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European Journal of Advances in Engineering and Technology, 2023, 10(7):11-19

Research Article ISSN: 2394 - 658X

Preparation and Characterization of Cotton Linter Cellulose Reinforced Urea Formaldehyde Resin Polymer Composites

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ABSTRACT

Natural resources in the field of fibre reinforced polymer composites with their new range of applications illustrate an important basis in order to meet the ecological purpose of creating eco-friendly materials. Because of their low density, light weight, low price, ease of processing and good mechanical performance; cotton linter, a garbage product of textile/garment industries and cotton mills may offer a real alternative to the reinforcing fillers. A study on the preparation of polymeric composite materials using raw cotton linter cellulose (CLC), chemically treated CLC such as bleached, alkylated & acetylated (microcrystalline cellulose, MCC) as reinforcing materials with urea formaldehyde (UF) resin as binder has been made via solution casting method. Evaluation of the prepared samples (raw CLC-UF, alkylated CLC-UF, bleached CLC-UF and acetylated MCC-UF composites) were carried out by means of scanning electron microscope (SEM), water uptake percentage and biodegradability tests. In case of morphological behavior, morphological results neatly showed that when UF resin is reinforced with the different cotton linter cellulose, morphological change taken place depending on different type of cotton linter cellulose fibre. The chemically treated cotton linter cellulose reinforced UF composites showed the gradual decreasing water uptake percentage as the order: alkylated CLC-UF composite < raw CLC-UF composite < bleached CLC-UF composite < acetylated MCC-UF composite, which supports a better fibre-matrix interaction. The biodegradability result showed the lesser biodegradability rate of bleached CLC-UF composite and acetylated MCC-UF composite comparing with other composites. The obtained results suggest that MCC can be a foremost substance for the reinforcement of high-performance polymer composites.

Key words: Cotton Linter, Microcrystalline Cellulose, Urea Formaldehyde Resin, Water Uptake Test, Biodegradability Test

INTRODUCTION

Bio-based industrial residues are increasing day by day. Due to increasing cost of their management the disposal of these residues is a big challenge imparting waste management. That is why, the use of bio-based industrial residues as industrial raw material is now an advanced trend among entrepreneurs [1]. Utilization of such residues in accordance with the environmental requirements has motivated many young researchers [2-3]. Cotton linters (CL) are such types of bio-based raw materials that have long been recognized as providing cellulose furnishes suitable for the production of superior cellulose derivatives. Raw cotton linters have been considered as an excellent source of high molecular weight cellulose for over 80 years. It is commonly known to us as linters which are short fiber residues left on the cottonseed after the longer staple fibers are removed by ginning [4]. It is an important source of cellulose collected from textile garbage by processing from cotton mills as well as factories directly. In every year, a lot of cotton linter is produced from textile mills and factories in Bangladesh. It is considered in the whole world as a valuable cellulosic raw material for paper manufacture, for the purpose of converting to cellulose derivatives and for regenerated fibers [5]. Long Zhao et al. in their study

