

# Scanning Electron Microscope Probe of Low Density Polyethylene Doped with Alumina Nanocomposites

\*A. Aliyu<sup>1</sup>, U. Ahmadu<sup>2</sup>,  
A. A. Abdelmalik<sup>1</sup>, S. S. Maidawa<sup>1</sup>  
<sup>1</sup>Department of Physics,  
Ahmadu Bello University,  
Zaria, Nigeria

<sup>2</sup> Department of Physics,  
Federal University of Technology,  
Minna, Nigeria  
Email: aliyuelokene@gmail.com

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## Abstract

Nano-sized kaolin also known as alumina was synthesised from local kaolin and subsequently suspended in low density polyethylene (LDPE) to produce nano-polymer for application in electrical insulation. The internal behaviour of the prepared polymeric insulation was studied to understand the structure of the nano-polymer. The alumina was prepared by acid leaching via digestion, precipitation, peptization and drying. The prepared alumina was subsequently used to dope the LDPE at varying percentage compositions. The structural properties of LDPE/ $\gamma$ -alumina with LDPE as a host matrix were investigated by Scanning electron microscopy (SEM). The SEM images revealed a heterogeneous dispersion of nano particle layers in the LDPE matrix owing to the nature of the dispersed alumina. Uneven dispersion of nano particles was evident due to agglomeration of alumina.

**Keywords:** Alumina, low density polyethylene (LDPE), nanocomposites, agglomeration,

## INTRODUCTION

Low density Polyethylene recently gained some popularity as power cable insulation when it was first introduced in 1960s, due to its low cost, better electrical properties compared to paper-oil insulation, mode of processing, moisture and chemical resistance, and low temperature flexibility (Andrews *et al*, 2006). Polymer/clay nanocomposites are an important class of organic-inorganic composites in which clay platelets are dispersed at a nanometric level in the polymer matrix, acting as a reinforcing phase (Silvia *et al*, 2013). With a very small addition of clay into the polymer matrix while achieving nanometric dispersion, the resulting nanocomposites exhibit substantial improvements in its physical properties (Ray *et al*, 2003; Grandjean *et al*, 2006). This is because the properties of nano-composite materials depend not only on the properties of the parent constituent material or matrix but also on their morphology and interfacial characteristics (Rao *et al*, 2004). Therefore, there is no doubt that inorganic mineral fillers has helped in increasing polymer performance properties, such as mechanical, structural and electrical.

Previously, Priya and Jog (2002) and Priya and Jog (2003) were the first to prepare Polymer-clay nanocomposites. They observed from the modified Cloisite used that the addition of organically modified clay resulted in the formation of the  $\beta$ -phase instead of the  $\alpha$ -phase upon melt processing. Usually the general class of clay are hydrophilic in nature and are stacked

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\*Author for Correspondence

together. In order to effectively disperse the clay into an organic polymer matrix and increase compatibility, it is usually necessary to modify the hydrophilic character of the silicate into organophilic character, so as to make it hydrophobic in order to ease its dispersion into the polymer matrix.

In general, the material properties of polymer nanocomposites are superior to the pure polymer matrix or composites with larger sized inclusions. The effects of the nanoparticles are dependent on many variables but especially upon the relative crystalline or amorphous nature of the polymer matrix as well as the interaction between the filler and matrix. Liqiang and Seong, (2008), in their work, preparation and characterization of polyethylene (PE)/clay nanocomposites by *in situ* polymerization with vanadium-based intercalation catalyst successfully carried out complete exfoliation of clay during Vanadium-based Ziegler-Natta polymerization of ethylene. Even though the mechanical properties of the PE/clay nanocomposites have a noticeable improvement when the clay was exfoliated and well dispersed in the PE matrix, improvement in the electrical properties was not considered. Owing to the high need of enhancement of dielectric property of flexible polymeric materials with nano-ceramics, it will be imperative to look into how much of its structure has been altered during the course of adding the nanoparticles. Ching *et al* (2012) in their work characterization of mechanical properties: low-density polyethylene nanocomposites using nanoalumina particle as filler obtained a SEM micrographs of LDPE/alumina at 0, 1 and 5wt% of alumina loading. Also Youyuan *et al* (2017), in their work effect of nanoparticles on the morphology , thermal and electrical properties of low density polyethylene after thermal aging was able to establish that there was variation of structures after aging with with different dopant. The structural analysis was necessary principally to evaluate the morphology and the extent of dispersion of the nano-particles to ascertain the microstructure of the alumina. It was also important to determine the mechanism by which the nano-particles interact with the polymer matrix.

