Effect of alkali treatment on tensile properties of short madar fibre reinforced polyester composites

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Abstract
Natural fibre reinforced polymer composites were fabricated with madar fibre as reinforcement and polyester as matrix. The madar fibres were treated with 10% NaOH solution (Mercerization) for surface modification. The composite samples were made with different weight proportions (2.5% to 15%) of both untreated and treated fibre in polyester according to ASTM standards using hand layup technique and tested for tensile properties. From the results, it was seen that there was improvement in the tensile strength of untreated madar fibre reinforced polyester composite to a value of 23.33MPa with increased fibre content up to 10% and with further addition of fibres there was drop in the strength. There was a significant outcome of alkali treatment on tensile strength of composite. The strength of alkali treated madar fibre reinforced composite was increased to a value of 26.23MPa with increase in the fibre content up to 15% which is 12.43% higher than that of untreated fibre reinforced polyester composite. There is increase in the modulus of untreated madar fibre reinforced polyester composite to a value of 493.23MPa with increased fibre loading up to 10% and with further fibre loading the value is reduced. It was also depicted that the modulus of treated fibre reinforced composite increased to a value of 444.57MPa with increased fibre loading up to 15% but the value is 9.86% less compared to that of untreated fibre composite. The % elongation of untreated fibre reinforced polyester composite decreased with increase in fibre content and was same with the treated fibre reinforced polyester composite also.

Key words: Madar fibre; Alkali Treatment; Polyester; Tensile Strength;

1. Introduction
From the past three decades the natural fibre reinforced composites have become promising substitutes to glass fiber reinforced polymer composites in several applications. It is experimentally proved that adding of natural fibres to the polymer improves strength, stiffness, thermal and acoustic insulating properties of that polymer composite and it is also found that these composites have high resistance to fracture and biodegradability[1-2]. Several natural fibres such as sisal, coir, bamboo, flax, jute, hemp etc. are used as reinforcing materials for thermosetting and thermo plastic composites [3-5].

The cellulose of natural fibre contains large quantity of hydroxyl group and gives hydrophilic property to natural fibre when utilised to reinforce hydrophobic matrices. Hence the effect is a poor resistance to moisture absorption and very poor interface leading to reduction in strength of composite [6]. To overcome such difficulties and to increase the bonding strength of fibre, chemical treatments are used. Among various treatments, treatment with alkali solution is one of the best among various...
treatments on natural fibres [7-9]. The significant modification done by alkali treatment is the interruption of hydrogen bonding in the network structure of fibre to increase the surface roughness [10]. It is also depicted that the treatment of natural fibre with NaOH will lead to the ionization of the hydroxyl group to alkoxide [11].

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\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O} - \text{Na} + \text{H}_2\text{O}
\]

Chemical treatment on natural fibre at different curing temperatures and times were examined [12]. Jute fibres were treated with 5% NaOH solution for 2 to 8 h at 30°C. The results depicted an increase in the modulus by 79% after 8 hours of treatment respectively. Also there was fibre pullout when cured between 0 and 4 h, whereas there was transverse fracture with minimum fibre pullout during 6 and 8 hours of treatment.

Another important parameter for improving strength of composite is fibre length. The composites were made with fibres of different lengths (5, 10, 15, and 20 mm) and weight ratios (8, 12, 16, and 20%) in the epoxy resin and tensile strength was evaluated [13]. The results showed improvement in tensile strength and modulus with increase in fibre length up to 15mm and weight ration up to 12%. It was also observed that the maximum tensile strength and modulus for the fibre length of 5mm and fibre content of 12%. In another study, the composites were fabricated using kenaf fibres in unsaturated polyester resin [14]. The resin was mixed with 2.5mm and 5mm kenaf fibres with varying fibre weight fraction (2.5 and 5%). The results showed that 5 mm length of fibre in the composites has higher strength than 2.5 mm length of fibre.

Present work is aimed at fabrication of composite with untreated and alkali treated madar fibre as a reinforcement in polyester matrix. The composites were fabricated with varied weight fractions of treated and untreated fibres by means of hand layup technique, and tested for tensile properties. The effect of chemical treatment on the fibre and fibre weight fraction on the tensile properties were depicted.

2. Experimental work
2.1. Materials Used
2.1.1. Polyester
Polyester is a type of polymer which contains the ester functional in their main chain. Their well-known applications include clothing, plastic water, food packaging and carbonated soft drinks bottles. It is often known as polyethylene terephthalate (PET). polyester is purchased from Bindu Agencies, Vijayawada.

2.1.2. Catalyst
The catalyst is the agent that is mixed directly in to the polyester in a composition of 1.2ml for 100gm of resin. The name of the catalyst used for curing of polyester is Methyl Ethyl Ketone Peroxide [MEKP].

2.1.3. Accelerator
Accelerator is also directly mixed with the polyester in combination with the catalyst MEPK. The job of the accelerator is to make the process faster. The name of the accelerator used is cobalt octate (C\text{16}H\text{30}CoO\text{4}).

2.1.4. Madar fibre
Madar fibre is a natural fibre extracted from the stem of Calotropis gigantean plant (figure 1). Calotropis gigantean is a medium sized bush that grows up to a length of 4m with usually waxy
exterior and abundant milky sap. The stem is ash colored, smooth, branching almost from the base of the plant. The leaves appear grey-green, opposite, waxy, thick and rounded-ovate.

![Figure 1. Madar Plant (Calotropis Gigantea)](image)

### 2.2. Methodology

#### 2.2.1. Extraction of fibres

The Madar fibres were extracted by retting process. The stem is cut into certain small length pieces and soaked in water for nearly 20 days and later they were beaten gently with mallet to extract the fibres from the stem. Prior to the development of the composites, the fibres were washed thoroughly and cut to 5mm length.

