

# Zea Mays Husk Reinforced Epoxy Composites

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## **Abaract**

*This work is focused to explore the potential of zea mays husk (ZMH) as reinforcement in the epoxy composite. Most of work that had been carried out globally on zea mays stover is especially on zea mays husk and that too primarily for ethanol production and upto some extent as reinforcement for composite fabrication after fiber extraction. A novel effort has been made to use ZMH in film form for reinforcement.*

*Standard epoxy resin and hardener are used as matrix and pretreated ZMH as filler to form the laminate composites. The effect of alkali treatment conditions on ZMH is optimized in terms of ZMH and the respective composite properties. Characterization of ZMH is carried out by XRD, SEM and mechanical properties. ZMH laminate composites are also characterized with SEM and DMA. SEM images confirmed the partial delignification of ZMH after pretreatment. Fractured samples of composite reinforced with NaOH treated ZMH evidenced good interphase between matrix and filler as compared to neat epoxy. DMA test showed promising result for ZMH reinforced composites. Flexural modulus of ZMH reinforced composite is found to be 3.45 GPa as compared to 2.43 GPa for neat epoxy composites. Damping coefficient ( $\tan \delta$  value) of reinforced composite is higher than neat epoxy composites.*

**Keywords-** Lignocellulosic fibres, Epoxy, Laminate Composites, SEM, DMA

## **1. Introduction**

Use of natural fillers as reinforcement in polymer matrices is encouraged now a days in order to compensate the rising demand of synthetic polymers for making composite materials in various technical applications. There is a shift in trend from high performance engineering materials towards cost effective 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 investigated as alternative filler with the polymer matrices to prepare reinforced composites [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 (pallet form by sd-fine Chemicals Ltd.), Acetic Acid (sd-fine Chemicals Ltd.), Afra Silicone spray (Aerol Formulations Pvt. Ltd) is used for easy removal of the composites after curing from the mold. Customized moulds are prepared for fabrication of composites from Central Institute of Hand Tools, Jalandhar, India.

**2.2 Alkali Pretreatment-** For optimizing the pretreatment process, 3 level - 3 factor Box Behnken design of experiment is used. Alkali concentration, treatment temperature and time are considered as factors in the experiment. In all cases 1:40 MLR is maintained. 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 FTIR spectra of the treated and untreated ZMH are obtained using Agilent 630 FTIR spectrophotometer in the  $400\text{ cm}^{-1}$  to  $3900\text{ cm}^{-1}$  wave number range. SEM analysis is carried out by JEOL (6510LV). DMA tests are performed using DMA1 dynamic mechanical analyzer (Mettler Toled) in 3 point bending mode.

## 3. RESULTS & 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 Figure1. Maximum breaking strength of 35.5 KPa is obtained at 15 g/l of NaOH concentration, 30 minutes time and at  $30^{\circ}\text{C}$  whereas least is around 10 KPa at 20 g/l of NaOH concentration, 60 minutes time and at  $50^{\circ}\text{C}$ . 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 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 (20g/l) and time (60 min) are kept constant, elevated temperature (from  $30^{\circ}\text{C}$  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 minutes time.