## **MATERIALS AND METHODS**

### **Materials**

Low density Polyethylene(LDPE) (commercial grade), Hydrochloric Acid (HCl) of M. W., 36.48 and purity 35.38% was supplied by BDH Chemical Ltd, Poole England. Ethanol (Absolute) of purity 99.7% was gotten from Johnson Solomom (Export) Ltd, London, England. NaOH of M. W. 40.0g/mol was also supplied by BDH. Polyvinyl Alcohol (PVA) of 98% purity was supplied by BDH Raw kaolintic clay sample. Toluene Analar-M.W. 92.14 and 0.86g/mol .Alum (commercial grade), potash (commercial grade)

### **Sample Preparation**

#### **Preparation of Alumina From Kaolin by Acid Leaching**

Nanokaolin synthesis from kaolin has proved to be very challenging especially through acid leaching process owing to the various acids involved. The safe handling of the acids requires a researcher to wear protective agents and handle the acids with utmost care in order to avoid laboratory accidents. It appears to be the only option available in this case due to the ready to be used nature of the acids. The processes are Digestion, Precipitation, Peptization/Gelling/Capping/surfactant and Drying

### **Digestion**

Kaolin was first calcined for 4 hours at 600°C in a furnace. 150g of calcined Kaolin was digested with 250 ml HCl using a KDM Style Control Mantle under reflux at the boiling point (108.6 °C) of HCl for 70 mins. The mixture will now be precipitated.

### **Precipitation**

The digested Kaolin was allowed to sediment by ageing it in the refrigerator for 18 hours. The sedimented solution was decanted and then filtered to yield a clear solution. The filtrate was precipitated by the addition 25g of NaOH while keeping the temperature at about 10°C. NaOH was added to the clear solution to neutralize it leading to the formation of aluminium hydroxide precipitate in the basic medium. The gelatinous precipitate of the hydrated alumina was further filtered under vacuum using a vacuum pump, washed repeatedly five times with distilled water and finally with absolute ethanol. The precipitate was then dried, ashed at 400°C for one hour in a muffle furnace to burn any carbonaceous materials. The crude nanokaolin formed was stored in a desiccator.

### **Peptization/Gelling**

A small amount of the crude nanokaolin was weighed into a crucible and dried further for 1 hour at 400°C. 10ml of 70% nitric acid was then added to 18.100g of the hydrated alumina in a 500ml beaker. This was done with constant and vigorous stirring until it turned almost translucent. More distilled water was then added to make it up to 100ml. In a separate beaker, 5g of polyvinyl alcohol (PVA) was dissolved in 100ml of water and boiled with stirring until the colloids completely dissolves. The PVA acts as *surfactant* to control hydrolysis. After the dissolution, it was kept in the refrigerator to in order to get chilled. Both the hydrated alumina and the dissolved PVA were mixed together and stirred for 40 minutes. This process of adding PVA to alumina and mixing is called *capping*.

After the capping, ammonia was added to make the PH to 8. At this point the solution turns into a thick gel due to condensation. Again the mixture was finally stirred for 40 minutes to age the sample and poured on a filter paper and allowed to dry at 60°C in a hot oven

### **Drying**

The dried sample is broken to pieces and calcined in a furnace at 600 °C for 4 hours to burn out the remaining carbonaceous materials such as PVA and volatile ammonium nitrate contaminants. The process also helps to crystallize the alumina (Nanokaolin).

## **MODE OF OPERATION OF SEM**

### **Scanning Electron Microscopy (SEM)**

Dispersion of the silicate layers in the polymer matrix was investigated by using SEM (Scanning electron microscopy (Phenom ProX) from the fractured surface of the tensile samples. The primary goal of using SEM was to determine particle size for interphase analysis, and to determine particle dispersion (Ciprari, 2004). It also reveals local topography in the early stage of the growths of a crystal.

