

Effect of Chemical Treatments on the Physicochemical and Tensile Properties of Cow Hair Fibre for Low Load Bearing Composites Development

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Abstract: This research has investigated the influence of H₂O₂, KOH and NaOH treatments on the physicochemical and tensile properties of cow hair fibres for composites applications. The chemical treatments for this present study were prepared with predetermined molar concentrations of 0.10, 0.15 and 0.2 M respectively. The physicochemical properties of the cow hair fibres were established using proximate analysis and the tensile properties of the fibres were evaluated with the aid of an Instron universal testing machine. From the results of the proximate analysis, it was observed that, with increase in the concentration of the H₂O₂ treatment there was increase in the crude protein and moisture contents and decrease in the ash content of the fibres. However, the KOH and NaOH treatments showed an opposite trend. Also, from the tensile test results, it was observed that the chemical treatments aided the enhancement of ultimate tensile and yield strengths of the fibres in comparison with the untreated fibres while tensile strain at break was enhanced by 0.1 and 0.2 M of KOH as well as 0.2 M of NaOH treatments. The results of this research have shown that proper chemical treatment can enhance the properties of cow hair fibres for composites applications.

Keywords: Animal Fibre, Alkaline and Oxidative Treatments, Physicochemical Constituents, Mechanical Behaviour, Composite Applications

1. Introduction

Nowadays, topical advancements in materials processing techniques have efficaciously occasioned in a drastic change in the development of novel engineering materials [1]. Contemporary materials processing routes such as, laser surface melting, electric discharge machining, chemical vapour deposition, thin film technology, and nanotechnology have immensely revolutionized the types of engineering materials emerging from the laboratories of materials scientists and engineers worldwide [2-5]. These new materials offer a broad spectrum of advantages such as aesthetics, low density, biomimicry, non-toxicity and topnotch performance under the harshest service environments be it acidic, basic, and cryogenic or elevated temperatures [6-10]. However, most of these processing techniques are unbearably

extortionate, environmentally hazardous and are not relatively available to materials scientists and engineers globally [11].

Albeit, some researchers in the developed countries have been able to establish that the shortcomings concomitant with these new materials are overridden by their fundamental applications and unique functionalities during service life [12]. Quite the opposite, researchers from the developing countries who stalwartly believed that these shortcomings can be progressively vanquished are now congregating their ingenious research efforts in seeking alternatives solutions to develop renewable, sustainable, cost effective, biodegradable, and highly functional engineering materials [15-17]. This mounting convergence in their research efforts has culminated in the proliferation of published research articles on the potential applications of natural fibres, dispersoids and

particulates as the reinforcing phase in composites, instead of utilizing orthodox synthetic materials such as kevlar, glass, carbon and aramid fibres [18-21].

Hitherto, bulk of the works of materials scientists on natural fibres for composites development has been intensive on vegetal fibres and this has irrefutably yielded in very few literatures available on animal fibres. In fact, these very few literatures available on animal fibres primarily focused on their potential applications in the civil and textile industries [22-25]. Animal fibres include feather, hair, wool and silk which are gotten from animals such as chickens, turkeys, spiders, llamas, alpaca, dogs, horses, rabbits and cows [26]. This present study lays its emphasis on cow hair fibre. Cow hair is gotten from the skin of cows and is made up of keratin protein like most other animal hair fibres which accounts for their engineering properties [27,28]. Oladele et al (2014) reported in their work that the mechanical behavior of cow hair fibre-reinforced high density polyethylene polymer matrix composite under tensile and flexural loadings was improved with random orientation of short cow hair fibres dispersed in the matrix [29]. Another investigation carried out by Oladele et al (2015) also revealed that appropriate chemical treatment on the cow hair fibre prior the composites development, was able to enhance the mechanical behavior of cow hair fibre-reinforced high density polyethylene polymer matrix composites under flexural loading [30].

Despite the fact, that natural fibres from both plants and animals possess some atypical integral advantages over synthetic fibres, such as low energy consumption, carbon dioxide sequestration during materials processing, ecofriendliness, relative availability and cost effectiveness [31], they have also been documented to have high susceptibility to microbial degradation, high moisture absorption, weak interfacial adhesion and poor wettability between the matrix materials and the reinforcing fibres [32]. Nevertheless, studies of researchers have revealed that these discouraging drawbacks can be moderated with the appropriate chemical treatments [33]. M. S. Huda et al (2013) used sodium hydroxide, 10 % maleinized polybutadiene rubber, and a silane-coupling agent to treat fibres of poultry feathers and documented an improvement on thermal performance of fibres and the mechanical properties of the composite developed [34]. Also, according to Lakshmanan (2014) the influence of various chemical treatments such as thioglycollic acid reduction, sulphytolysis, formic acid, morpholine, NaOH and protease enzyme treatment on Angora rabbit fibres was able to improve the physicochemical, mechanical and morphological properties of the fibres [35]. This present study has investigated the influence of H_2O_2 , KOH and NaOH treatments on cow hair fibres.

