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Tensile Properties and Fractographic Analysis of Low Density Polyethylene Composites Reinforced with Chemically Modified Keratin-Based Biofibres

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Abstract

This research has investigated the tensile properties and fractography of animal fibre-reinforced low density polyethylene composites. The composites were synthesized by hot compression moulding using chemically modified white and black cow hair biofibres as the reinforcing phase of composites. Alkaline solutions of varying molarities were used to prepare the chemical treatments in this present study. Tensile properties of the developed composites were evaluated based on molarities of chemical treatment and % fibre loading. Scanning electron microscopy was used to characterize the morphologies of the fractured surfaces of composites. Obtained tensile test results revealed significant enhancement in the tensile properties of composites, with the optimum combination of tensile properties presented by 2 wt% white cow hair biofibre reinforcement treated with 0.15 M sodium hydroxide. Observations from the fractographic analysis of the developed composites revealed shearing of the polymer matrix at the fibre-matrix interface and no fibre pullout behaviour.

Keywords

Animal Fibre, Alkaline Treatment, Fibre-Matrix Interface, Mechanical Behaviour, Polymer Matrix Composites

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1. Introduction

Mammalian hairs especially the human hair have been reported by many authors and researchers to exhibit very good physical and mechanical properties, which in turn account for their intrinsic ability to undergo appreciable mechanical stressing and various types of chemical and thermal treatments without sustaining permanent damage [1]-[3]. This unique behaviour of hair has been attributed to the presence of structural proteins which are essentially keratin in the hair fibre [4]. Hair has been defined as a filamentous biomaterial consisting principally of proteins, most especially keratin [5]. These keratins are scaffolding proteins that combine to form a network of intermediate filaments in the cytoplasm of epithelial cells and their foremost function is fundamentally to provide structural maintenance for cells and tissues [6]. Remarkable works of some researchers have shown that the response of hair fibres to mechanical stresses is highly dependent on the stability of the structure of the cortical keratin of hair fibre and this stability is at times affected by heat and chemical treatments depending on the degree of application [7] [8]. Recent research findings have also shown that appropriate chemical treatments can significantly improve the structural integrity of hair fibres without deteriorating their intrinsic properties [9] [10]. In recent times, these attractive properties of hair fibres have impelled materials scientists especially from the developing countries to reassess the economic importance of hair fibres, and in doing so, they are able to unlock a new vista for the industrial applications of hair fibres, as reinforcements for the synthesis of novel composite materials [11] [12].

Historically, the development of hair fibre reinforced composites dated back to the BC ages, when goat and horse hair fibres were used to reinforce masonry mortar and plaster [13] [14]. However, this application of hair fibres as reinforcements was ephemeral owing to the advent of mineral and synthetic fibres in the ensuing millennia [15] [16]. Although these new fibres demonstrated phenomenal improvement in their applications and in a short time saturated the fibre market, the limitations associated with them in terms of availability, synthesis, pecuniary viability and ecological impact have ultimately obligated materials scientists and engineers of this contemporary era to revisit the prospective applications of animal fibres for composites development [17]-[19]. This revisitation on animal fibres particularly keratin-based fibres for composites development has manifested in quite a remarkable but not exhaustive number of investigations with encouraging results [20] [21]. Dwivedi *et al.* used human hair fibre to reinforce polypropylene and documented a significant improvement in the mechanical properties of the developed composites; the work of Oladele *et al*. also showed an improvement in the flexural properties of cow hair fibre reinforced high density polyethylene composites [22] [23]. This present study is aimed at investigating the efficiency and suitability of utilizing cow hair fibres as the reinforcing phase in low density polyethylene polymer matrix.

