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Dynamic Mechanical Behaviour of Coir and Coconut Husk Particulate Reinforced Polymer Composites: The Effect of Exposure to Acidic Environment

David O. Obada, Laminu S. Kuburi, David Dodoo-Arhin, Yongdan Hou, Muyideen B. Balogun and Mahmud Muhammad

Abstract

This chapter describes an experimental investigation into the dynamic mechanical properties of coir and coconut husk particulate reinforced polymer composites which were prepared by the hot press method. The composite was immersed in a strongly acidic environment of pH 2.2 for a period of 14 and 28 days (14P and 28P). Values of storage modulus at different vibrational frequencies recorded for the polymers at low temperatures where the molecules are still tightly compressed and the region of first solid state transitions are: Control sample (CS)—913.18, 984.18 and 979.94 MPa; 14P—505.54, 492.47 and 473.60 MPa and 28P—282.25, 298.70 and 285.36 MPa at 2, 5 and 10 Hz, respectively. While values of loss modulus at different vibrational frequencies are: CS—113.32, 109.43 and 107.62 MPa, 14P—92.92, 92.92 and 101.93 MPa and 28P—46.08, 45.61 and 45.18 MPa at 2, 5 and 10 Hz, respectively. Degradation of the mechanical properties was ascribed to the penetration and absorption that occurred between the acid solution and the composite constituents (matrix, filler, and fiber). It was found that frequency variation influenced the dynamic mechanical properties of the polymer composite at the points of measurement.

Keywords: particulate fillers, mechanical integrity, corrosion

1. Introduction

Recently, polymers have become increasingly important for an array of applications by reason of several attractive properties which includes light weight, the ease of processing and its affordability. Fiber reinforced polymers are also attractive as well, due to their biodegradability, high stiffness and strength, good corrosion resistance and many other properties which are important from the tribological point of view, for instance, low coefficient of friction etc. [1–6]. These natural fibers consist of jute, hemp, sisal, coir, banana among others [7].

Among the natural fibers, it is pertinent to note that coir is essentially utilized in view of its low cost, durability among other advantages [8]. Coir has been accounted for as having the highest extension at break among common natural fibers, which allows it absorb strain more than other fibers [9–11]. Reinforcing polymer, for example, polyethylene with natural fiber/particulates to produce polymer composites has gained significant consideration because of their intrinsic properties [12]. Glass fiber has been an interesting option as reinforcement, however, despite the fact that glass fiber reinforced plastics have high quality, their fields of use are exceptionally restricted on account of its inherent higher cost of production [13]. By reason of this, natural fiber reinforced composites have been suggested for use industrially in the off-shore oil and gas industry, in addition to application as composite pipelines, for storage tanks, fluid handling and chemical processing equipment, etc. Therefore from the economic point of view, natural fiber and lignocelluloses, for example, coir and coconut shell powder has remarkable properties as reinforcement/fillers in plastics. Coconut shell particles have become essential as a reinforcement material because of some natural properties like high strength and high modulus [14]. It is worth noting that increment in coconut shell content improves the mechanical strength and water absorption properties of polymer composites, nonetheless, it lessens the stretching at break [15].

In a variety of industrial applications, composite materials are under the attack of corrosive environments (acidic and alkaline). In this way, it is pertinent to note that the influence of exposure or attack of polymer materials to corrosive environments may be quite difficult to notice. It is possible that the material may seem normal but in actual sense may have become embrittled and the mechanical properties may have deteriorated. These mechanically stressed polymers which are exposed to corrosive environments may have crack initiation on the surfaces which propagates by reason of the inherent stresses or with continued exposure to chemical attack. The degradation pathway could occur when the acid, salt or alkaline solution diffuses through the surface and reaches the laminates of the polymer or a situation where the solution penetrates the laminate through micro cracks or other deficiencies/imperfections which may have resulted during the processing stage of the polymer.

Nonetheless, by reason of the continued usage of fiber reinforced polymers (FRPs) and the ambiguities in environmental conditions, etc., the degradation of FRPs in corrosive environments have not been examined in detail. A lot of studies on the durability of FRP composites have laid much emphasis on glass, aramid and carbon composites [16]. Generally, carbon based FRPs are not affected by most corrosive environments. In line with this, to the best of our knowledge, scanty reports are available for the degradation resistance of natural FRPs. For instance, Sindhu et al. [17] studied the reduction of the mechanical properties of coir/polyester and glass/polyester composites under the action of different solvents and environmental weathering. These properties increased with increased aging, but the mechanical properties of samples aged by water, acid and environmental weathering displayed a decrease in their properties. In another study, the resistance of basalt fibers in alkaline solution was noticed to be better than that of glass fibers while the acid resistance was found to be poorer [18, 19].