The underlying impetus that stimulated the selection of cow hair for this research is the relative abundance of cow hair in Nigeria [36, 37]. According to a report by GEMS (Growth and Employment in States) about 7 million cattle are slaughtered annually in Nigeria and the hair is getting rid of by singeing since a lot of people consume the skins of

cows in Nigeria [38-40]. This act of unconscious waste and environmental pollution caused by the burning of the cow hair can be curbed if the cow hair fibres are channeled towards the development of animal-fibre reinforced composites. The economic importance of the cow hair will also be more than the production of felt cushions and shoe brushes which are major crafts in northern Nigeria. The chemicals used for this study were chosen due to their low cost.

2. Experimental

2.1. Materials

The principal materials utilized for this research were the hairs scraped from the tails of white Fulani cows (Zebu breed) procured from a local abattoir in Akure, Ondo state, South-West, Nigeria. The chemicals were procured from Pascal Scientific Akure, Ondo State, Nigeria.

2.2. Experimental Methods

2.2.1. Chemical Treatments of the Cow Hair Fibres

The cow hair fibres procured from a local abattoir in Ondo State were thoroughly washed with detergents and rinsed with tap water and distilled water ensued by drying at room temperature for 24 hours to remove impurities and blood stains from the fibres. The cleaned fibres were separated into two portions for chemical treatments and controlled experimentations. The fibres to be chemically treated were dipped in containers containing the chemicals of varying molar concentrations of 0.1, 0.15 and 0.2 M for the H_2O_2 (pH = 11), NaOH (pH = 10) and KOH (pH = 10) treatments respectively. The chemical treatments were placed inside a shaker water bath maintained at 50 °C for 4 hours to effectively remove lipids from the surfaces of the fibres in order to improve wettability and interfacial adhesion between the fibres and any matrix material where they may be used. The chemically treated fibres were later rinsed with tap water and distilled water to remove any residual chemicals left on the surfaces of the fibres and this was ensued by sun drying for 5 days.

2.2.2. Proximate Analysis

The physicochemical constituents of the cow hair fibres were manually determined using proximate analysis in accordance with AOAC standard (2001) [41]. The crude protein, moisture, and ash contents of the cow hair fibres were evaluated.

2.2.3. Crude Protein Determination

This involved three stages namely; digestion, distillation and titration.

(i). Digestion

About 0.5g of sample was weighed into 500 ml kjeldahl flask. Concentrated H_2SO_4 (10 ml) and selenium catalyst were added and boiled until the sample turned into clear solution. It was cooled and made up to 50 ml with distilled

water. The sample was stored in a bottle.

(ii). Distillation

To carry out distillation, 5 ml of 2 % H_2BO_3 (Boric acid) was put into the conical flask and 2 drops of mixed indicator (0.198 g bromocresol green plus 0.132 g methyl red into 200 ml alcohol) were added. The receiving flask was positioned so that the tip of the condenser tube was below the surface of the boric acid. The 5 ml of digested sample was pipetted into the condenser's cup and 10 ml of 40 % NaOH was added. This was then washed down with distilled water. The joints were tightened and distillation was done till a volume of 50 ml was reached in the receiving flask.

(iii). Titration

The distillate was titrated with 0.1 ml HCl until the end point (pink colour) was reached.

$$\% \text{ Nitrogen} = \frac{\text{Titre Value} \times 0.1 \text{ M HCl} \times 0.014 \times 100 \times \frac{V_1}{V_2} \times 100}{\text{Weight of Sample}} \quad (1)$$

Where,

V_1 = volume of digest (50 ml)

V_2 = volume of digest used (5 ml) and

% Crude protein = % Nitrogen \times 6.25

(iv). Moisture Content Determination

This was done using the oven-drying method. Cleaned, dry and well-labeled petri dishes were weighed (W_1). About 5 g of each sample were weighed into the dishes (W_2) and transferred into the oven maintained at 105 °C for 3 hours. After the three hours, they were transferred into the desiccators to cool and then weighed (W_3). This process was continued every hour until a constant weight was obtained.

$$\% \text{ Moisture} = \frac{\text{Loss in Weight of Sample } (W_3 - W_2) \times 100}{\text{Weight of Samples before drying } (W_2 - W_1)} \quad (2)$$

(v). Ash Content Determination

Dry and clean crucibles were weighed (W_1) and their respective weights recorded and, about 1g of the samples was added and weighed again (W_2). The crucibles and contents were placed into the muffle furnace at 600 °C until a light grey colour of ash was obtained. The crucibles were removed and allowed to cool in the desiccators and then weighed (W_3).

$$\% \text{ Ash} = \frac{\text{Weight of Ash } (W_2 - W_3) \times 100}{\text{Weight of Sample } (W_2 - W_1)} \quad (3)$$

To ensure accuracy and reliability of test results, the analyses were replicated twice.

