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Research Article

Water Absorption Behavior of Teff (*Eragrostis tef*) Straw Fiber-Reinforced Epoxy Composite: RSM-Based Statistical Modeling and Kinetic Analysis

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Recently, reinforced polymeric composites prepared from natural fibers have received a significant interest among the researchers because of its appreciable sustainability, environmentally friendly, and low cost. However, one particular issue, that is, hydrophilic property, still needs to be addressed for its successful applications. Since the hydrophilic tendency of natural fibers is extremely undesirable, it leads to the quick degradation of fiber-based polymer composites. Hence, the fiber property, hydrophilic nature, is influenced by the presence of noncrystalline and voids part of these fibers that significantly influences the polymer matrix adhesion. Hence, it is very important to understand the water absorption behavior of reinforced fiber composites. In this study, a crop residual material specific to Ethiopia, teff straw (*Eragrostis tef*), was used as fiber material. The fiber was treated with 1% NaOH followed by 1% CH_2=CHCOOH at room temperature for improving the bonding strength between the fiber and polymer, which leads to suppress the water absorption. The investigation on mathematical model for water absorption property at different fiber loadings (4%, 8%, 12%, 16, and 20%) was carried out, and the analysis on the kinetic behavior of water absorption was also investigated. In addition, the response surface-based statistical modeling which correlates water absorption, fiber loading, and time has been analyzed.

1. Introduction

In recent days, a significant awareness has been increased to look for the materials not only with excellent properties but also having appreciable sustainability with economically feasible [1]. Commercially, the cellulosic fibrous feedstocks are used as major raw materials to prepare reinforced polymer matrixes for the development of composite materials which are derived from both woody and nonwoody sources. Woody supplies are collected from forests, whereas nonwoody sources are obtained from sources other than forests, such as recycled fibers, sugarcane bagasse, nonwoody plant straws, rice husk, agroindustrial byproducts, and agricultural leftovers. In order to overcome the environmental issues from deforestation, nonwoody raw materials are gaining the huge interests for production of fiber pulps in composite material sectors since they have several benefits of being readily grown, high growth rate, comparatively high cellulose content, a low amount of lignin, high volume generation in modern irrigation, and requirement of small amount of fertilizer for mass production [2]. Undeniably, the approach with respect to the smart use of full natural resources with integrated multiproduct valorization is always most welcomed. Under this concept, the study of underutilized resources, including residues and by-products from existing nonwoody crops, attain a renewed interest for fibrous production. In elsewhere, different natural fibers such as wheat straw, bagasse, jute, and pineapple were investigated to be used as reinforcement in polymer matrix.

Teff hay or straw is hard stem part of the teff plant which is known to be an indigenous lignocellulose biomass. In Ethiopia, teff crop is cultivated most extensively. At present, a significant volume of teff and its straw is produced annually in different provinces of Ethiopia. It is well-known cereal crop culverted and staple food in Ethiopia. Hence, teff straw is most widely available in almost all agricultural places of Ethiopia. Once the grains of teff seeds were separated, the teff straw was considered an agrowaste. Lot of researches were carried out in terms of beneficial utilization of teff straw. Earlier, teff straw was investigated by Devnani and Sinha [3] to develop a light-weight composites reinforced by epoxy. In another study, Abraham has examined on the mechanical properties reinforced concrete made by the teff straw [4]. However, still there is a room for research on the use of teff (*Eragrostis tef*) straw- (TS-) based-reinforced polymeric composite materials. Teff is one of Ethiopia's most well-known indigenous crops; once the seeds are harvested from the crop, a significant volume of TS from post-harvest is obtained as agricultural residue. Hence, TS is widely available, inexpensive, and most abundant in Ethiopia [5]. TS is the dry stalks part of teff hay. The studies on chemical composition of TS revealed that it composed with 37.1% cellulose content, 28.99% hemicellulose, 17.85% lignin, and 8.55% extractives. Such a substantial amount of cellulosic and hemicellulosic contents makes that the TS can be a potential feedstock for natural fiber-based polymeric composite materials.

From the previous studies, it was observed that one particular issue, that is, the compatibility between the hydrophobic nature of the polymer matrix and hydrophilic nature of the natural fibers, is the responsible factors for unsatisfied mechanical and thermal properties of polymeric composite materials. Various researches were carried with different treatment methods to attain an improved quality of composites towards abovementioned issue. Keeping this view, a most important behavior of composite materials, water absorption, can be an imperative aspect to produce a superior quality composite [6, 7].