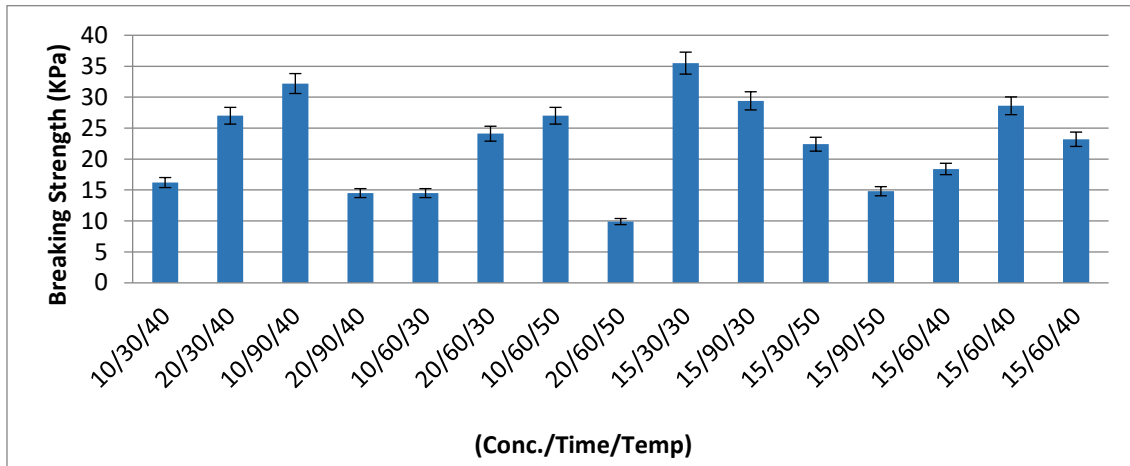


Figure1: Breaking Strength of ZMH after pretreatment at different levels of Concentration /Time/Temperature

**3.2 XRD Results-** Figure 2 presents the comparison of X-ray diffraction patterns of alkali treated and untreated ZMH exhibiting four main reflection peaks at  $2\theta = 16.12^\circ$ ,  $21.67^\circ$ ,  $34.92^\circ$  and  $44.35^\circ$ . The different X-ray diffraction spectrum depends on proportion of two crystalline forms[13]. The highest peak at  $2\theta = 21.67^\circ$  correspond to the  $I_{200}$  plane represents the both crystalline and amorphous material. The lowest peak height at  $2\theta = 18.28^\circ$  corresponds to the  $I_{AM}$  crystallographic plane and represents only the amorphous part.

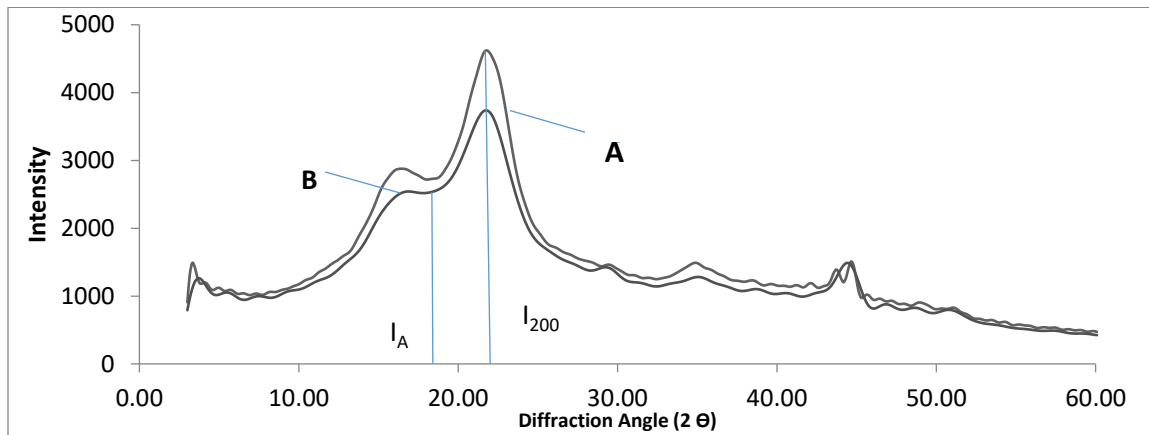


Figure2: XRD pattern of ZMH under varying treatment conditions viz. A. 20/30/30 B. Untreated

**3.3 SEM Analysis-** SEM images of pretreated and untreated samples are shown in Figure3. Array of numerous polygon type structures can be observed from Figure-3a on the surface of untreated ZMH, While in Figure-3b the ZMH after pretreatment is quite different in appearance. The major changes which can be clearly observed

are the shrinkage in the ridges (polygon shapes) and delignification (marked with ovals) of the film. Delignification is a result of the breakage of lignocellulosic bonding due to 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].

