

Properties of Corn Husk Fibre Reinforced Epoxy Composites Fabricated Using Vacuum-assisted Resin Infusion

Koay Seong Chun,^{1*} Tengku Maimunah,¹ Chan Ming Yeng,²
Tshai Kim Yeow³ and Ong Thai Kiat⁴

¹School of Computer Science and Engineering, Faculty of Innovation and Technology,
Taylor's University Lakeside Campus, No. 1, Jalan Taylor's,
47500 Subang Jaya, Selangor, Malaysia

²Centre of Engineering Programmes, Imperium International College (formerly HELP
College of Arts and Technology), 6.01, Level 1–7, Kompleks Metro Pudu,
1 Jalan Metro Pudu 2, Fraser Business Park, 55200 Kuala Lumpur, Malaysia

³Faculty of Engineering, University of Nottingham, Jalan Broga,
43500 Semenyih, Selangor, Malaysia

⁴Faculty of Engineering and Technology, Tunku Abdul Rahman University College,
Jalan Genting Kelang, Setapak, 53300 Kuala Lumpur, Malaysia

*Correspondence author: seongchun.koay@taylor's.edu.my

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ABSTRACT: *Corn husk is a non-fruit part of the corn and an agricultural waste. This research focuses on fabricating composite material from the corn husk fibre by using a vacuum assisted resin infusion (VARI) technique. The corn husk fibre was extracted using water retting method and treated with alkali and bleaching treatments using sodium hydroxide (NaOH) and hydrogen peroxide (H₂O₂). Then, those fibres were made into non-woven fibre mat using water laid method. The fibre mat was infused with epoxy resin to make a composite. The mechanical result has shown that the composite with 20% of fibre has higher tensile strength and modulus compared to neat epoxy. Both treatments have removed the non-cellulose content, such as hemicellulose and lignin. Moreover, the treatments also promoted better interfacial adhesion between fibre and matrix. Thus, the NaOH treated and H₂O₂ treated composites showed approximately 56% and 82%, respectively, higher tensile strength than untreated composite. Both treated composites were also stiffer and exhibited lower water absorption compared to untreated composite.*

The composite made from fibre that treated with H_2O_2 have shown the highest tensile strength and modulus approximately to 28 MPa and 2605 MPa.

Keywords: Corn husk fibre, epoxy, composite, vacuum assisted resin infusion, corn husk composites

1. INTRODUCTION

Nowadays, natural fibre reinforced composites have attracted more attention compared with synthetic fibre reinforced composites. This is because natural fibres exhibit several benefits over synthetic fibre, such as being cost-effective, readily abundant, lightweight and having high mechanical strength to weight ratio. In the automotive industry, the applications of natural fibre reinforced composites are increasing rapidly such as in the production of door trim panel. Other than that, natural fibre reinforced composites have also been used in construction and aerospace industries.¹ Essentially, a natural fibre reinforced composite is considered a green material as the source of natural fibre is renewable and this composite also has a smaller impact to environment when applied for energy recovery.² Recent research works have shown that many natural fibres from agricultural waste have been utilised to produce composite materials, such as durian husk fibre, corn husk fibre and pineapple leaf fibre.³⁻⁶ From the literature, the corn husk fibre was fabricated into composite materials by mixing with recycled plastic material.⁵ However, the study of fabricating composite from corn husk fibre using vacuum assisted resin infusion (VARI) technique has not been reported in the open literature.

Corn is the largest planted crops in the world. World corn production for years 2019 and 2020 was approximately 43,731 million bushels.⁷ The production of corn is expected to increase gradually each year with the growth of technology in the agricultural industry. Increase in corn harvesting also in turn produces higher generation of wastes in the form of corn husk, corn leaves and stalks. Corn husk is the protective leaves that cover the ear of a corn and contains mainly lignocellulosic fibre. From a previous study, the corn husk fibre was used as reinforcement in producing thermoplastic composite material.⁵ However, the study about the utilisation of corn husk fibre in producing composite materials using VARI technique is rarely reported in the literature.