The first of the polyolefins, Low Density Polyethylene or LDPE, was originally synthesized some fifty years ago by the high pressure polymerization of ethylene. Its relatively low density arises from the presence of a small amount of branching in the chain, on about 2% of the carbon atoms. This gives a more open structure. It is translucent to opaque, robust enough to be apparently unbreakable and at the same time quite flexible; it may be used at temperatures up to 95˚C for short periods and at 80˚C continuously [24]. Despite competition from new contemporary polymers, LDPE continues to be an important plastic grade. In 2013, the worldwide LDPE market reached a volume of about 33 billion dollars (USD) [25]. Applications of LDPE are extensively seen in low load bearing materials such as general purpose tubing, pipette washing equipment, wash bottles, plastic bags for computer components, wire and cable insulations, utensils, playground slides and toys [26]. Despite the vast areas of applications presented by LDPE, its associated drawbacks include low strength and stiffness, susceptibility to stress cracking, flammability, high gas permeability particularly CO_2, poor temperature capability and poor weathering resistance [27]. Numerous approaches to progressively surmount these limitations utilizing varieties of reinforcements from microfibres to nanotubes in the matrix of LDPE are voluminously available in existing literature [28]-[30]. Nevertheless, the utilization of cow hair fibres as a potential reinforcement in alleviating these unpromising limitations has not been documented and this in particular, contributes to the novelty of this research.

The relative abundance of cow hair fibres in Nigeria [31], their unsanitary methods of disposal [32], and the exigent compulsion to appreciably promote and concurrently encourage the development of low cost polymer matrix composites in Nigeria, with a view to moderating the dependence of the nation's polymer-consuming industries on foreign polymers [33], are the stimulating objectives for selecting cow hair fibres in this present study. In addition, the selection of sodium hydroxide or NaOH and potassium hydroxide or KOH for the purpose of this research is due to their relative availability, cost effectiveness and successful treatment history on natural

fibres [34].

2. Materials and Method

2.1. Materials

2.1.1. Cow Hair Fibres

White cow tail hair fibres, Zebu breed and black cow tail hair fibres, N'Dama breed were sourced and procured from abattoirs in Akure, Ondo State, Nigeria. The average age of cows from which the hair fibres were scraped is 2 years ± 6 months. The cows were raised in North-Central Nigeria under free-range system where the temperature across the year varies between 22.55˚C ± 0.423˚C in the wet season and 33.54˚C ± 0.23˚C in the dry season [35].

2.1.2. Chemical Treatments

The chemicals used for preparing the treatments were sourced and procured from Pascal Scientific Akure, Ondo State, Nigeria.

2.2. Methods

2.2.1. Chemical Treatment of the Cow Hair Fibres

As-procured black and white cow hair fibres of average diameters 130 ± 30 µm were thoroughly washed with tap water and detergents to remove impurities such as blood stains, oil and grease that stuck to the fibre surface. This was ensued by rinsing with distilled water and drying at a temperature of 26˚C ± 2˚C for 5 days. The fibres were cut into 45 mm fibre length to develop two-dimensional near isotropic composite structures. In order to enhance the surface topography and hydrophobicity of the fibres, for appreciable mechanical bonding between fibres and the LDPE polymer matrix, fibres were dipped into predetermined molar concentrations of 0.05, 0.10 and 0.15 M of KOH and NaOH treatments having pH values of 8, respectively. Subsequently, the chemical treatments containing the fibres were heated in a shaker water bath maintained at 50˚C for 4 hours to further enhance the adhesive property of fibres.

2.2.2. Development of the Cow Hair Fibre-Reinforced LDPE Composites

The animal fibre-reinforced LDPE composites and the control test samples were synthesized by hot compression moulding technique. To produce the composites, the matrix and the fibres were mixed together in predetermined proportions as shown in **Table 1**.

The materials were weighed on an electronic weighing balance prior their pouring into tensile test moulds, made of steel. To achieve homogeneous mixture of the fibres within the LDPE matrix and a near isotropic structure of the LDPE composites, mixing was done with a mixing spoon. Afterwards, the filled moulds were placed inside a compression moulding machine maintained at 160˚C for 5 minutes. Upon full compaction of the developed test samples, they were extracted from the mould in a warm condition and allowed to cure in air. The same compositions were used for all the developed composites.

