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Synthesis of Trimethylol Urea Composite from Alkali Treated Tea Waste Fiber: Water Uptake Profile

Fidelis Timothy Tizhe
Student, Department of Chemistry, Modibbo Adama University of Technology, Yola, Adamawa State, Nigeria
Louis Hitler Muzong
Student, Department of Chemistry, Modibbo Adama University of Technology, Yola, Adamawa State, Nigeria
Barminas Jeffery Tsware
Professor, Department of Chemistry, Modibbo Adama University of Technology, Yola, Adamawa State, Nigeria
Ansa Eyo Bassey
Assistant Lecturer, Department of Pure and Applied Chemistry, University of Calabar, Calabar, Cross River State, Nigeria
Bisong Emmanuel Asu
Student, Department of Pure and Applied Chemistry, University of Calabar, Calabar, Cross River State, Nigeria
Akakuru Ozioma Udochukwu
Assistant Lecturer, Department of Pure and Applied Chemistry, University of Calabar, Calabar, Cross River State, Nigeria
Philip Monday
Student, Department of Chemistry, Modibbo Adama University of Technology, Yola, Adamawa State, Nigeria

Abstract:
In our continuous research to further characterized composite of tea waste fiber from untreated and treated tea waste (UTWF and TTWF).Different fiber weights (2g, 3g, and 4g) with trimethylol urea (TMU) were prepared. The water uptake test was carried out at room temperatures and at different temperatures. The result indicated a significant difference from the different fiber weights used, 3g UTWF show the highest water uptake while 4g UTWF showed the lowest water uptake. For the TTWF composite, 4g TTWF composite exhibited the highest water uptake while 3g TTWF show the lowest water uptake values. TMU with zero fiber content showed lowest water uptake throughout compared to the composite from both the UTWF and TTWF using different fiber weights. Ash and moisture content of the untreated and treated tea waste fiber (UTWF and TTWF) was determined as well. The ash content of UTWF was higher than that of TTWF. While the moisture content of the UTWF was also higher than that of TTWF.

Keywords: *Tea waste, trimethylol urea, composite, water absorption.*

1. Introduction

Tea is one of the major drink in the world. Consumption of ready-made tea, which packed into cans and bottles, has been increasing remarkably in recent years. Beverage companies manufacturing various tea drinks produce various tons of tea leaf waste annually. On world scale, Nigeria is a medium producer of tea crop, being favoured by the humid condition in the southern Nigeria. They produce large quantities of chaff, which are burnt. Some literature have shown that, these tea waste may be considered as a valuable protein source consisting of 22~35% of crude protein (CP) (Kondo *et al*., 2004). While tea waste contains high CP, it is known to contain a high proportion of tannins (Kondo *et al*., 2004). The by-product from tea leaf (TL), otherwise known as spent tea leaf (STL) may be used as an alternative feed resources for ruminant animals. The by-product may be utilized by ruminants, which are capable of converting fibrous feed into human edible food as milk and meat. Although, the gas production from the *in vitro* fermentation is a nutritional waste, it remains one of the reliable means to measure quality of feeds (Fievez *et al*., 2005). Apart from losing the economic value of the waste through burning, a huge amount of capital is expended in disposing it. In some places, the waste constitutes environmental hazards through indiscriminate dumping and incineration and some youths within the domain of the company used it as intoxicant (smoking as cigarette). Having considered the above negative impact of the waste, not only on the environment but also on human, this waste tea was converted into a valuable industrial. The objective of this research was to evaluate how this tea waste was transformed in a natural fiber composite followed by weight loss and water uptake determination of the composite. Natural fiber composites are emerging as realistic alternatives to glass and carbon-reinforced composites in many applications. Shortcomings associated with natural fibres have to be overcome before using them in polymer composites. The most serious concern with natural fibres is their hydrophilic nature due to the presence of pendant hydroxyl and polar groups in various