In this context, this research aims to explore the potential of producing fibre mat from corn husk fibre and turn it into a composite material with the aid of VARI technique. VARI technique is a composite fabrication technique in which the vacuum pressure is used to drive liquid resin flow and the voids in an evacuated fibre mat are infused with liquid resin. After the liquid resin is cured and turned

into solid resin matrix, the resin binds the fibres into a rigid composite. The VARI technique is a cost-effective method for producing better surface finish and lower void defect composite parts suitable for low-volume production compared to the traditional technique such as the hand lay-up method. Furthermore, a high fibre-to-matrix ratio of the composite can be produced by using VARI.⁸ Chin et al. prepared non-woven fibre mat from chrysanthemum waste and infused it with epoxy resin using VARI technique.⁹ They reported that the non-woven composite fabricated using VARI technique exhibited high mechanical strength and modulus. The fibres extracted from corn husk were short fibres and they can be made into non-woven mat. Therefore, the VARI technique is ideal for fabricating the corn husk fibre reinforced epoxy composite.

Natural fibre absorbs a significant amount of moisture, has poor thermal stability, shows variation in quality, poor interfacial adhesion, and less compatibility with hydrophobic polymer.⁴ This is because the natural fibre comprises strong polarised hydroxyl groups in its chemical structure, thus exhibiting hydrophilic characteristic.¹⁰ The poor interfacial bonding between natural fibre and polymer matrix is a main issue that influences the mechanical strength of composite materials.¹¹ Hence, it is necessary to implement chemical treatment for improving the adhesion between fibre and polymer matrix. The alkali and bleaching treatments were usually used to pre-treat the fibre before fabricating into composite material. These treatments were effective in removing wax, hemicellulose and lignin in the fibre. The pre-treated fibre with either alkali or bleaching treatment usually has better adhesion with the polymer resin.^{4,9}

In this research, the corn husk fibre was pre-treated with alkali and bleaching treatments before made into non-woven fibre mat. Then, the fibre mat was infused with epoxy resin to create composite using VARI technique. This study focuses on the effect of the fibre treatments on the mechanical, morphological and water absorption properties of the composite.

2. EXPERIMENTAL

2.1 Raw Material

In this research, a low viscosity grade of epoxy resin (A) and hardener (B) (mixing ratio AB 2:1 by weight) were purchased from PiCarbon Fibre (M) Sdn. Bhd., Malaysia. The density and viscosity of the epoxy resin are 1.14 g cm^{-3} and $0.7 \text{ Pa}\cdot\text{S}$, respectively at room temperature. Corn husk wastes were collected from a wet market in Kelana Jaya, Selangor, Malaysia. Sodium hydroxide (NaOH) pellets and hydrogen peroxide (H_2O_2) were purchased from R&M Chemicals, Malaysia.

There were several components in VARI such as peel ply, vacuum bag, vacuum hose, gum tape and releasing wax were purchased from PiCarbon Fibre (M) Sdn. Bhd.

2.2 Preparation of Corn Husk Fibre

The water retting method was implemented for extracting the corn husk fibre following the method by Tahir et al.¹² The corn husks were fully immersed in a plastic container filled with tap water. This process would take approximately 15 days at room temperature to complete the extraction of corn husk fibre from the corn husk. After a few days, the softened corn husks were scratched using a plastic scraper to peel off the fibres from the husk. After the fibres disintegrated from the corn husks, they were washed with tap water for a few times to remove contaminants from the husk. The corn husk fibres were dried in an oven with temperature of 60°C. The dried corn husk fibres were further chopped into short fibres by using a miniature grinder. The chopped corn husk fibres were sieved using a stainless-steel mesh with mesh size of 5 mm. The average length of the short fibres was 15–20 mm. From Figure 1, the fibres were made into a fibre mat through the wet laid method. The mass of the fibre mat was 20 g and the size of the wire mesh frame used for wet laid was 15 cm × 15 cm. After the process, the fibre mat was dried for 12 h in an oven with temperature of 60°C until the fibre was fully dried.



Figure 1: Preparation of corn husk fibre mat.