2.2.4. Tensile Testing

The tensile properties of the cow hair fibres were evaluated with the aid of an INSTRON 1195 universal testing machine at a fixed crosshead speed of 10 mmmin⁻¹ at a temperature of 25 \pm 2 °C. The test samples were prepared according to ASTM D412-83 [42]. The computer automatically generates the required data and graphs as the tests are being carried out. To ensure accuracy and reliability of tensile test results, three repeatability tests were performed for each determined tensile property of the cow hair fibres.

3. Results and Discussion

3.1. Physicochemical Properties

3.1.1. Crude Protein Content

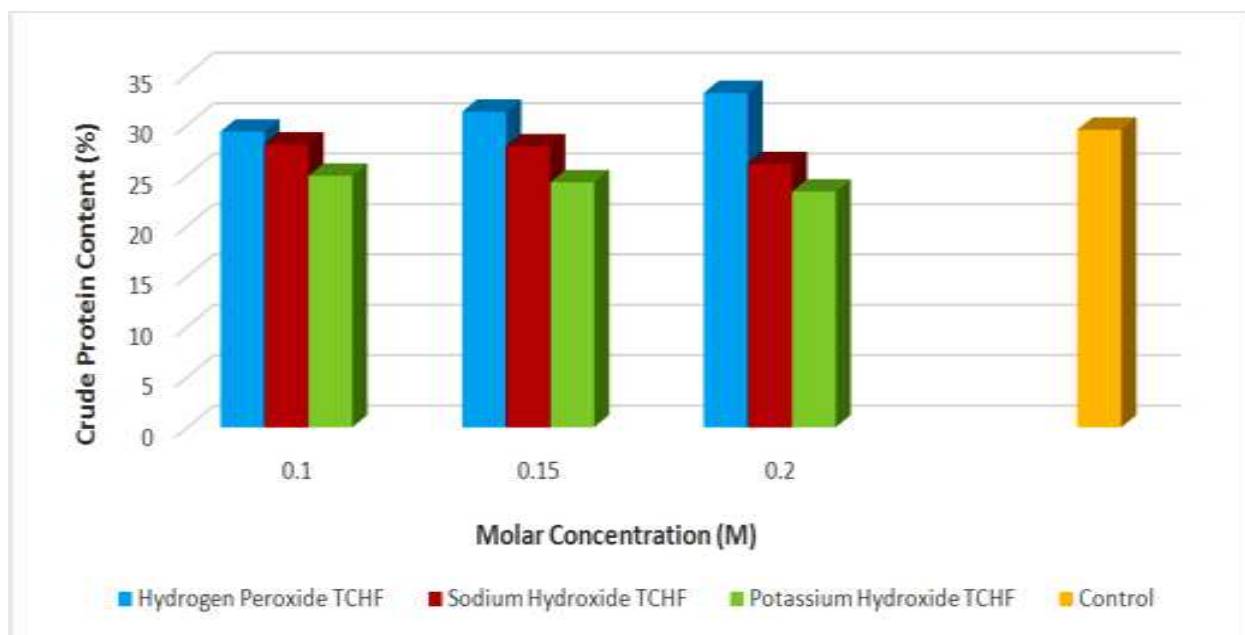


Figure 1. Variation of crude protein content with molar concentrations of the chemical treatments for the cow hair fibres.

The variation of crude protein content with molar concentrations of the chemical treatments for the cow hair

fibres is presented in Figure 1 above. The results revealed that there is a progressive increase in the crude protein

content of the H_2O_2 treated cow hair fibres (TCHF) with increase in molar concentration and a regression was observed in the case of the NaOH and KOH TCHFs respectively. The cow hair fibre (CHF) treated with 0.2 M H_2O_2 gave the superlative result in terms of crude protein content while the CHF treated with 0.2 M KOH gave the least enhancement. Of all the chemical treatments, only the CHFs treated with 0.15 and 0.2 M H_2O_2 have crude protein contents that are higher than that of the control test sample i.e. the untreated CHF. Goud et al (2011) in their investigation on the effect of H_2O_2 treatment on protein content and catalase activity in *Cajanus cajan* (L.) Millsp leaves reported a similar progressive trend in the protein content of the leaves with lower molar concentrations of H_2O_2 treatment [43]. According to Wolfram et al (1970) the perhydroxy anion (HO_2^-) is the predominant reactive species during H_2O_2 treatment of hair fibres. The abundance of sites in hair keratin which might yield to a nucleophilic attack by this ion precludes any firm prior assignment of a specific interaction. The reaction between keratin and H_2O_2 is confined mainly to the cysteine residues. The decrease in cysteine is almost quantitatively matched by a corresponding increase in cysteic acid [44]. The work of Robbins (2012) has also shown that the primary chemical differences between H_2O_2 treated hair and unaltered hair are lower cystine content, a higher cysteic acid content, and lower amounts of tyrosine and methionine in the treated hair. He said these results support Zahn's original conclusion that the reaction of H_2O_2 with human hair protein occurs primarily at the disulfide bonds. Fewer total