constituents, which can lead to poor adhesion between fibres and hydrophobic matrix polymers (Rong *et al.*, 2001, Bledzki & Gassan *et al.*, 1996). The hydrophilic nature of the fibre surface lead also to high moisture up take for the natural fibres which can seriously lower the mechanical properties of the fibres themselves. Lack of good interfacial adhesion, low degradation temperature, and poor resistance towards moisture make the use of natural fibre reinforced composites less attractive than
synthetic fibre (glass, carbon, aramid, etc.) that have been up to now the only choice for
reinforcing polymeric composites, due to their superior mechanical properties. However, the production of composites reinforced with synthetic fibres and matrices requires a large amount of energy which is only partially recovered with incineration of fibre reinforced composites. This has once again drawn the attention towards natural fibres due to their environmental advantages. It has been demonstrated that the energy needed for production of natural fibres is, on average, more than half of the amount needed for synthetic fibres Thus, the renewed interest in the natural fibers, due to their lightweight, nonabrasive, non irritating, combustible, nontoxic, biodegradable properties (Saheb & Jog,1999), low energy consumption for production, budget zero CO_2 emissions if burned, low cost, main availability and renewability compared to synthetic fibres, has resulted in a large number of applications to bring it at par and even superior to synthetic fibers.
Because of such properties natural fibers are fast emerging as a viable choice as reinforcing material in composites (kalia et al., 2009). Even if natural fibre has a very low energy consumption for production compared to other synthetic fibre, such as glass or carbon, careful environmental impact evaluation must be take in consideration in order to make the right choice. In fact, the validity of "green" case for substitution of synthetic fibre by natural ones is dependent on the type of reinforcement and related production processes.

2. Materials and Methods

2.1. Materials

Urea, formaldehyde, sodium dihydrogen phosphate, sulphuric acid and sodium hydroxide pellets were reagent grade products from the British Drug House (BDH). The waste tea fiber also known as spent tea leaf (STL) was collected from a tea making company, located in Taraba State of Nigeria. All materials were used as received.

2.2. Treatment of Tea Waste fiber

The fiber was treated with alkaline solution and this treatment is also known as mercerization, NaOH concentration of 3% was used as it has been reported improve the tensile strength and the flexural strength of the fibres (Rout J *et al.*, 2001). The waste tea fibre was soaked in 3% of NaOH for 24 hours at room temperature as shown on the reaction. After that, the waste tea fibre were rinsed and dried under the sun. Therefore fibres were randomly spread within the matrix.
$\text{FIBER-OH} + \text{NaOH} \rightarrow \text{FIBER-O-Na}^+ + H_2O$.

2.3. Proximate Analysis of Both the UTWF and TTWF

2.3.1. Determination of Ash Content

The method reported by National agency for food and drug administration and control (NAFDAC) for the analysis of food samples was adopted for the determination of the ash content. In brief, a dry crucible dish (w_1), was weighed and 1g of the waste tea fiber was spread into the dish and weighed (w_2). Then it was transferred into a muffle furnace at 1000^{o}C until fully ash (colour changes to gray) was obtained and weighed (w_3). Triplicate determinations were made for each sample (UTWF and TTWF) and the average value recorded.

$$\%\ \text{Ash} = \frac{(W3-W1)}{(W2-W1)} \times 100$$

2.3.2. Determination of Moisture Content

The method reported by National agency for food and drug administration and control (NAFDAC) for the analysis of food samples was adopted for the determination of the moistures content. In brief, a dry crucible dish (w_1), was weighed and 1g of the waste tea fiber was spread into the dish and weighed (w_2). Then the dish and its content was transferred into an oven at 105^{o}C to dry for 3 hours. The dish was removed and weighed (w_3). Triplicate samples (UTWF and TTWF) were used and average value taken.

$$\%\ \text{Moisture} = \frac{(W2-W3)}{(W2-W1)} \times 100$$

2.4. Synthesis of Tirmethylol urea

Trimethylol urea was prepared by the method described by Barminas and Osemeahon, (2006). One mole (6.0 g) of urea was reacted with three moles (24.3 ml) of 37% (w/v) formaldehyde using 0.2 g of sodium dihydrogen phosphate as catalyst. The pH of the solution was adjusted to 6 by using 0.5 M H_2SO_4 and 1.0 M NaOH solutions. The solution was then heated in a thermostatically controlled water bath at 70°C. The reaction was allowed to proceed for 2 h after which the sample was removed and kept at room temperature (30°C). The UF samples used in this experiment were obtained by removing 125ml of resin from the synthesized UF resin and was mixed with the tea waste using different weights (2g, 3g, and 4g) both the untreated and the treated tea waste fiber (UTWF and TTWF).

