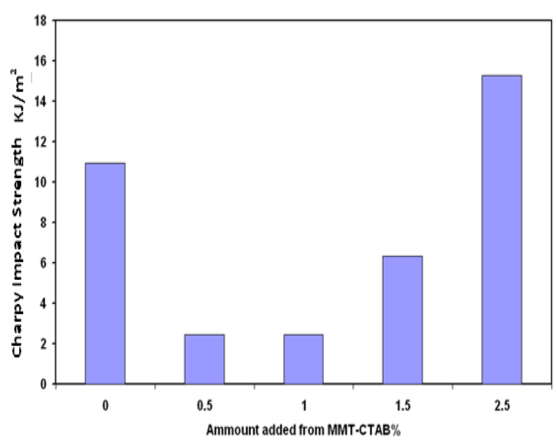


Fig.(11) The change in notched Izod impact strength with the amount of MMT-CTAB increase .

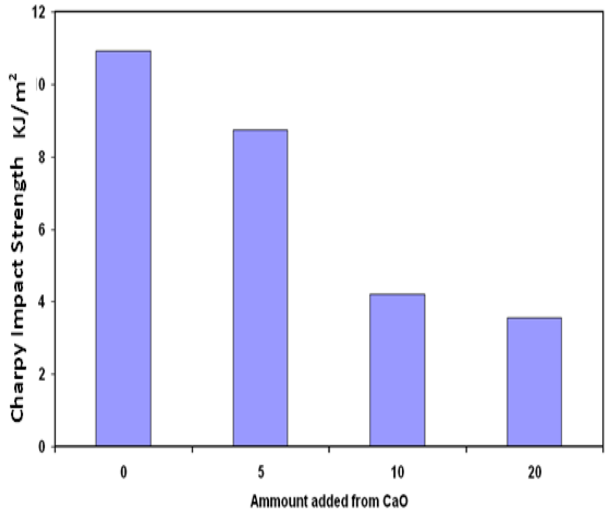


Fig.(12): The change in notched izod impact strength with the amount of CaO loaded with RSF / HDPE composites.

3.6 Hardness:

Hardness is an important test for different applications of rice straw /high density polyethylene/MMT-CTAB composites. The change in the Hardness with loaded amounts from MMT-CTAB on the RSF/HDPE composites was studied. The obtained results are shown in Fig (13).hardness of the composites increased with increasing the nano-particles MMT-CTAB in the polymer composites. From fig (14) hardness increases as amount of calcium oxide increase.

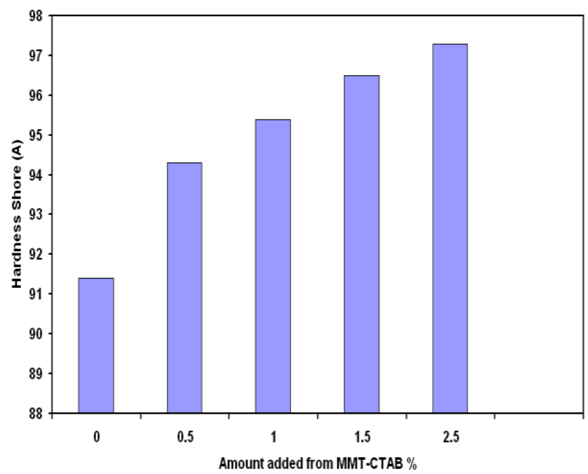


Fig.(13) The change in Hardness (Shore A) with different amount of MMT-CTAB.