micromoles of amino acids per gram of hair are found in treated hair than in unaltered hair most likely because of addition of oxygen to the sulfur containing amino acids and the solubilization of protein or protein derived species into the treatment bath [45]. The result of this study revealed that it is possible that the increase in the molar concentration of H_2O_2 facilitated the formation of more amino acids from the oxidation of sulfhydryl ($-\text{SH}$) groups contained in the keratin of the CHFs hence, increase in the crude protein content. Also, the underlying chemistry behind the regression observed in the crude protein content of the CHFs treated with NaOH and KOH treatments can be seen in the work of Ameen et al (2014) when they treated *Jatropha curcas* leaf with NaOH. They attributed this occurrence to possible loss of some amino acids in the fibres due to high concentration of the alkaline treatment. These amino acids are released into the treatment bath as the polypeptide bonds are broken down by the potassium or sodium hydroxide during alkaline treatment of the fibres [46]. Alkaline hydrolysis generates sodium and/or potassium salts of free amino acids; oligopeptides (small chains of amino acids) are also generated as intermediates in the reaction. Some amino acids, such as arginine, asparagine, glutamine, and serine, are destroyed, while others are racemized; i.e., the molecules are structurally modified from a left-handed configuration to a mixture of left-handed and right-handed molecules [47].

3.1.2. Moisture Content

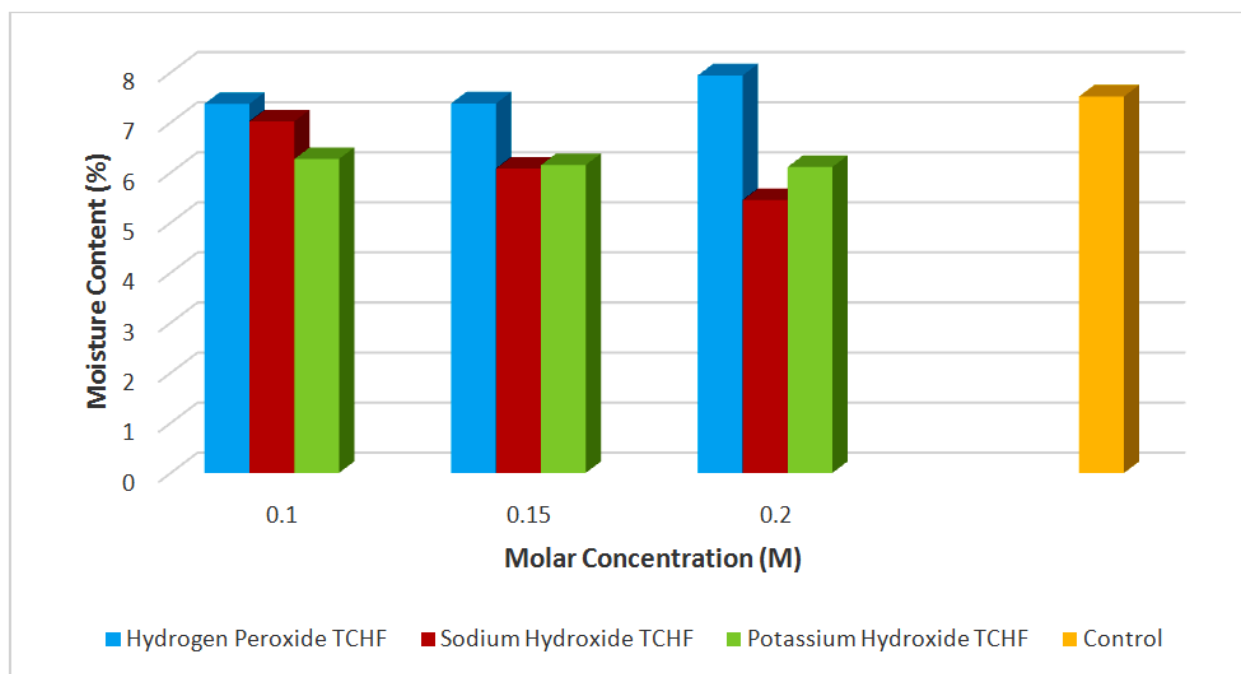


Figure 2. Variation of moisture content with molar concentrations of the chemical treatments for the cow hair fibres.