2.5. Preparation of Composite

The fiber was reinforced with Tirmethylol urea (TMU) resin after the mercerization process, the TMU resin serve as the matrix. The TMU resin helps to bind the individual component together. But this requires series of chemical treatment for this bonding between the matrix and waste tea fiber to be effective, because natural fibres are inherently incompatible with nonpolar-hydrophobic thermoplastics, such as polyolefins. Moreover, difficulty in mixing because of poor wetting of the fibres with the matrix is another problem that leads to composites with weak interface (John & Anandjiwala, 2008). The fiber in form of mats were randomly oriented in a mould using different fiber weights (2g, 3g, and 4g) both the (UTWF and TTWF) and pressed into a mat of different thickness (10, 20, and 30mm). The thickness observed in treated tea waste fiber (TTWF) tend to be higher than the untreated tea waste fiber (UTWF). TMU resin, which served as a matrix medium, was mixed with the tea waste fiber and poured into a square plastic container of 10cmx10cm in dimension which served as a mould. After being cured for 48 hours, the CFRP composites were removed from the mould for characterisation as seen in Fig 1.

A:Treated tea waste composite B: Untreated tea waste composite

Figure 1: Treated (A) and unreated (B) tea waste composite

2.6. Characterization of Composite

2.6.1. Determination of Water Uptake at Room Temperature and at Different Time

The modified tea bag method reported by (Barminas and Osemeahon, 2005.) was adopted. This involved the immersion of samples (2g, 3g, and 4g) for both the UTWF, TTWF composite and TMU with zero fiber in excess distilled water inside a polyethylene bag. The assembly was pre-weighed and hermitically sealed and left at different time (2h, 4h and 24h) at 30 ^{0}C to attain equilibrium. At the end of the equilibrium period, excess solution was carefully sucked out using a micro-syringe and tea bag was reweighed (Barminas*et al.*, 2005).

2.6.2. Determination of Water Uptake at Different Temperatures

The process was also repeated for the same sample after been subjected to different time and temperatures kept in an oven, using modified tea bag method reported by (Barminas and Osemeahon, 2005.) was used. This involved the immersion of samples (2g, 3g, and 4g) for both the UTWF and TTWF in excess distilled water inside a polyethylene bag. The assembly was pre-weighed and hermitically sealed and left for 24h at 30^{o}C to attain equilibrium. At the end of the equilibrium period, excess solution was carefully sucked out using a micro-syringe and tea bag was reweighed (Barminas*et al.*, 2005).

Percentage water uptake was calculated as follows;

$$\text{Water uptake (\%)} = \frac{(W_s - W_g)}{W_g} \times 100$$

where

W_g =weights of the dry sample

W_s =weights of the wet sample

3. Results and Discussion

3.1. Proximate Analysis of Both the UTWF and TTWF

3.1.1. Determination of Ash Content

The ash content obtained from UTWF is higher than that of the TTWF due to loss of inorganic components during processing of the raw material used in making the tea. The difference between the value of ash content obtained from both the UTWF and TTWF is (0.08%). This can be seen on (Table 1).

Fiber	Ash Content	Temperature (^{O}c)
UTWF	2.22±0.01	1000
TTWF	2.15±0.01	1000

Table 1: The ash content for both UTWF and TTWF at a temperature of 1000^{o}C

3.1.2. Determination of Moisture Content

In Table 2, the moisture content obtained from UTWF is higher than TTWF. This may be due to the pendants hydrophilic hydroxyl groups present in the cellulose of the UTWF than TTWF. Perhaps, the hydrophilic hydroxyl groups present in the cellulose of the TTWF had been reduced during the chemical treatment thereby increasing fibers moisture resistance property (John and Anandjiwala, 2008).

$\text{FIBER-OH} + \text{NaOH} \rightarrow \text{FIBER-O-Na}^{+} + H_2O$.

Scheme 1: Mercerization of the tea waste fiber

Furthermore, the chemical treatment of the fibers as illustrated by scheme 1 lead to the formation of pores on the fiber surface, increases the number of reactive sites and allowing better wetting property between the matrix and fiber.The difference between the value of moisture content obtained from both the UTWF and TTWF is (0.02%).

Fiber	Moisture Content	Temperature (^{O}c)
UTWF	0.14±0.01	105
TTWF	0.12±0.01	105

Table 2: The moisture content for both UTWF and TTWF at a temperature of 105°C

3.2.2. Determination of Water Uptake

The water absorption in natural fiber thermoplastic composite is mainly due to the present of lumens, fine spores and hydrogen bonding sites in the natural fiber, the gaps and flaws at the interfaces and the micro cracks in the matrix that occurred during the fabrication process (stokke and Gardner 2003). The water absorption of different composites prepared using (2g, 3g, and 4g)) both the UTWF and TTWF as a function of time after 2h, 4h, and 24h immersion in water are presented in Figs 2, 3, and 4

All the composite show an increased in water uptake both the UTWF and TTWF composite but the amount of water uptake varies among the composite. From the result, UTWF composites showed a high uptake of water after 2h immersion than the TTWF, which may be attributed to the high content of lignocelluloses present in the UTWF composites as observed only for 2g and 3g. This can be seen in the Figs 2 and 3.

