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Research Paper

Acetylation and Mercerization Treatments on the Flexural and Hardness of Raffia Palm Fibre Reinforced Phenolic Resin Composite

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Abstract
Advantages of light-weightlessness, environmental friendliness, availability and affordability top the reasons for the consistent attraction natural fibers offer both the industry and research sectors. Most natural fibers are byproducts with no developed economic values apart from soil nutrients as organic matter. In this study, the need to expand the economic potentials of Raffia Palm fiber (RPf) was evaluated in terms of its flexural and hardness contributions as a reinforcement in a hot compression-molded Phenolic resin matrix composite. Locally harvested, extracted and retted RPf were treated in 10% NaOH and 5% Acetic Anhydride solutions at oven-drying temperatures of 70^oC and 30^oC respectively. RPf length and weight fraction in the composite were varied. Results showed that at fixed 60mm fiber length, percentage rise in breaking strengths of 33.73% and 32.56% were recorded for NaOH and Acetic anhydride treatments respectively in addition to 1.74% breaking strength of mercerization higher than the acetylation. Also, Rockwell hardness values of 277HRM and 256HRM at 60mm fiber length show a 7.58% superior performance of NaOH overacetylation. These results demonstrate the potentials ofRPf in sports, manufacturing and construction industries for military and ballistic parts, composite in fire-resistant plumbing, heat-insulating mines, electric insulators, durable kitchenware and toys.
Keywords –*Fiber, Acetylation, Mercerization, Palm, Phenolic,Raffia, Resin, Composite.*

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I. Introduction

In the last few years, there has been stringent consumers' awareness towards new productsfrom renewable sources. Some factors have led the consumer towards environmentally friendly products like green marketing, new directives on recycling, social influence and change of cognitive values.To introduce new products in a sustainable and responsible way, composite materials are being developed and redesigned aiming to improve and to adapt traditional products as reported in Mitra, 2014 (1).The overdependent on synthetic and fossil-base byproducts has consequential effects on health and the environment as detailed inUchegbulam, et al., 2022 (2). Hence, it is high time industrialization starts exploiting agricultural byproducts like plant-based fibers relegated as wastes.Some of the issues of natural fibresare the scattered information, differences in reported mechanical properties, lack of standards for both producers and users of these materialsregarding methods of collection, treatment, processing and post-processing. Natural fibre performance composites depend directly on the fibres counting, length,shape, arrangement and also the interfacial adhesion with the matrixaccording to AL-Oqlaand Salit, 2017 (3).The twist angle also, is responsible for thecohesion of the fibres and yarn strength up to a certain point. The maximum fibre strength decreases due to the increase in obliquity,as stated by Bar, et al., 2018 (4). Moreover, the increase of the fibre twist angleis correlated with a decrease of fibre-resin bond strength, lower permeability and consequently poormechanical propertiesaccording to Shah et al., 2013 (5). In determining the maximum applied load in the one-dimensional architecture, the twist angle and the level of alignment of continuous-filament yarns play a significant role.The unidirectional composites tend to be weaker for this reason, in the transverse directionsas stated by AL-Oqla and Salit, 2017 (3) and Bar, et al., 2018 (4). Given theseattributes, for a known state of stress, these anisotropic structures can exhibit at least 3 to 4 times bettermechanical properties than their isotropic counterparts.In particular, matcomposed with randomly chopped fibres (whiskers) does not have any preferential stress directionbut they are the preferable choice for large-scale production due to the high availability, ease and costeffectiveness when manufacturing complex parts of isotropic natureaccording to AL-Oqla and Salit, 2017 (3). Despite the reality that natural fibre composites widely used in the automotive industry, yet, there are endless applications in the textile, medical, healthcare and pharmaceuticals, home and personal care, food and feed additives, construction and furniture, packaging, pulp and paper, bioenergy and biofuels industries. As the demand for light-weight and environmentally friendly raw materials are in higher demands, natural fibre-based composites hold significant potential for several industries. Studies indicate that natural fibre composites can contribute to a cost reduction of 20% and weight reduction of 30% of an automotive part as stated in Huda, et al., 2008 (6).