two kinds of cotton linter-based adsorbents were synthesized by grafting dimethylaminoethyl methacrylate on cotton linter [6]. Cotton linter is a staple source MCC which is known as cellulose microcrystal or crystalline cellulose. It is generally produced from the acetylation of cellulosic fiber to remove the amorphous region of cellulose [7]. The MCC is porous, insoluble in water, possess high cellulose content percentage, high aspect ratio (>1000) and high crystallinity [8-9]. Now-a-days, producing cellulose microcrystals is an interesting use for cotton linter. The increasing interest and their unique properties led to as intensive research in the field of composite materials. On account of its small size, high surface area and aspect ratio, MCC is a potential reinforcing material with the benefits of being derived from renewable resources, biodegradable and biocompatible. Rizka Yulina et al. stated cotton wastes from cotton yarn spinning mills has been utilized as an alternative resource for the production of MCC, an important ingredient in food, cosmetic, pharmaceutical industries etc. [10]. Rahman et al. have been prepared MCC from cotton linter for the composite fabrication with UF resin [11]. Pizzi et al. [12] in their book explained that UF resins, beside their low cost, have many advantages such as good solubility in water, ease of usage under different curing conditions, quick curing reaction in the hot press, excellent thermal properties and resistance to abrasion. Paul et al. [13] addressed a comparative study of the PF and UF particleboards from wood waste for sustainable environment and concluded that the property of the particleboards is a function of the percentage composition of the binder (resin) and the filler (sawdust). Alonso et al. [14-15] stated that in the wood composite industry; UF and PF are the two most commonly used binders but the two major disadvantages of using formaldehyde-based resins are formaldehyde emission which is harmful to human health and petroleum for producing formaldehyde and it should be limited. Bledzki et al. [16-17] studied that the use of synthetic fibers as reinforcements like glass, Kevlar and carbon fibers are very expensive and natural fibers like abaca, areca, jute, cotton, sisal, coir etc. are used as reinforcements in polymer composites because of numerous advantages like flexibility during manufacturing, available in abundance, light in weight, cheaper, high strength to weight ratio, renewable, recyclable as well as biodegradable. Adhikari et al. [18-19] described that natural fibers are emerging and superior substitutes compared to glass and other inorganic fiber-based fillers for reinforcement in polymer composites from economic and ecological points of view. Sekhar et al. [20] narrated that the properties of a composite are greatly influenced by the properties of the fiber (filler) and the interfacial bonding of fiber and matrix. The chemical bonding plays a significant role in the bonding process between the matrix and fiber in a composite. Fu S.Y.et al. [21] stated that the particles size, particle-matrix interface adhesion and particle loading on composites have notable effects on the mechanical properties. Alomayri et al. [22] fabricated the cotton fiber (CF) reinforced polymer composite and the effect of water absorption on the mechanical properties of the composite is evaluated. Crop-residue based particleboards have also shown high water absorption and thickness swelling. This is associated with the increase in porosity of the crop residues particleboards which increases the water absorption and thickness swelling [23]. Alam et al. [24] in their research, proposed that three types of single layer particleboard i.e., branch-waste mixed particleboard, bamboo branch particleboard and bamboo wastes particleboard were manufactured with 15% UF resin. Physico-mechanical properties of the manufactured particleboards were evaluated and results showed that the physical properties i.e., density, moisture content, water absorption and thickness, swelling of bamboo branch-waste mixed particleboard were better than bamboo branch particle board and bamboo waste particleboard. Sellers et al. [25] stated that the demand for particleboards in the sectors of housing construction and furniture has continued to elevate. Therefore, the feasibility of using fast-growing trees and agricultural residues as well as textile wastage as raw materials for particleboard production has been explored by a number of researchers. UF is a commercial resin and cotton linter is inexpensive biodegradable filler which is regionally available. Thus, cotton linter/UF compounds may be of interests as an environmentally friendly choice of material currently used in many applications. The objective of this study is to isolate microcrystalline cellulose from cotton linter cellulose by combined methods i.e.; acetylation and ultrasonication. Various CLC (raw CLC, alkylated CLC & bleached CLC) and acelytated CLC (MCC) were cast off as filler to fabricate composites with UF resin of filler content ranging from 1 to 8 % depending on the weight by easy processing conditions and cheap machinery. Morphological properties of different CLC-UF and MCC-UF composites were investigated by means of SEM as well as the physical properties which were characterized by water uptake and biodegradability tests. This study reallocates the use of cotton linter cellulose (raw, bleached & alkylated) and MCC as filler for CLC/MCC-UF composites. According to the forward presented results, these composites have improved surface morphology and physical properties,

costing less than other composites. It is also worth mentioning that the use of cotton linter cellulose to produce valuable composite products is an income source for the poor population and it also reduces the environmental pollution of garment/textile regions in Bangladesh.

MATERIALS AND METHODS

The textile garbage was collected from garment factories at Dhaka district, Bangladesh. All chemicals used in this study were analytical reagent grade purchased from Sigma Aldrich.

Preparation of Cotton Linter Cellulose

Processing of textile garbage into cotton linter consists of three steps; collection of textile garbage, sorting and ginning. At first, textile garbage is collected from garment factories, textile mills and dumping yards. After collection of textile garbage the solid particles and any other particles without fabrics part are separated from it. Finally, the fabric part was ginning to obtain cotton linter. The cleaned long fibers and cleaned short fibers are separately collected by collector [26]. The removal of impurities such as dirty materials and gummy substances from textile cotton linter is called scouring and it was carried out by the use of surface active agent like soda and detergents. Cotton linter fiber was coursed in a solution containing 6.5 gm of Jet powder and 3.5 gm of soda per liter at 70-75˚C for 30 minutes in a large beaker. The ratio of the CLC to solution was 1:50. After scouring, the CL was thoroughly washed with distilled water and dried in the open air and finally stored in desiccator.