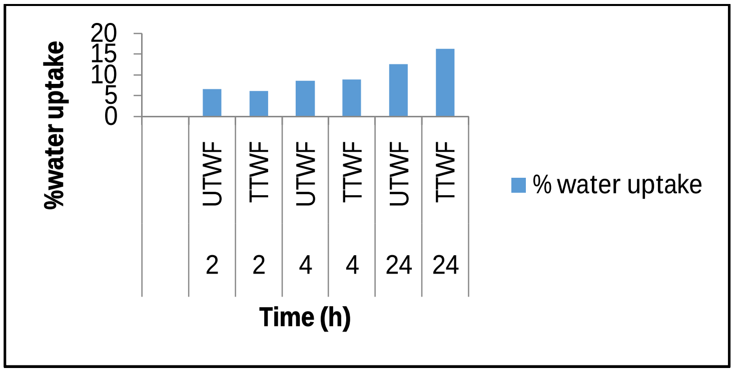

Figure 2: Show change in %water uptake of 2g for both UTWF and TTWF composite

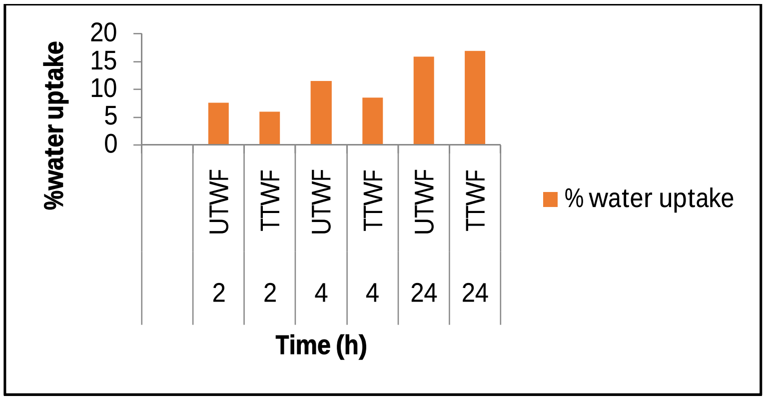

Figure 3: Show change in %water uptake of 3g for both UTWF and TTWFcomposite

But 4g composite of TTWF absorbed more water than 4g UTWF composite, this is due to low moisture content of TTWF and high moisture content of UTWF as shown in Figure 4.

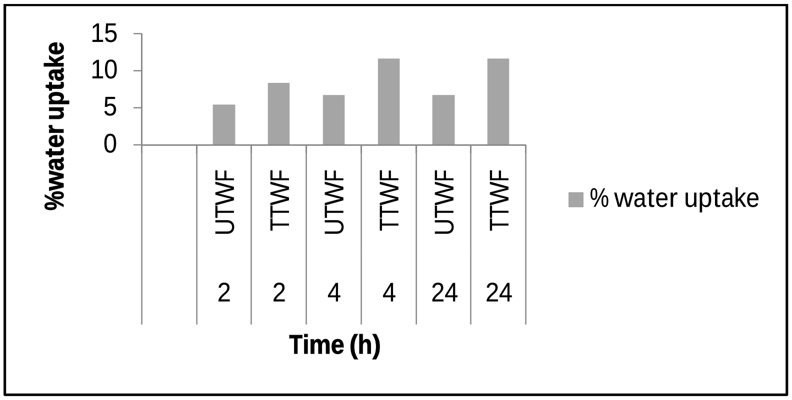

Figure 4: Show change in %water uptake of 4g for both UTWF and TTWF composite

However, as the time increases from 4h to 24 the water uptake tend to decreases for UTWF and increases for TTWF. This is because of reduced pore space and low accessible hydrophilic hydroxyl groups within the UTWF. The increased in water uptake for TTWF is as a result of high compatibility of TTWF with the polymer and moreover, because the matrix is polar, which tend to allow the water to penetrate into the composite (i.e. high diffusion coefficient) gradually as seen on the Figs 2, 3 and 4. These differences can attribute to the anatomical properties of the UTWF relative to the TTWF composites. The packing arrangement of fibers and chemical treatment of the TTWF reduces fiber diameter, thereby increasing the aspect ratio, which leads to development of a rough surface topography that result in better fiber/matrix interface adhesion and an increase in properties (Kalia *et al*., 2009) than the UTWF composite. This make TTWF to absorbed more water to UTWF as reported by (Var and Oktem, 1999) that hardwood absorb more water than softwood, because the cell cavities are larger in hardwoods, the cell wall are thinner and more permeable, and the openings of the pits are larger than in softwoods.