Two different types of chemical treatments such as alkali and bleaching treatments were carried out to pre-treat the corn husk fibre before made into fibre mat. For alkali treatment, the fibres were soaked in 5% of NaOH solution for 4 h at room temperature.⁵ A study by Koay et al. reported that the alkali treatment with NaOH was effectively enhanced the mechanical properties of the corn husk fibre reinforced

recycled polystyrene composite.⁵ Besides, the bleaching treatment with H_2O_2 was carried out by following the method used by Wong and Chan, in which a single bleaching treatment was used that effectively removed hemicellulose and lignin content from fibre.⁴ After bleaching treatment, the colour of the fibre changed from light brown to off white. The fibres were soaked into 5% H_2O_2 solution and heated at a temperature of 70°C for 60 min. The pH of H_2O_2 was controlled at 11 by adding 1 mol of NaOH solution. The fibres were washed using tap water until the pH value reached 7. After treatment, all the treated fibres were filtered and washed with distilled water until the pH became neutral. Then, the treated fibres were also made into a fibre mat using a wet laid method mentioned in this section.

2.3 Preparation of Composites

All composites were prepared using VARI technique. Before that, releasing polyvinyl alcohol (PVOH) gel coat was done on a $25\text{ cm} \times 25\text{ cm}$ mould base made from Perspex. The purpose of applying this PVOH gel coat was to easily remove the composite from the vacuum bag after curing. Then, the VARI process was set up as shown in Figure 2. Before the infusion process, the epoxy resin was mixed with hardener with the mass ratio of 2:1 and manually stirred for 1 min. Next, the fibre mat was infused with the epoxy resin assisted by 1 bar of vacuum pressure. Once the fibre mat was fully infused with resin, it was sealed off in a vacuum condition and left to cure at room temperature for 24 h. Lastly, the composite was removed from the vacuum bagging and post cured in an oven at 60°C for 12 h. The mass of the composite was approximately 100 g and weight ratio of epoxy/fibre was 80/20.

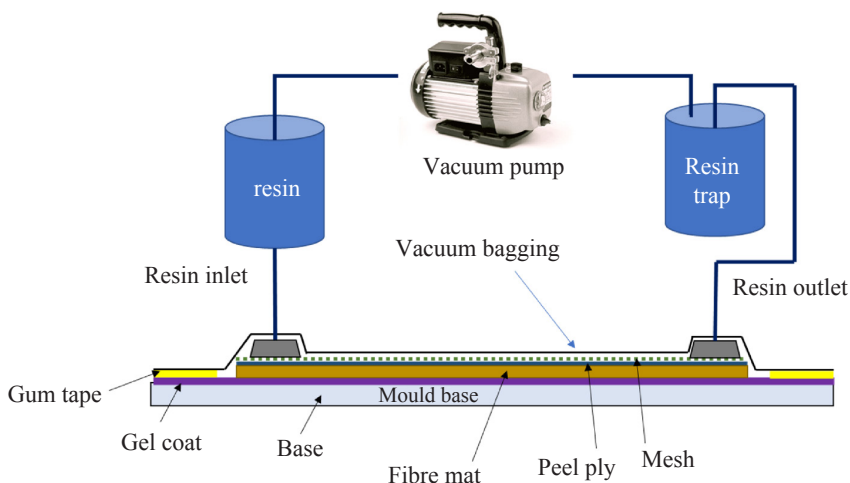


Figure 2: Setup of VARI technique for fabricating the composite.

2.4 Testing and Analysis

Fourier-transform infrared (FTIR) analysis was used to analyse the chemical components of the treated and untreated fibres. The FTIR analysis was carried out by using Perkin Elmer Spectrum 100 spectrometer machine (from Perkin Elmer, Waltham, United States) and the attenuated total reflection (ATR) technique was used. The scanning spectra range was 4000 cm^{-1} to 650 cm^{-1} with a resolution of 4 cm^{-1} . All the fibres were scanned with 32 scans.

Prior to tensile test, all the composites were cut into dumbbell specimens with dimension as stated in ASTM D638 type IV. Each of the composite sample was cut by using a laser cutter with 80% power and 150 units of speed. All the dumbbell specimens were tested for its tensile properties using Instron® Universal Testing Machine (model 5569, Norwood, United States). The testing method made reference to the ASTM D638 standard. The crosshead speed was fixed at 5 mm min^{-1} and the load cell of 15 kN was used. Seven specimens of each composite sample were tested to obtain an average result for its tensile strength, tensile modulus and elongation at break.