According to Gill [20], the versatile high performance applications of natural fiber composites, like coir, can replace glass and carbon fibers. It has also been noticed that a considerable level of research have focused mainly on the performance of glass FRP composites in highly corrosive environments. Amaro et al. [21] subjected glass FRP samples in HCl and sodium hydroxide (NaOH) solutions at room temperature (25 °C) for 12, 14 and 36 days. This was followed by conducting experiments on the degradation of mechanical properties. It was concluded that the flexural and impact

strength of the composites reduced with an increase in exposure time with the effect of exposure to the alkaline solution more pronounced than that noticed for the acidic solution. Stamenović et al. [22] investigated the influence of corrosive environments (acidic and alkaline) on the tensile characteristics of glass FRP pipes. It was shown that increasing the pH value of the alkaline solution further degraded the mechanical integrity of the pipe samples, while the samples subjected to the acidic solution provoked an increase in tensile strength and modulus, and decreasing pH values led to a more significant increase. These results from the study of Stamenović et al. [22] corroborate findings of Sindhu et al. [17], where the effects of various corrosive conditions on the mechanical properties of GFRP were investigated. The tensile strength and modulus increased as residence time in acidic solution increased.

On the other hand, Tripathy [23] studied the mechanical properties and interfacial properties of jute fiber filled epoxy resin. It was observed that the moisture intake by natural fibers, insufficient adhesion between untreated fibers and the polymer matrix, led to fiber pull-out with time [24]. Gilbert and Lee [25] investigated the influence of environmental conditions on the mechanical properties of short fiber reinforced composites. The relationship between moisture, acid, and alkali attacks were determined and the chemical properties were evaluated. Potts et al. [26] investigated the tensile properties of short coir reinforced composites. The tensile characteristics were found not to be dependent of fiber length, although the ultimate tensile strength showed some improvement at 10 mm fiber length.

Despite the volume of fiber reinforced polymer composites under investigation, most of the research efforts have been focused on either the characteristics of these polymers or the basic properties of the different phases that make up the composite [12, 27–29]. In our previous work [12, 30], we identified a coir length which enhanced the mechanical and dynamic mechanical (viscoelastic) properties of our developed coconut husk filled composites. Hence, the present work focused mainly on the evaluation of the dynamic mechanical characteristics of the fabricated composite on exposure to an acidic environment. We have considered dynamic mechanical properties of the samples at low temperatures where polymer molecules are tightly compressed and where the first solid-state transitions occur.

2. Materials and methods

Coir (fiber) which was extracted from coconut husk was cleaned with water to remove contaminants and dried at room temperature for 48 h. The dried fibers were soaked in 5 wt.% NaOH solution at room temperature (27 °C) for 30 min as a fiber treatment procedure. The treated coir was subsequently washed with distilled water to remove retained alkali. Furthermore, washed fibers of 30 mm length were open air dried for a day and afterwards dried at 60 °C in a hot air oven for 8 h. Further composite processing is reported elsewhere [30].

2.1 Exposure conditions

Three test conditions were selected in this study to investigate the short term effect of the exposure of the fabricated coir reinforced polymer composite to an acidic medium. According to Mahmoud and Tantawi [31], who investigated the effects of various aggressive acids including HCl, H₂SO₄, HNO₃, and H₃PO₄ on glass FRP composites, H₂SO₄ had a more pronounced effect than the other acids used in their experiments. Additionally, H₂SO₄ is one of agents which FRP components are generally exposed to. Therefore it was used as the acid solution for this study. The condition is an H₂SO₄ solution with pH 2.2 at room temperature. Exposure periods

of 14 and 28 days (14P and 28P) were considered. The sample not exposed to the acid solution was used as control.

2.2 Dynamic mechanical properties of corroded polymer specimens

Dynamic mechanical analysis (DMA) was used to investigate the viscoelastic properties of the exposed polymer samples as reported in our previous study [30]. The frequencies under which DMA measurements were made in this study are: 2, 5 and 10 Hz. The loss and storage moduli at low temperatures where polymer molecules are tightly compressed and where the first solid-state transitions occur were analyzed.

3. Results and discussion

3.1 Storage modulus (E')

The results in **Figures 1– 3** for control, 14P and 28P samples, respectively, demonstrated the trademark drop in modulus around the first transition stage from elastic to viscous of the composite which can be ascribed to energy release with gradual increase in temperature [32]. Values of storage modulus recorded for the polymers at the point of interest were: Control Sample (CS)—913.18, 984.18 and 979.94 MPa; 14P—505.54, 492.47 and 473.60 MPa and, 28P—282.25, 298.70 and 285.36 MPa at 2, 5 and 10 Hz, respectively.

