



## Fabrication and mechanical/thermal properties of composites from cotton linter and urea formaldehyde resin

Moshiur Rahman<sup>1</sup>, G M Arifuzzaman Khan<sup>1</sup>, S M Abdur Razzaque<sup>1</sup>, M Ahsanul Haque<sup>1</sup>, M A Gafur<sup>2</sup> & M Shamsul Alam<sup>1, a</sup>

<sup>1</sup>Polymer Research Laboratory, Department of Applied Chemistry and Chemical Engineering, Islamic University, Kushtia 7003, Bangladesh

<sup>2</sup>Pilot Plant and Process Development Centre, Bangladesh Council for Scientific and Industrial Research, Dhaka, Bangladesh

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The aim of this study is to prepare composite from commercial valueless cotton linter cellulose (CLC) of textile mills and garment industries, and urea formaldehyde (UF) resin. The alkali-treated cellulose (ATC), bleached cellulose (BLC) and microcrystalline cellulose (MCC) have been prepared from CLC. A novel fabrication method of CLC, ATC, BLC and MCC reinforced with urea formaldehyde resin has been developed. These composites are subsequently subjected to evaluation of their mechanical (tensile, flexural, hardness) and thermal (TGA, DTA, DTG) properties. The MCC-UF composites show the highest tensile strength (TS), flexural strength (FS) and Vickers micro hardness number (VHN) having the corresponding values 48.09 MPa, 34.05 MPa and 521.33 VHN respectively. The lowest mechanical values are found for CLC-UF composites (TS 32.96 MPa, FS 21.28 MPa and VHN 201.00). It is also revealed that the mechanical properties of the composites increase with the increase in fibre loading up to 5% and beyond this loading, these values are decreased. The morphological changes in flexural fractured surface are clearly observed by scanning electron microscopy measurement. The thermal stability of the composites is influenced by the filler content. The thermal stability of MCC-UF composite is found slightly higher than those of other composites.

**Keywords:** Composite, Cotton linters, Microcrystalline cellulose, Mechanical properties, Thermal properties, Urea formaldehyde resin

### 1 Introduction

Nowadays, the demand of sustainable eco-friendly materials has obtained much popularity due to rise in environmental awareness towards the people. Cellulose and its derivatives are suitable resources for the replacement of hazardous synthetic materials owing to their biodegradability, availability, cost-effectiveness and versatility in applications<sup>1</sup>. The natural fibres such as cotton, jute, hemp, flax, including agricultural biomass residue, etc. face the huge requisition of cellulose every year<sup>2,3</sup>. In addition, a good quantity of cellulose can also be achieved from recycled sources. Cotton linter is a renowned source of excellent quality cellulose. Huge amount of long-staple fibres have been discarded from ginning process<sup>4</sup> every year from textile mills and garment factories. So far, a little fraction of discarded cotton linter is used to prepare the value-added product, like paper, nanocellulose<sup>5,6</sup>, cellulose derivatives, etc<sup>7</sup>. The nano-structured cellulose (cellulose nano whisker,

micro fibrillated cellulose, microcrystalline cellulose, etc.) has now become popular for its usability in membrane and biomedical engineering materials. Microcrystalline cellulose (MCC) possesses porous crystalline structure formed by removing the amorphous part of cellulose by hydrolysis reaction<sup>8,9</sup>. Due to small size, high surface area, insolubility in water and high aspect ratio, MCC is promising reinforcing material of biocomposites. Composites from MCC reinforced with various polymer provide strength, rigidity and biodegradability, as already reported<sup>10-12</sup>.

