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PERFORMANCE EVALUATION OF MERCERIZATION AND ACETYLATION ON HARDNESS OF RAFFIA PALM FIBRE

Obinichi, N[1] and Uchegbulam, I.[2] *
[1]Department of Mechanical Engineering, University of Port Harcourt, Choba, P.M.B., 5323, Nigeria.
[2]Production Technology, School of Science Laboratory Technology, University of Port Harcourt, Choba, Nigeria.
*Corresponding author. E-mail address: uche.aberdeen.ac.uk@gmail.com (Uchegbulam, I.).

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ABSTRACT

As the demand for environmentally friendly products rises and global awareness on reducing toxicities in manufacturing increases, the exploitation of plant-based raw materials like raffia palm fiber requires closer attention. In this study, the effect of sodium hydroxide and acetic anhydride concentration on the hardness of raffia palm fiber at varying drying temperature and fiber length were investigated. The treatments were carried out using 10% sodium hydroxide solution and 5% acetic anhydride at oven drying temperatures of 70°C for mercerized fiber and 50°C for acetylated fiber with varying fiber length of 50mm, 60mm, 70mm and 80mm. Findings show hardness values of 257HR and 370HR for 10% NaOH at 70°C and 5% acetic anhydride at 50°C respectively showing a 30.38% rise in Rockwell Hardness value in the acetylated fibers over the mercerized cohorts. Remarkably, these results were obtained at ideal fiber lengths of 60mm in both treatments. Hence, acetylation treatment at 5% acetic anhydride concentration and 50°C oven-drying temperature using 60mm fiber length offered optimal hardness value for raffia palm fiber. This is highly recommended for industrial commercialization for composites in the automobile, construction, manufacturing, medical, sports, oil and gas industries. In addition, the results from this study will open new economic values for Nigerian Raffia Palm fiber as potential reinforcement material for both domestic and international markets and applications.

Keywords: acetylation, mercerization, Acetic anhydride, sodium hydroxide, Rockwell hardness.

INTRODUCTION

The importance of natural fibers, which has been used for hundreds of years in order to meet human needs such as clothing and sheltering has considerably reduced through the use of synthetic fibers toward the end of the 1900s. The increasing environmental concerns and depletion of petroleum resources have increased the importance of natural fibers once again and have stimulated researchers and industries to use sustainable fibers instead of conventional synthetic fibers (Cicala et al., 2010). The need to meet the needs of the rapidly increasing population with basic necessities points to the direction of natural raw materials from plants like fibers (Uchegbulam et al., 2022). Besides mechanical and physical properties (such as good specific modulus values, low density), considerable toughness properties of natural fibers, low cost, recyclability, nontoxicity, and easy accessibility properties are also attractive aspects of natural fibers and these properties give an opportunity to use natural fiber reinforced composite products in various industries such as automotive, building, and furniture. Natural fibers can be classified based on their origin into: animal, mineral and plant fibers. Plant fibres have become the most commonly accepted fibres by the industry and the most analysed by the research community. This is mainly due to the short growth period, renewability and wider availability (Cicala et al., 2010). The vegetable fibres are composed of cellulose, hemicelluloses and lignin, which can be extracted from bast, leaf, seed, fruit, wood, stalk and grass/reed. Moreover, there are some relevant previous studies suggesting that some fibres may have a potential ability to work as reinforcement candidates in the near future. For example, roselle (*hibiscus sabdariffa*), sugarcane (*saccharum cilliare*), pine, bagasse, henequen, alfa, among others are listed (Pickering et al., 2016; Bharath and Basavarajappa, 2016; Dittenber and GangaRao, 2012; Naldony et al., 2016). The natural fibres usually have several benefits over synthetic fibres such as availability, low cost, low density, acceptable modulus-weight ratio, high acoustic damping, low manufacturing energy consumption, low carbon footprint and biodegradability. Some authors state the evidences for clear benefits, for example, natural fibres cost much less and require much less energy to produce than traditional reinforcing fibres like glass and carbon (Huda et al., 2008). However, natural fibres have negative aspects due to their low consistency of properties and quality. These fibres have higher variability of physical and mechanical properties, higher moisture absorption, lower durability, lower strength and lower processing temperature (Pickering et al., 2016; Rohan et al., 2018; Cheung et al., 2009; Carvalho et al., 2016). The large variety of properties is mainly dependent upon plant species, growth conditions and method of fibre extraction. Moreover, properties depend on the fibre cell geometry of each type of cellulose and its degree of polymerization (Ho et al., 2012). It should be noted that linear cellulosic macromolecules are linked by hydrogen bonds and are closely associated with hemicelluloses and lignin which confer stiffness to fibre. Not only holds fibres together but also the cellulose within the fibre cell wall (Chen, 2014). Some authors also named natural fibers as stem fibres, because they are obtained from the pseudo-stem of the plant, that is, from the outer cell layers of the stem. The bast fibres are the most widely used non-wood lignocellulosic fibres due to their superior technical characteristics and ease of extraction from raw resources (Verma and Jain, 2016), usually by retting and manual extraction techniques (Srinivasababu, 2015). The manual extraction method offers high quality fibers but the process can be a lengthy and laborious task (Praful and Lanjewar, 2017). The general natural fibre life cycle phases are extraction, processing, fabrication, use, disposal and recycle. Within this cycle, there are several limiting factors for a large-scale production and use of fibres. These factors affect several stages of the natural fibre’s life cycle: large variability of soil composition and morphology, fibres hydrophilic nature, degradation by microorganisms, service life and sunlight. The physical and chemical properties of natural fibres are linked to plant source, cultivation location, climate conditions, harvest window, use of Genetic Modified Organisms (GMO), pesticides and fertilizers (Hodzic and shanks, 2014; Franck, 2005). Usually, natural fibres require several treatments to overcome some of the mentioned limitations so as to improve fibre-matrix interfacial adhesion. Several techniques have been reported, such as water-repellent chemicals, coupling agents and heat treatments, by modifying the surface morphological, topological properties, roughness and water absorption index of the fibres (Kabir et al., 2012; Ku et al., 2011; Bousfield et al., 2018). As a result, research and technological effort has been reported fostering the improvement of crops quality and fibres performance on technical and economical perspective, aiming to provide new solutions and applications (Todor et al., 2018). Despite the referred limitations, fibres are taking a growing interest by researchers.