In addition, TTWF composite and TMU show gradual increased in water uptake as the immersion time increases from (2h, 4h, and 24h) as shown in the Figs 2, 3 and 4, while for TMU can seen in Fig 5

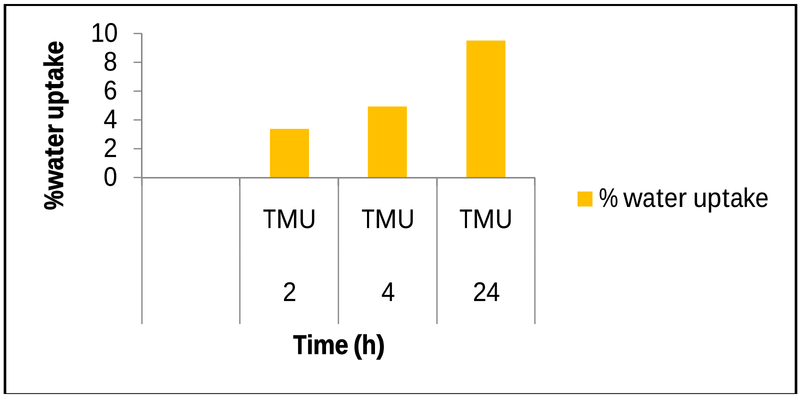

Figure 5: Show change in % water uptake for TMU with zero fiber

Generally, it can be seen that the 3g TTWF composite absorbed more water than the 2g TTWF and 4g TTWF composites. This may be due to the high compatibility of the 3g TTWF with the matrix. From all the result obtained for both UTWF and TTWF, TMU (with zero fiber) show lower water uptake through out the immersion time (2h, 4h, and 24). This may be due to stiffness and rigidity nature of the TMU which is incompatible with the hydrophilic water as can be seen in Fig 6 Fig 6 on the other which shows the combination of all the composite prepared from both UTWF and TTWF using (2g, 3g, and 4g) fiber as well as TMU.

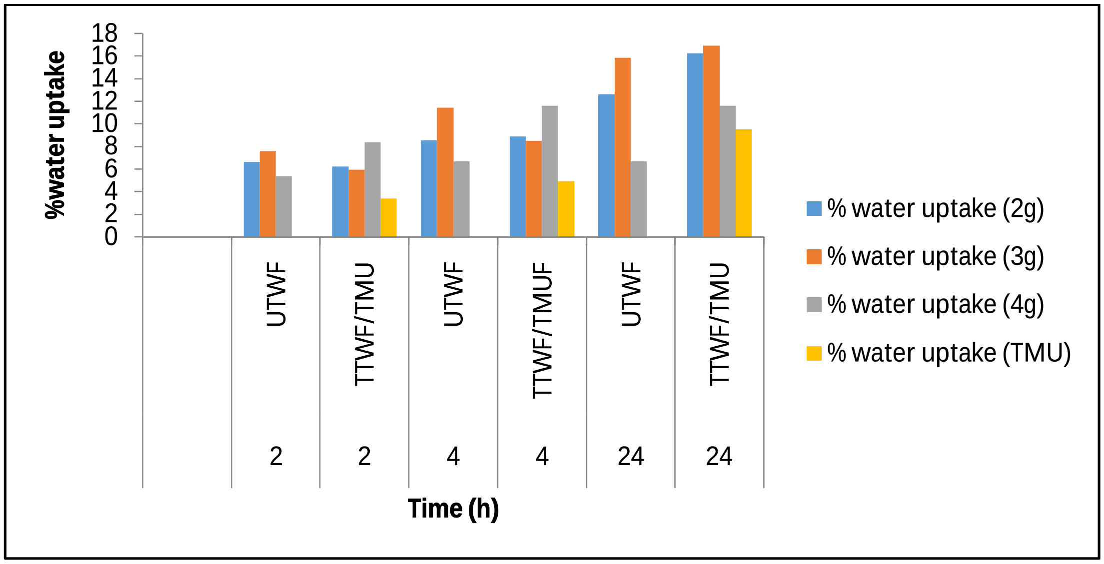

Figure 6: Show change in %water uptake of (2g, 3g and 4g) both UTWF, TTWF and TMU Change

Hydrophilic nature of fibre surface due to the presence of pendant hydroxyl and polar groups in various constituents, lead to poor adhesion between fibres and hydrophobic matrix polymers (John *et al*., 2008, Kalia *et al*., 2009). Furthermore, moisture content decrease significantly fibre's mechanical properties. That is why; cellulose and hemi cellulose are mostly responsible for high water absorption of natural fibres since they contain numerous accessible hydroxyl groups as in case of UTWF. It can also be seen that water absorption values of composites from 2g and 4g TTWF after 4h and 24h immersion in water were higher than those of composites from 2g and 4g UTWF. The high moisture content in the UTWF may be attributed to the higher cellulose and pentosans contents respectively.