In this light, the present study seeks to explore the potentials of Raffia Palm fiber as a reinforcement in Phenolic Resin matrix composites with the focus on the flexural properties and hardness values at different chemical treatments.

II. Materials and Methods

2.1 Materials

The materials used in this study were: Extracted and treated Raffia palm fiber, pH meter, electronic weighing scale as shown in figure 2.1. Others used in extraction and treatment include: Electric oven, graduated cylinder, plastic bucket, plastic cup, glass beaker, distilled water, Universal testing machine (UTM) and 3.10kg Polylactic Acid pellets.

Figure 2.1: Extracted Rafia palm fibre (a) Digital weighing balance and pH used (b) Phenolic Resin used (c)

III. Methods

Matured Raffia palm fronds were sourced from an ancestral swampy forest in OmuigweAluu, a rural community in Ikwerre local Government area of Rivers State, Nigeria. The pseudo-stems were cut from the trunk and retted by soaking in water for 2 days so that the fibers can be separated from the heavily laden organic matter. The retted trunks were washed in distill water until the organic matter was minimal. The fibers realized were dried under the sun for 7 days before they were separately soaked in 10% NaOH solution with another batch soaked in 5% acetic anhydride solution at ambient conditions to remove the organic matter contents. These chemical immersion of the fiber bundles in the different treatment solutions within 60 minutes at the different operating temperatures. The clear fibers obtained which were free of organic matter were thoroughly rinsed in water until the pH level remained at 7.00 ± 0.02 ready for fiber treatment.

- **Fiber Treatment**

The refined fibers soaked in reagents and rinsed in distilled water were separately mercerized in 10% sodium hydroxide solution by immersing the fibers in the alkaline solution for 3 hours. The fibers were removed from the treatment reagents and rinsed severally in distilled water. The residual waste wastes were tested for pH values intermittently after each rinsing process until the pHremained 7.00 ± 0.02. This set of fibers were oven-dried at 70^oC before they were preserved in a desiccator ready for hardness and tensing testing. This was repeated for the Raffia Plam fiber bundles acetylated in 5% acetic anhydride for 3 hoursand rinsed in distilled water until the pH values remained constant at 7.00 ± 0.02. Like in the mercerized process, the acetylated fibers were oven-dried at 50^oC before they were preserved in a desiccator ready for hardness and tensing testing.Two sets of Raffia Palm fibers were prepared: the batch for Flexural test and another for Hardness test. The hardness test batch were cut to varying fibre length of 50, 60, 70 and 80mm while the set for flexural test were maintained at 60mm length but will be loaded into the mold at varying fiber fractions by weight.

Composite Preparation

The polyoxybenzyl methylene glycol anhydride also known as phenol-formaldehyde resin, Phenolic powder or simply Bakelite was obtained from Mile 3 industrial market at Port Harcourt Nigeria. It was supplied as pellets of 150µm and density of 0.00143g/mm^3. The 60mm long treated Raffia Palm fibers were embedded into the resin in varying weight percentages of 0, 10, 20, 25, 30, 35 and 40% labelled as R_0, R_{10}, R_{20},R_{25}, R_{30}, R_{35}, and R_{40}. Thesamples were set in a metallic mold of inner dimension: 60×15×10mm before hot pressing. The four other molds of length 50, 60, 70 and 80mm were used to mold the composites for 50, 60, 70 and 80mm fiber lengths for hardness test.The hot compression molding was carried out at 170^{o}C with the heat supplied by a heating mantle while the 40bars of pressure was generated via a hydraulic mechanism in the mounting press. The smooth polished surface was an advantage as there was no need for mold lubrication to free the molded compacts. This produced a hard thermosetting plastic composite ready for testing.