In the present study, a crop residual material, TS, was used as fiber feedstock to prepare a composite material. The fiber was treated with NaOH followed by acrylic acid at room temperature for improving the bonding strength between the fiber and polymer and to suppress the water absorption. The investigation on mathematical model for water absorption property at different fiber loadings was carried out. The kinetic analysis on water absorption was also established. In addition, the response surface-based statistical modeling which correlates water absorption, fiber loading, and time has been analyzed. In order to get the interaction between water absorption and fiber loading, an emphasis has been given for statistical modeling using response surface methodology (RSM) with aim of appropriate valorization of the TS towards reinforced polymeric composite production.

2. Materials and Methods

2.1. Teff Straw and Chemicals. Teff straw (TS) was collected from the nearby agriculture province of Addis Ababa, Ethiopia. The TS was chopped by manually to have the average length of TS 5-10 mm. Further, it was washed thoroughly using distilled water to remove unwanted waste impurities and soil debris. Then, it was subjected to dry using hot air oven for 48 h at 650°C. This material was named as untreated TS fiber; hereafter, the same is called as UTSF [8]. A standard epoxy resin (AW106) and hardener as curing agent (HV953IN) were obtained from Mexico Araldite Pvt. Limited Addis Ababa, Ethiopia. NaOH was procured JIGRA ETH Chemicals Pvt. Limited, Addis Ababa, Ethiopia. Acrylic acid was obtained from AE Chemicals Trading Plc. Pvt. Limited, Addis Ababa, Ethiopia.

2.2. Surface Treatment of TS Fibers. Clean TS fibers were under taken to soak in 1% NaOH for 45 min followed by 1% acrylic acid for 45 min. This treatment was carried out at room temperature. The treatment was carried out at ratio of 20 : 1 for the liquor to fiber. Further, the treated TS fibers were again washed thoroughly using distilled water for removing the remaining chemicals that used for surface treatment. Then, treated TS fibers kept in hot air oven (75°C for 48 h) to dry by removing the moisture. These fibers were named as TTSF and considered as treated TS fibers [9].

2.3. Preparation of UTSF and TTSF Composites. As per the recommendation from the supplier, the hardener and epoxy resin were mixed in the ratio of 4 : 5. Then, UTSF and TTSF were mixed separately to prepare the reinforced polymeric composites. Each mixture was stirred using mechanical agitator at 1800 rpm for 15 min. The mixture was observed to be uniform and homogeneous. Further, the mixture was poured in a mold with the size having $250 \times 250 \times 10\,mm^3$. Then, it was allowed to cure at room temperature for 24 h. For the fabrication purpose, hand layup technique was carried out to prepare the composites obtained from UTSF and TTSF. In order to avoid sticking of epoxy fiber mixture to the wall of the mould, a clean polythene sheet was used [10]. A dead weight of 20 kg was kept on the mould for two days. The composites were carefully withdrawn from the mould for water absorption test. From the results, it was apparent that the composite prepared from TTSF had lowered water absorption. Hence, further water absorption analysis has undertaken with different fiber loadings using TTSF. Abovementioned composite preparation technique was used for different fiber loadings (4%, 8%, 12%, 16, and