3.2.3. Determination of Water Uptake at Different Temperatures (30°C,40°C, 50°C, 60°C and 70°C) and Immersion for 24h

Statistical analysis indicated that temperature had significant influence on water absorption of the composites ($P<0.05$). Fig 7 show the water absorption of UTWF, TTWF composites and TMU with zero fiber as a function of temperature after 24h immersion in water. The diffusion coefficient is the most important parameter for water absorption as it shows the ability of solvent molecules to penetrate inside the composite structure. It had been shown that Water absorption of the composite increases with temperature. Temperature activates the diffusion process. The high absorption of water may be due to the micro cracks develop on the surface and inside the material and/or natural fiber swelling due to moisture, as well as the resulting fiber debonding from the matrix due to the moisture and high temperature environment (Penthapulakkal and sain 2007). For 2g UTWF composite show higher water uptake than that of 2g TTWF composite after immersion in water for 24h. The high water uptake of 2g UTWF composite may be due to the micro cracks develop on the surface of the composite as a result of thermal degradation than the 2g TTWF composite. This can be seen in the Fig 7.

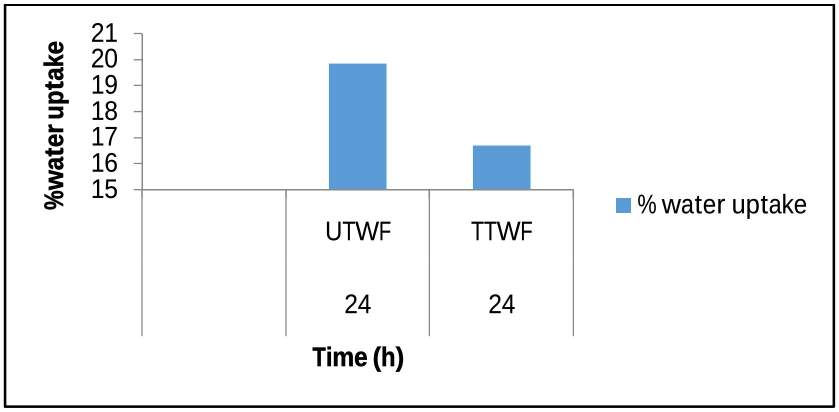

Figure 7: Show change in %water uptake of 2g for UTWF and TTWF composite after oven at temperature (30°C, 40°C, 50°C, 60°C, and 70°C) for 24h

For 3g UTWF composite show higher water uptake than that of 3g TTWF composite after immersion in water for 24h.The higher water uptake of 3g UTWF composite was due to the micro cracks developed on the surface of the composite as observed in the case of 2g UTWF composite above. This is as a result of thermal degradation observed in 3g UTWF composite than the 3g TTWF composite, meaning that that TTWF has a lower thermal degradation than UTWF. This can be seen in the Fig 8

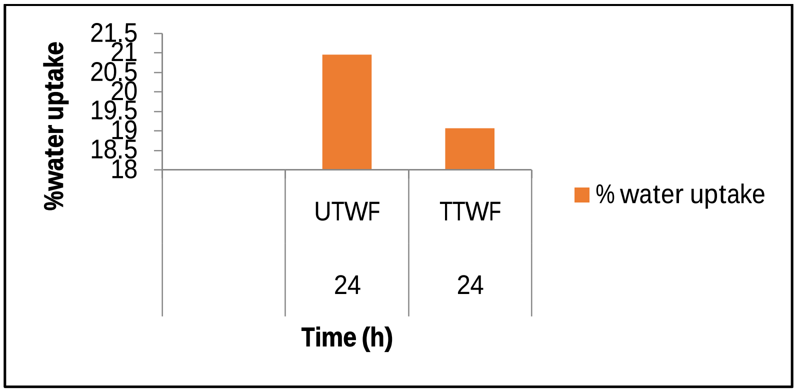

Figure 8: Show change in %water uptake of 3g for UTWF and TTWF composite after oven at temperature (30^oC, 40^oC, 50^oC, 60^oC, and 70^oC) for 24h