2.2.3. Tensile Testing

The tensile properties of the test samples were evaluated using an Instron universal testing machine, Model 1195. The tests were performed at a fixed crosshead speed of 10 mm min^{-1} at 25˚C. Test samples were prepared according to ASTM D412 standard [36]. To ensure accuracy of test results, three repeatability tests were performed on the test samples for each evaluated tensile property.

2.2.4. Fractographic Analysis

The fracture behaviour of the developed LDPE composites was studied with scanning electron microscopy, Model

Table 1. Composition of the cow hair fibre-reinforced LDPE composites.

Composition	wt%	wt%	wt%
LDPE	98	96	94
Fibre	2	4	6

JEOL JSM-6480LV. The samples were thoroughly cleaned, air-dried and coated with 100 Å thick platinum in a JEOL sputter ion coater and observations were made at 20 kV. Also, to enhance the conductivity of the composite samples, a thin film of platinum was vacuum-evaporated on the test samples prior the microphotography.

3. Results and Discussion

3.1. Tensile Properties

3.1.1. Tensile Stress at Peak

The variation of tensile stress at peak with molar concentrations of the chemical treatments for the cow hair fibre (CHF)-reinforced LDPE composites and the unreinforced LDPE polymer (control) is presented in **Figure 1**. Observations from this result revealed that increase in molar concentration of the NaOH treatment is directly proportional to increase in the ultimate tensile strengths of most of the LDPE composites reinforced with NaOH-treated CHFs and for the KOH treatment there is no direct relationship between increase in molar concentration of the KOH treatment and enhancement in the ultimate tensile strengths of the LDPE composites reinforced with KOH-treated CHFs. Also, with respect to % fibre loading it was observed that there is no consistency in the trend of the LDPE composites relative to tensile stress at peak. This is a clear indication that the fibre population is effectively supported by the LDPE matrix. Categorically, all the developed LDPE composites demonstrated better performances with respect to tensile stress at peak in comparison with the unreinforced LDPE polymer. The superlative performance is given by the LDPE composite reinforced with 0.15 M NaOH-treated 2 wt% white cow hair fibre (WCHF) and it is 186.65% better than the unreinforced LDPE polymer. The least enhancement is given by the LDPE composite reinforced with 0.15 M KOH-treated 4 wt% black cow hair fibre (BCHF) and it still supersedes the tensile stress at peak of the unreinforced LDPE polymer by 24.99%. This significant enhancement in ultimate tensile strengths of the developed LDPE composites can be attributed to improved hydrophobic nature and surface topography of the chemically treated CHFs which in turn manifested in a very strong interfacial adhesion between the CHFs and the LDPE polymer matrix [37]. The work of many research scholars on the mechanical behaviour of fibre-reinforced polymers have shown that appreciable interfacial adhesion between fibre and matrix allows large fraction of the applied stress to be transferred by shear loading at the fibre-matrix interface via the matrix to the fibres and this in turn increases the magnitude of plastic deformation the material can sustain before fracture [38] [39]. The result of this research is in conformity with their analytical explanation. Furthermore, the effect of fibre length and fibre orientation adopted for the purpose of this research is undoubtedly responsible for the outstanding performance of the LDPE composites. Longer fibre lengths have been reported to give significant enhancement in tensile properties, [40] Amuthakkannan *et al*., investigated the effect of fibre length on the mechanical properties of basalt fibre-reinforced polymer matrix composites and documented a superlative improvement in the tensile strength of the composite reinforced with 50 mm basalt fibres in comparison with shorter length of fibres used for their investigation. They attributed this phenomenon to the reduction in the number of fibre ends which serve as sites for localized stress concentration and crack initiation points in the matrix of the material [41]. Also, according to the works of some authors on the mechanics of fibre-reinforced plastics, random orientation of discontinuous fibres evenly dispersed in the matrix of a material either gives the material a three-dimensional or two-dimensional near isotropic structure depending on the fibre length [42] [43]. For an isotropic material it is assumed that stress distribution across the material structure is uniform [44]. Two-dimensional near isotropy adopted for this research could have correspondingly contributed to the observed enhancement in tensile strengths of the developed composites.