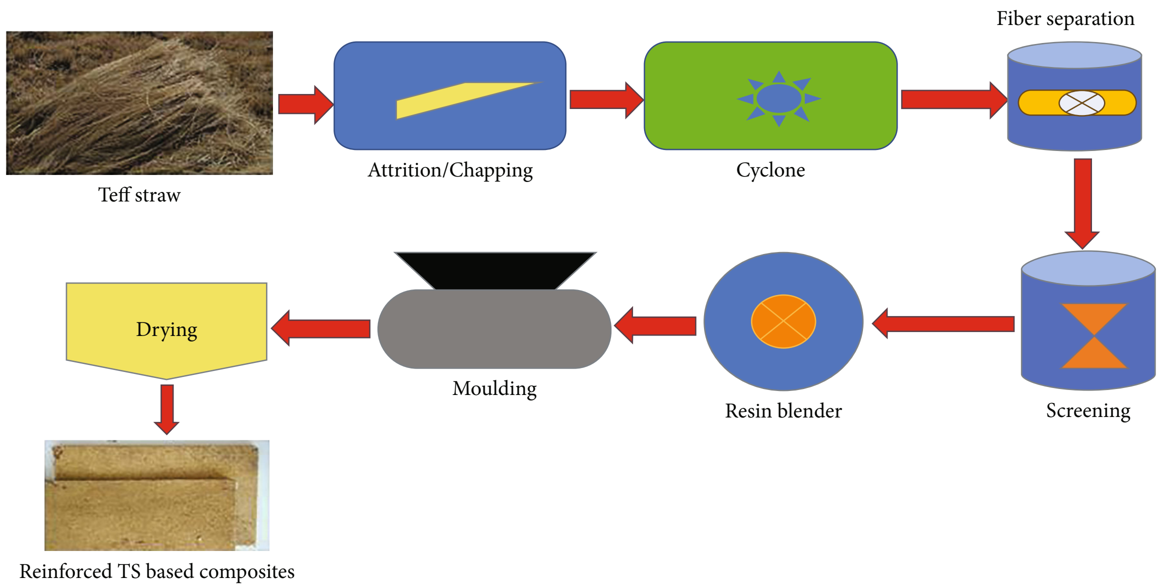

FIGURE 1: Schematic flow of TS-based composite preparation.

20%) of TTSF. Further, they were subjected to water absorption test. The schematic flow of TS-based composite preparation is presented in Figure 1.

2.4. Water Absorption Test. The standard method, ASTM D570, was adopted to analyze the water absorption of TTSF epoxy composites. For that, a sample with 25.4 × 76.2 mm thickness by the wide of composite material was taken [11]. Water absorption of composite was determined using Equation (1).

$$W(\%) = \frac{M - N}{N} \times 100. \quad (1)$$

In Equation (1), M refers the dry initial weight of the sample. N denotes the weight of the sample after immersion in distilled water. W is water absorption rate in percent.

2.5. Kinetic Approach for Water Absorption Behavior. The studies showed that the water absorption in polymer composites can be explained based on Fickian as well as non Fickian concept [12]. While dealing the water absorption behavior on polymer composite, the diffusion coefficient should be considered which is the most important criterion. Diffusion coefficient is defined as the ability of penetration of the water molecules to inside the any sample structure. In order to understand the diffusion behavior, Equation (2) can be used.

$$F = \frac{mt}{mx} = kt^n. \quad (2)$$

In Equation (2), F refers the water absorption in percentage at time any time "t," and "mt' denotes the maximum % of water that can be absorbed. The constants, n and k, are known to be kinetic constants which are useful to understand the water absorption behaviors. By applying the natural logarithm for Equation (2), the resulted equation can be presented as Equation (3).

$$\log F = \log \left(\frac{mt}{mx}\right) = \log k + n \log (t). \quad (3)$$

From Equation (3), a plot, log (F) vs. log (t), was generated which gives the value of n and k using statistical analysis. The n value provides the insight of the diffusion behavior. Accordingly, if the n value is 0.5, the diffusion can be called Fickian mechanism [13]. If the n value is less that 0.5 but close to 0.5, the diffusion can be classified as pseudo-Fickian mechanism. Furthermore, diffusion coefficient (D) was calculated using the following correlation (4).

$$F = \left(\frac{4}{n}\right)\left(D^{0.5}\frac{t^{0.5}}{n^{0.5}}\right). \quad (4)$$

2.6. RSM Statistical Modeling and Analysis. The two independent factors, fiber loadings and time, were under taken for further statistical modeling to correlate the water absorption behavior using response surface method (RSM) through CCD. Table 1 presents the rage of the factors used in this study.

In this study, the Design-Expert® (version 12.0.0) software was employed for CCD combination of experiments and RSM optimization [14]. The sequence of experimental run was undertaken in randomized in order to minimize the effects of the uncontrolled factors. The factors were correlated using a quadratic model. This quadratic model is presented in Equation (4):

$$WA = L_0 + \sum_{j=1}^{2} L_j P_j + \sum_{j=1}^{2} L_{jj} P_j^2 + \sum_{i} \sum_{<j=2}^{2} L_{ji} L_i L_j + s_i. \quad (5)$$

Table 1: The rage of the factors used in this study.

Name of the factor	Minimum	Maximum	Coded low	Coded high	Mean	Std. Dev.
TS loading	1.89	23.11	-1	+1	12.50	6.12
Time	1.59	4.41	-1	+1	3.00	0.8165

Table 2: Parameter values for TTSF-reinforced epoxy composites with different fiber loadings.