- **Composite Testing**

The flexural testing of the composite produced was carried out by the three-point bending test. The compacts were loaded on two supports at 10mm each from the edges. The machine was set at uniaxial load of 5KN as a ramp of 5mm/min. The breaking stress at fracture of each sample was measured and recorded accordingly. This was carried out in triplicates with the average breaking stress presented.

Likewise, Rockwell hardness testing in line with ASTM D785 standard in 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, fiber, etc. This was done using an indenter ball of 6.35 mm diameter and 100kgf load with the hardness values of the composites taken as the average from fourrandomly selected points on the sample surface following the procedure in Uchegbulam, et al., 2019 (7). Five hardness tests were run for each fiber length and these were labelled as replicatesR_1, R_2, R_3, R_4 and R_5. These measurements were recorded at the end of dwell time of 10 seconds started counting once the indenter ball made a direct contact with the sample surface. Meanwhile, all hardness tests were carried out at ambient conditions.

IV. Results and Discussion

The results of the Flexural test conducted on 14 samples: 7 each for the mercerized and acetylation fibres respectively are presented in table 1 while the graphical presentation was shown in figure 2.

Table 1: Effect of fiber weight fraction of Raffia Palm fiber in Phenolic resin on the breaking stress of Raffia Palm fiber reinforced Phenolic resin composite.

Weight Fraction of Fiber in composite (%)	**10% NaOH solution**	**5% Acetic anhydride solution**
	Treatment at 70^{o}C for 60mm fiber length	**Treatment at 30^{o}C for 60mm fiber length**
0	44.52	44.52
10	49.29	47.83
20	58.67	55.31
25	64.12	63.11
30	67.18	64.74
35	64.99	66.01
40	64.43	65.23

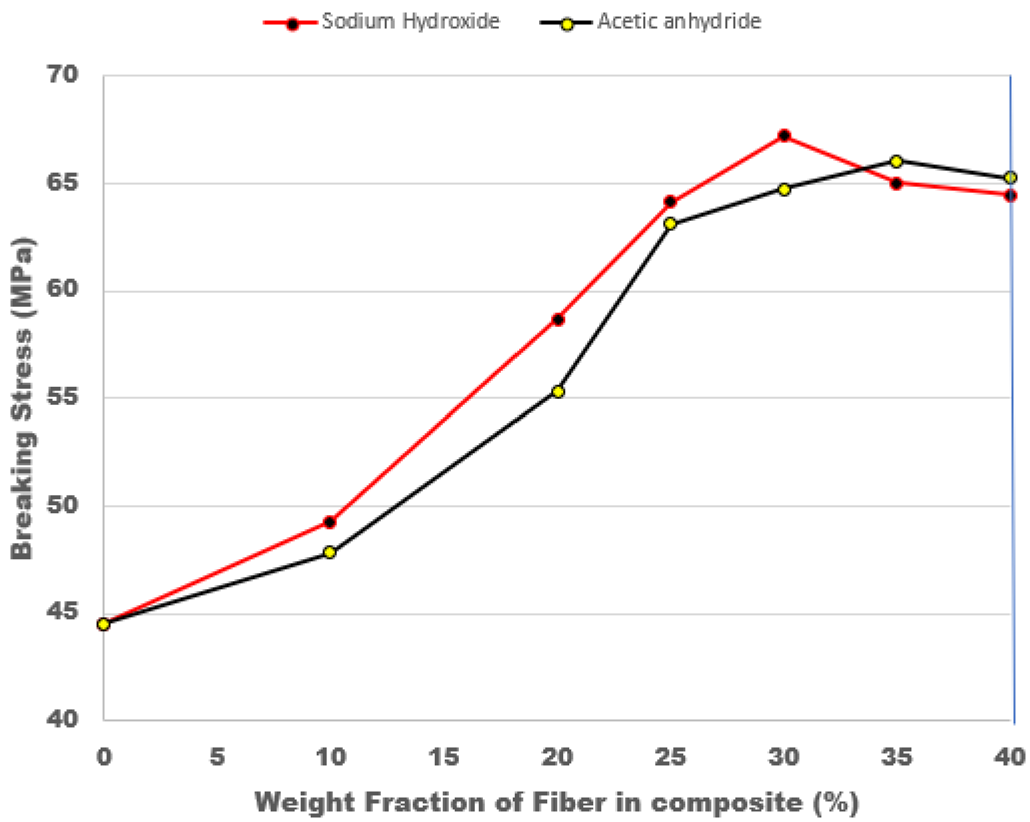

Figure 1: Effect of Raffia Palm fiber weight fraction in Phenolic resin matrix composite.