The tensile fracture specimens of the composites were examined for its morphological properties using field emission scanning electron microscope (FESEM) equipment, model FEI Quanta 400 (ThermoFisher Scientific, Hillsboro, United States). Prior to the analysis, all specimens were coated with an ultrathin layer of gold to avoid charging during the scan. The electron acceleration was set at 20 kV.

The water absorption test of the composite was carried out according to ASTM D570 standard. The composites were cut into dimension of $30\text{ mm} \times 30\text{ mm}$. The specimens were dried in an oven at 60°C and weighted. The weight of the dried specimens, W_d was measured using electronic balance. The specimens were fully immersed in distilled water at room temperature with various time durations of 24 h, 48 h, 72 h, 96 h and 120 h. After that, the specimens were taken out from the container and then wiped with clean dry cloth to remove excess water. The specimens were weighed again until the weight of specimens became a constant value. The electronic weighing scale was used to measure the weight of the specimens after being immersed, W_a . The percentage of water absorption of the composite was calculated using Equation 1.

$$\text{Percentage of water absorption (\%)} = \frac{W_a - W_d}{W_d} \times 100\% \quad (1)$$

3. RESULTS AND DISCUSSION

3.1 FTIR Analysis

Figure 3 displays the FTIR spectra of untreated NaOH and H_2O_2 treated CHF. Table 1 lists the main functional group of corn husk fibre. A strong and broad absorption peak in the wavelength range between 3200 cm^{-1} and 3600 cm^{-1} was observed, which was due to the stretching vibration of O-H that belongs to hydroxyl groups in fibre.¹³ Moreover, the peaks observed at 2923 cm^{-1} and 2850 cm^{-1} were attributed by the C-H stretching vibration.⁴ A peak located at 1733 cm^{-1} was observed and it was attributed to the C=O stretching of ester carbonyl vibration in lignin.¹⁴ The peak intensity at 1736 cm^{-1} has significantly reduced after the corn fibre treated with NaOH. This was because the alkali treatment has removed some of the lignin content on corn husk fibre, which was in agreement with the result by Koay et al.⁵ It can also be seen that the peak at 1733 cm^{-1} has disappeared after the corn was fibre bleached with H_2O_2 . This was due to the removal of lignin content on corn husk fibre.⁴ The removal of lignin also can observe through the change of the fibre colour.