Preparation of Alkylated Cellulose and Bleached Cellulose from CL

Alkali treatment of scoured CL was carried out by using 17.5 % NaOH solution. The CL was coursed in 17.5 % NaOH solution at 40˚C for 4 hours in a large beaker by heating mantle. The ratio of the fiber to solution was 1:50. After 4 hours, the CL was thoroughly washed with 2.0 % glacial acetic acid three times, then washed with distilled water and dried in the open air and finally stored in desiccator.

Bleaching of mercerized CL was carried out by using 0.7 % $NaClO_2$ solution. Mercerized CL was poured in 0.7 % $NaClO_2$ solution at 90˚C for 90 minutes in a large beaker by heating mantle. The ratio of the CL to solution was 1:50 and the pH of the solution 3.99 was adjusted by buffer solution (acetic acid and sodium acetate buffer). After 90 minutes, the fiber was thoroughly washed with 0.2 % sodium metabisulphite solution, then washed with distilled water and dried in the open air and finally stored in desiccator.

Preparation of MCC from CL

The preparation method of MCC was reported in the previous paper [27]. Several steps have been carried out to obtain MCC from cotton linter. In this case 10 gm of each bleached cotton linter was suspended in a solution of 12 N Sulfuric acids. The fibre-liquor ratio was maintained at 1:50 (w/v). The suspension was then placed on a magnetic stirrer and stirred up to 6 hours by a magnetic bar. After hydrolysis, the white powder like MCC was formed which was filtered and washed thoroughly with distilled water. The obtained MCC was kept in acetone and sonicated for 12 hours in an ultrasonic bath.

Fabrication of Different CLC-UF and MCC-UF Composites

Technical grade urea and formaldehyde (F/U ratio 2:1) were taken in a pyrex beaker. The mixture was stirred at 40˚C with a hot plate magnetic stirrer up to all the urea goes to solution. The pH was adjusted to 8.2-8.3 by adding 20 % sodium hydroxide solution. Then the solution was heated at 80˚C for 1.5 hours. The pH of solution was again adjusted into 4.5 by adding dilute acetic acid. To enrich F/U ratio 1:1, excess urea was added into the solution and temperature was maintained at 80˚C for 1 hour. The viscose solution of UF resin prepolymer was formed. The predetermined amount of different CLC or MCC and other ingredients such as polyvinyl alcohol (1.5 %), magnesium stearate (1 %) and hexamine (1.5 %) were added into UF prepolymer maintaining the temperature 80˚C. After 1.5 hours, the semi-solid CLC-UF or MCC-UF composites were formed. The composites were casted onto bakelite mould and then subjected to ageing in an electric oven at 40˚C for 4 hours. Then the solid composites were being cured at room temperature for 7 days.

CHARACTERIZATION TECHNIQUES

SEM Test

Scanning electron microscope photographs of virgin UF resin, various CLC-UF and MCC-UF composites were captured using JEOL JSM-7600F, Japan. In this case, the samples were coated with gold using the sputtering technique and the images with 1500 and 2000× magnifications were taken for each sample.

Water Uptake Test

The composite specimens were used for water absorption test immersing in water in a 1 litre beaker at room temperature and continued for longer period of time to reach at equilibrium. At regular interval, the specimens were taken out from water and wiped with filter paper to remove surface water and weighed with digital scale (DHAUS Analytical plus). The samples were re-immersed in water to permit the continuation of sorption until saturation limit was reached up to 24 hours. The weighing was done within 30 seconds in order to avoid the error due to evaporation. The percentage of water content (M_t) was determined using the following equation [28]:

$$M_t\,(\%) = \frac{W_t - W_0}{W_0} \times 100$$

Where, W_t, weight of the sample at time t; W_o, initial weight of the sample

In all cases, a protective gel coat (araldite) was applied on the cut side to prevent penetration of water from it. Weight measurements were taken periodically up to 24 hours, initially with a periodicity of one measurement per 4 hours during the first 12 hours and later with a longer periodicity since the mass changes were not as large as during the initial hours. Three replicates of each specimen were used reporting the average of the measurements.