The variation of moisture content with molar concentrations of the chemical treatments for the CHFs is presented in Figure 2 above. The result revealed a progression in the moisture content of the H_2O_2 TCHFs with

increase in the molar concentration of the H_2O_2 treatment. This is a clear indication that the chemical treatment was effective enough in removing the lipids and other hydrophobic layers covalently bonded to the surface of the

CHF thereby exposing its hydrophilic cortex which in turn resulted in increase in water absorption capacity of the H_2O_2 TCHFs [47] and this increased with increase in molar concentration, oxidative treatment such as hydrogen peroxide treatment have been reported by Baghdadli and Luengo, (2008) to increase the hydrophilic character of hair [48]. For the NaOH and KOH treatments, a reduction in moisture content with increase in molar concentration was observed in the CHFs. The treatments possibly rendered the cow hair fibres more hydrophobic by depositing hydrophobic coatings formed during the treatment on the surfaces of the CHFs thereby reducing the chances of exposing the hydrophilic cortex and this increased with increase in molar concentration, hence reduction in water absorption capacity of the CHFs which accounted for the reduction in moisture content of the KOH and NaOH TCHFs. Also the reduction in the moisture content of the NaOH and KOH TCHFs can be attributed to the reduction of the hydrophilic hydroxyl groups

within the fibre structure caused by the chemical reducing potential of the KOH and NaOH treatments which can break down the alkali sensitive hydroxyl groups present within the structure of the CHFs, this reaction drives out water molecules from the fibre structure and hence reduction in the hydrophilic power of the CHFs [49]. A glaring consistent trend in moisture reduction with increase in molar concentration was observed with the NaOH TCHFs and for the KOH TCHFs the reduction in moisture content with increase in molar concentration is almost negligible. The 0.2 M H_2O_2 TCHF has the highest moisture content in comparison with all the other test samples while the least moisture content is given by the 0.2 M NaOH TCHF. Only the 0.2 and 0.15 M H_2O_2 TCHFs have higher moisture contents than the untreated CHF.

3.1.3. Ash Content

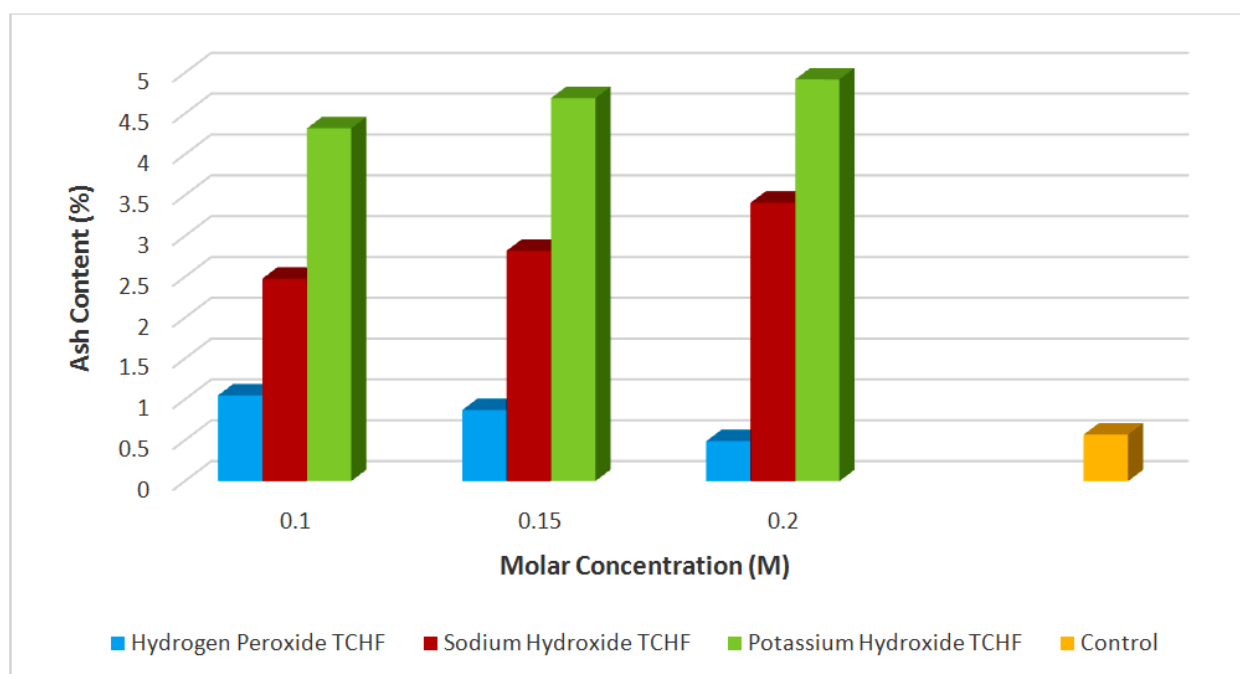


Figure 3. Variation of ash content with molar concentrations of the chemical treatments for the cow hair fibres.