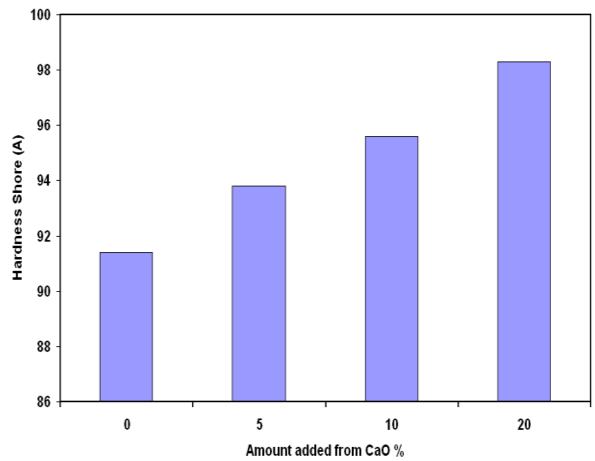


Fig. (14) The change in hardness (shore A) with different amount of CaO.

3.7 Water uptake:

The moisture penetration into polymer matrix composites is explained by three different mechanisms [32]. The first type the direct diffusion of water molecules into the matrix and with much lesser extent, into the fibers second is the capillary form of flow of water molecules along the fiber-matrix interface followed by its diffusion into the bulky polymer matrix. It tends to occur preferentially along the polymer-filler interface. However, this is expected to happen only when the debonding between the fibers and the matrix occurred, and the debonding which occurred is often as a result of water attack at the interface. The third mechanism is the transport of water through micro cracks or other forms of micro damage in the matrix, such as pores or small channels already present in the material or expanded by water due to swelling. Thus, each of them becomes active only after the occurrence of specific damage to the composites. The activation of these mechanisms is distinguished in increasing both the rate and the maximum capacity of moisture absorption in an auto accelerative manner. [33].In this study moisture uptake gives the water absorption characteristics of the rice straw/HDPE/MMT-CTAB composites. The obtained results for the water uptake with time are shown in Fig(15).which Indicates that nanoclay modified with CTAB reduces the affinity for water absorption.

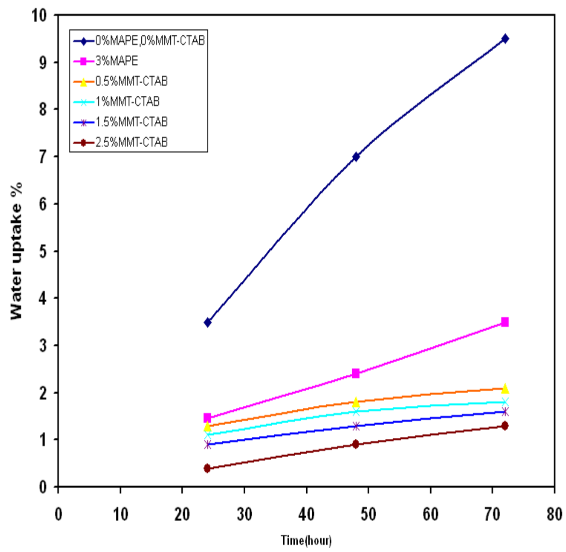


Fig.(15) The change in the water uptake %with time.

3.8 Thermal stability of the prepared Rice Straw /HDPE composites.

For studying the thermal stability of the prepared polymer composites, the Thermogravimetric analysis (TGA) was carried. The obtained results for the weight loss% changed with temperature were recorded and the obtained results are shown in Fig(16).From the figure it can be detected that at lower temperature until 200°C there is no large difference between the prepared composites formed from RSF/HDPE in presence of MAPE as compatibilizer and that after adding 2.5%Nanoclay-CTAB,also when adding 10% CaO instead of nanoclay, compared with pure PE.

In the second part of the curves in the range of 220-425 °C large difference between the different types of composites. It was found the most stable for the pure PE, followed by the composites formed in presence of CaO, than that containing nanoclay modified with CTAB and the lowest stable composites, is that formed from RSF /HDPE only when it lost 3% by weight at the beginning of this region and reached to 30% at 425 °C .For PE the loss weight was 0% at 220 °C and reached to 10% at end of this region. For the third region between 425 °C up to 500 °C .Opposite behavior was detected; the loss weight for pure PE increased ,but the different type of polymer composites, the residue present was at about 11%.From the Thermogravimetric study it can be concluded that ,the thermal stability of pure PE is higher than that of any other type of prepared polymer composites up to high temperature (above 220 °C) but at normal temperature for using of polymer composites instead of rice straw (up to 220 °C) there is no difference between Pure PE and the prepared RSF/HDPE composites. The thermal decomposition of each sample took place in a programmed temperature range of 25°C to 600°C.According to TGA analysis, results the increase of rice straw fiber level increases the thermal degradation of composites in early stage, but decreased in late stage [34].