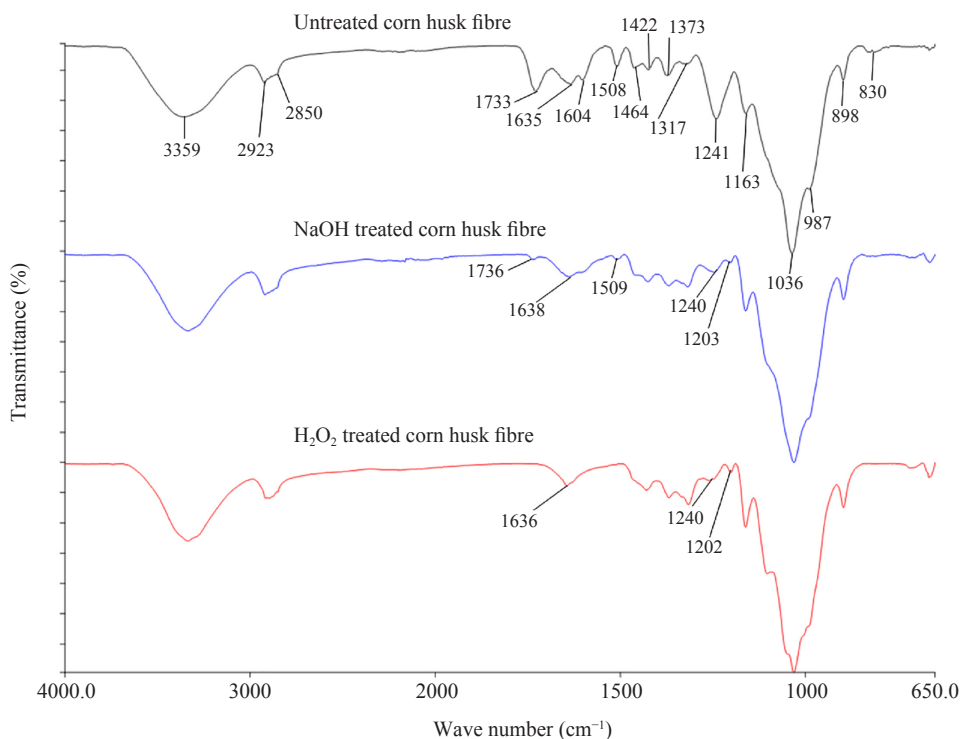


Figure 3: FTIR curves of untreated, NaOH treated and H_2O_2 treated corn husk fibre.

Table 1: Main functional group of corn husk fibre.

Wave number (cm ⁻¹)	Functional group
3200–3600	Hydroxyl groups (O-H) in fibre
2923–2850	C-H stretching vibration
1733	C=O stretching of ester carbonyl vibration in lignin
1635–1604	Carbonyl group in hemicellulose and carbonyl aldehyde in lignin
1508	Conjugated C-O group from aromatic skeletal in lignin
1464	C-H group from lignin
1442	Stretching of CH ₂ from cellulose
1373, 1317	C-H group vibration from cellulose
1241	C-O groups from acetyl group of lignin
1150–950	Anti-symmetrical vibration of C-O-C groups in cellulose
898	C-H bending of cellulose
830	C-H stretching of lignin

Figure 4 shows the brown colour of untreated specimen and the colour was changed to yellowish brown colour after treatment with NaOH solution. The change of colour indicated that part of the lignin was removed after NaOH treatment. Furthermore, the colour of specimen changed to off white after bleaching with H₂O₂ and this is because the majority part of lignin had been removed from the fibre. The peak recorded at 1635 cm⁻¹ was observed in untreated fibre, which was related to carbonyl group in hemicellulose and carbonyl aldehyde in lignin.¹⁵ The intensity of the peak was reduced after the alkali treatment, as part of the hemicellulose and lignin had been removed from the corn husk fibre. For H₂O₂ bleached corn husk fibre, the absence of peak measured at 1635 cm⁻¹ indicated that the hemicellulose and lignin were mostly removed after the bleaching process. The intensity of peak recorded at 1508 cm⁻¹ for untreated fibre also decreased after alkali treatment and the peak also absence after undergoing bleaching treatment. The intensity of peak at 1464 cm⁻¹ was also reduced after alkali and bleaching treatment. Besides, the peak found at 1422 cm⁻¹ was due to the stretching of CH₂ from cellulose. Other peaks at 1373 cm⁻¹ and 1317 cm⁻¹ were also an indication of C-H group vibration from cellulose.¹⁶ Peak found at 1241 cm⁻¹ is observed in untreated corn husk fibre, which is attributed to C-O groups from acetyl group of lignin.¹⁷ As can be seen, the intensity of the peak was remarkably reduced due to the removal of the lignin after the alkali treatment and the bleaching treatment. A broad peak range between 950 cm⁻¹ and 1150 cm⁻¹ was caused by the anti-symmetrical vibration of C-O-C groups in cellulose. Besides, the C-H bending of cellulose was also recorded at peak 898 cm⁻¹. The C-H stretching of lignin was represented by the last peak located at 830 cm⁻¹.⁵ This intensity of the peak at 830 cm⁻¹ has disappeared in the spectrum after alkali treatment and bleaching treatment. This indicated the removal of lignin from the corn husk fibre.