For 4g UTWF composite show low water uptake than that of 4g TTWF composite after immersion in water for 24h, unlike 2g and 3g UTWF and TTWF composite, only UTWF composite that show higher water uptake. The low water uptake of 4g UTWF composite may also be due to less micro cracks develop on it surface as a result of thermal degradation than the 4g TTWF composite. Another factor that is responsible for the low water uptake of 4g UTWF composite than 4g TTWF composite may be due to fiber content within the polymer matrix even though same weight of fiber was used during the fabrication of the composite. This is because the thickness observed in TTWF was 10mm higher that of UTWF, this perhaps make the matrix to be more exposed to degradation due to increase in the thickness of the TTWF than UTWF and hence packing arrangement (i.e. accommodation) within the matrix becomes problematic. This increased in thickness was also observed in both 2g and 3g TTWF than the UTWF, but in the case of 2g and 3g TTWF the matrix was able accommodate them. This can be seen n the Fig 9.

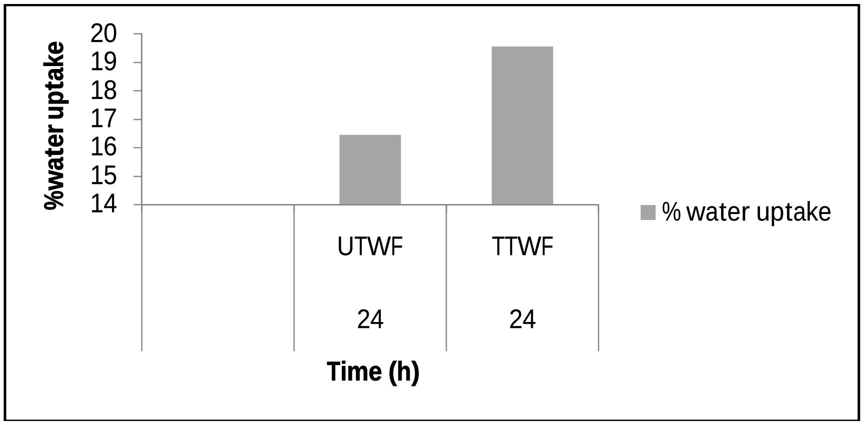

Figure 9: Show change in %water uptake of 4g for UTWF and TTWF composite after oven at temperature (30^oC, 40^oC, 50^oC, 60^oC, and 70^oC) for 24h

For the TMU with zero fiber show the lowest water uptake than all the composite prepared either from UTWF or TTWF composite, this is because of the hydrophobic nature of TMU which is incompatible with the hydrophilic water. This can be seen in the Fig 10.

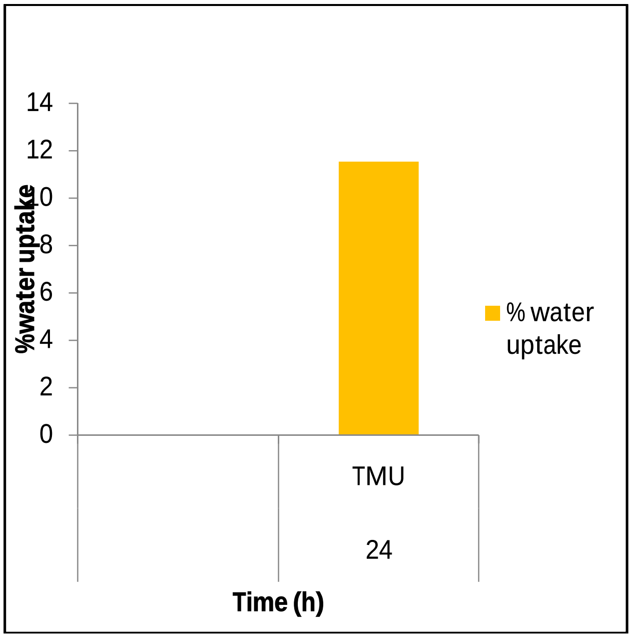

Figure 10: Show change in %water uptake for TMU after oven at temperature (30°C, 40°C, 50°C, 60°C, and 70°C) for 24h

From all the results obtained at different temperatures, the TTWF composites showed lower water absorption compared to the UTWF composite with the exception of 4g. The extent of water absorption of TTWF composite was about 3.15%, and 1.89% less than that of UTWF (2g and 3g) fiber composites respectively for 24h immersion. 4g TTWF composite show higher water uptake than UTWF composite as explained above on Fig 5.The composite prepared from both the UTWF and TTWF using (2g, 3g, and 4g) as well as TMU with zero fiber at different temperature is been summarized as seen in Fig 11.