Composite of cellulose fibre with thermoplastic and thermo setting resin has added a new dimension to people's comfortable life. Thermoplastics are widely used as matrix for cellulose fibre due to easy processing and high mechanical properties. Thermosetting plastics also hold good mechanical properties along with tremendous heat resistant properties. Formaldehyde based thermosetting resins, such as urea formaldehyde (UF) and phenol-formaldehyde (PF), are popular binders due to cheap, easy and convenient curing, excellent heat

<sup>a</sup> Corresponding author.  
E-mail: s.alam@acce.iu.ac.bd

and abrasion resistance<sup>13,14</sup>. Wood based UF resin composites, such as particle board, fibre board, oriented strand board, laminated veneer lumber, plywood, etc. show high mechanical and thermal properties<sup>15,16</sup>. The existence of strong chemical bonding between wood cellulose and resin might give high mechanical strength. Therefore, UF resin laminated composite is prominent for high load bearing application. Though UF is used in laminated composite manufacture but so far very little work has been found on short fibre impregnated UF composites preparation and characterization. The composite fabrication technique with thermosetting polymer especially for sticky UF resin is a big challenge for non-volatile, non-gluing and insoluble filler. To prepare high strength compact composite, proper wetting of filler in matrix and homogeneous filler distribution is needed. The conventional composite fabrication techniques, namely injection molding, compression molding, hand layout, pultrusion, etc. are not found efficient for UF composites<sup>15,16</sup>. Moreover, celluloses are unstable when subjected to high temperature composite fabrication with commercial UF resin powder. Therefore, the main objective of this study is to develop convenient method of composite fabrication with short cellulose fibre and UF resin.

Cellulose polysaccharide is constructed by anhydroglucose unit linked with  $\beta$ -1,4-glycosidic bonds. Due to the presence of hydroxyl groups in cellulose chain, microfibrils are attached together with strong intramolecular hydrogen bond as well as intermolecular van der Waals forces. For this reason, cellulose shows inertness when it combines with polymer matrix. Chemical pretreatment of cellulose is very effective to improve adhesion between cellulose and polymer<sup>17-19</sup>. In addition, filler size and filler loading on composite have noteworthy effect on the mechanical properties<sup>20-22</sup>. Richard *et al.*<sup>23</sup> showed in their study that the amount of fibre loading is crucial to produce a composite with high mechanical properties. Prasad *et al.*<sup>24</sup> showed that the increase in fibre loading as well as chemical treatment of the natural fibre increases the mechanical properties of the composites like tensile and flexural properties. A number of studies also showed that the presence of very high fibre content in composites decreases the mechanical strength<sup>25-27</sup>. This may happen due to higher fibre fraction, which cannot distribute homogeneously and make coagulation in composite, thereby decreasing the strength. The aspect ratio of

fibre has also great influence on the properties of cellulose reinforced polymer composites<sup>28-30</sup>. Composites of microcrystalline cellulose (MCC) have high aspect ratio which exhibit higher mechanical properties than regular cellulose<sup>10</sup>. In this study, cotton linter cellulose (CLC) has been treated with alkali as a bleaching agent to prepare MCC. The CLC, alkali treated cellulose (ATC), bleached cellulose (BLC) and MCC were mixed with urea-formaldehyde prepolymer before curing step of composite.

## 2 Materials and Methods

### 2.1 Materials

The cotton linters were collected from the local textile industries in Pabna, Bangladesh. The analytical grade chemicals and reagents were purchased from Sigma Aldrich.

### 2.2 Methods

#### 2.2.1 Preparation of ATC, BLC and MCC

The preparation methods of ATC, BLC and MCC have been reported in our previous studies<sup>5,31</sup>. Firstly, CLC was washed with distilled water to remove dirty materials and water soluble components. The washed cellulose sample was then treated with 17% sodium hydroxide solution at room temperature (30°C) for 3 h to prepare ATC. The fibre-to-liquor ratio was maintained at 1:50. The ATC was washed with water for minimum 10 times to remove the alkalinity. The ATC was dried in open air and also in an electric oven at 60°C temperature.

Then the ATC was bleached with 0.7% sodium chlorite solution for 90 min at 90°C by maintaining fibre-to-liquor ratio at 1:50. The pH of solution was adjusted at 4 by adding dilute acetic acid and acetate buffer (10 vol% of solution). As soon as the reaction was completed, BLC was washed with distilled water and treated by 2% sodium metabisulphite solution for 20 min to stop the oxidation. Finally, the BLC was washed and dried in an electric oven.