Biodegradability Test

All dried composite samples were weighted out and then placed them under 4 inches depth of the soil mixture containing bacteria. The mixture consisted of leaves, paper waste, cow manure, food waste, composting seeds, urea, wood waste and water [29]. Then the degradation of the samples were observed after the intervals of 20, 40 and 60 days. For this purpose, the samples were picked up from soil on fixed date and then washed with water. Then the samples were dried in an electric oven at 105°C for 10 minutes to remove water and then 60°C for 48 hours to remove moisture and then again weighted out.

The biodegradability percentages of the tested samples were calculated by the following formula:

$$\text{Biodegradation (\%), } B = \frac{W_i - W_d}{W_i} \times 100$$

Where, W_i, initial dry weight of the tested sample in gm; W_d, dry weight after degradation of the tested sample in gm.

RESULTS AND DISCUSSION

The surface morphology of various CLC-UF and MCC-UF composites (plain & fracture surfaces) prepared by reinforcing with 5 wt. % of different CLC and MCC with UF are observed using SEM as shown in Fig. 1(a) and 1(b). The surface areas of all types of composites are continuous and compact. But a few little flakes formed on the surface of the fiber layer are alkylated CLC-UF composite. Composites are prepared with chemically treated CLC. The surface of the composites changed noticeably, provoked by acid hydrolysis. The microroughness of the surface is developed on the surface of raw CLC-UF and alkylated CLC-UF composites. This surface structure is the base of hydrophobicity. Water penetrates into the fibre bundle and causes the breaking down of the composite fibre bundle into finer fibrils due to decrease in bundle coherence when subjected to flexural loads. It can also be observed that extensive fibre pulls-out and no evidence were found of resin adhering to the filler which are an indication of poor resin-filler adhesion. In contrast, prior to exposure to water, SEM micrographs showed almost no filler pull-out and undamaged filler bundle which were attached to the fibre surface of cotton linter. These observations are indicative of strong bond between the filler and the resin in bleached CLC-UF and MCC-UF composites as shown in Fig. 1(a-b). Bezerra et al. [30] observed that the SEM images of poly (ε-caprolactone) (PCL)/cotton linter (1wt. %) in where, a matrix/fibrils morphology could be seen clearly; upon fracture some fibrils remained attached to PCL matrix while others were detached from it. In general cotton linters fibrils were well dispersed in PCL matrix promoting the good properties of PCL compounds. SEM images of PCL/cotton nanolinter (5 wt%) showed a particulate structure embedded within PCL matrix which is verified, the acid hydrolysis to which raw cotton linter was subjected changed its shape from fibrils to particle. Particle aggregates were obtained probably due to the lower miscibility between PCL and cotton nanolinter. Fig. 1(a-b) showed the images of MCC-UF composites, a drastic structural change was verified which aws occurred by acid hydrolysis of CLC. Clearly, matrix-fibrils morphology could be seen; upon fracture some fibrils remained attached to UF matrix while others were detached from it. In general, CLC and MCC fibrils were well dispersed in UF matrix promoting the good properties of bleached CLC-UF and MCC-UF composites as mentioned above. SEM images of MCC-UF composites (5 wt. % MCC loading) showed a

homogeneous structure embedded within UF matrix which was verified, the acid hydrolysis to which raw CLC was subjected changed it shape from fibrils to particle as earlier described. Filler particle aggregates in case of raw CLC-UF and alkylated CLC-UF composites are not uniform due to the lower miscibility between binder UF and filler CLC; so, they aren't strong enough to compromise the mechanical performance and biodegradability properties. Therefore, acetylated MCC-UF composites have smooth surface, no micropore and fibre distribution is which well compared to other composites. The water uptake percentage of composite depends on the composition and processing history of the composite sample. The water uptake values increased for all CLC-UF and MCC-UF composite specimens as the CLC and MCC fibre content increased.