In Figure 3 above, the variation of ash content with molar concentrations of the chemical treatments for the CHFs is presented. The results revealed a decrease in the level of ash content with increase in molar concentrations of the H_2O_2 treatment. The lowest level of ash content was observed in the 0.2 M H_2O_2 TCHF which is also the only sample with ash content lower than that of the control sample. The result showed that the optimum molar concentration of chemical treatment for reducing ash content of the hair fibre is 0.2 M H_2O_2 . Considering NaOH and KOH treatments, the results showed a progressive increase in the ash contents for the CHFs with increase in molar concentrations. The results also revealed that for each molar concentration, the KOH TCHFs showed higher ash contents than the NaOH TCHFs. Some researchers have been able to identify that the ash content of

cattle hair contains the following element: boron, barium, calcium, copper, iron, lead, potassium, magnesium, manganese, phosphorus, silicon, silver, sodium and zinc [50].

3.2. Tensile Properties

3.2.1. Tensile Strength at Maximum Load

The variation of tensile strength at maximum load with molar concentrations of the chemical treatments for the CHFs is presented in Figure 4 below. The result showed a progressive increase in the ultimate tensile strengths of the H_2O_2 TCHFs with increase in molar concentration and an inconsistent trend was observed with the KOH and NaOH TCHFs. However, the ultimate tensile strengths of all the chemically treated samples were significantly enhanced, with the 0.2 M H_2O_2 TCHF having the superlative performance

with a tensile stress at maximum load which is 57.15 % better than the untreated CHF and the 0.15 M NaOH TCHF having the lowest value which is still 36.57 % better than the untreated CHF. With respect to the KOH treatment the optimum result is given at 0.1 M while the optimum result for the NaOH treatment is at 0.2 M. This is a clear indication that the basic hydrolysis of the cortex keratin of the CHFs by the KOH and NaOH treatments and the solubilization of the cortex keratin of the CHFs by the oxidative H₂O₂ treatment at 50 °C for 4 hours resulted in an improved structural integrity of the cortical keratin of the chemically treated CHFs, hence improved plasticity of the TCHFs under tensile loading. It is available in existing literatures that the mechanical behavior

of hair fibre is fundamentally dependent on the orderliness of the structure of hair cortex [51]. Also the improvement in the ultimate tensile strength of the TCHFs might be due to the breaking of the covalent bonds between the lipids and the surfaces of the CHFs leaving a stronger and stiffer fibre which in turn also improved the maximum stress the TCHFs can sustain under tensile loading. Rosa et al (2009) investigated the effect of chemical treatment on coir fibers and the report about their research findings revealed that alkali treatment was able to improve the tensile strength of the coir fibres which is in agreement with the result of this present study [52].The superlative performance is observed with the 0.2 M H₂O₂ TCHF.