Ten gram of BLC was suspended in a solution of 12N sulfuric acid. The fibre-to-liquor ratio was maintained at 1:50 (w/v). The suspension was then placed on a magnetic stirrer and stirred upto 6 h by a magnetic bar. Later, this suspension become white powder (MCC), filtered and washed thoroughly by copious of distilled water. The MCC was kept in acetone and sonicated for 12 h in an ultrasonic bath.

### 2.2.2 Fabrication Method

Technical grade urea and formalin (F/U ratio 2:1) were taken in a beaker. The mixture was stirred at 40°C with a hot plate magnetic stirrer until all the urea goes to the solution. The pH of the solution was adjusted at 8.2–8.3 by adding 20% sodium hydroxide solution. The solution was then heated at 80°C for 1.5 h. Again, the pH of the solution was adjusted at 4.5 by adding dilute acetic acid. To enrich F/U ratio to 1:1, additional urea was added into the solution and the temperature was maintained at 80°C for 1 h. The viscous solution of UF resin pre-polymer is formed. The predetermined amount of filler (CLC, ATC, BLC and MCC), polyvinyl alcohol (1.5%), magnesium stearate (1%) and hexamine (1.5%) were added into UF pre-polymer, maintaining temperature at 80°C. After 1.5 h, the semi-solid composites are formed. The composite was cast onto bakelite mould and then subjected to ageing in an electric oven at 40°C for 4 h. Then the solid composites were cured at room temperature (30°C) for 7 days.

## 2.3 Characterization

### 2.3.1 Tensile Strength Test

The tensile properties of composites were determined by a universal testing machine (Hounsfield UTS 10KN) (H10KS). The tensile specimen was prepared according to the ASTM method<sup>32</sup>. To carry out the tensile strength, the single composite was bonded to a rectangular hole of 6 inch length. The gauge length for each specimen was 6 inches. The cross-head speed of the tensile testing machine was 0.8 mm/min. The average result of tensile strength was taken from 10 measurements. The tensile strength of the specimen was calculated by the following formula<sup>33</sup>:

$$\text{Tensile strength} = \frac{\text{Applied load}}{\text{Cross sectional area of load bearing area}}$$

### 2.3.2 Flexural Strength Test

For the determination of flexural properties of manufactured composites, the rectangular bar shaped molded composites were directly used. This method utilized the three-point stacking framework applied to the upheld beam. The flexural sample was set up as indicated by ASTM D790M, 3 point stacking. The sample measurement was 125×10×6-8 mm and support beam was 96 mm. The test speed was taken as 2.6 mm/min. The strength was determined for any

place of the load deflection through the following equation<sup>34</sup>:

$$\text{Flexural strength} = \frac{3FL}{2bd^2}$$

where  $F$  is the load at fracture point (N);  $L$ , the length of the support span (mm);  $b$ , the width of tested specimen (mm); and  $d$ , the thickness or depth of tested specimen (mm).

### 2.3.3 Vickers Microhardness Test

This technique is formulated based on the optical measurement system. It follows the ASTM E-384 procedure while using a specified light load by a diamond indenter to verify the hardness value. Commonly used loads are 5, 10, 30 and 50 kg, but the range can be enlarged if necessary<sup>35</sup>. The following formula was used for calculating the Vickers Hardness Number (VHN) of different tested samples:

$$\text{VHN} = 1854.4 \times \frac{P}{d^2}$$

where  $P$  is the applied force (load) in kg; and  $d$ , the diagonal length of the impression in  $\mu\text{m}$ .

### 2.3.4 Thermal Behavior (TGA, DTG, DTA) Analysis

Thermal behavior of various composite samples was analyzed by the TG/DTA 6300 analyzer with operated EXSTAR 6000 STATION, Seiko Instrument Inc., Japan. Twenty milligram sample is required for each analysis. The sample was heated up consistently at a pace of 0.01°C/min from 25°C to 600°C under a persistent progression of nitrogen at 50 mL/min. To get perfection, the analysis was done twice for each specimen.

### 2.3.5 Scanning Electron Microscopy Measurement

Scanning electron microscope (SEM) photographs of various specimens 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.