The result from figure 1 show that at a fixed fiber length of 60mm, the breaking stress increased consistently as fiber content increased until the maximum breaking stress before a decline. Also, the results show that NaOH treatment made a higher peak breaking stress than the Acetic anhydride treatment. Both NaOH and Acetic anhydride treatments made remarkable increase in breaking stress compared to the molded sample without fiber reinforcement. Percentage rise in breaking strengths of 33.73% and 32.56% were recorded for NaOH and Acetic anhydride treatments respectively. Interestingly, the breaking strength of NaOH treated composites was 1.74% higher than that of the Acetic anhydride treatment. Also, maximum breaking strength was noticed at 30% and 35% Raffia Palm fiber reinforcements for the NaOH and Acetic anhydride treatments respectively.

Results from the hardness test conducted on 10 samples: 5 each for the mercerized and acetylation fibresrespectively were presented in tables 2 and 3 respectively while the graphical presentation are shown in figures 2 and 3.

Table 2: Results of Rockwell Hardness Scale M (RHM)test carried out in 10% NaOH solution at 70^{o}C for different treated fiber lengths.

Fibre Replication	HRM @50mm	HRM @60mm	HRM @70mm	HRM @80mm
R_1	88	117	115	100
R_2	152	174	183	89
R_3	152	277	163	161
R_4	131	189	154	111
R_5	145	210	174	128

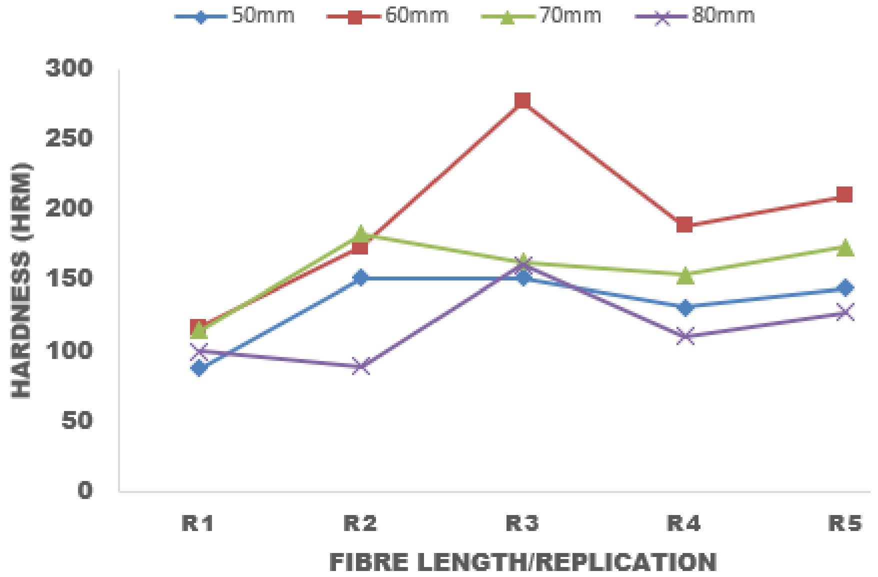

Figure 2: Rockwell Hardness numbers of Raffia Palm fibers mercerized in 10% NaOH solution at oven-drying temperature of 70°C at different fibre lengths.

Table 3: Results of Rockwell Hardness Scale M (RHM) test carried out in 5% Acetic anhydride solution at 30°C for different treated fiber lengths.

