Zea Mays Husk Reinforced Epoxy Composites

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1 Introduction

Use of natural fillers as reinforcement in polymer matrices is encouraged nowadays in order to compensate the rising demand of synthetic polymers for making composite materials in various technical applications. The focus has been shifted from high performance engineering materials toward socio-economic and efficient utility-based products. Numerous natural agro-based lingo-cellulosic fibers such as kenaf, sisal, coir, jute, hemp, rice husk, wheat straw, bamboo, banana fiber, etc., have been used as alternative reinforcement with the polymer matrices [1–5]. Delignification of zea mays has been carried out in different ways by researchers for fiber extraction [6–10], but in current work partial delignification of ZMH is done and used as reinforcement in the epoxy matrix.

2 Materials and Methods

2.1 Materials

Araldite Epoxy Resin AW 106 and Hardener HV-953 IN, manufactured by Huntsman International (India) Pvt. Ltd. are used as matrix for making the composites. ZMH is procured from local market. NaOH is used in pallet form by sd-fine Chemicals Ltd. for the alkali treatment. Customized moulds are prepared for fabrication of composites from Central Institute of Hand Tools, Jalandhar, India.

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2.2 Alkali Pretreatment

The pretreatment process is optimized by using, 3 factors (alkali concentration, treatment temperature and time)- 3 level, Box Behnken design of experiment. ZMH is immersed in alkali concentrations (10, 15 and 20 g/l) at 1:40 MLR solution for given time (30, 60 and 90 min) and (30, 40 and 50 °C) temperature. After the alkali treatment ZMH are washed with 5% acetic acid solution to neutralize the samples followed by drying. Fabrication of Composites is done by hand lay-up method.

2.3 Characterization Techniques

The XRD is done using Empyrean diffractometer made by Melvern Panalytical. The diffraction angle (2θ) is taken in the range of 5°–60°. SEM analysis is carried out by JEOL (6510LV) at 500x magnification level. DMA tests are performed using DMA1 dynamic mechanical analyzer (Mettler Toledo) in 3 point bending mode.

3 Results and Discussion

3.1 Effect of Pretreatment on Tensile Strength of ZMH

The effect of the pretreatment on breaking strength of the ZMH is shown in Fig. 1. Maximum breaking strength of 35.5 kPa is obtained at 15 g/l of NaOH concentration, 30 min time and at 30 °C whereas least is around 10 kPa at 20 g/l of NaOH concentration, 60 min time and at 50 °C. The reduction in breaking strength at higher levels of NaOH concentration, time and temperature is due to enhanced delignification at elevated temperature, time and NaOH concentration [11]. However, reason for lowered breaking strength in case of 10/30/40 (16.2 kPa) as compared to at 20/30/40 (27 kPa) and within 15/60/40 may be attributed to the natural variation in the thickness of ZMH which is inevitable. Defibrillation is observed at elevated temperature and time, which resulted in the loss in weight of the ZMH after pretreatment. If concentration (20 g/l) and time (60 min) are kept constant, elevated temperature (from 30 to 50 °C) is playing a vital role in further increasing the weight loss by 22% [12]. The optimized pretreatment conditions are 30 °C temperature, 20 g/l NaOH concentration and 30 min time.