## 3 Results and Discussion

The tensile strength is the major tool to demonstrate the mechanical properties of synthesized composites. It doesn't rely on the size of the test sample. However, it depends on the techniques of sample preparation, surface defects and the temperature of the environmental and material tests. In this context, the variation in tensile properties with filler loadings of 1, 3, 5 and 8 wt% have been

evaluated for the composites prepared with CLC, ATC, BLC and MCC respectively. Therefore, the tensile strength was measured in terms of different fibre loadings (Fig. 1).

Figure 1(a) shows the tensile strength of all specimens which increases with the increase in fibre loading up to 5% and then decreases with the further increase in fibre loading (8%). The low fraction of fibre (beyond 5%) might be uniformly distributed into

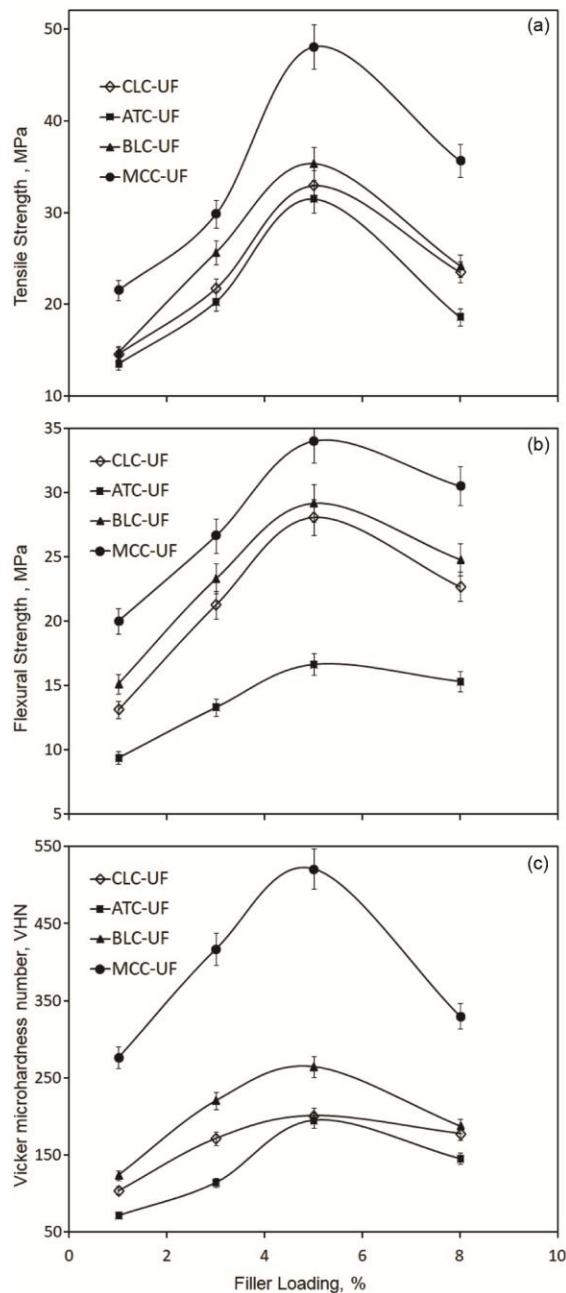


Fig. 1 — Mechanical Properties (a) tensile strength, (b) flexural strength and (c) Vickers micro-hardness number of CLC-UF, ATC-UF, BLC-UF, MCC-UF composites

