Effect of elevated temperatures on flexural strength of hybrid Napier/glass reinforced epoxy composites

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Abstract. The effects of elevated temperatures on the flexural strength of hybrid Napier/glass reinforced epoxy composites were investigated. Hybrid composites laminates were fabricated using untreated, 5%, or 10% alkali-treated Napier fibres with woven E-glass fibres and epoxy resin. The composites were manufactured using a vacuum infusion process; the volume fraction of the Napier, glass fibres and epoxy resin were 24%, 6% and 70% respectively. When tested at room temperature (RT), the maximum flexural strength was recorded for the hybrid composites with the 5% alkali-treated Napier fibres. When the test temperature greater than 60°C, the matrix cracking and delamination were occurred due to the temperature that approached the glass transition temperature (Tg) of the composites, which resulted in a reduction of the flexural strength. The fracture surface morphologies indicated that the 5% alkali-treated Napier fibres improved the fibre-matrix interfacial bonding of the hybrid Napier/glass reinforced epoxy composites.

Keywords: Hybrid composites; natural fibres; flexural strength; elevated temperatures; epoxy.

1. Introduction
Lately, the practice of natural fibres for reinforced composites has become a promising idea to emerge “green” based economy in industrial application. Natural fibres are completely renewable resources, environmentally friendly, low-cost, high biodegradability and possessed comparable mechanical properties as synthetic fibres[1–4]. Their weight also light with preeminent specific modulus and required little energy during processing; less wear and tear, which all these advantages have brought natural fibres as a solid reinforcing agent in polymer composites for various applications both in engineering and technology fields [5]. Plant-based natural fibre reinforced composites are mostly from plant fibres like jute, sisal, kenaf, hemp, Napier, ramie, pineapple leaf, abaca leaf, sugarcane, bamboo and so on [3-5]. Due to severe environmental problems and a high concern for the world plus easy availability and eco-friendly of natural fibres, many researchers are compelled to investigate the use of these continuous resources as an alternative to traditional polymer composites manufactured from synthetic fibres [9]. Therefore, the exploitation of natural fibres is improved remarkably and has substantially generated the interest in the study for the development of plant-based polymer composites [7–9].
However, the elevated temperature on the composites brought a serious impact towards the mechanical properties of the composites[13]. For example, increasing the temperature of surrounding can impair the structure of a composite which will create various damages such as intra-ply damage, matrix cracking, interlaminar cracks, fibre breakage and subsurface delamination [14]. The fact is natural fibres have less thermal stability. Thus, they tend to degrade at lower temperature [15]. An interesting technique to limit this problem is through hybridization rather than using chemical treatment since it has the disadvantage of being expensive. Concerning the hybrid composites which consist two kinds of fibres, the most virtue relies on the fact that the benefits of a fibre could surpass the lack of another fibre [15].

In this study, experimental analysis was implemented to analyze the flexural strength of hybrid Napier/glass reinforced composites. The tests were carried out in room temperature (RT) and also at elevated temperatures. By thoroughly elucidating and analyzing the mechanical properties of the composites which are cost-effective and environmentally friendly, this study is expected to produce supportive manifestation for further application and development.

2. Materials and methods

2.1. Materials
The hybrid composites consisted of Napier’s fibres, woven E-glass fibres and epoxy resin. Napier grass was gained from a local plantation located at Bukit Kayu Hitam, Kedah, in Northern Peninsular Malaysia. Water retting process was practiced to facilitate the extraction of fibres from the Napier’s stem internodes. By using a mallet, the stems were chopped into smaller portion at the early step. After that, the short stems were left in water for 2-4 weeks to ease the separation of the fibres from the skin and the cellulose. The woven E-glass fibres were ordered from a local supplier and prepared in the form of mats with dimensions of 300 × 300 × 15 mm per layer. EpoxAmite 100 series resin was used as a matrix, and mixed with a hardener at a ratio of 3:1.

2.2. Composite fabrication
The hybrid Napier/glass-reinforced epoxy composites were fabricated using a vacuum infusion method. The volume fractions of the Napier fibres, glass fibres, and epoxy resin were 24%, 6%, and 70%, respectively. The hybrid composite samples consist of either untreated, 5%, or 10% alkali-treated Napier fibres. The reinforcement plies were laminated above a glass mould. By using a high vacuum pump machine, the matrix resin was infused into the lamination plies. The in-mould pressure was ensured to be below than 2,000 Pa in preparation for the matrix resin to be infused into the mould. Then the composites were left at room temperature for 12 hours to cure. In the end, rectangular plates with dimensions of 300 × 300 × (3.2 ± 0.4) mm$^3$ were produced. Afterwards, post-curing was carried-out in an oven at 80 °C for 2 hours under air circulation [17]. Lastly, by using a Dremel 4000 tool, the plates were cut to prepare specimens for flexural testing according to the related standards.

2.3. Flexural testing
Flexural tests via three-point bending tests, was carried-out on specimens with dimensions of 125 × 13 × (3.2 ± 0.4) mm$^3$, in accordance with ASTM D790-1 [18]. The specimens were loaded with the recommended span-to-depth ratio of 16:1. Flexural tests were implemented in the Instron environmental chamber at RT (25°C) and at elevated temperature of 40, 60 and 80°C. The tests on the specimens were conducted using a universal micro tester with a load cell of 2 kN at a crosshead speed of 2.5 mm/min [20,21]. Five replicated specimens of each composite were tested for every different temperature. For those stated temperatures, the flexural stress–strain curves were plotted and recorded.