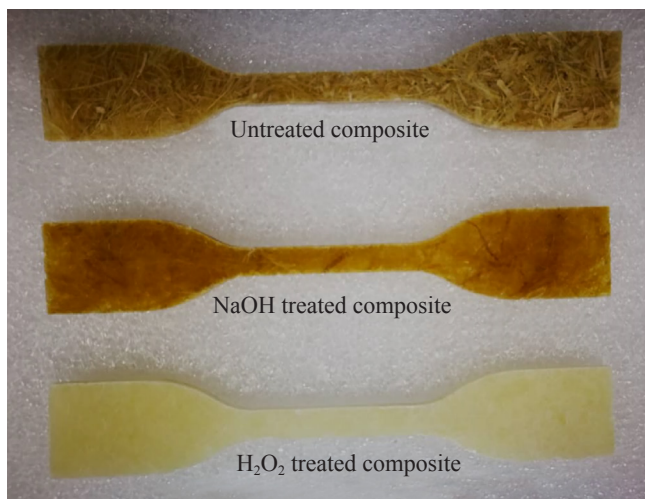


Figure 4: Specimens of untreated, NaOH treated and H_2O_2 treated composites.

3.2 Tensile Properties

Figure 5 displays the tensile strength of neat epoxy, untreated, NaOH and H_2O_2 treated epoxy/CH fibre composites. Based on the results, the untreated epoxy/CH fibre composite exhibited about 23% and 41% higher tensile strength and tensile modulus, respectively compared to neat epoxy. This indicated that the presence of CH fibre has reinforced the epoxy matrix. Koay et al. also found that the addition of the CH fibre has acted as reinforcement in recycled polystyrene based composite.⁵ However, the untreated epoxy/CH fibre composite showed higher tensile strength as compared to recycled polystyrene/CH fibre composite. This might be because the fibre mat was infused with low-viscosity resin under vacuum condition when the composite was prepared with VARI technique. As this technique was able to ensure a better wettability between fibre and matrix, the composite prepared via VARI technique will have a better fibre-matrix bonding than the composite prepared by using compression moulding. Furthermore, the tensile strength and tensile modulus of H_2O_2 treated composite were higher than untreated and NaOH treated composites. For NaOH treated composite, the tensile strength and tensile modulus were approximately 56% and 33%, respectively higher than the untreated composite. The untreated composite was expected to have lower mechanical strength compared with treated composite. This was because the untreated fibre usually exhibits weak interfacial adhesion with matrix as compared to treated fibre in composite. Moreover, the presence of micro-voids in untreated composite might act as stress concentrator that decreases the mechanical strength. The micro-voids were formed during the VARI process.

For NaOH treated fibre, part of the lignin and hemicellulose was removed from the fibre, resulting in a rough fibre surface as illustrated in Figure 6(a). The rough fibre surface was able to form better mechanical interlock after wetting with epoxy resin. Therefore, a stronger mechanical interlocking at fibre-matrix interfacial enhanced the mechanical properties of composite. As displayed in Figure 6(c), the H_2O_2 bleaching process caused fibrillation on the CH fibre. This was because most of the lignin and hemicellulose were effectively removed from the CH fibre. Thus, the treated CH fibre surface became rougher and was fibrillated in comparison with untreated fibre. Then, the adhesion between epoxy and fibrillated fibre surface was enhanced. For this reason, the H_2O_2 treated composite exhibited a better tensile strength and modulus compared to untreated and NaOH treated composites. There are several research studies reporting that the alkali and bleaching treatments would enhance the interface bonding between fibre and matrix, thus improving the stress transferring at interfacial region and eventually produces composites with better mechanical properties.^{6,9,18}