3.1.2. Tensile Stress at Break

The variation of tensile stress at break with molar concentrations of the chemical treatments for the LDPE composites and the unreinforced LDPE is presented in **Figure 2**. Observations from this result showed almost a similar trend as was observed in **Figure 1**. The peculiar difference in this case is that the superlative performance is given by the LDPE composite reinforced with 0.10 M NaOH-treated 2 wt% WCHF and is 309.5% better than the unreinforced LDPE polymer. The least enhancement is given by the LDPE composite reinforced with 0.15 M KOH-treated 4 wt% BCHF and it is still better than the unreinforced LDPE by 5.835%. The tensile stress at break of the LDPE composite reinforced with 0.15 M NaOH-treated 2 wt% WHCF which has the highest ultimate tensile strength improved by 67.73%. This significant improvement in the fracture strengths of the LDPE

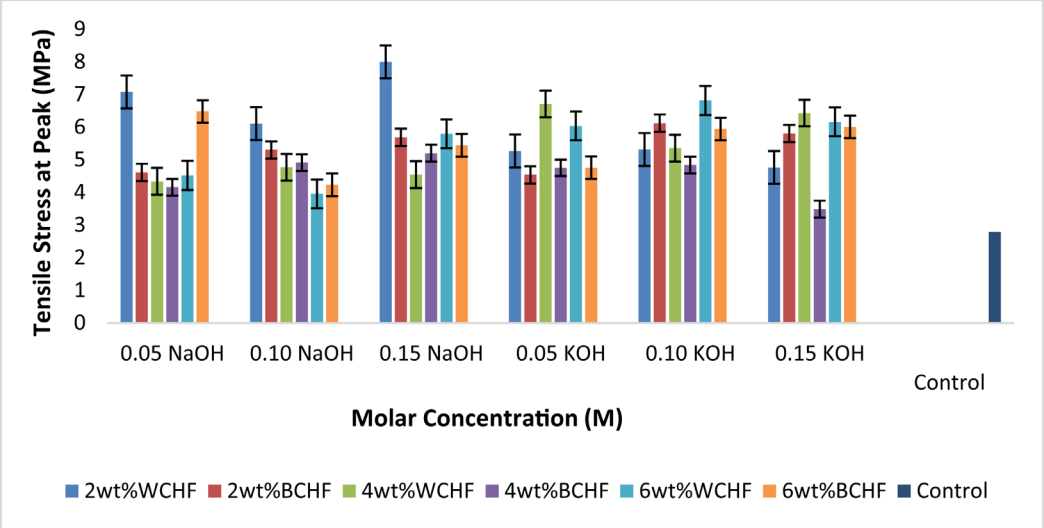


Figure 1. Variation of tensile stress at peak with molar concentrations of the chemical treatments for the LDPE composites and the unreinforced LDPE (control).

