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Comparative studies on physico-mechanical properties of composite materials of low density polyethylene and raw/calcined kaolin



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ABSTRACT

The paper describes the preparation of the composite materials of low density polyethylene (LDPE) as the base mixed separately with raw kaolin and the same calcined at 800 °C under the same variation in weight percentage using single-screw extruder and a mixing machine operated at a temperature between 190 and 200 °C. Some of the mechanical and physical properties such as Young's modulus, elongation at break, shore hardness and water absorption were determined at different weight fractions of filler (0, 2, 7, 10 and 15%). It was found that the addition of filler increases the mechanical properties. Absorption test was done in water at different immersion times for different composites. The degree of water absorption of composite materials was found to decrease with increasing wt% of kaolin filler (0–15%) according to Fick's law. Calcined kaolin produces better mechanical properties than raw kaolin.

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1. Introduction

Materials are classified as metals, alloys, ceramics, glasses, composites, polymers, and semiconductors [1]. Ceramics can be defined as inorganic crystalline materials, probably the most nature derived materials obtained from kaolins after proper thermal treatment. The natural colouring of kaolins is due to the presence of mineral impurities-either phyllosilicates or chromophore elements in the crystalline lattice of the kaolinite [2], such as Fe in isomorphic substitution with octahedral Al [3]. When kaolinite is calcined [4,5], it is transformed into metakaolinite, a material which also has industrial applications. Metakaolinite features a highly disordered structure with remnants of the Si-O networks, those appear as an outcome of dehydroxylation of Al-O-OH networks followed by reorganization [6,7]. Also important is the change of hexa-coordinated Al to tetra-coordinated Al [8]. The calcination of kaolin at 650–700 °C [9–11] may favourably modify the properties of resiliency, opacity and dielectric character, and as the chemical surface becomes more compatible with organic systems, it finds wide applications [12]. The change of colour upon calcination may be due to changes in the oxidation state of the heavy elements, like

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Fe, or to the loss of part of the structural iron from the crystalline structure [12,13].

India has high quality kaolin clay deposits at Patel Nagar, Birbhum District, West Bengal that possesses good potentiality to produce metakaolin. Metakaolin ($Al_2Si_2O_7$) is a highly reactive pozzolan made through the calcination of a clay rich in kaolin. The reactivity of metakaolin depends on various factors, such as temperature, heating rate and cooling regime. The optimal temperature window is 700–800 °C, although the dehydroxylation of clay is known to begin above 500 °C [13].

$$Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O \rightarrow Al_2O_3 \cdot 2SiO_2 + 2H_2O \uparrow$$
(1)

The thermal transformation of kaolinite, which has been the subject of a large number of investigations [14–17], has proved that the heating parameters such as temperature, heating rate and time, as well as cooling parameters, significantly control the dehydroxylation process.

Composites are made from different types of materials. They provide unique combination of mechanical and physical properties that cannot be found in any single material. They are light in weight, hard, ductile, high temperature resistant as well as shock-resistant materials [18,19]. Low density polyethylene is defined by a density range of 0.910–0.940 g/cm³. It is unreactive at room temperature, except towards strong oxidizing agents. Having more branching, this polyethylene exerts weak intermolecular forces, lower tensile strength, and higher resilience. Also, since its molecules are

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Table 1Chemical analysis of kaolin clay.

$\begin{tabular}{ c c c c c c } \hline Sl. no. & Name of chemical & China clay (%) \\ \hline 1 & Silica (SiO_2) & 44.45 \\ \hline 2 & Alumina (Al_2O_3) & 37.75 \\ \hline 3 & Ferric oxide (Fe_2O_3) & 0.73 \\ \hline 4 & Platinum oxide (PtO) & 0.20 \\ \hline 5 & Calcium oxide (PtO) & 0.20 \\ \hline 5 & Calcium oxide (CaO) & 1.28 \\ \hline 6 & Magnesium oxide (MgO) & 0.17 \\ \hline 7 & Potassium oxide (MgO) & 0.08 \\ \hline 8 & Sodium oxide (Na_2O) & 0.08 \\ \hline 8 & Sodium oxide (Na_2O) & 1.04 \\ \hline 9 & LOI & 14.30 \\ \hline Total & 100.00 \\ \hline \end{tabular}$				
	Sl. no.	Name of chemical	China clay (%)	
2 Alumina (Al ₂ O ₃) 37.75 3 Ferric oxide (Fe ₂ O ₃) 0.73 4 Platinum oxide (PtO) 0.20 5 Calcium oxide (CaO) 1.28 6 Magnesium oxide (MgO) 0.17 7 Potassium oxide (K ₂ O) 0.08 8 Sodium oxide (Na ₂ O) 1.04 9 LOI 14.30 Total 100.00	1	Silica (SiO ₂)	44.45	
3 Ferric oxide (Fe ₂ O ₃) 0.73 4 Platinum oxide (PtO) 0.20 5 Calcium oxide (CaO) 1.28 6 Magnesium oxide (MgO) 0.17 7 Potassium oxide (K ₂ O) 0.08 8 Sodium oxide (Na ₂ O) 1.04 9 LOI 14.30 Total 100.00	2	Alumina (Al ₂ O ₃)	37.75	
4 Platinum oxide (PtO) 0.20 5 Calcium oxide (CaO) 1.28 6 Magnesium oxide (MgO) 0.17 7 Potassium oxide (K ₂ O) 0.08 8 Sodium oxide (Na ₂ O) 1.04 9 LOI 14.30 Total 100.00	3	Ferric oxide (Fe ₂ O ₃)	0.73	
5 Calcium oxide (CaO) 1.28 6 Magnesium oxide (MgO) 0.17 7 Potassium oxide (K ₂ O) 0.08 8 Sodium oxide (Na ₂ O) 1.04 9 LOI 14.30 Total 100.00	4	Platinum oxide (PtO)	0.20	
6 Magnesium oxide (MgO) 0.17 7 Potassium oxide (K2O) 0.08 8 Sodium oxide (Na2O) 1.04 9 LOI 14.30 Total 100.00	5	Calcium oxide (CaO)	1.28	
7 Potassium oxide (K ₂ O) 0.08 8 Sodium oxide (Na ₂ O) 1.04 9 LOI 14.30 Total 100.00	6	Magnesium oxide (MgO)	0.17	
8 Sodium oxide (Na20) 1.04 9 LOI 14.30 Total 100.00	7	Potassium oxide (K ₂ O)	0.08	
9 LOI 14.30 Total 100.00	8	Sodium oxide (Na ₂ O)	1.04	
Total 100.00	9	LOI	14.30	
		Total	100.00	