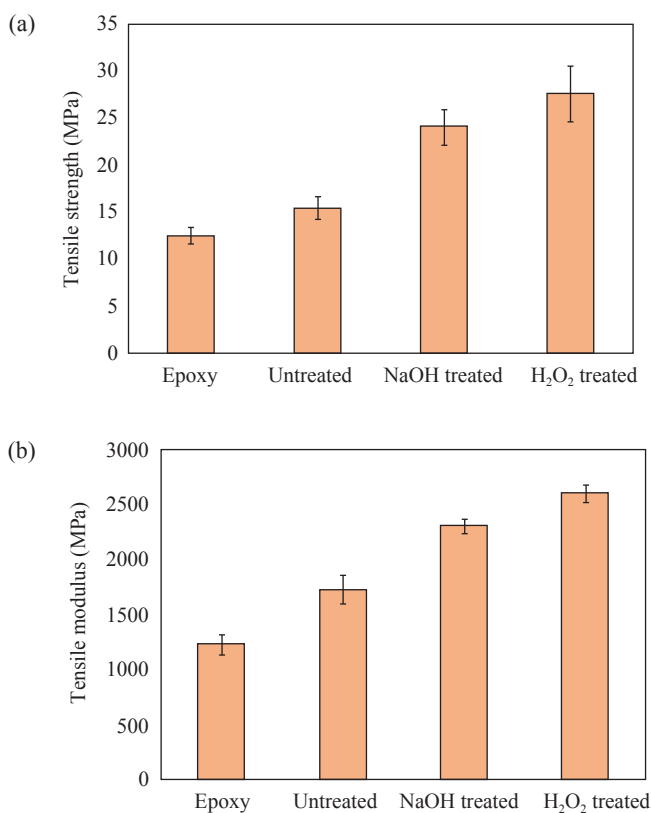


Figure 5: Illustration of (a) tensile strength, and (b) tensile modulus of untreated, NaOH treated and H_2O_2 treated composites.

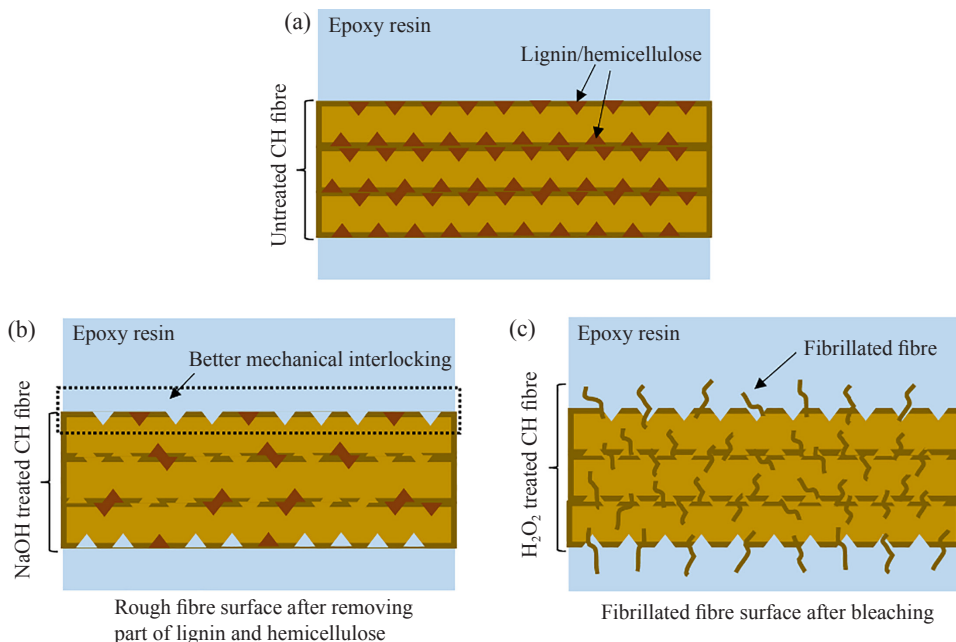


Figure 6: Proposed schematic of interaction between epoxy matrix and fibres.

3.3 Morphological Properties

Figure 7 shows the SEM micrographs of untreated fibre, NaOH treated fibre and H_2O_2 treated fibre, respectively. From Figure 7(b), the fibre exhibited a scalloped surface and surface roughness was obviously seen from Figure 7(b) after NaOH treatment. This was due to the removal of lignin and hemicellulose caused by the surface modification of the CH fibre with NaOH. Furthermore, the fibre had undergone fibrillation during bleaching treatment with H_2O_2 and this fibrillation happened due to the fact that most of the lignin and hemicellulose was removed during the process. The micro fibrils attached on the corn husk fibre were clearly observed in Figure 7(c).

