Effect of alkaline treatment on mechanical properties of woven ramie reinforced thermoset composite

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Abstract. Ramie fibres are one of the strongest bast fibres with valuable potential as reinforcement in a composite. In this study, the laminated composite of ramie reinforced plain woven was prepared through hand lay-up process. Prior to moulding, the ramie reinforced woven fabric was treated by soaking the fabric in 5% NaOH solution for 1 hour at room temperature. The effects of alkaline treatment on ramie reinforced woven epoxy resin and ramie reinforced woven unsaturated polyester (UPE) resin were investigated in terms of tensile, flexural, and impact properties. Five samples of both treated ramie (TR) composite and untreated ramie (UR) composite were tested and the results were averaged for comparison with one another. Based on the results obtained, the effects of 5% (w/w) concentration of alkaline solution treatment for 1 hour at room temperature are insignificant to the tensile, flexural, and impact properties. The result of UR and TR composite was compared to the composite for pure epoxy resin and UPE resin. Based on the finding, the data obtained shown no improvement in tensile properties. However, in the case of the flexural properties are enhanced except flexural modulus of the TR-epoxy composite. Impact strength only gets better when reinforcing with the untreated ramie. On overall, the TR ramie have an admirable mechanical when reinforced with the epoxy resin than UPE resin.

1. Introduction

Natural fibres are advantageous when used as a reinforcement material for the improvement of properties such as the improvement in strength, fatigues, and impact [1]. Unlike synthetic fibres, natural fibres also favourable for its characteristics such as environmentally friendly, low density, biodegradable, abundant availability, renewable, and cost effective as compared to synthetic fibres. However, several issues hindered the wide applications of natural fibres such as poor interfacial adhesion between polar hydrophilic fibre and nonpolar hydrophobic matrix and poor wetting of the fibre with the matrix polymer which resulted in a composite with weak interface [2]. The polar hydrophilic of natural fibres causes dimensional change in the composite [3].

To improve the major drawback of natural fibres as reinforcement materials, previous studies have introduced two methods of surface modification, either by physical or chemical treatment [2, 4, 5].
The main purpose of both treatments on natural fibres is to enhance the composite interface and enhance the adhesion between natural fibres. The common treatments that have been performed previously include alkaline treatment, silane treatment, sea water treatment, and hot water treatment. Among them, the alkaline treatment is frequently performed due to the low cost incurred and simplicity of the process [6]. Despite that, the alkaline treatment enhances the mechanical properties of the composites by the removing of hemicellulose and wax at the fibre surface [6]. The alkaline treatment produces a rough surface on natural fibre which consequently increases the fibre-matrix adhesion through mechanical interlocking. The alkaline treatments have been performed previously on various kind of natural fibre such as jute, kenaf, sisal, banana, hemp, banana, and ramie in the variable form of reinforcement [7-9]. The effects of treatment can either be positive or negative depending on many factors such as the type of treatments, a number of treatment agents, and the properties of natural fibres [10]. Results from the previous study mostly produced positive effects of alkaline treatment on natural fibre [5, 11, 12]. However, several adverse effects have also been reported [7, 13, 14]. The alkaline treatment of ramie woven was less discussed previously as compared to the other form of reinforcements, such as unidirectional and short directional, thus providing less information regarding this process.

Therefore, the present work focused on the effects of alkaline treatment on ramie reinforced woven epoxy resin composite. The results of tensile, flexural, and impact testing for both untreated ramie (UR) composite and treated ramie (TR) composite were compared with the data from pure/neat epoxy and polyester resin sample and are discussed in details.

2. Materials and methodology

Ramie fibre, in the form of plain woven fabric, which was purchased from Tazdiq Engineering Sdn Bhd. Malaysia was selected as the main reinforcement material. The size of ramie fabric was 40 × 40 cm. Four plies of ramie fabric were used in each laminated composite. The matrix polymer that was selected in this study was an epoxy resin and unsaturated polyester (UPE) resin. The epoxy resin, UPE resin and hardener were purchased from Revertex (Malaysia) Sdn. Bhd. The NaOH solution was supplied from Sigma-Aldrich, United States of America.