matrix; as a result the composites with lower fibre loading show better fracture resistance against tensile load. Among all the samples, 5% fibre loaded composites show maximum tensile strength, such as 32.96 MPa for CLC-UF composite, 31.51 MPa for ATC-UF composite, 35.34 MPa for BLC-UF composite and 48.09 MPa for MCC-UF composite which is higher than the conventional UF-bonded particle board (7-15 MPa) and fibre board<sup>36,37</sup>. The variation in tensile strength of prepared composites with chemically treated fibre (ATC, BLC and MCC) can be elucidated based on the changes in fibre composition and structure. It is unknown that the lignin and hemicellulose present in cellulose fibre are eliminated by chemical treatment. Therefore, the relative percentage of cellulose in fibre is increased. Consequently, the fibre exhibits more hydroxyl groups on the surfaces. The presence of hydroxyl group is favorable for bond formation with matrix. Again, the fibre diameter is reduced due to defibrillation of fibre. As a result, it enhances the aspect ratio and stress transfer capacity followed by the improvement in tensile strength. Besides, 8% filler loaded composites show the lowest tensile strength. The cellulose could be aggregated into the composites at this ratio which reduces the strength of the composites. In other words, cellulose filler is not dispersed homogeneously in the matrix at the 8 wt% loading. Kamath *et al.*<sup>38</sup> showed that the bonding between cotton fibres with polymer binders is satisfactory when composites are made from good mixture of fibre and this increases the tensile strength. Among the composites, MCC-UF composite represents the highest tensile strength. The small size of MCC could be well-dispersed in UF matrix. Pereira *et al.*<sup>39</sup> also stated that the surface quality and internal bond strength are higher with small particles. Again, acid treatment of fibre during MCC preparation increases surface roughness which might provide better interlocking, i.e. improved fibre-matrix adhesion, thereby increasing the tensile strength. However, ATC-UF composites exhibit low tensile strength. Usually ATC fibre contains more -OH groups on its surface, which may lead to a stronger interfacial adhesion between fibre and matrix. However, it has also been found that the diameter and the aspect ratio of the ATC is smaller than that of CLC, resulting in a less entanglement of ATC fibre<sup>40</sup>. The less entangled ATC fibre could reduce the tensile strength of ATC-UF composites. Similar observation

is found for mercerized cellulose-polyethylene oxide composite in the previous studies<sup>41</sup>.

The change in morphology of the chemically treated fibre based composites is clearly observed in Fig. 2. In composite, the chemically treated fibre is found more compatible than in raw fibre with UF resin. It can be explained that the huge number of cellulose microfibrils exposed towards the surface in treated fibres, which causes more interaction of UF resin with fibres. From the SEM micrographs (Fig. 2) of CLC-UF, ATC-UF, BLC-UF and MCC-UF composites, it is clear that the MCC-UF composite surfaces are relatively smoother, highly compact with the absence of void space and micro-pore as compared to other CLC-UF composites.

Figure 1(b) shows the flexural strength of CLC-UF, ATC-UF, BLC-UF and MCC-UF composites with the variation in filler content (fibre loading %). It is observed that all types of composites show good resistance against bending stress. The cellulose fibre have ability to resist bending forces in composites, which were reported in previous studies<sup>42</sup>. In dry condition, the flexural strength increases when filler content is increased up to 5% and then it decreases due to poor interfacial bonding, resulting in void

spaces between the fibre and matrix (UF), generating a weak structure. At 5% fibre loading, fillers are uniformly dispersed within matrix, considering low fibre fractures and uniform stress transfer through the composites. It is also assumed that flexural strength of composites in all fibre loading are found greater for MCC-UF composites than the other composites. The flexural strength values of MCC-UF composites are found 20.02, 26.66, 34.04 and 30.53 MPa for 1, 3, 5 and 8 wt% MCC loading respectively. Srinivasa *et al.*<sup>43</sup> stated that the flexural strength could increase due to removal of outer fibre surface, with the increase in cellulose content and interfacial adhesion. Moreover, the MCC distribution with UF resin provides good flexural strength. Besides, ATC-UF composites are shown as the lowest flexural strength. That could be due to the hydrophilic nature of the composite samples, which affects the interfacial adhesion between fibre and matrix and creates fibre debonding, leading to a decrease in mechanical properties.