This study is reporting the hardness value of raffia palm fibers. By using mercerization

(Sodium Hydroxide treatment) and Acetylation (fiber preparation using acetic anhydride) methods. The focus was on the effects of oven-drying temperature and the contributions of fiber lengths ~~were~~ on the hardness of raffia palm fibers. This was aimed at increasing the market value of this local raw material to both domestic and international applications.

MATERIALS AND METHODS

The materials used are raffia palm, pH meter and electronic weighing scale as shown in Figure 1. Other apparatus used in extraction and treatment include: Electric oven, graduated cylinder, plastic bucket, plastic cup, glass beaker, distilled water etc.

Figure 1: (A) Extracted Rafia palm fibre (B) Digital weighing balance and pH used

The raffia palm fronds were collected from the forest in swamp area of Omuigwe Aluu, Ikwerre local Government area of Rivers State, Nigeria. The matured fibre was cut, extracted, gathered, and retted. The fibers obtained were sun-dried for 7 days. After sun-drying, mercerization by soaking for one hour in 10% NaOH solution and acetylation at a soaking time of 90mins (1hour 30mins) in 5% acetic anhydride solution was carried out. These were done in other to get rid of organic matter locked up in the fresh plantain frond and were carried out at ambient temperature. The fibres were thoroughly washed in distilled water several times to get rid of all organic matter and the pH was measured at the end of each washing process until pH neutrality was achieved.

- **Fibre Treatment**

The fibres obtained after soaking and washing in distilled water until pH neutrality was achieved were treated. A separate batch of the Raffia palm fiber was mercerized in 10% sodium hydroxide solution by immersing the fibres in the alkaline solution for 3 hours. The fibres were retrieved and rinsed severally in distilled water measuring the pH value of the residual water after each rinsing period until pH neutrality was observed and this was labelled as Batch 1. Another set of the washed fibers were acetylated in 5% acetic anhydride for 3 hours. The fibers were retrieved and rinsed severally in distilled water while routinely measuring the pH value of the residual water after each rinsing period until pH neutrality was observed and this was labelled as Batch 2.