2.1. Alkaline treatment of woven ramie

Washed. The ramie reinforced woven fabric was soaked in purified water for 6 hours to remove the impurities and dried in the oven at 60 °C for 24 hours. The simply washed fabric is referred as untreated ramie fabric (UR).

Treatment with alkaline solution. The fabric was soaked in a 5% NaOH solution for approximately 1 hour. The alkaline treatment for ramie reinforced woven fabric was performed at room temperature, followed by purification using water. Litmus paper was used to check the neutrality of the fabric. The fabric was then dried at 60°C for 48 hours. This fabric is referred as treated ramie fabric (TR).

2.2. Sample preparation of laminated composite

The composite specimens were prepared by using hand lay-up technique. At first, the realising agent was applied on the surface of the mould to easily remove the composite after curing. The epoxy resin and hardener were mixed according to the ratio of 10:1 in a beaker. The epoxy resin mixture was then stirred using a rod to ensure no air was entrapped inside the solution. Firstly, the UR fabric was placed on top of the mould and the epoxy resin mixture was poured on top of it. The hand roller and scraper were applied to remove entrapped air bubbles. The process was repeated until the desired layering with the size of 4 was achieved. All the plies were laid up at 0° directions. After completed, the dead weight of 10 kg was applied on top of the mould and cured at room temperature for 24 hours. Upon completion, the composite was then carefully removed from the mould. The same fabrication process was repeated on another sample of UR, except by using a different resin which was UPE resin. For comparison purposes, similar fabrication process was also repeated on the sample of TR fabrics. Finally, the UR and TR laminated composite were cut by using diamond cutter into the desired dimension according to the selected standard of American Society for Testing and Materials (ASTM) based on each testing such as tensile, flexural, and impact testing.
2.3. Mechanical testing

Tensile testing. The Universal Testing Machine (UTM) model of AGI 100KN Shimadzu was used to perform the tensile testing. ASTM D-638 type I was used for preparing the sample having the dimension of 165 × 19 × 3.2 mm. The cross-head speed was set at 1 mm/min and the test was conducted at 25±20 °C with 50% humidity. Five replicated samples were used in the test to obtain a more statistical reliability.

Flexural testing. The samples for flexural testing were prepared according to the ASTM D-790, having the dimension of 127 × 12.7 × 3.2 mm. The tests were carried out at an average relative humidity of 50% and at a temperature around 25±20 °C. A cross head with a constant speed of 2.5 mm/min were applied by a load on two support span with the length of 48 mm. The test was repeated for five times to acquire the average value and the standard deviation.

Impact testing. The notched Charpy impact testing was performed by using the Izod impact tester. All the samples were notched and prepared according to the ASTM D-256 having the dimensions of 64 × 12.7 × 3.2 mm. The tests were performed at 25±20 °C with 50% humidity. The average of 5 tested samples was calculated for statistical reliability.

3. Result and discussion

3.1. Tensile properties

The results of tensile properties from mechanical testing are illustrated in Figure 1. Based on Figure 1, the neat epoxy resin and UPE resin have higher tensile strength. The addition of UR into epoxy resin and UPE resin did not improve the tensile strength and the tensile modulus. The tensile strength for neat epoxy resin drop from 51.37 MPa to 44.22 MPa, which about 13.9% reduction, while the neat UPE decreases from 37.26 MPa to 30.87 MPa that represents about 17.1% reduction. At the same time, the tensile strength of the neat epoxy resin reduced from 51.37 MPa to 26.56 MPa, with 48.3% reduction when reinforced with TR fabric. Furthermore, the tensile strength of the neat UPE resin also drops to 24.68 MPa with 33.7% reduction when reinforced with TR fabric. The tensile modulus also revealed a similar result as shown by the result of tensile strength. The tensile modulus of the neat epoxy resin decreased from 1.81 GPa to 1.52 GPa and 0.65 GPa when reinforced with UR and TR, respectively. Meanwhile, of the tensile modulus of the neat UPE resin drops from 1.53 GPa to 1.36 GPa and 1.09 GPa when reinforced with UR and TR, respectively.