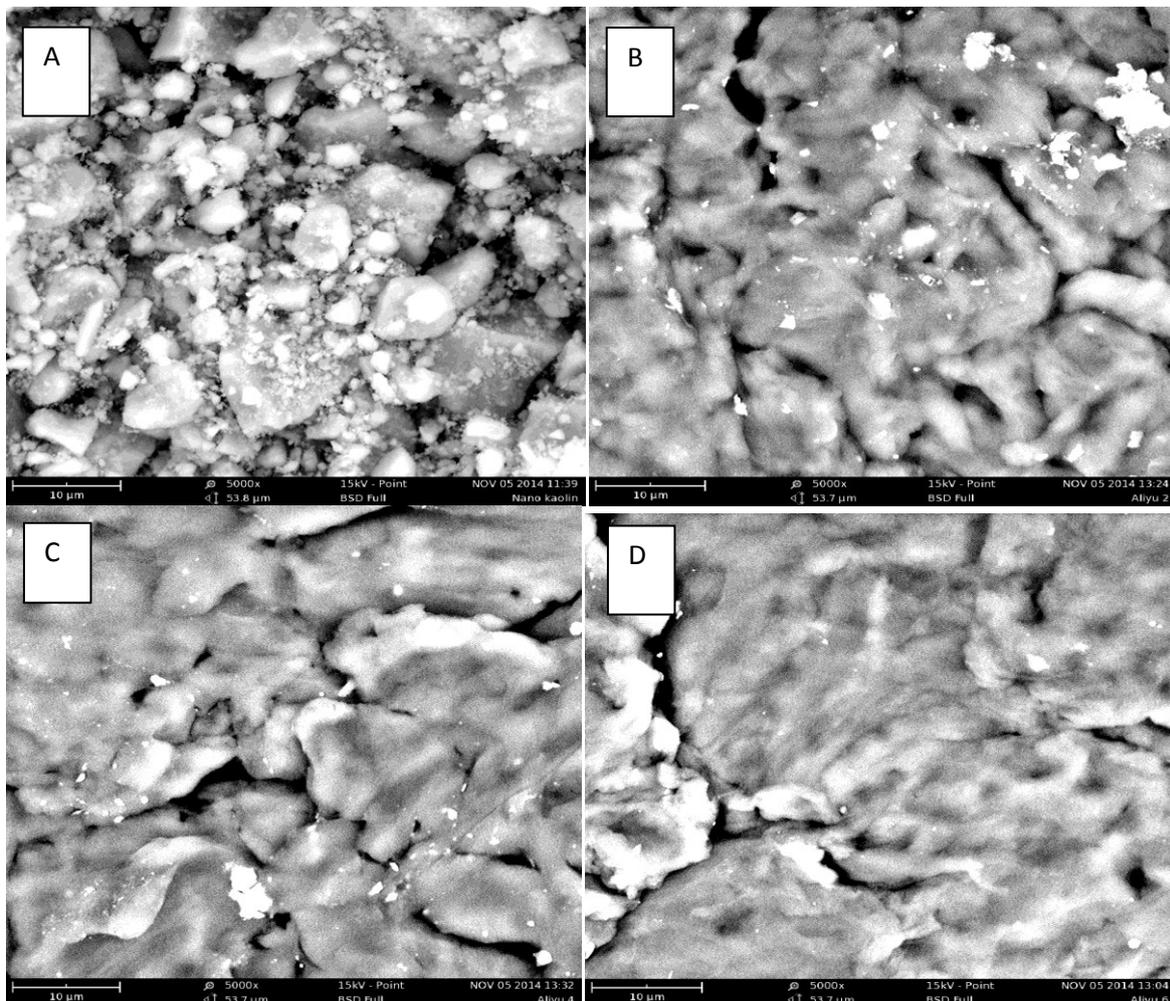
Some unspecified quantity of sample was placed on a double adhesive sticker placed in a sputter coater machine for 5sec before placing the sample in the SEM to give the sample a conductive property, and this will not affect the quality of the images. Sample stub was fixed on a charge reduction sample holder and was then changed into the machine column thereafter the doors of the SEM was closed.

Machine was allowed to stabilize for some seconds, the parameters to be used were then set. 15kV was used in imaging the sample set at 1000x magnification and was focused using a rotary knob until a clear and proper image was produced in a NavCam mode, this was then transferred to an electron imaging mode. The image was then transferred to Phenom suite software where fibermetric/pore measurements were carried out after contrasting/ brighten, after the spots were all analyzed.