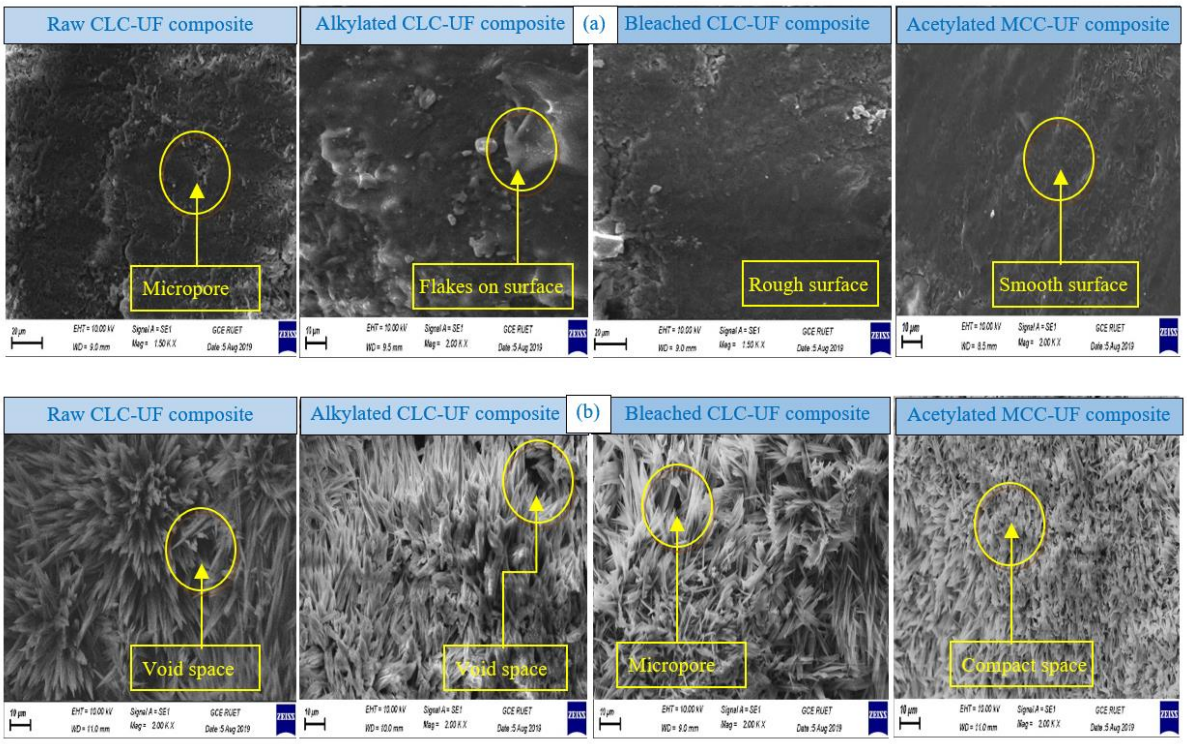

Fig. 1(a) SEM images of different CLC-UF and MCC-UF composites (Plain surface) and (b) SEM images of different CLC-UF and MCC-UF composites (Fracture surface)

From Fig. 2 it is evident that the water uptake percentage of UF resin composition: 99, 97, 95 and 92 % which exhibited better resistance to water than larger counterpart. However, 99% resin composition of CLC-UF and MCC-UF composites showed better resistance to water, with the UF composite having 97, 95 and 92 % respectively. It is also found that the water uptake percentage of MCC-UF composite is less than different CLC-UF composites. For 5 % CLC and MCC loading, the water uptake percentages of raw CLC-UF, alkylated CLC-UF, bleached CLC-UF and MCC-UF composites value is decreased except alkylated CLC-UF composites. This is an indication that the cohesive and binding forces of MCC-UF composite are stronger and more sustainable than other CLC-UF composites. Asha [31] showed that the percentage of water absorptivity increases as the percentage of rice husk in the particleboard increases. For 20% of rice husk, the water absorption was 2.63 % and for 25 % of rice husk, the water absorption is 6.67 %. Zhong et al. [32] showed that the water absorption of composite with 30 wt. % sisal fiber (SF) is only 0.98 wt. %. This can be attributed to the greater adhesion between the fiber and matrix, the low water absorption nature of the matrix. It is also observed that with the increase of SF content from 30 to 70 wt. %, the water absorption rockets. This might be due to the poor distribution of fibers in the composite which result in the formation of fiber lumps. These lumps require more forces to deform during hot pressing and as a result more stresses are built up in the composites [33]. It can be seen that the water absorption increases with the increase of fibre content in CLC-UF and MCC-UF composites.