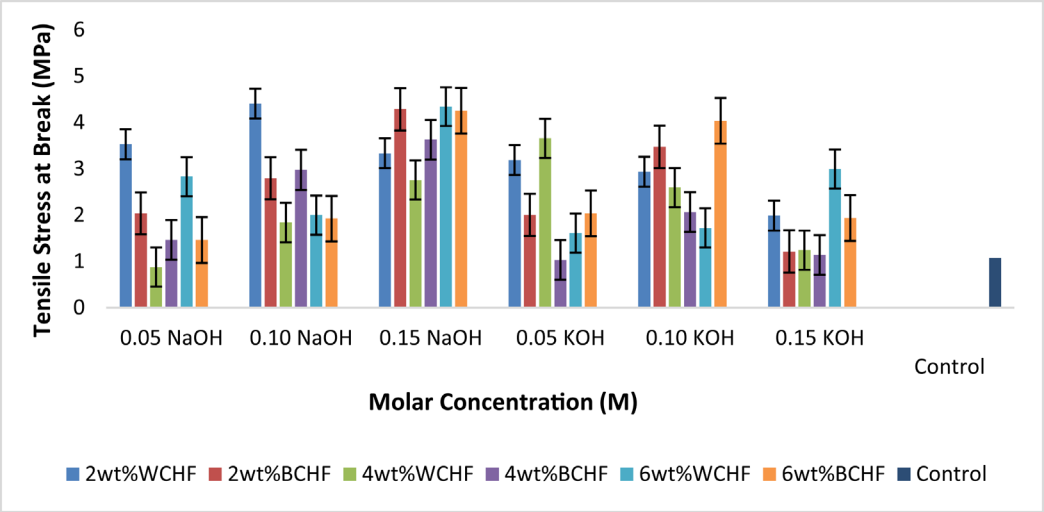


Figure 2. Variation of tensile stress at break with molar concentrations of the chemical treatments for the LDPE composites and the unreinforced LDPE (control).

composites can be attributed to the key factors discussed comprehensively under **Figure 1**.

3.1.3. Tensile Modulus

The variation of tensile modulus with molar concentrations of the chemical treatments for the LDPE composites and the unreinforced LDPE is presented in **Figure 3**. Observations from this result revealed that increase in molar concentration of the NaOH treatement favored increase in tensile moduli of the LDPE composites reinforced with 2 - 4 wt% CHFs and at 6 wt% fibre loading, the optimum enhancement is at 0.05 M. For the KOH treatment, the tensile moduli of all the LDPE composites increased with increase in molar concentration from 0.05 M to 0.10 M. The trend of the consistency is interrupted at 0.15 M. The superlative enhancement is presented by the LDPE composite reinforced with 0.10 M KOH-treated 6 wt% WCHF and it has a tensile modulus which is better than that of the unreinforced LDPE by 160.70%. The least enhancement is given by the LDPE composite reinforced with 0.05 M NaOH-treated 4 wt% WCHF with a tensile modulus that supersedes that of the unreinforced LDPE by 5.203%. The tensile modulus of the LDPE composite reinforced with 0.15 M NaOH-treated 2 wt% WCHF which has the highest ultimate tensile strength improved by 119.04%.

3.1.4. Tensile Strain at Break

The variation of tensile strain at break with molar concentrations of the chemical treatments for the LDPE composites and the unreinforced LDPE is presented in **Figure 4**. Observations from this result revealed that there is no consistency in the trend of increase in molar concentrations of both chemical treatments and increase in strain to fracture of the developed LDPE composites. The superlative performance is demonstrated by the same LDPE composite with the highest ultimate tensile strength, its strain to fracture is 80.86% better than that of the unreinforced LDPE. The least enhancement is given by the 0.10 M KOH-treated 2 wt% WCHF reinforcement with 4.35% betterment in comparison with the unreinforced LDPE.

3.2. Fracture Behaviour

The fracture behaviour and failure mode of the LDPE composites are presented in **Figure 5**. The results revealed shearing of the LDPE composite at the fibre-matrix interface which implies that the applied tensile stress