Sl No.	Fiber loading	n	k	$D \times 10^{12}$ (m^2/s)
1	4%	0.3123	-0.73631	3.47
2	8%	0.4314	-0.9871	4.71
3	12%	0.4184	-1.0627	5.45
4	16%	0.3572	-0.8126	6.82
5	20%	0.3265	-0.8542	7.32

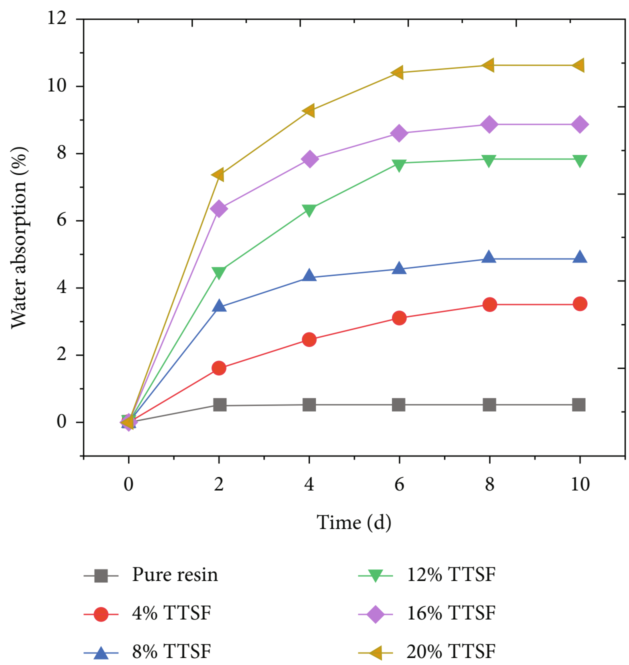

Figure 2: The influence of TTSF loading on water absorption for reinforced epoxy composites.

In the present study, there were 13 runs of experimental plan (Table 2). The water absorption was fed as the response for each combination which was taken as the mean value of duplicate. The results obtained from the 13 combinations of runs were used to fit the model for developing the interrelationship between the fiber loading and time [15]. The model developed by RSM was investigated to check the statistical significance using the ANOVA methodology.

3. Results and Discussions

3.1. Influence of Fiber Loading on Water Absorption and Kinetics. In order to understand the properties of the com-

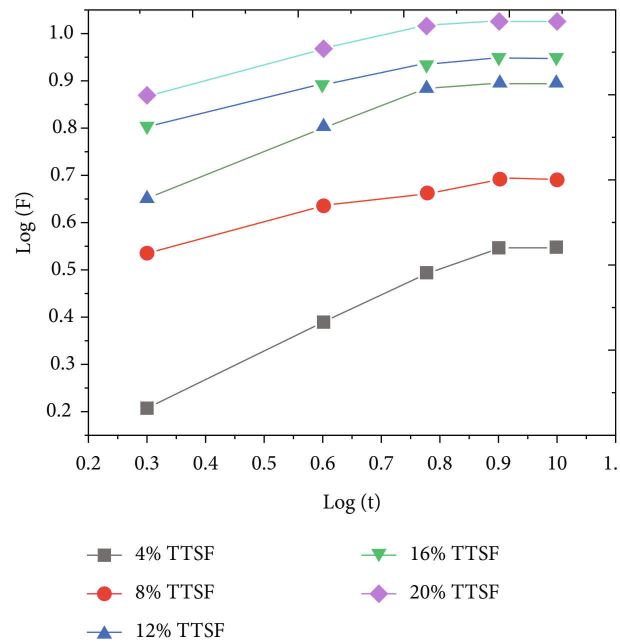

Figure 3: The kinetic plot for water absorption on different TTSF fiber loadings of reinforced epoxy composites.

posite materials, water absorption should be considered seriously [16]. Prior to the experiments, different TTSF loading range was examined. Using these results, it was observed that the range of TTSF varied from 4 to 20%. In the present study, fiber (TTSF) loading was varied (4%, 8%, 12%, 16, and 20%) at fixed level of hardener and epoxy resin. Based on the water absorption property, Figure 2 was constructed which illustrates the water absorbed (%) by the TTSF-reinforced composite material at different fiber loadings. From the results, it was observed that the composites showed higher percent of water absorption compared to pure epoxy resin. The trend of water absorption seemed to be increased with fiber loading [17, 18].

Figure 3 presents the fitting of experimental data to Equation (3) as kinetic analysis. From the best fit plot, the value of k and n for the TSSF-reinforced composites with different fiber loadings is given in Table 1. From the results (Table 2), it is apparent that TTSF composites follow the mechanism of pseudo-Fickian diffusion. It was observed that n value for all the fiber loadings was close to 0.5. From such a mechanism of pseudo-Fickian diffusion, it is cleared that rate of water diffusion is comparatively less than polymer segment mobility. Thus, equilibrium in polymer can be achieved rapidly. The diffusivity values for the TTSF were observed to be low that explicates that the water absorption

Table 3: CCD matrix and corresponding water absorption for RSM.