Figure 1(c) shows the Vickers Micro-hardness Number (VHN) of CLC-UF, ATC-UF, BLC-UF and MCC-UF composites. This technique can be endorsed as the effect of filler content in composites. The MCC-

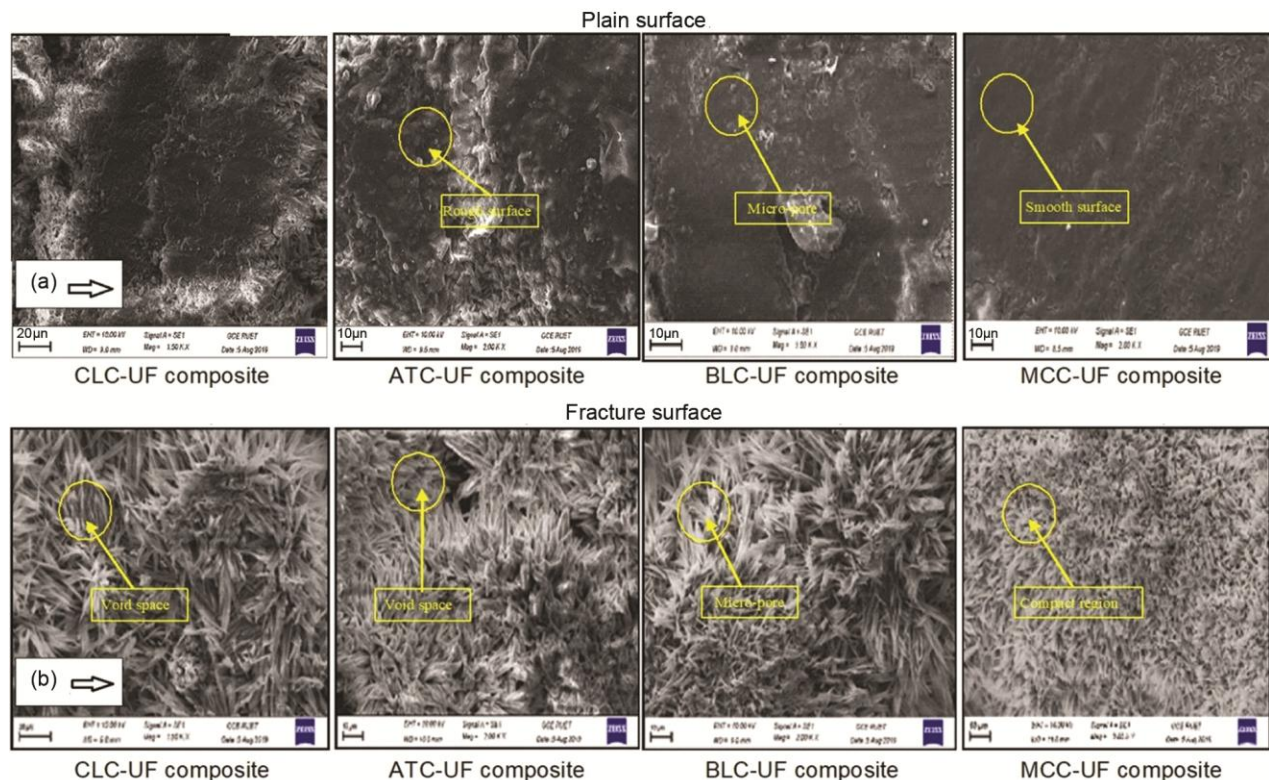


Fig. 2 — SEM images of (a) plain surfaces of CLC-UF, ATC-UF, BLC-UF and MCC-UF composites and (b) fracture surfaces of CLC-UF, ATC-UF, BLC-UF and MCC-UF composites