2.4. Morphological observation
The fractured surface morphologies of the hybrid Napier/glass-reinforced epoxy composites were observed using a field-emission scanning electron microscope (FESEM). For FESEM observation,
section of the fractured test specimens was cut. Platinum was then coated over the cutting section before scanning. The scanned images were acquired with accelerating voltage of 3–5 kV.

3. Results and discussion

Figure 1 shows the maximum stress curves for the different states of the hybrid Napier/glass reinforced epoxy composites for the untreated, 5% and 10% of alkali-treated that were subjected to the flexural test in RT and under certain elevated temperatures. Based on Figure 1, it can be simply concluded that the stress curves become increasing non-linear while the temperature is increased. The figure apparently showed that the hybrid composites when tested at RT and 40°C, the recorded stress was at the high for all composite states and significantly differed to those results obtained and recorded at 60 and 80°C which much lower. This situation is due to the increased temperature that softens the epoxy matrix which caused the structure of the specimens to be ductile and eventually failed at higher strain due to higher temperature.

The maximum stress recorded for hybrid composites with untreated Napier’s fibres were at 69, 67, 33 and 15 MPa for RT, 40, 60 and 80°C respectively. From these values, it can be observed that the flexural strength at RT and 40°C were very similar due to the fibre reorientation which happened during the softening of the matrix. On the other hand, at greater temperatures, the characteristics of the matrix resin crucially effected the total potential of the hybrid Napier/glass reinforced epoxy composites. This is substantially proved by the decrement of the flexural strength at the testing temperature of 60°C, showing there was 50% reduction in strength than the result gained at room temperature.

The maximum stress recorded for hybrid composites with 5% of alkali-treated Napier fibres at testing temperatures of RT and 40°C compared to the results acquired for the untreated hybrid composites. This is due to the excellent interfacial adhesion among the fibre and the matrix resin. The fact is alkali treatment caused the removal of the hemicellulose, waxes, lignin and other impurities. In the other word, this condition would offer a better fibre/matrix adhesion instead of causing a rough surface on the Napier fibres. Besides, the alkali treatment also led to fibre fibrillations which increased the effective surface area between the fibres and the matrix. As a consequence, the flexural strength of the composites improved indirectly. This has been verified by some researchers that apparently validated the increment in the composites’ flexural strength of using alkali-treated natural fibres is because of the greater interface between the fibres and the matrix after the treatment [22–26]. However, a lower flexural strength was recorded for hybrid composites with 10% of alkali-treated Napier fibres. This decrement of flexural strength can be clarified because of the loss of hemicellulose and lignin at the interfibrillar region, thereby causing the fibres to be less dense and rigid. Das et al. [26] claimed that the decrement of long-chain cellulose molecules at the natural fibre interfaces in right concentration of alkali treatment will reduce fibre breakage and lead to optimal flexural strength of the composites. Though, when the composites were tested at elevated temperatures of 60 and 80°C, the flexural strength values recorded for the composites with the untreated Napier fibres were higher compared to the hybrid composites with 5% and 10% of alkali-treated Napier fibres.

Following test at RT, it was observed that brittle failure occurred at the centre of the specimens, causing in the formation of rough fracture surfaces. Similar fracture surfaces were indicated at elevated temperatures for both untreated and treated Napier fibres hybrid composites. At testing temperature of 40°C, the fracture surfaces were rough with noticeable fibres breakage. Though, part of the fracture surfaces exhibited a smoother profile if comparing to those of the specimens tested at RT. On the other hand, the resulting fracture surfaces of the specimens were rougher when tested at temperature of 60°C compared to the specimens tested at RT and 40°C, showing that the specimens failed because of the softening of the polymer matrix. This could be observed from the specimens that failed more gradually with progressive failure and more pull-out of the Napier fibres happened [27]. Eventually, when the specimens were tested at 80°C, high levels of defectiveness were noticed due to the crack propagation that hindered by the fibres, propagating at different points of the specimens,
thereby numerous cracks were visible. This is specified by a gradual decrement of stress of the composites with the increasing temperature as showed by the figure 1.

\[ \text{Figure 1. Maximum flexural stress behaviour of the hybrid Napier/glass reinforced epoxy composites subjected to RT and elevated temperatures for different state of composites.} \]

4. Fracture surface morphology
The fracture surface morphologies for the different state of hybrid Napier/glass reinforced epoxy composites which subjected to flexural tests at RT and elevated temperatures shown by Figure 2. The surface morphologies of the hybrid composites with the untreated Napier fibres, when tested at RT, was rough and demonstrated typical long fibres pull-out as shown in Figure 2 (a). When tested at 40°C, matrix cracking has been observed as illustrated in Figure 2(b). When the specimens were tested at 60°C, voids because of to the fibres pull-out were visible as well as the porous structure like in the Figure 2(c), indicating that the testing temperature was approaching the $T_g$ value of the composites. Lastly, delamination of glass fibres was observed in the fracture surface image of the specimens that were tested at 80°C as figured in Figure 2 (d).

The effects of elevated temperature on the flexural strength of hybrid Napier/glass reinforced epoxy composites were investigated. Untreated, 5% and 10% alkali-treated Napier fibres were used to fabricate the hybrid composites. The highest maximum flexural strength recorded following test at RT was given by the hybrid composites with 5% alkali-treated Napier fibres. All hybrid composites with untreated and treated Napier fibres showed maximum flexural strength when tested at RT and 40°C. However when the samples tested at elevated temperatures of 60°C and 80°C, the voids due to fibres pull out and delamination occurred. Consequently, the damages reduced the flexural strength of the composites since the temperatures approached and exceeded the glass transition temperature ($T_g$) of each composite. This situation was due to the softening of the matrix, leading to poorer interfacial bonding between the Napier fibres and the matrix.
5. Conclusion

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