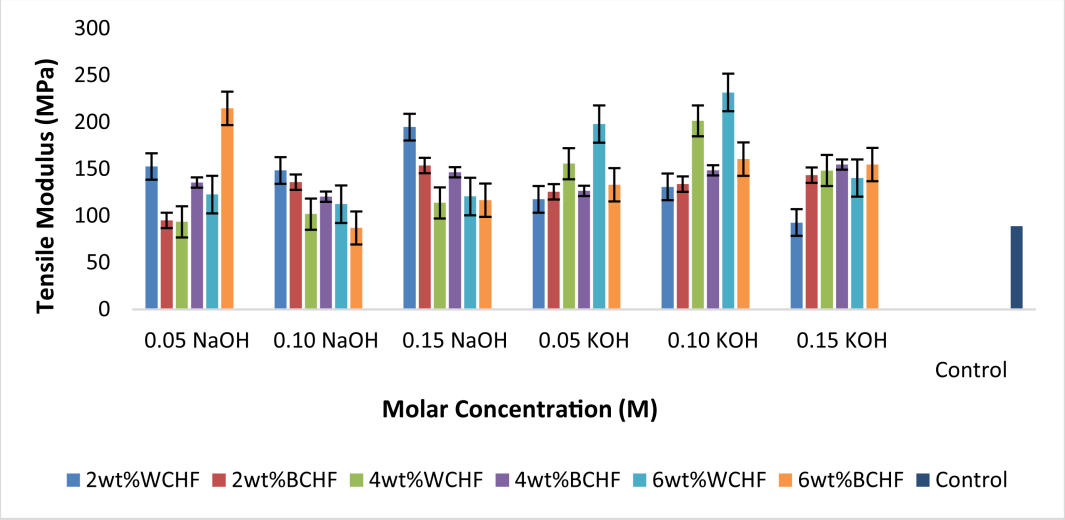


Figure 3. Variation of tensile modulus with molar concentrations of the chemical treatments for the LDPE composites and the unreinforced LDPE (control).

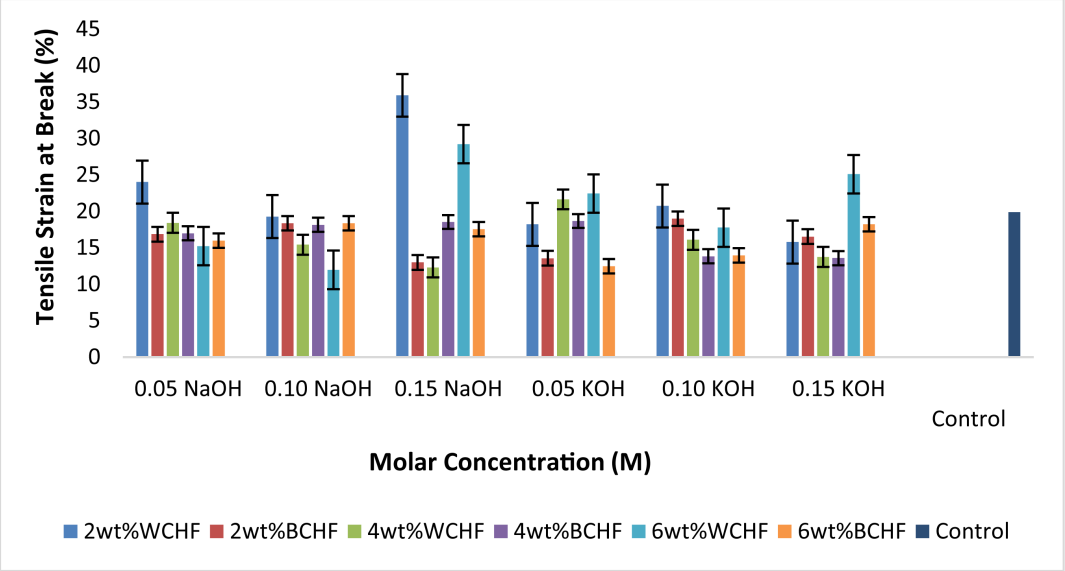
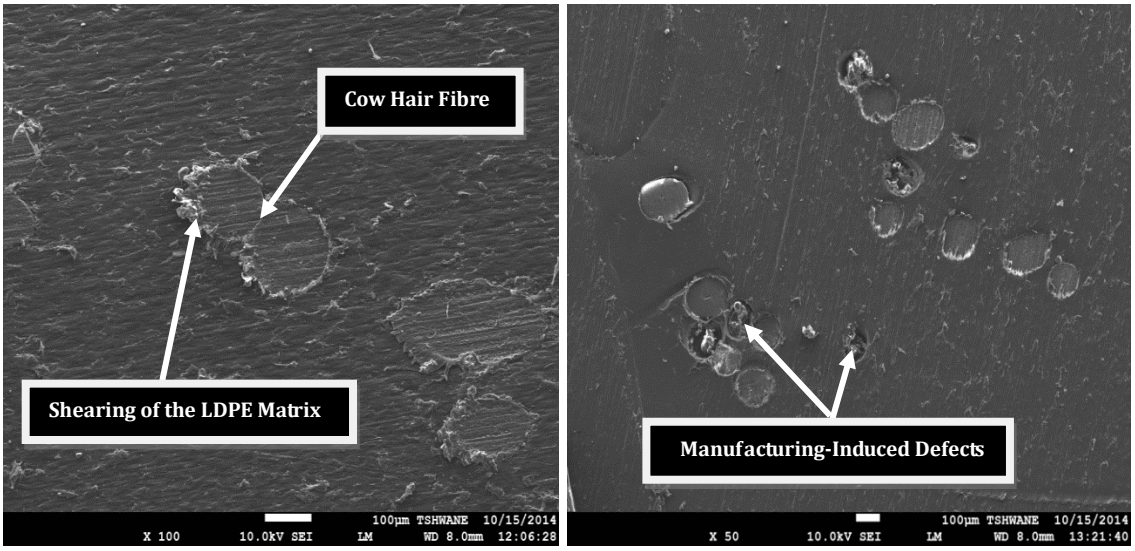