## RESULTS AND DISCUSSIONS ON THE SEM ANALYSIS

### Scanning Electron Microscope (SEM)

The SEM techniques studies the structural changes that occur on the surface by giving an insight into the structural arrangements in this case of polymer surface, absorbents and interfacial solvent molecules in the electrical double layer. It also allows the study of the charge transfer reactions and absorption processes occurring at the surface of electrified double layer to be altered. Each of the samples reported was subjected to the same procedures to get the desired structure. The structures obtained using SEM measurements are shown in fig 3.1:



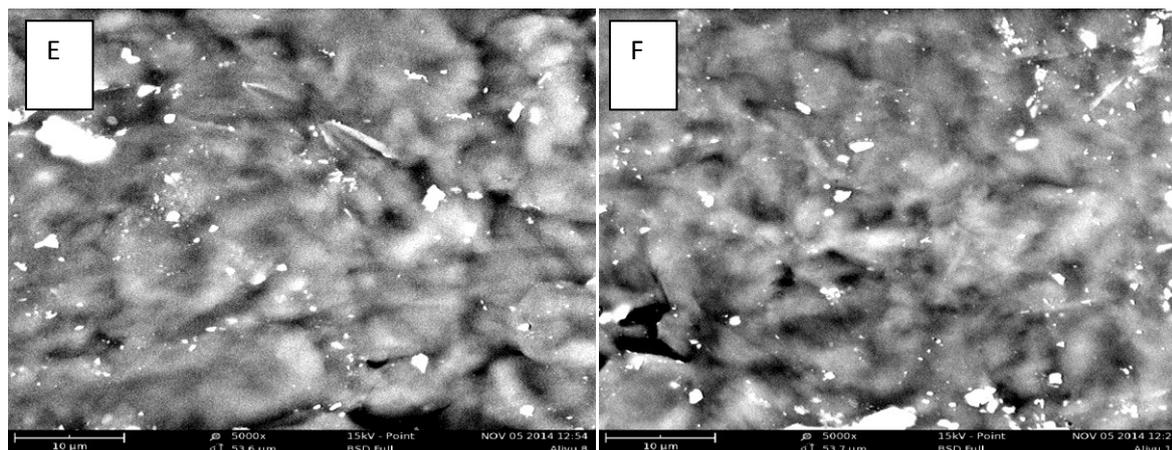


Fig 3.1: Backscattered SEM images of (A) alumina from kaolin synthesised by acid leaching, (B) LDPE doped with 1.0% wt Alumina, (C) LDPE doped with 2.0% wt Alumina, (D) LDPE doped with 3.0% wt Alumina, (E) LDPE doped with 4.0% wt Alumina, (F) LDPE doped with 5.0% wt Alumina; ; all images were taken at 15kV and with magnifications (5000x).

In fig 3.1 (B&C), the nanoparticles used in doping was manifest showing that the material is more of intermediate than exfoliated or intercalated nanocomposites. This is due to the percentage increase in the addition of the alumina to the LDPE from 1.0wt to 2.0 %wt of alumina which appeared to be partially dispersed. And this is expected to be more pronounced as the addition of alumina increases gradually to 5.0 %wt. In such doping mechanism, many particles of varying sizes are visible, appearing as small bright dots, fairly well dispersed across the image. There seems to be no obvious effect on the morphology by considering the amount of agglomeration on the fracture surfaces observed by SEM from 4.0%wt to 5.0 %wt doping.

Generally, the SEM images of the samples indicate that the particles are unevenly dispersed throughout the LDPE samples. Areas with no visible particles were found in each sample. This uneven dispersion occurred while using poly(methyl metecrylate)/alumina and it was explained to be due to ultimate strain and stress that results in the samples (Ash *et al*, 2001).

## CONCLUSION

Alumina was prepared via acid leaching with kaolin. The kaolin as raw materials demonstrated the potential to produce alumina with the use of acid.

The morphology and microstructures of the samples have been examined by scanning electron microscopy. An intermediate nanocomposite material was produced. The SEM images of the samples indicate that the particles are unevenly dispersed throughout the LDPE samples; areas with no visible particles were found in each sample. It's very clear that from SEM images produced when zoomed that the amount of whitish substances which are the nanoparticles, becomes more visible and the pore spaces becomes a little bit pronounced. This is because of the percentage increase in the addition of the nanomaterial to the LDPE from 0.0%w/w to 5.0%w/w

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