less tightly packed and of inferior quality of crystalline character, its density is lower [1,18]. Many workers investigated the physico-mechanical properties of filled polyethylene with additives. Vlasova [19] studied how the degree of filling and the nature of the fillers do influence the properties of polyethylene composites obtained by polymerizing ethylene on the surface of the filler particles. He found that the breaking stress and relative elongation at rupture with rise in the degree of filling diminish, while the elasticity modulus on stretching and the toughness of the polyethylene-kaolin composites rise. The scales of change in the properties of the composites with the degree of filling are determined by the nature of the filler. Hindryckx [20] studied the interfacial adhesion in polyethylene-kaolin composites using a stearic acid and maleic anhydride-grafted polyethylene as potential interfacial agents with consideration of the efficiency of a minosilane-surface-treated kaolin. They found that the melt index of the enhanced interfacial filler-polymer had decreased. Comparing with other low molecular weight additives such as stearic acid and aminosilane, they also concluded that anhydride-grafted polyethylene was very efficient in improving the impact resistance of HDPE kaolin composites even at low contents. Al-Neamee [21] investigated the mechanical and physical properties of polyethylene that are reinforced with alumina. She found that mechanical properties increase with increase of wt% of alumina and the absorption water decreased with the addition of filler. The objective of this study is to prepare the composite materials fabricated from a low density polyethylene as base and different weight fractions of raw and calcined kaolin of Patel Nagar by single-screw extruder to make affordable materials with the desired physico-mechanical properties.

2. Experimental

2.1. Materials

The present study shows that starting clay is high quality kaolin, with alumina content of about 60%. Kaolin clay was collected from Patel Nagar and Muhammad Bazaar, Birbhum District, West Bengal, India. Before being characterized, the samples were dried up to 0.5% moisture content, crushed and milled. Chemical compositions of two different types of raw clays were determined by silicate method (Table 1). Commercial low density polyethylene was supplied by E. Merck. The melt flow index and the density of the material were 20 g/10 min and 0.9235 g/cm³, respectively.

Table 2

Extrusion parameters.

2.2. Composite synthesis

In this work, mechanical mixing was used to prepare composite materials from low density polyethylene/kaolin using extruder machine. Low density polyethylene (LDPE) was mixed with kaolin filler under various compositions (0, 2, 7, 10 and 15%). The mixture was then fed into 25 mm single-screw extruder. The machine was connected with electric heater to melt the materials by shearing between the barrel and the smooth cylindrical part of the screw. Finally the flat-end part of the screw acted as a pump forcing the molten composite materials to come out. The barrel temperature was monitored and controlled by thermostat. The die temperature was also controlled by a thermostat and was adjusted, together with barrel temperature to yield uniform output. The extrusion parameters are listed in Table 2. The monofilament of about 2 mm diameter produced by the extruder at screw speed limit of 0-50 rpm was found to be uniform and opaque. The monofilament was then cooled after which it was cut in the form of square like grids of 3-4 mm length. The sheet was prepared by pressing the grids between hydraulic plates at 190 °C for LDPE and LDPE/kaolin. A pressure of 10 MPa was applied for 5 min to allow the composite to melt and the molten mass spread out between plates. The pressure was released and the mould sheet was quenched in water at room temperature.

Melt flow index (MFI) was determined using CEAST modular line flow indexer ASTM D1238 applying a load of 5 kg at 200 $^{\circ}$ C.

3. Characterization techniques

3.1. Tensile strength test

It can be defined as the maximum tensile sustained by the material being tested to its breaking point [17].

Tensile strength =
$$\frac{F}{A}$$
 (2)

where F = force applied in N and A = cross-section area in mm [17]. The tensile strength values were determined by using tensile test according to ASTM (D638) [21]. Tensile properties were obtained using Zwick Universal Testing Machine. The cross-head speed was 10 mm/min, and the standard literature for tensile test, Dumbbell-shaped was the general purpose test piece for plastic materials according to ASTM (D638); so test Specimens type 1434 with a thickness of 4 mm was used in this work. Elongation was measured by using Zwick machine. The measurement was carried out according to the test specification of ASTM (D638).

Elongation

$$= \frac{\text{final} \times \text{gauge} \times \text{length} - \text{original} \times \text{gauge} \times \text{length}}{\text{original} \times \text{gauge} \times \text{length}} \times 100 \quad (3)$$

3.2. Impact strength test (IS)

The degree of resistance of a polymeric material to impact loading is a matter of concern in some applications [17]. Charpy test was used to determine the impact strength of the polymeric material; the samples of impact test were notched by (Notch instrument)

Polymer and clay	Temperature (°	Temperature (°C)			
	Zone 1	Zone 2	Zone 3	Zone 4	
Low density polyethylene (LDPE) LDPE/kaolin	150 150	170 190	220 250	190 220	50 50

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