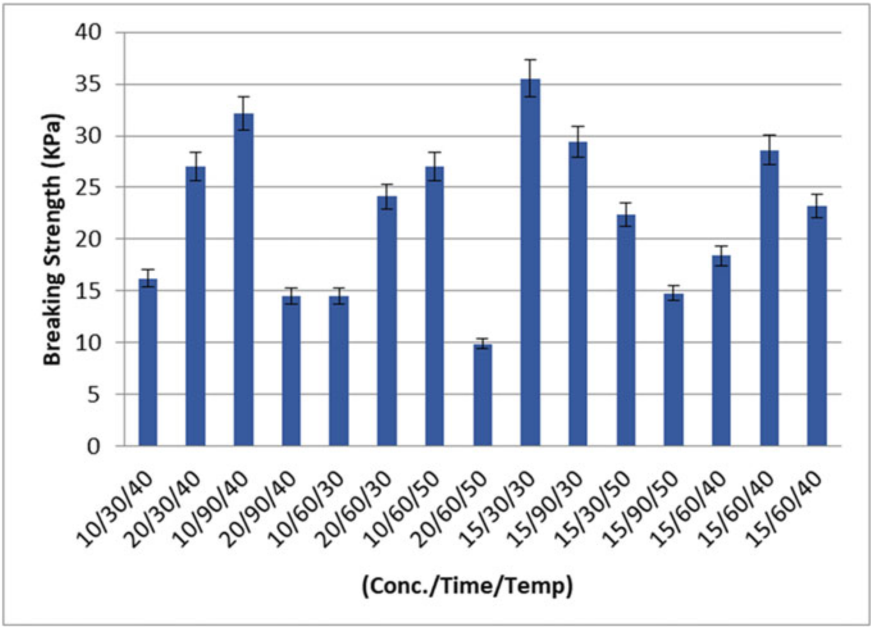


Fig. 1 Breaking strength of ZMH after pretreatment at different levels of concentration /Time/Temperature

3.2 *XRD Results*

Figure 2 presents the comparison of X-ray diffraction patterns of alkali treated and untreated ZMH exhibiting two main reflection peaks at 2θ = 16.42° and 21.87°. The different X-ray diffraction spectrum depends on proportion of two crystalline forms [13]. The highest peak at 2θ = 21.87° corresponds to the I_{200} plane represents the both crystalline and amorphous material. The lowest peak height at 2θ = 18.43° corresponds to the I_{AM} plane and represents only the amorphous part. The crystallinity of the treated sample is increased as compared to untreated one by 20.46% due to removal of lignin and partial hemicellulose.

3.3 *SEM Analysis*

SEM micrographs of pretreated and untreated samples are shown in Fig. 3. Array of numerous polygon type structures can be observed from Fig. 3a on the surface of untreated ZMH, while in Fig. 3b the ZMH after pretreatment appears somewhat different. There is shrinkage in the ridges (polygon shapes) and delignification of the film. Delignification is a result of the breakage of lignocellulosic bonding due to

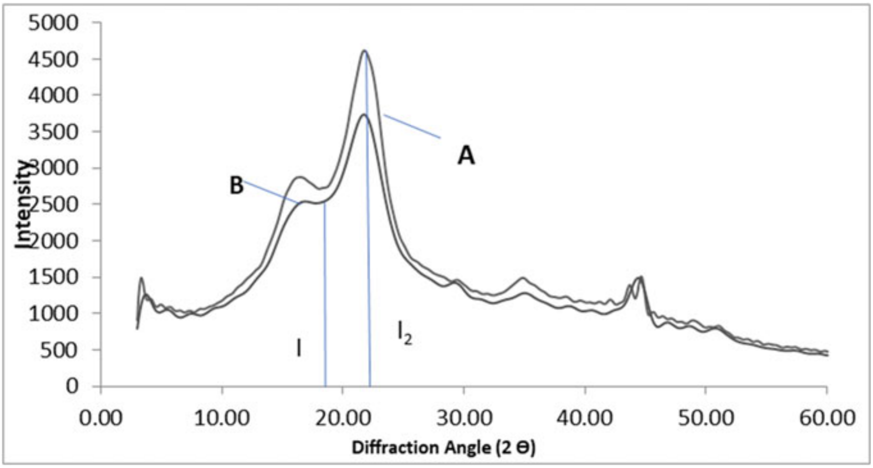
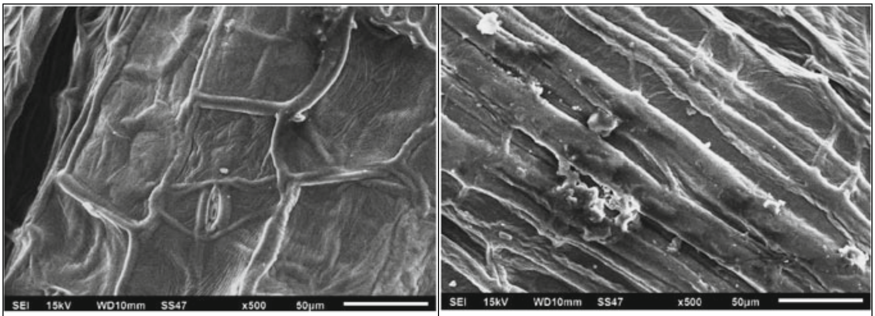