The values of E' for the CS (**Figure 1**) increased with the frequency until 5 Hz and a slight decrease is observed at 10 Hz, while values of E' for 14P sample (**Figure 2**) decreased with frequency until the highest frequency of 10 Hz. In addition, values of E' for 28P samples (**Figure 3**) increased with the frequency and a slight decrease is observed at 10 Hz. A possible explanation for these are: for the control sample, the tendency of an increase in E' under a higher frequency and comparatively lower value of E' for composite subjected to 10 Hz, can be ascribed to increase in molecular mobility of the polymer chains in the first solid

Figure 1.
Storage modulus curves of control sample under varying frequencies.

Figure 2.
Storage modulus curves of 14P sample under varying frequencies.

Figure 3.
Storage modulus curves of 28P sample under varying frequencies.

state transition phase [33]. The continued decrease of storage modulus for the 14P sample can be ascribed to a gradual degradation of the storage modulus due to the influence of exposure to the acidic environment which causes a reduced grip of the tightly bound polymer molecules, allowing them to flow more as compared to polymer molecules in the control sample. It also means at every point of frequency variation, there was an increase in molecular dynamics of the polymer chains because the molecules can move with the force which results in a decline in storage modulus. A similar trend of storage modulus variation at each point of frequency change as observed for the control sample was noticed for the 28P sample. However a decrease in the storage modulus was observed at every corresponding frequency variation, which further explains the effect of exposure to acidic environment

to which the degradation in the fiber/filler/matrix interface can be ascribed, and subsequently allows tightly bound molecules to move with the applied force and cause a decline in the ability of the composite to store energy.

3.2 Loss modulus (E'')

The results in **Figures 4–6** for control, 14P and 28P samples, respectively, show that upon frequency variation, the values of loss modulus (E'') decreased, but an exception was noticed in this trend for the 14P sample. Values of loss modulus recorded for the polymers at low temperatures where the molecules are still

Figure 4.
Loss modulus curves of control sample under varying frequencies.

Figure 5.
Loss modulus curves of 14P sample under varying frequencies.

Figure 6.
Storage modulus curves of 28P sample under varying frequencies.

tightly compressed and the region of first solid state transitions were: CS—113.32, 109.43 and 107.62 MPa; 14P—92.92, 92.92 and 101.93 MPa and 28P—46.08, 45.61 and 45.18 MPa at 2, 5 and 10 Hz, respectively. These results indicate that the acid solution provoked degradation in the fiber/filler/matrix interface which may be associated with the occurrence of fractures under stress conditions. These results corroborates findings of Stamenović et al. [22], which suggested that the most significant influence of corrosive solutions was on the fiber-matrix connection and this influence directly reduces the load carrying capacity of composites.

The possible pathways for the reduction in storage and loss moduli after exposure to the acidic environment can be explained or associated with the penetration and absorption that occurred between the acid solution and the composite constituents (matrix, filler and fiber). The solution most likely penetrates through the polymer matrix and has the possibility of separating out in micro-cracks. These assertions have also been buttressed by [31, 34]. Also similar pathways can be attributed to the degradation of the fiber/filler/matrix interface which is caused by the penetration of the acid solution through cracks which may have gained entry through voids in the matrix [17, 22, 35]. According to Hammami and Al-Ghilani [36], the degradation of the mechanical properties of polymers can occur in two stages. Firstly, the polymer is degraded under actions which are as a result of the diffusion of water and the presence of hydrogen ions. Secondly, the fiber itself can be degraded which results to cracks appearing on the surface of the fiber. This to some extent affects the resistance of the composite to stresses. Moreso, according to Stamenović et al. [22], a degradation of the mechanical properties can be associated with the fiber-matrix interface where the acid immersion of the composites promotes the deficiencies of the stress carrying capacity of the polymer composites.

4. Conclusions

The dynamic mechanical properties of coir and coconut husk particulate reinforced composites immersed in an acid solution over periods of 14 and 28 days

were investigated. The dynamic mechanical analysis of the composite not subjected to the corrosive medium was also tested for comparison. The degradation pathway was clarified on the basis of the experimental results. The major conclusions are as follows:

1. The composites exhibited relatively little resistance to corrosion in acid solution by reason of the reduced storage and loss moduli after immersion in the acid solution.
2. In terms of mobility of polymer chains, an increase in frequency or stress loading caused a sharp decline in storage and loss moduli of the immersed samples due to the reduced grip of the tightly bound polymer molecules, allowing them to flow more as compared to polymer molecules in the control sample.
3. Degradation of the mechanical properties can be associated with the penetration and absorption that occurred between the acid solution and the composite constituents (matrix, filler and fiber).
4. It is recommended that the developed composites can be applied in areas where the composites are not exposed to load carrying corrosive environments.

Conflict of interest

The authors declare no conflicts of interest.

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