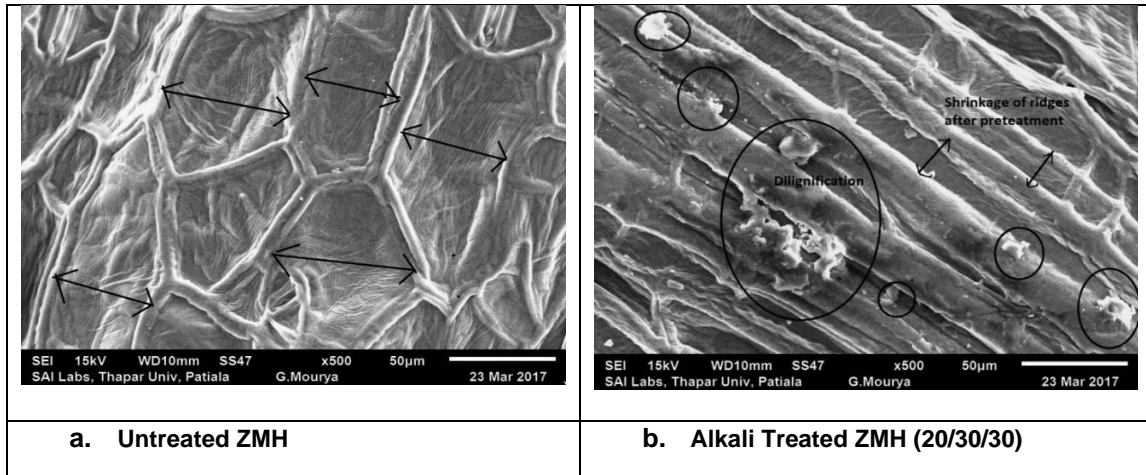


Figure3: SEM images of untreated & pretreated CHF

**3.4 Dynamic Mechanical Analysis** - Figures 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 3.45 GPa at 27 °C to 0.34 GPa at 48 °C, i.e. 90% decrease in storage modulus.

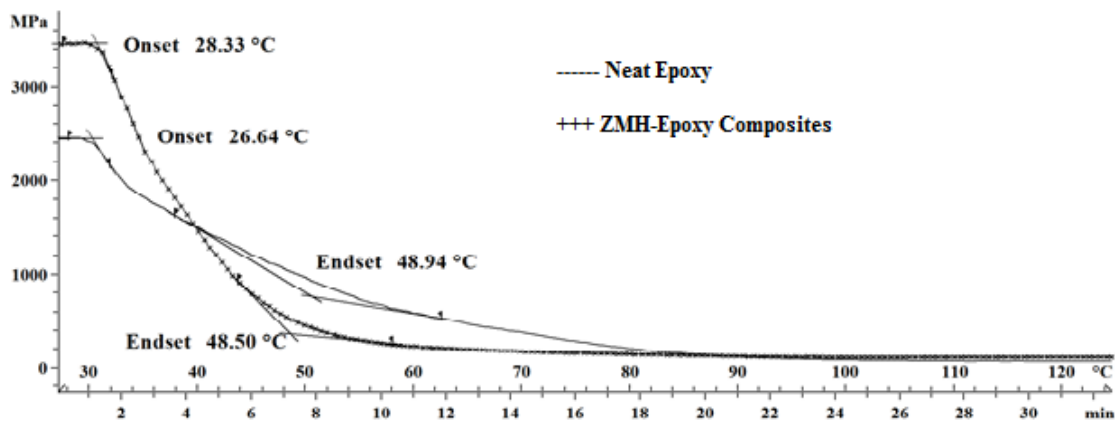


Figure4: Storage Modulus of A0 (100% Epoxy) and A1 (ZMH reinforced Epoxy Composite)

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, 17].

**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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