- **Fibre Testing**

The treated fibres were oven-dried prior to Rockwell hardness testing in line with ASTM D785 standard. The Rockwell hardness test was carried out with the results recorded and analyzed. The treatment was carried out using and 5% acetic anhydride at two different oven drying temperatures of 70°C and 50°C for fibre with varying fibre length of 50, 60, 70 and 80mm. Rockwell Hardness Scale M (RHM) at scale factors of N130 and h500 was chosen for this purpose in this study because it is the best method to measure the hardness of soft materials like plastics, Bakelite, fibre, etc. To do this, an indenter ball with a diameter of 6.35 mm and a load of 100kgf were used. The hardness values of the fiber samples were computed as an average of three areas on each sample. All tests were conducted at ambient conditions and was carried out in triplicates to ensure precision. All the measurements were taken at a dwell time of 10 seconds after the indenter ball had made direct contact with the sample.

RESULTS AND DISCUSSION

The results of the hardness test conducted on mercerized and acetylation fibres are presented in Tables 1 and 2 respectively while the results analysis is shown with line chats in Figures 1 and 2.

Table 1: Results of hardness test on different fibre lengths treated with 10% NaOH solution and dried at 70^{0}C using Rockwell Hardness Scale M (RHM).

Fibre Replication	HRM @50mm	HRM @60mm	HRM @70mm	HRM @80mm
R1	125	92	104	168
R2	114	96	126	115
R3	89	257	114	163
R4	109	148	115	149
R5	118	204	119	153

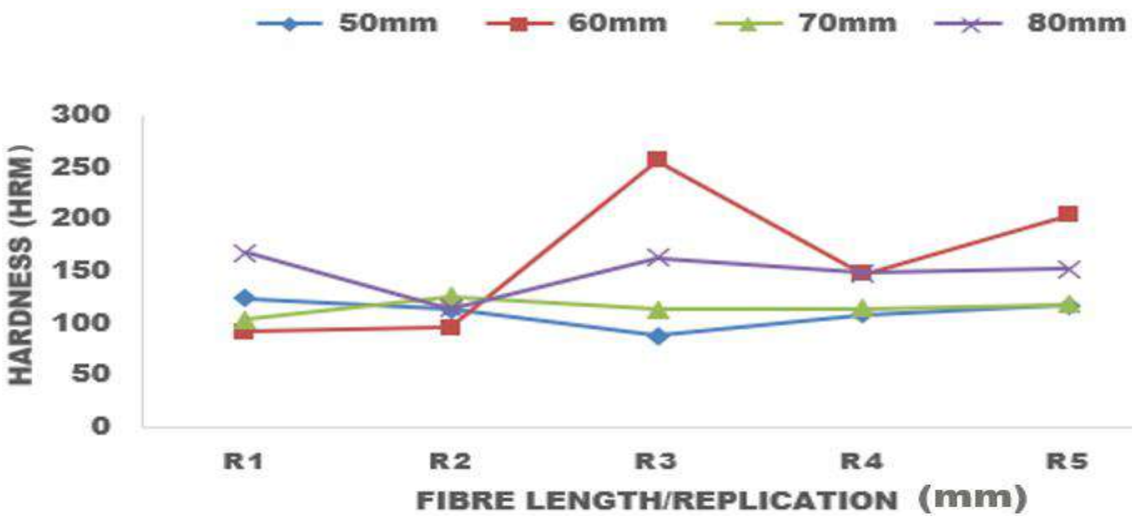

Figure 1: Rockwell Hardness numbers of Raffia Palm fibres mercerized in 10% NaOH solution at oven-drying temperature of 70°C at different fibre lengths.

Table 2: Results of hardness test on different fibre lengths treated with 5% Acetic anhydride, and dried at 50°C using Rockwell Hardness Scale M (RHM).