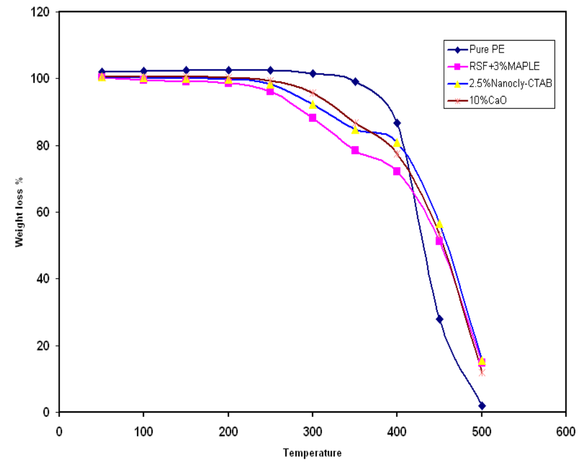
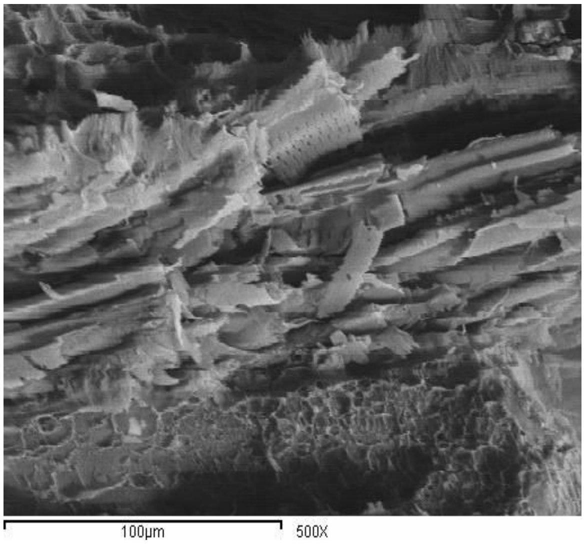


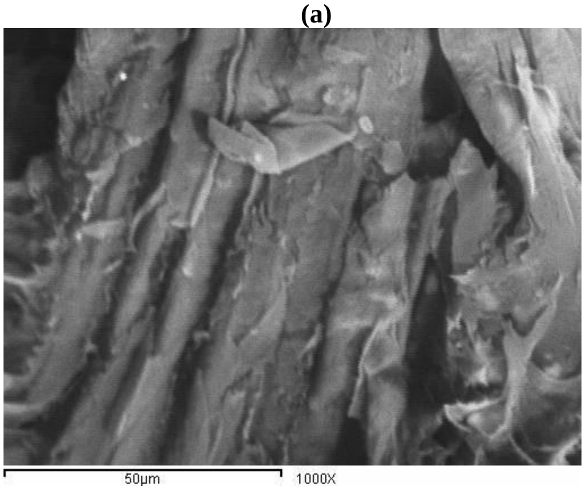
Fig. (16) Relation between Weight loss % at different Temperatures.