UF composites with 1, 3, 5 and 8 wt% filler loading display 96.33, 276.33, 417.33, 521.33 and 330.00 VHN respectively. It is evident that the VHN values increase with the increase in filler content in composites up to 5 wt% fibres loading<sup>44</sup>. In other studies, Pokhrel *et al.*<sup>45</sup> showed that the hardness property of composites increases upto 30% fibre loading. The hardness value decreases in all ATC-UF composites as compared to other composite samples. This happens due to the weak interface between the UF matrix and the ATC, caused by the water absorption due to hydrophilic nature. Dhakal *et al.*<sup>46</sup> reported that the hardness of fibre-reinforced composites decreases with the increase in water absorption caused by the hydrophilic nature of the fibres, which leads to build a weak fibre-matrix interface. In case of 8 wt% filler loading, VHN of CLC-UF, ATC-UF, BLC-UF and MCC-UF composites is lower than those of 1, 3 and 5 wt% filler loading. This might be due to presence of unbounded fibre in the composite, which losses the interface between UF matrix and CLC/MCC filler. This leads to the relaxing of the fibre-matrix adhesion, resulting in reduced composite properties, like hardness. Sharma *et al.*<sup>47</sup> shows that the hardness of UF resin-based composite decreases while impregnating various types of cellulosic fibres in that composite. In this case, the fibre-matrix interfacial bond can be weakening if additional unbounded fibre remains present in the composite. For this reason, the hardness of prepared MCC-UF composites is higher than those of other composites.

Thermal stability of CLC-UF, ATC-UF, BLC-UF and MCC-UF composites is investigated by thermogravimetric analysis (TGA, DTA and DTG) [Figs. 3(a-c)]. Figures 3(a)-(c) represent the TGA, DTG, and DTA curves respectively. Different CLC-UF and MCC-UF composites show similar behavior in TG plots; around 100°C is observed as the first stage weight loss due to water release; and the corresponding values are 5.2, 4.1, 3.8 and 5.2% for 5% filler i.e. CLC, ATC, BLC and MCC loaded composites respectively. In second and third stage, degradation is verified at around 320°C and 550°C respectively [Fig. 3(a)].

In Figure 3(b), DTG plots for CLC-UF, ATC-UF, BLC-UF and MCC-UF composites are indicated. The peak between 170°C and 390°C is associated with pyrolysis of cellulose, lignin and hemicelluloses with the corresponding weight loss of 2.6, 1.7, 1.3 and

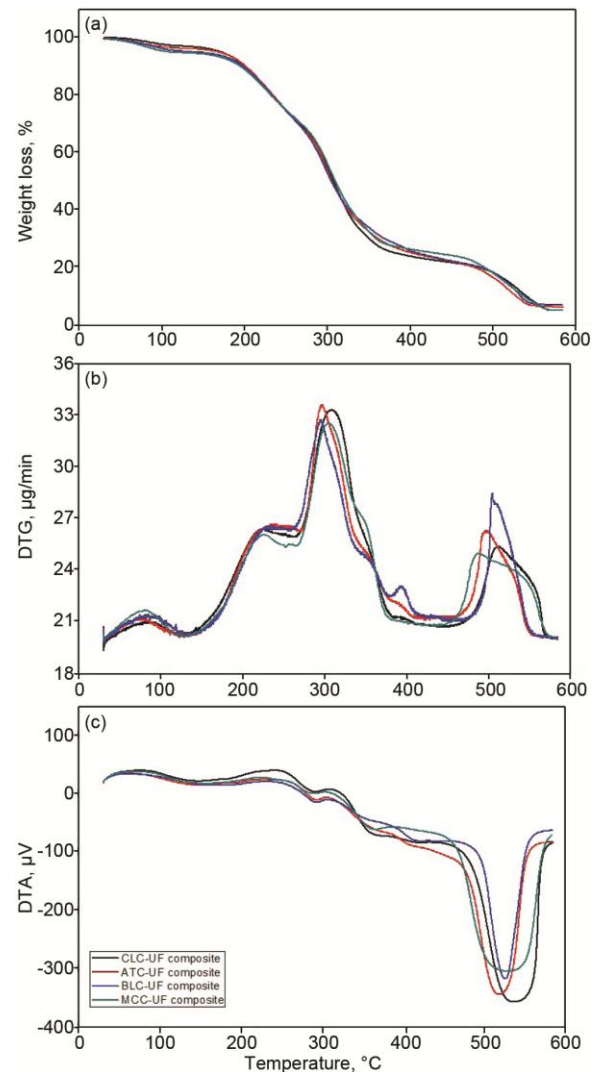


Fig. 3 — Images of (a) TGA curves, (b) DTG curves and (c) DTA curves of CLC-UF, ATC-UF, BLC-UF and MCC-UF composites

2.1% for CLC-UF, ATC-UF, BLC-UF and MCC-UF composites respectively. The third stage is generated due to the degradation of residuals taking place at around 600°C. From TG and DTG plots, it is verified that the acid hydrolysis does not change abruptly in the thermal stability of CLC-UF, ATC-UF, BLC-UF and MCC-UF composites. The residual char contents at the end of thermal analysis are 25.6, 19.8, 14.3 and 8.5% for CLC-UF, NaOH treated CLC-UF, BLC-UF and MCC-UF composites respectively (Table 1).