The results obtained from tensile testing have good agreement with the previous study that performed alkaline treatment on ramie fibre. As reported by Choi et al. [4], the tensile strength and tensile modulus of the composite decreased when treated with alkaline treatment. A similar finding was reported by Goda et al. [14] which compiled the report of ramie fibre treated with 5% NaOH reinforced biodegradable polymer. It concludes that the alkaline treatment on ramie fibre causes an insignificant improvement on the tensile strength but only a slight change in the tensile modulus. In the other work, Goda et al. [13] evaluated the effects of alkaline treatment with 25% (w/w) concentrations on ramie fibre with and without applying load condition. In the case of without load applied, the tensile properties of treated ramie fibre show insignificant improvement. On the contrary, the tensile strength recorded differently in other cases. Nevertheless, both conditions recorded a reduction in the tensile modulus.
3.2. Flexural properties

The flexural properties of composites are shown in Figure 2. In the case of neat epoxy resin, the flexural strength increased from 45.53 MPa to 47.21 MPa and 46.05 MPa when reinforced with UR and TR, respectively. Similarly, the reinforced of UR and TR also improved the flexural modulus of neat epoxy resin from 1.15 GPa to 1.51 GPa and 1.43 GPa, respectively. As for neat UPE resin, the results obtained are different. The UR reinforced UPE improve the flexural strength of the neat UPE resin from 41.86 MPa to 44.89 MPa. However, the value for flexural strength obtained when the neat UPE resin was reinforced with TR was only 27.95 MPa. Nonetheless, both neat epoxy and neat UPE resin show significant effects on flexural modulus when reinforced with UR and TR fabrics. The UR composite is better than TR composite since it exhibited higher strength resistance. The TR composite is more brittle compared to the UR composites. Therefore, the TR composite will break first before deformation took place.
3.3. Impact properties
The variation of impact properties of UR and TR reinforced epoxy resin and UPE resin was presented in Figure 3. The addition of UR to the epoxy resin and UPE resin has higher influences on the impact of composites as compared to the addition of TR. The UR reinforced epoxy resin increased the impact strength of the neat epoxy resin by up to 29% and UR reinforced UPE resin enhance the impact strength of the neat UPE resin for up to 127%. However, the impact energy decreased around 32% and 40% for TR reinforced epoxy resin and TR reinforced UPE resin, respectively as compared to the neat epoxy and UPE resins. Based on the result, it can be concluded that the TR composites are more brittle compared to the UR composites. The brittle behaviour of TR composites caused by lesser fibre pull-out where the dissipation of impact energy is unable to perform. By comparing the findings with He et al. (2008)[15], the alkaline effects from the alkaline treatment of ramie fibre on the impact properties are analogous since no improvement was reported.

![Impact strength graph](image)

**Figure 3.** Impact properties of woven ramie thermoset composite.

4. Conclusions
The effects of a number of alkaline treatment on the mechanical properties of ramie reinforced woven epoxy resin and unsaturated polyester resin was explored. Based on the experimental results, the following conclusions were drawn:

- The ramie woven laminated composites treated with 5% (w/w) concentrations for 1 hour at room temperature does not improve the mechanical performance of the laminated composites for all samples. However, there are slight improvements in flexural strength and flexural modulus for the epoxy composite
- When compared to UPE composite, the epoxy composite is stiffer and brittle when reinforced with UR and TR
- The incorporation of UR composite into having significant properties than TR composite.

The scanning electron microscopic (SEM) should be performed on sample fracture to gain a better insight into the behaviour of ramie woven in the composite. Several steps should be taken into considerations to improve the development of ramie reinforced woven polymer matrix in the future. On the other hand, the immersion time and concentrations of the alkaline treatment need to be reconsidered. This is because both the duration and concentrations play a significant role in predetermining the mechanical performance of the treated woven fabric. To increase the mechanical performance of woven ramie laminated composite, it is suggested that the variable of alkali treatment such as the concentration should be defined at lower values. Through optimisation of the variable, the
alkaline treatment will unleash its full potential not only to improve the interfacial adhesion strength between natural fibre and polymer matrix but at the same time eliminate the void formation and enhance the overall mechanical performance

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