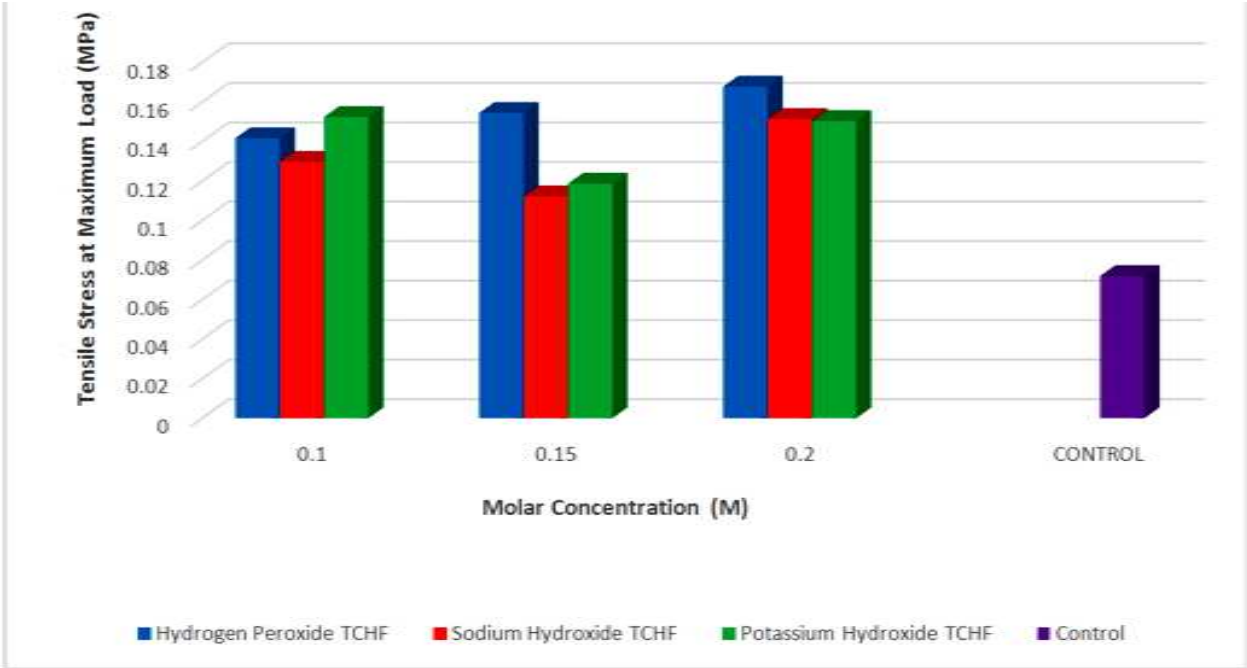


Figure 2. Variation of tensile strength at maximum load with molar concentrations of the chemical treatments for the cow hair fibres.

3.2.2. Tensile Stress at Yield

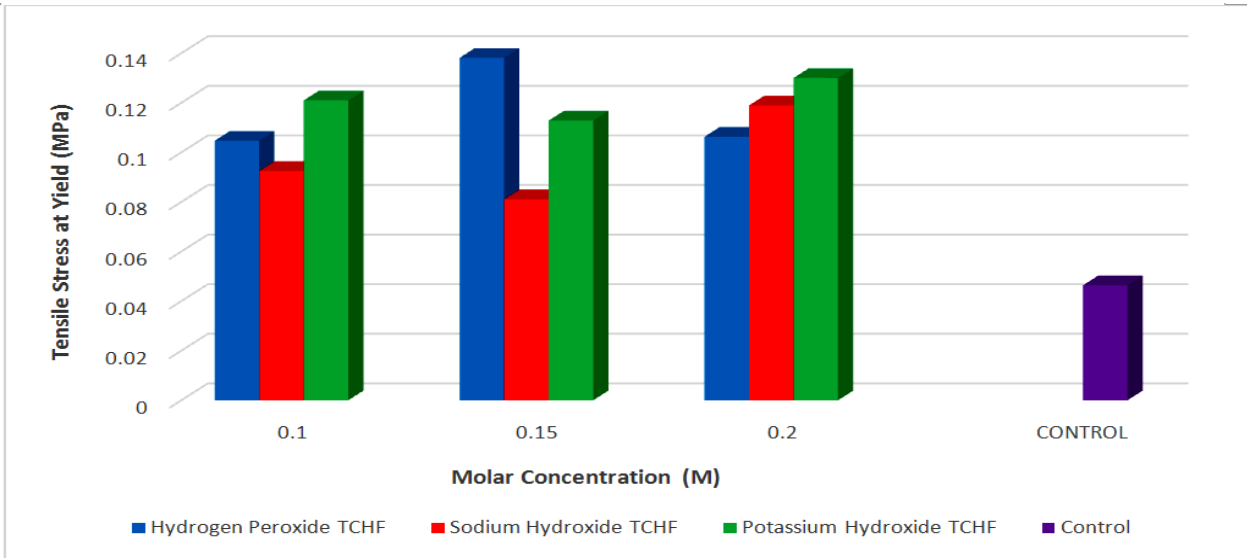


Figure 3. Variation of tensile stress at yield with molar concentrations of the chemical treatments for the cow hair fibres.

Figure 5 above shows the variation of tensile stress at yield with molar concentrations of the chemical treatments for the CHF. Observations from this result revealed an inconsistency in the trends of all the TCHFs with respect to increase in molar concentration and tensile stress at yield. Similarly, as observed in Figure 4, all the chemically TCHFs gave better tensile stress at yield in comparison with the untreated CHF. The superlative performance of the results can be seen with the 0.15 M H_2O_2 TCHF which has a yield strength that supersedes that of the untreated CHF by 66.43 % and the least enhancement in yield strength is observed with the 0.15 M NaOH TCHF which is still better

than the untreated CHF by 42.77 %. The result also revealed that the optimum performance with respect to the KOH and NaOH treatments is at 0.2 M. This is a clear indication that thermochemical treatments was able to reduce the strength of the chemical bonds such as cystine, hydrogen and salt bonds in the TCHFs, hence improvement in their elastic behaviour. These various chemical bonds have been reported by McKay (2015) to be responsible for holding strands of protein (in this case, the strands of the cortical keratin) in the hair fibre together [53].