ATC-UF composite shows slightly different TG and DTG plots to that of CLC-UF composite with plots showing decomposition in one event. The weight loss started at around 300°C and the maximum degradation rate is observed at around 520°C in DTG plots. These results proclaim that the thermal stability

Table 1— TGA, DTG and DTA data of CLC-UF, ATC-UF, BLC-UF and MCC-UF composites

Sample	TGA			DTG		DTA	
	Temp. range °C	Weight loss, %	Residual char, %	Temp. range, °C	Rate of wt. loss, mg/min	Temp. range, °C	Peak types
CLC-UF composite	38.5-123.7	4.8		118.6	0.095	172.5	Endothermic
	123.7-342.6	1.9	25.6	313.4	3.242	322.7	Exothermic
	342.6-512.2	67.7		531.5		540.3	Endothermic
ATC-UF composite	33.6-119.3	5.2		117.9	0.078	167.4	Endothermic
	119.3-324.6	2.6	19.8	298.7	2.123	309.5	Exothermic
	324.6-516.2	72.4		511.7		512.6	Endothermic
BLC-UF composite	44.1-134.2	4.1		133.2	0.064	174.6	Endothermic
	134.2-343.6	1.7	14.3	320.1	1.663	315.8	Exothermic
	343.6-549.7	79.9		527.5		531.8	Endothermic
MCC-UF composite	36.4-142.7	3.9		141.7	0.160	178.3	Endothermic
	142.7-329.4	1.2	8.5	327.3	3.916	326.5	Exothermic
	329.4-566.3	86.4		578.6		542.1	Endothermic

of ATC-UF composite does not significantly change through chemical treatment. But TG plots of MCC-UF composites present decomposition in one stage, starting at around 330°C; this decomposition is visualized in DTG plots also as a peak with maximum height at around 556°C. MCC-UF composites present TG and DTG plots, which are quite dissimilar to that of CLC-UF, ATC-UF and BLC-UF composites. These results express that the thermal stability of MCC-UF composite is greater than those of other composites due to small size of filler (MCC), which is proved by the Fig. 2(b). Several researchers showed that the small particle size of filler had great effect on thermal stability of composites. Goodrich *et al.*<sup>48</sup> stated that the high thickness (2.0mm) bearing composites exhibit high thermal stability than the low thickness (0.6mm) composites. This happens because of small-sized fibres which can generate more surface orientation with the UF resin to develop the decomposing process. Alaei *et al.*<sup>49</sup> also showed that the small-sized dispersed phase displays good thermal behavior as compared to the big-sized counterpart of composites. The dispersion of particles in the composites is also an important factor which contributes to the enhancement of thermal stability. The fibre distribution in the MCC-UF composite is more homogeneous than other composites [Fig. 2(b)]. So, the thermal stability of the MCC-UF composite is higher than those of other prepared composites.

#### 4 Conclusion

This study states mechanical and thermal properties of CLC-UF, ATC-UF, BLC-UF and MCC-UF composites. The mechanical values of MCC-UF composites are found higher as compared to CLC-UF,

ATC-UF and BLC-UF composites. The composites show the highest mechanical strength at 5% filler loading. For filler loading of above 5%, mechanical strength has been found to be decreasing due to agglomeration of fibres. The SEM observations confirm that the MCC is well-dispersed in UF polymeric matrix. In case of thermal properties, MCC reinforced UF composites show the better thermal stability as compared to untreated CLC as well as ATC and BLC reinforced UF composites.

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