Fibre Replication	HRM @50mm	HRM @60mm	HRM @70mm	HRM @80mm
R1	153	157	89	82
R2	199	196	144	157
R3	145	370	119	113
R4	166	241	117	117
R5	145	182	125	132

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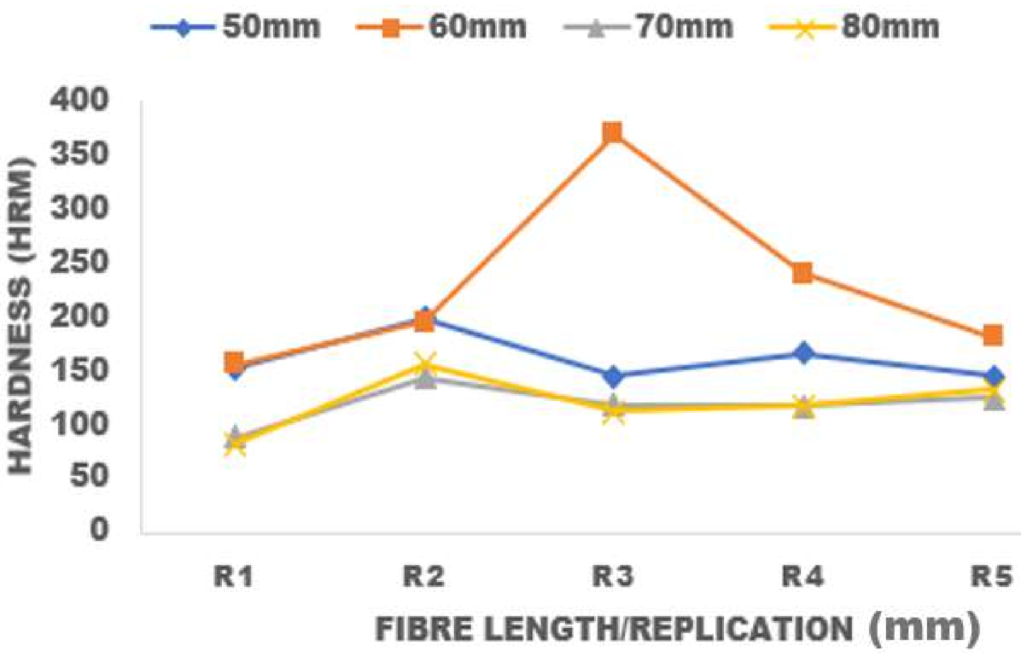

Figure 2: Rockwell Hardness numbers of Rafia Palm fibres mercerized in 5% Acetic anhydride solution at oven-drying temperature of 50^{o}C having different fibre lengths.

The result obtained from the hardness test carried out in 10% NaOH and 5% Acetic Anhydride solutions at two oven drying temperatures using different lengths of the treated fibre were presented. Optimal hardness of **257HRM** and **370HRM** were recorded from the mercerized (treated in 10% NaOH solution at oven-drying temperature of 70^{o}C) and acetylated (treated in 5% acetic anhydride solution at oven-drying temperature of 50^{o}C) respectively. Remarkably, these optimal results were obtained from raffia palm fiber of 60mm length.

Furthermore, the results demonstrate a higher Rockwell Hardness value in the acetylated treatment than in the mercerized fibre conditioning. This shows a **30.54%** increase in Rockwell Hardness value in M-Scale at 60mm raffia palm fibre length. Since structural hardness is a direct approximation of the ultimate tensile strength (UTS) of materials. For instance, the UTS of materials can be estimated by multiplying the hardness value by a factor of 3.3 (Moore and Booth, 2015). Based on this, the degree of hardness of materials was recommended by Pintaude, (2022) for quality control as a formidable measure of the deformation profile of materials. Hence, composites made from harder reinforcement fibres will offer both stronger and less abrasive components especially in machines usually exposed to high frictional contact. Therefore, this result is recommendable in structural applications where extended machine service life is anticipated.

CONCLUSION

The potential use of natural fibres, especially as commercial composite reinforcements depend largely on the extent of regulating their characteristics. In this study, the degree of resistance to indentation and abrasion via hardness testing was carried out to offer increased market value of Nigerian Raffia Palm fibre. Results from this study have demonstrated the economic viability of this fibre as potential reinforcements in composites for the military, automobile, construction, manufacturing, medical, sports, oil and gas industries to exploit. This investigation has identified acetylation using 5% acetyl anhydride at 50°C for 60mm fiber lengths as optimal treatments for increasing the hardness of Raffia Palm fibre.

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