3.2.3. Tensile Strain at Break

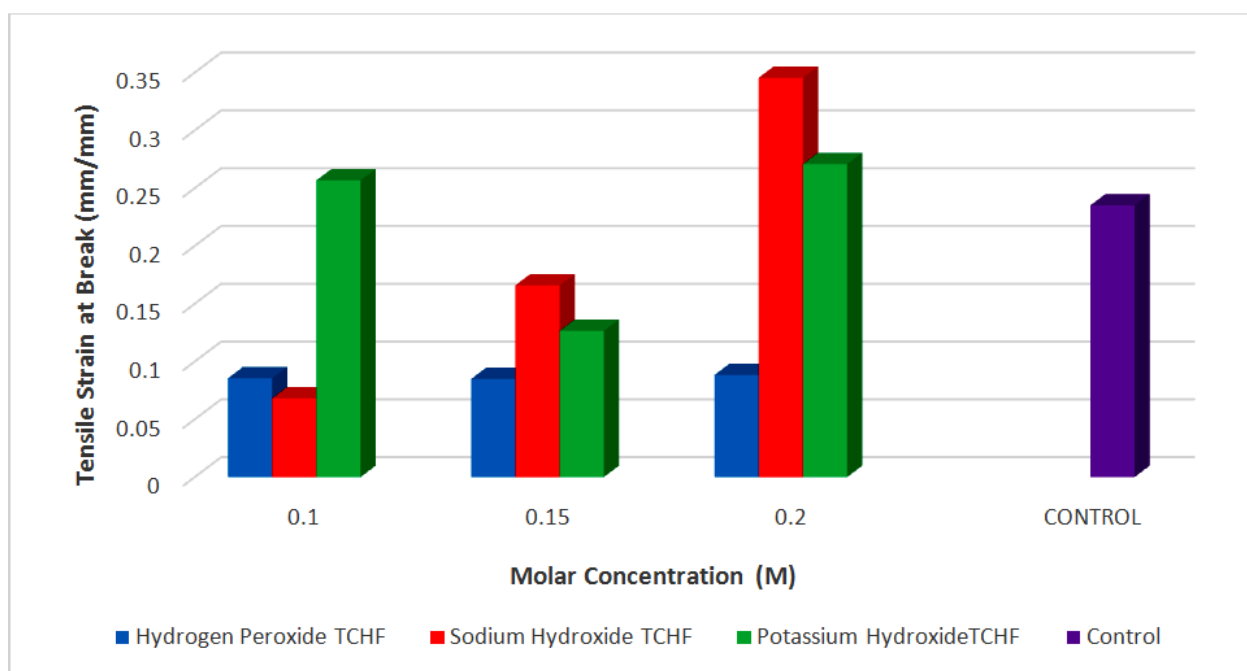


Figure 4. Variation of tensile strain at break with molar concentrations of the chemical treatments for the cow hair fibres.

The variation of tensile strain at break with molar concentrations of the chemical treatments for the CHF is presented in Figure 6 above. Observations made from the results revealed that, the effect of H_2O_2 treatment on the CHF is irrespective of molar concentration did not enhance the elongation at break of the CHF; however, increase in molar concentration did not affect the tensile strain at break of the H_2O_2 TCHFs. There appears to be a constant tensile strain at break of about 0.085 ± 0.003 % for all the H_2O_2 TCHFs. For the NaOH TCHFs a progressive increase in tensile strain at break with increasing molar concentration was observed with the 0.2 M NaOH TCHF giving the best value of elongation before failure which supersedes that of the untreated CHF by 31.94 %. As for the KOH TCHFs there was no consistency in the trend of increasing molar concentration with tensile strain at break. However, the optimum tensile strain at break with the KOH treatment is given at 0.2 M. According to some authors, it is expected that increase in the yield strength of a material is very likely to bring about decrease in the ductility of the material [54] which is what is apparent with most of the chemically TCHFs in this present study, notwithstanding

the 0.1, 0.2 M KOH and the 0.2 M NaOH TCHFs demonstrated exceptional behaviours.

4. Conclusions

This work has been carried out in order to further encourage the use of natural fibres, particularly animal fibres which have not been extensively exploited as their vegetal counterparts for engineering applications and also to promote the selection of renewable ecofriendly materials for composites development; thereby adding value to what is otherwise regarded to as wastes. From the research findings, it was observed that:

- Chemical treatments is suitable for the modification and enhancement of the physicochemical and hence, the tensile properties of animal fibres. This was the case because in all the properties examined, it was observed that, chemically treated cow hair fibres gave the best results.
- Among the selected chemicals used, H_2O_2 was observed to be the best followed by NaOH and KOH respectively.

The results revealed that H₂O₂ treatment is always at variance with the NaOH and KOH treatments.

- The use of higher concentration of the chemicals between 0.15-0.2 M aids the best enhancement of the investigated properties.

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