Fibre Replication	HRM @50mm	HRM @60mm	HRM @70mm	HRM @80mm
R_1	81	117	110	82
R_2	83	94	144	138
R_3	84	256	123	231
R_4	83	156	126	150
R_5	80	210	131	195

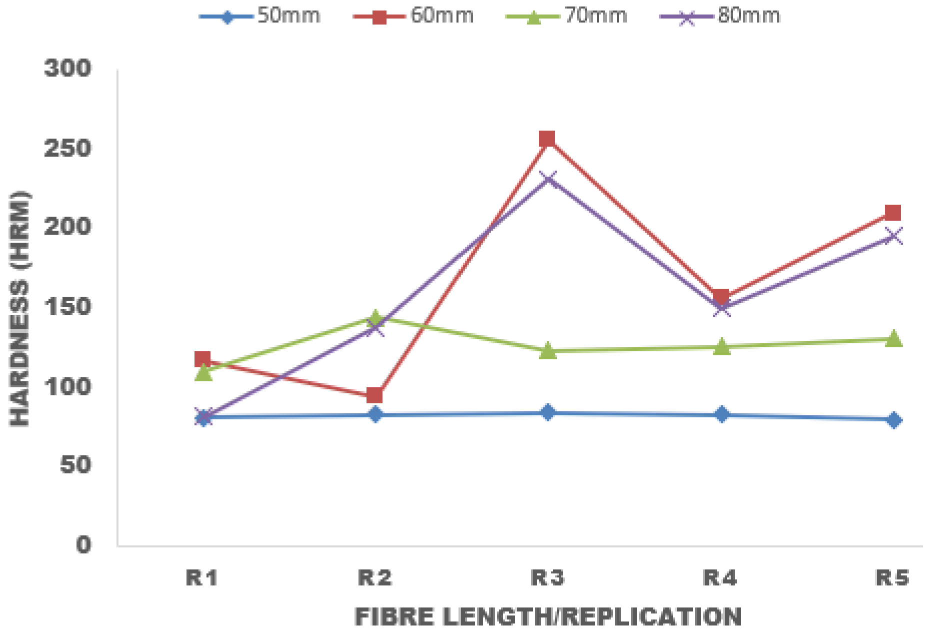

Figure 3: Rockwell Hardness numbers of Raffia Palm fibers mercerized in 5% Acetic anhydride solution at oven-drying temperature of 30°C at different fibre lengths.

The result obtained from the hardness test carried out in 10% NaOH and 5% Acetic Anhydride solutions at oven-drying temperature of 70°C and 30°C respectively were presented in figures 2 and 3 respectively.These results show that the increasing length of Raffia Palm fiber from 50 to 80mm affected the

Rockwell hardness of the Phenolic resin matrix composites tested. Maximum Rockwell Hardness values of 277 and 256 were recorded all at 60mm fiber length. This also reveals that 10% NaOH treatment was 7.58% more effective than 5% Acetic Anhydride fiber treatment in terms of Rockwell hardness values.

V. Conclusion

Natural fibers as reinforcements in polymer matrix composites is continuously explored. Instead of merely left as agricultural byproducts abandoned as wastes, the advantages of being lightweight, environmentallynon-hazardous, readily available and affordable offer tremendously potentials as reinforcements for polymeric based composites. This study explored the flexural and Rockwell hardness values of composite formulated from the Raffia Palm fiber. Results showed that increasing quantity of Raffia Palm fiber consequently increased the flexural properties and Hardness values of the composite molded.At a fixed fiber length of 60mm, percentage increments in breaking strengths of 33.73% and 32.56% were obtained for the NaOH and Acetic anhydride treatments respectively. Also, a 1.74% rise in breaking strength was realized for the NaOH mercerization higher than the acetylation of Acetic anhydride treatment. Remarkably, at 60mm fiber length, Rockwell hardness values of 277HRM and 256HRM were obtained for the NaOH and acetic anhydride treatments respectively showinga 7.58% superior performance of NaOH over acetic anhydride treatments. These results show that Raffia Palm fibers can compete favorably with both other natural and synthetic fibers in Poly matrix composite molding.

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