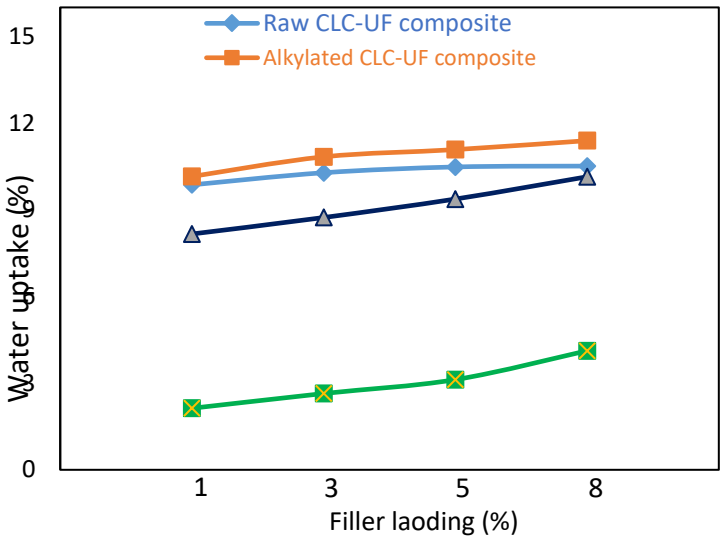

Fig. 2 Water uptake percentage of different CLC-UF and MCC-UF composites

The increase of water absorption is due to the hydrophilic nature of natural fibre and the greater interfacial area between the fibre and the matrix [34]. The water absorption of all specimens was high in the early stages of exposure, after which it slowed down and reached saturation level after prolonged time, following a Fickian diffusion process. The initial rate of water absorption and the maximum water uptake increase as the fibre loading increases in all natural fibre composite samples [34]. When natural fibre-reinforced composite is exposed to moisture the water uptake percentage increases due to the hydrophilic nature of fibre. For this reason cotton linter fibre absorbs water and becomes swell. As a result, micro-cracking of the CLC-UF composite occurs. Graupner et al. [35] stated that the high cellulose content in cotton fibre absorbs extra water that penetrates the interface through these micro-cracks, creating swelling which lead to composite failure. The more the composite cracks, the more capillarity and transport via micro-cracks become active. The water absorption capacity of composite materials can be changed by changing the filler as well as binder ration of composites. Tomalang et al. [36] also described that higher cellulose content of bamboo is mainly responsible for the water absorption of particleboard. So, the water uptake studies show composites with higher fibre and more void content display higher water uptake percentage. This is due to the hydrophilic nature of fibre and more void space in composites. The chemically treated CLC-UF and MCC-UF composites showed the decreasing of the water uptake percentage gradually by the order: Alkylated CLC-UF composite < raw CLC-UF composite < bleached CLC-UF composite < acetylated MCC-UF composites.