3.9 Scanning electron microscopy (SEM)

Samples were immersed in liquid nitrogen and fractured to ensure that the microstructure remained intact. Then samples were sputter coated by gold using a sputter-coater to avoid charging under the electron beam .Fig (17) and Fig (18).Represents the cross section of the specimens for RSF /HDPE / 2.5 %MMT-CTAB nanocomposites, RSF/HDPE/CaO composites respectively. It was found that fiber bundles are embedded in the matrics. Modified nanomontmorillonite added to rice straw fibers/HDPE composites improves dispersiability, compatibility and interfacial bonding between rice straw fiber and HDPE as seen from figure(17), bonding between the filler and the matrix polymer is strong, and this reflects good dispersion of Nanoparticles in the prepared composites. This indicates in the presence of compatibilizing agent (MAPE). Improved interfacial bonding leads to improved tensile property, which is reflected in the increased strength. It is seen in fig (17a) fiber pulled out due to fracture of the fiber after immersion in liquid nitrogen. Fig (18) shows the effect of calcium oxide in homogeneity of the composites also good dispersion of the particles of CaO that are more dispersed with rice straw and HDPE networks.

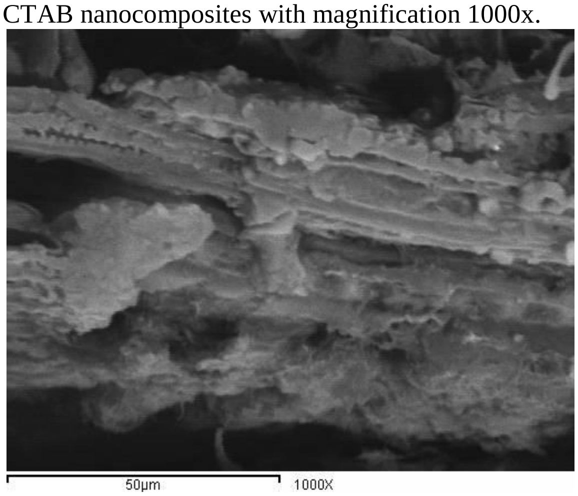


(a)

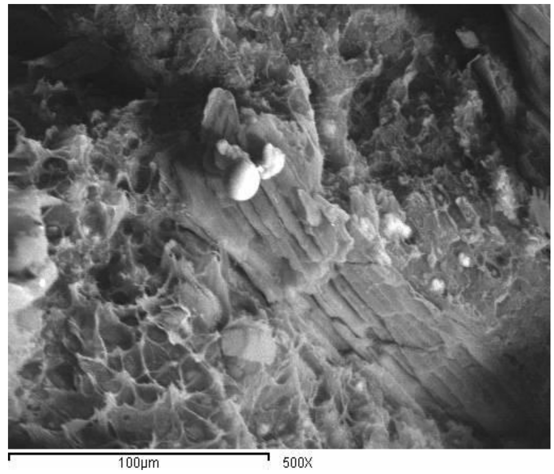


(b)

Fig. (17) SEM observations of the composites (a)RSF/HDPE/2.5%MMT-CTAB nanocomposites with magnification 500x (b) RSF/HDPE/2.5%MMT-CTAB nanocomposites with magnification 1000x.



(c)



(d)

Fig.(18)SEM observations of the composites (c) RSF/HDPE/CaO composites with magnification 1000x (d) RSF/HDPE/CaO composites with magnification 500x.

Conclusions:

The aim of this research was to study the rheological behaviors of HDPE/Rice Straw Fiber/MMT-CTAB nanocomposites in presence of MAPE as a coupling agent. The physico-mechanical properties were evaluated. Tensile strength, flexural strength and impact strength of the prepared polymer nanocomposites were found to be improved by increasing the loaded amounts of modified MMT Nanoparticles. Morphology was investigated by SEM which show modified nanomontmorillonite added to rice straw fibers/HDPE composites improves dispersiability, compatibility and interfacial bonding between rice straw fiber and HDPE. Also CaO has a good effect to improve the physico- mechanical properties when added in 10% of ratio.

Corresponding author
Abdel -Salam Sabbah I.
Department of Chemistry, Faculty of Science, Al-Azhar, Cairo, Egypt
emadsobhy2007@gmail.com

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