Run	TS loading (%)	Time (d)	Water absorption (%)
1	5	2	2.1
2	5	4	2.8
3	12.5	3	4.6
4	12.5	3	4.62
5	1.893398	3	1.2
6	12.5	3	4.6
7	20	4	9.1
8	12.5	3	4.63
9	12.5	4.414214	5.36
10	20	2	7.12
11	12.5	1.585786	4.6
12	23.1066	3	10.23
13	12.5	3	4.63

Table 4: The fit summary of different type of equations.

Source	Sequential p value	Lack of fit p value	Adjusted R^2	Predicted R^2
Linear	<0.0001	<0.0001	0.9639	0.9449
2FI	0.2716	<0.0001	0.9652	0.9414
Quadratic	<0.0001	0.0036	0.9996	0.9983 (suggested)
Cubic	0.0006	0.5228	1	0.9999 (aliased)

is getting lowered while TS has been subjected to treated. The similar observations were found by Li et al. [12] and Japić et al. [9].

3.2. RSM Analysis and Statistical Modeling. As we know, CCD and the BBD are the most widely used designs for the RSM experiment. In this experiment, the two most important parameters only have been taken for the investigation of interaction effects and optimization. Keeping this view, CCD experimental design only can be applicable for the response optimization. Hence, CCD has been considered. Using the RSM coupled with CCD (Table 3), the experimental results were adopted to develop model of equation to correlate the TTSF loading and time. In order to select the most suitable model of equation, the fit analysis on different equations has been carried out; the same is presented in Table 4 as fit summary. In this context, quadratic model was picked out for further response surface analysis. Accordingly, Equation (4) was developed. Based on the model development, response surface was generated with respect to TSSF loading and time; the same is presented in Figure 4. From the interactive analysis, it was cleared that the increasing TSSF led to increasing the water absorption tendency. Also, it was well apparent that the water absorption of prepared TTSF composite materials increased with increasing time. The interaction effect of TSSF loading and time showed a positive effect with the coefficient value, 0.060284 (Equation (4)). However, the p value for the interactive effect was observed to be less than 0.05, which exhibited a potential significance of the adsorption process.

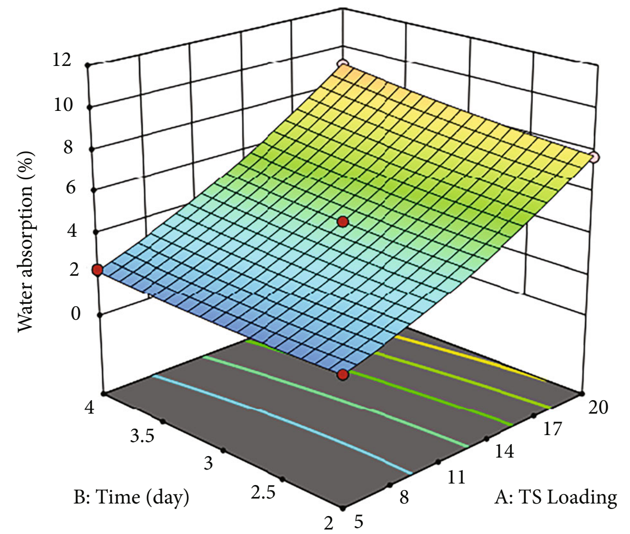

Figure 4: The 3D interactive effect of water absorption with respect to tome and TS fiber loading.

$$\begin{aligned}\text{Water absoption}(\%) = {} & +2.52878 + 0.060284(\text{TS loading}) \\ & - 0.969264(\text{time}) \\ & + 0.038333(\text{TS loading})(\text{time}) \\ & + 0.009802\,(\text{TS loading})^2 \\ & + 0.141375\,(\text{time})^2. \end{aligned} \quad (6)$$

4. Conclusion

The present study describes the influence of fiber loading on the water absorption property for the epoxy-reinforced polymer composite. Teff straw-based polymer composite was developed with different fiber loadings. It was observed that the chemical treated by NaOH followed by acrylic acid explicated less water absorption. Also, the experimental results showed that as TTSF increases, water absorption also increases. The kinetic analysis on the water absorption property revealed that the diffusion curve fitted well the pseudo-Fickian mechanism. In addition, the diffusivities were determined from the different fiber loadings. Further, the RSM-based statistical model was developed with respect to the CCD design, which gives the understandings of the interaction between the fiber loading and time for water absorption.

Data Availability

The underlying data supporting the results of this study were included in the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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