Figure 4. Variation of tensile strain at break with molar concentrations of the chemical treatments for the LPDE composites and the unreinforced LDPE (control).



(a) (b)

Figure 5. SEM Images of the fractured surface of composites after tensile loading (a) NaOH-treated CHF; (b) KOH-treated CHF.

stretched the LDPE polymer matrix more than the reinforcing CHFs [45] (**Figure 5(a)**). This is a clear indication that the developed CHF-reinforced LDPE composites are not susceptible to catastrophic failure under tensile loading. The image in **Figure 5(b)** shows manufacturing-induced defects that might have arose during the development of the composites due to air entrapment.

4. Conclusions

This investigation has been conducted to reassess the economic importance of cow hair fibres, establish their suitability and reinforcing efficiency in composite materials. From the research findings, it was observed that:

- Both treatments led to excellent performance in tensile properties. Considering the selected chemicals, tensile properties were better enhanced by treating the 2 wt% CHF reinforcement with 0.15 M NaOH and the 6 wt% reinforcement with 0.1 M KOH;
- The best molar concentration for the treatment of cow hair fibre intended for the reinforcing phase of LDPE was discovered to be 0.15 M for NaOH while 0.05 - 0.1 M was the best for KOH;
- The best % fibre loadings for these treatments laid between 2 - 4 wt% for NaOH and 6 wt% for KOH;
- Animal fibres should be treated with the best alkaline solution before they were used as reinforcement in composite materials development owing to the fact that chemical treatments had the potential of enhancing the properties of developed composites. The selected alkaline concentrations and the % fibre loading used revealed that high concentration and low fibre volume fraction favoured the use of NaOH while low concentration and high fibre volume fraction favoured the use of KOH treatment.

For engineering application where high tensile strength and high ductility of low density polyethylene are required, 0.15 M NaOH-treated WHCF reinforced-LDPE should be used in lieu of unreinforced LDPE, and in applications where moderate strength and high stiffness are required, 0.1 M KOH-treated 6 wt% WCHF reinforcement should be used.

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