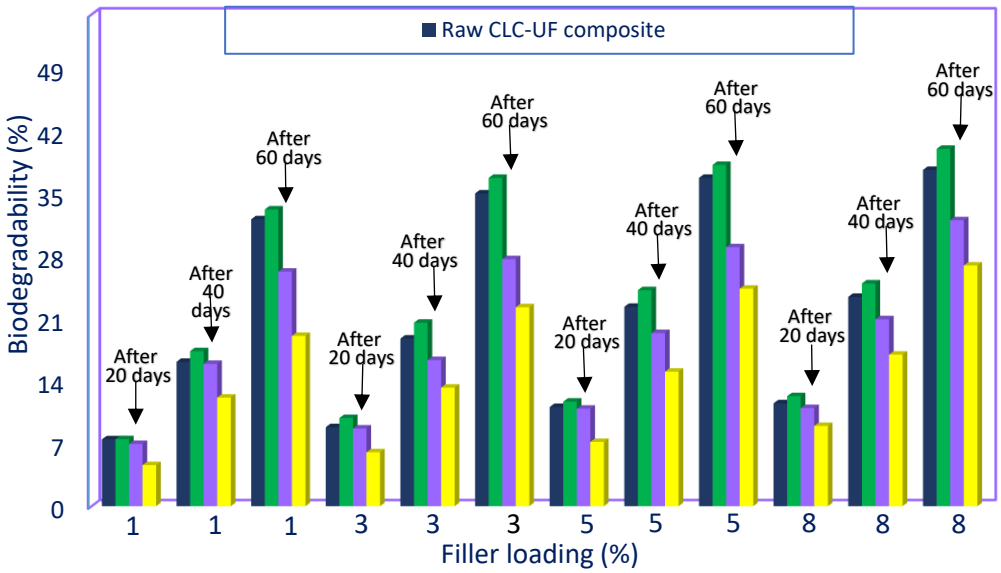

Fig. 3 Biodegradability percentage of different CLC-UF and MCC-UF composites

In polymeric substances biodegradation is like chemical degradation due to the action of naturally occurring microorganisms such as bacteria and fungi through enzymatic action into metabolic products of microorganisms. It is the chemical dissolution or breakdown of materials. It occurs because of enzymatic action and involves living micro and macro organisms. Many factors, such as chemical bonds, type of co-polymer, thickness, water uptake percentage and morphology can influence the rate of hydrolytic degradation in enzyme-mediated or non-enzyme-mediated conditions [37]. Fig. 3 showed the results of biodegradation from composting tests for different percentages of CLC-UF and MCC-UF composites. It is clear from this figure that in case of biodegradation test from composting, the amount is lost up to 7.52, 7.54, 7.00 and 4.62 % of its weight after the first 20 days in case of raw CLC-UF, alkylated CLC-UF, bleached CLC-UF and MCC-UF composites for 1 % fibre loading. Then the percentages of weight loss continued to increase over time until it reached up to 32.16, 33.25, 26.32 and 19.15 respectively after 60 days. During the biodegradation process, it is depicted in the Fig. 3 which represents a variation occurred in the weight loss of the composites.

Therefore, it is clear from these results that the weight loss of raw CLC-UF, bleached CLC-UF and MCC-UF composites are decreased gradually but in case of alkylated CLC-UF composite, the weight loss is little increased due to hydrophilic nature. It is also seen that the biodegradation rate increased with the increasing of CLC and MCC loading into the UF resin because the cellulose is easily attacked by microorganisms. Also, acetylated MCC-UF composite gave a lower weight loss (4.62 %) than other CLC-UF composites in case of 1% fibre loading. These results could be due probably to the high stability of chemical crystalline structure which is shown in Fig. 1(a-b). Shanta Pokhrel et al. [38] stated in their research, the water uptake of urea-formaldehyde and corn husk cellulose fiber composites increases with the increasing loading of cellulose which ultimately makes the composite more bio-degradable and eco-friendly. Lili Li et al. [39] showed that in soil and compost testing, which included multiple organisms and enzymes, the cotton fabric with softener had an accelerated degradation rate, while the cotton fabric with resin showed a relatively slow degradation rate. The results showed the lesser biodegradability rate of bleached CLC-UF and MCC-UF composites due to good interactions between molecular structures of resin and filler. The experimental results suggested that it is possible to make the less degradable composite with any natural fibre in order to improve its biodegradability properties and MCC could be utilized as less biodegradable and strong reinforcing filler in polymeric composite materials.

CONCLUSION

Natural fibre reinforced polymer composites are desirable and challenging materials to replace traditional materials and preface important environmental issues. The textile/garment by-product: cotton linter cellulose has less economic value that can be used to synthesize of valuable MCC to composite fabrication. Commercially available conventional wood based materials have some disadvantages. In this research paper, several physio-chemical properties such as surface morphology, water uptake properties and biodegradation behavior of composites produced from various CLC (raw, bleached & alkylated) and MCC (acetylated CLC) are largely influenced by the chemical treatment of CLC and change in the amount of UF resin which were used. From the SEM images it is confirmed that MCC reinforced UF composite has smooth surface, no micropore and fibre distribution which is well compared to other composites. Experimental data proved that the prepared different CLC reinforced UF and MCC reinforced UF composites don't have similar characteristics and quite different to other conventional composites. The water uptake percentage and biodegradability percentage of MCC reinforced UF composites are 3.12 and 24.42 whereas raw CLC reinforced UF composites are 10.48 and 36.77, bleached CLC reinforced UF composites are 9.37 and 29.04, alkylated CLC reinforced UF composites are 11.08 and 38.22 respectively for 5 % fibre loading in each case. So, the water uptake and biodegradability behavior of MCC reinforced UF composites are largely affected by the filler component MCC. Thus, it can be possible to inhibit the degradation of composites by controlling the binder content and improving the fiber properties. An outcome of this research will be a more economical and sustainable to particleboard which uses large quantities of softwood and hardwood (sawmill residue) and it is also expected to be readily accepted by the industry. It will also make a meaningful contribution to the sustainability of wood industry and the environment by reducing logging to produce flakes for softwood particleboard as well as environmental benefits of utilizing microcrystalline cellulose in particleboard.

Acknowledgements

We hereby acknowledge the assistance of Chairman, Department of Glass and Ceramic Engineering, Rajshahi University of Engineering and Technology (RUET), Bangladesh for instrumental measurements.

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