#### 2.2.2. Chemical treatment

The obtained fibres were soaked in 10% of aqueous sodium hydroxide solution for 6 hours. Then the fibres were washed for number of times with distilled water to eliminate any alkali adhering on their surface, then neutralized with dilute acetic acid and then finally washed again with distilled water, followed by drying at 70°C for 48 hours in hot air oven.

#### 2.2.3. Mould preparation

The moulds were prepared with rubber sheet of 3mm thickness which is cut to a size that can accommodate 5 specimens as per the ASTM D638 standard for tensile test (figure 2).

![Figure 2. Rubber moulds for sample preparation](image)
2.2.4. Specimen preparation

The samples were prepared in the moulds with untreated and alkali treated short madar fibres at different weight proportions (2.5%, 5%, 7.5%, 10%, 12.5% and 15%) in the polyester using hand layup technique. Catalyst and accelerators were also added for faster curing process. 5 samples of each weight proportions were fabricated.

2.3. Testing of composites

2.3.1. Diameter and density of fibre

Tool maker’s microscope is used to measure the diameter of both treated and untreated fibres. Density of fibre is determined based on Archimedes principle which states that the volume of fluid displaced is equal to the volume of any object which is fully immersed in that fluid.

2.3.2. Tensile test

These samples are tested for tensile property on a universal testing machine as per ASTM standards (ASTM D638). The samples are loaded on the machine and the maximum load is set to 200 kg and the load is applied and the samples were tested with a crosshead speed of 0.5 mm/min. The elongation and their respective loads are noted.

3. Results and discussion

3.1. Diameter and density of fibre

The physical properties such as diameter and density of untreated and alkali treated fibres were evaluated and depicted in the table 1. From the results it is seen that the alkali treatment on the fibre has reduced the diameter and density of fibres which is an indication of alkali effect of removal of hemicellulose and lignin from the fibre surface.

| Table 1. Physical properties of Madar fibre |
|------------------------------------------------|
| Diameter, mm | Density, gm/cc |
|----------------|----------------|
| Untreated Madar fibre | 0.237 | 1.45 |
| Alkali treated Madar fibre | 0.204 | 0.54 |

From figure 3, it was depicted that the tensile strength of untreated madar fibre reinforced polyester composite was increased with increased fibre loading up to 10% and with further addition of fibres there was decrease in the tensile strength. The maximum value attained for 10% fibre loading was 23.33 MPa. Also it was depicted that the strength of treated madar fibre reinforced composite was increased with increase in the fibre loading even up to 15%. The strength of treated fibre composite was 26.23 MPa which is 12.43% higher compared to that of untreated fibre reinforced polyester composite.

From the past research is fact that fibres are stronger than that of the matrix material, and as there is a sufficient fibre-matrix adhesion due to the presence of modified fibre (chemical treated), it is clear that with an increase in fibre content the composite strength can be improved. Also it was observed from the literature that alkali respond on the cementing materials of the fibre mainly hemicellulose which leads to the splitting of the fibre into finer filament leading to increase in the surface area of contact for bonding with matrix leading to increased strength. However, in case of untreated fiber composite the trend of increase in strength is only up to certain volume of fibre, and after that there is deficient polymer component in the composite for providing sufficient wetting of fibres. The increase in the fibre content beyond certain limit also led to reduction in fibre dispersion and fiber-fiber interaction than fiber-matrix interaction in the composites that may have further contribution to the decrease in the strength of composite.
From figure 4, it is observed that there is increase in the modulus of untreated madar fibre reinforced polyester composite to a value of 493.23 MPa with increased fibre loading up to 10% and with further fibre loading the value is reduced. Moreover the tensile modulus of treated fibre composite was increased to a value of 444.57 MPa with increased fibre loading up to 15% but the value is 9.86% less compared to that of untreated fibre polyester composite.

Figure 3. Variation of tensile strength of madar fibre reinforced polyester composite with fibre loading.

Figure 4. Variation of tensile modulus of madar fibre reinforced polyester composite with fibre loading.

Figure 5. Variation of % elongation at break of madar fibre reinforced polyester composite with fibre loading.
It is observed from figure 5 that the % Elongation at break of untreated fibres and treated fibres decreases as the fibre weight fraction increases. There is reduction in % elongation at break to 4.7% for untreated fibre reinforced composite with increase in the fibre content and is further reduced to 5.96% with addition of treated fibre in the polyester.

4. Conclusions
With the results obtained from the experimental procedure, the following conclusions are made.

- The tensile strength of untreated madar fibre reinforced polyester composite was increased with increased fibre loading up to 10% and with further addition of fibres there was decrease in the tensile strength. The maximum value attained for 10% fibre loading was 23.33MPa.

- It is also depicted that the tensile strength of treated madar fibre reinforced composite was increased to a value of 26.23MPa with increase in the fibre loading even up to 15% of treated fibre loading and the value is 12.43% high compared to that of untreated fibre reinforced polyester composite.

- There is increase in the modulus of untreated madar fibre reinforced polyester composite to a value of 493.23MPa with increased fibre loading up to 10% and with further fibre loading the value is reduced.

- Moreover the tensile modulus of treated fibre polyester composite has increased to a value of 444.57MPa with increased fibre loading up to 15% but the value is 9.86% less when compared with that of untreated fibre polyester composite.

- The % elongation of untreated fibre reinforced polyester composite decreases with increase in weight ratio and is same with the treated fibre reinforced polyester composite.

- Finally, it is concluded that, chemical treatments on fibre has produced the stronger and stiffer fibre with a good level of fibre separation leading to more surface area of contact and hence better interface with matrix. This has lead to better strength of the composite.

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