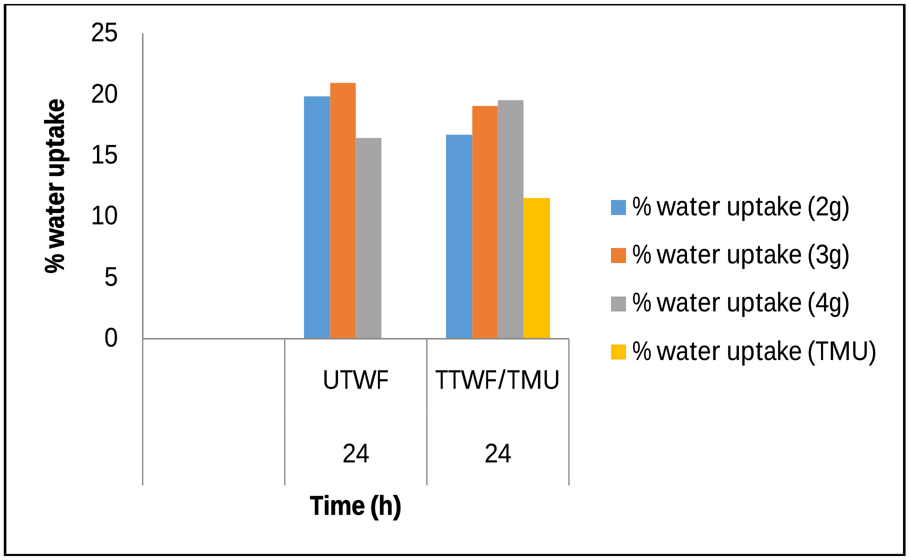

Figure 11: Show change in %water uptake for UTWF and TTWF composite using (2g, 3g, and 4g) as well as TMU with zero fiber at temperature (30°C, 40°C, 50°C, 60°C, and 70°C) for 24h.

The Mercerization process done on the tea waste leads to fibrillation which causes the breaking down of the composite fibre bundle into smaller fibres. Mercerization reduces fibre diameter, thereby increases the aspect ratio which leads to the development of a rough surface topography that results in better fibre/ matrix interface adhesion and an increase in mechanical properties (Kalia *et al.*, 2009). Moreover, mercerization increases the number of possible reactive sites, allows better fibre wetting and gets an effect on the chemical composition of the tea waste fibres, degree of polymerization and molecular orientation of the cellulose crystallites due to cementing substances like lignin and hemicelluloses which were removed during the mercerization process done on the tea waste fiber and can covalently bond to the TMU matrix of the fiber surface (Sanadi *et al.*, 2007).

The decrease of the water absorption therefore may be attributed to the occupation of free hydroxyl groups. The most cost effective benefit of improving moisture resistance along with the mechanical performance of natural fiber thermoplastic composites has been

found with coupling agents which play an important role in improving compatibility and adhesion between poplar natural fiber and non-poplar polymeric matrices. Bridges are formed with chemical bonds between the fiber and the matrix (Lu *et al.*, 2000).
At all temperatures, 4g TTWF composites showered the lowest water absorption, whereas 3g UTWF exhibited the highest water absorption was obtained with 24h immersion time as seen on Fig 4. The water absorption of the TMU was lower than those obtained from both UTWF and TTWF throughout the immersion time as can seen in Fig 4.
It can be deduced that water absorption of the composite increases with temperature, since temperature activates the diffusion process. From the above figures, UTWF, represent untreated tea waste fiber and TTWF represent treated tea waste fiber while TMU represent trimethylol urea.

4. Conclusion

Tea waste fiber reinforced trimethylol urea composites was successfully synthesized. A lot of research has been done on natural fiber reinforced polymer composites, but research on tea waste fiber polymer composites is very rare. Against this background, the present work has been undertaken, with an objective to explore the potential of the above said fiber polymer composites in which the weight loss was studied at different temperatures and time as well as water uptake determination of different fiber weights (2g, 3g, and 4g) used in producing the composites and also TMU with zero fiber. Proximate analysis, such as moisture content and ash content was determined as well for both the UTWF and TTWF. The synthesized composite material can be use to make indoor furniture's like table and the temperature of exposure should not exceed 40^{o}C, even though other parameters unique to the composite are yet to be characterized.

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