Fig. 2 XRD pattern of ZMH under varying treatment conditions viz. A. 20/30/30 B. Untreated



a. Untreated ZMH **b. Alkali Treated ZMH (20/30/30)**

Fig. 3 SEM images of untreated and pretreated CHF

pretreatment of ZMH with NaOH. Alkali treatment causes swelling which leads to an increase in the internal surface area because of which structural linkages between lignin and cellulose are separated leading to better fiber-matrix interphase [14, 15].

3.4 Dynamic Mechanical Analysis

Figure 4 show the DMA curve of storage modulus and temperature of ZMH reinforced epoxy composite and neat epoxy. The initial modulus of ZMH reinforced epoxy composite is 3.45 GPa which is around 42% more than neat epoxy. There is a marked decrease in storage modulus of ZMH reinforced epoxy composite from

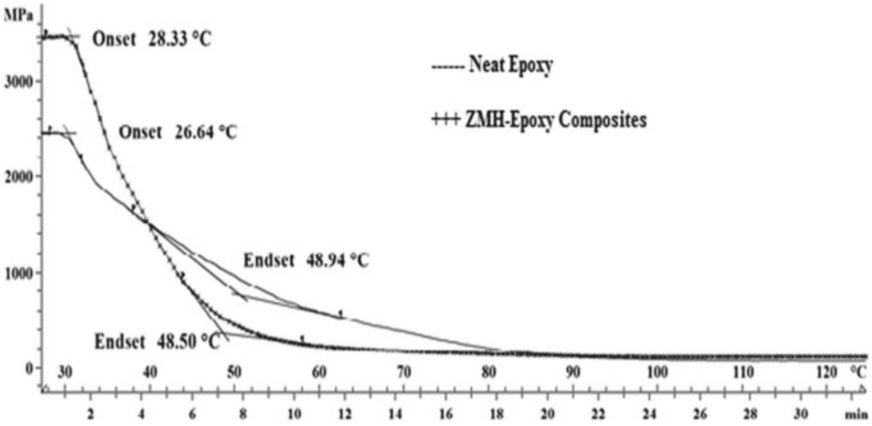


Fig. 4 Storage modulus of neat epoxy ZMH reinforced epoxy composite

3.45 GPa at 27 °C to 0.34 GPa at 48 °C, i.e. 90% decrease in storage modulus. This decrease is attributed to the softening of the polymer due to the increase in the chain mobility of the polymer matrix at high temperatures. Therefore the load bearing capacity is drastically reduced at elevated temperature [15]. Similar transition has been observed for neat epoxy but in this case, the decrease in storage modulus up to 90% has been delayed and achieved at around 75 °C [16].

4 Conclusion

ZMH can be used as reinforcement for polymer matrix. Pretreatment of ZMH is optimized (20/30/30) which resulted in surface modification of the ZMH and consequently enhanced the bonding between ZMH and epoxy. XRD spectra is governing the partial removal of lignin and hemicellulose because of which crystallinity of the ZMH is enhanced. DMA results have shown that storage modulus of the treated ZMH is 42% higher than untreated one which indicates that ZMH can be used for composites applications and could be explored further.

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