Figure 8 displays the SEM micrographs of tensile fracture specimens of untreated, NaOH and H_2O_2 treated composites. According to Figure 8(a), several micro-voids were found in the specimens. Then, there was a gap between epoxy matrix and the fibre. This indicated that the poor adhesion occurs between the matrix and the untreated fibre. These observations prove that the presence of micro-voids and poor interfacial adhesion deteriorated the mechanical strength of the composite. The fibre breakage was found in fracture specimen of NaOH and H_2O_2 treated composites. This observation shows that the fibre was shared as

part of the load subjected to the composite. Besides that, there was no gap found between the matrix and the NaOH treated fibre as illustrated in Figure 8(b). This is because the NaOH treated fibre has formed a better adhesion with the epoxy matrix. Thus, the tensile strength of the treated composite was higher than the untreated composite. On the other hand, the micro-fibrils found in H_2O_2 treated composites were embedded in the epoxy matrix and the micro-fibrils breakage can be observed as well. This indicates the presence of micro-fibrils acts load bearing in H_2O_2 treated composite. For this reason, it could be explained that the H_2O_2 treated composite exhibited a better tensile strength than NaOH treated composite. All the observations from SEM analysis supported the finding in the tensile test.

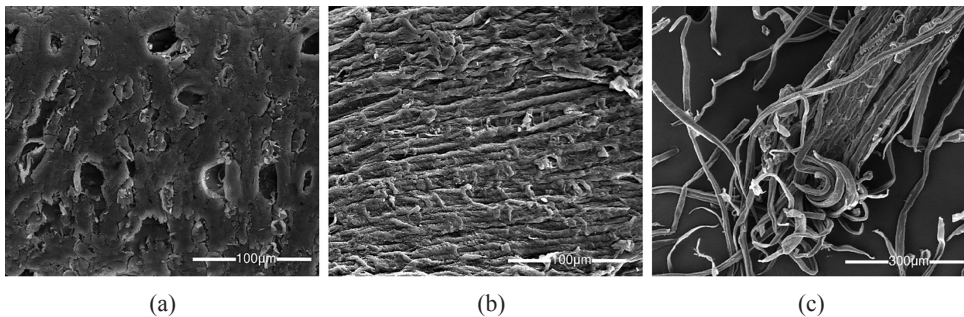


Figure 7: SEM micrographs of (a) untreated, (b) NaOH treated, and (c) H_2O_2 treated corn husk fibre.

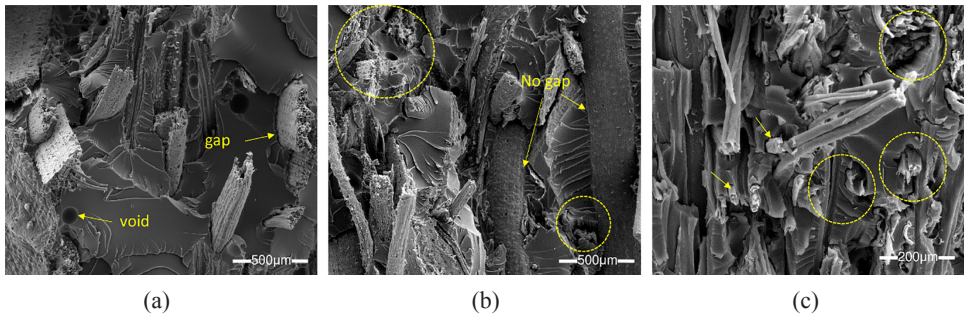


Figure 8: SEM micrographs of fractured specimens of (a) untreated, (b) NaOH treated, and (c) H_2O_2 treated composites.

3.4 Water Absorption

The water absorption of all composites was increased with the change of immersion time as displayed in Figure 9. This was due to the natural fibre itself comprising high amount of hydroxyl groups in the chemical structure. Hence, the

natural fibre might easily form hydrogen bonding with the water molecules.^{5,17} Besides, the water molecules would also diffuse into the composite and become trapped in micro gaps at interfacial region and micro-voids in the composite. From Section 3.3, there were micro-voids and micro-gap found in the untreated composite. Thus, these might be the reason for untreated composite posing a higher water absorption. Koay et al. also found that the recycled polystyrene/corn husk fibre composite shows a higher water absorption and it was due to the presence of micro-gaps caused by poor interfacial adhesion.⁵ As mentioned earlier, the NaOH and H₂O₂ treatments have significantly enhanced the adhesion between epoxy matrix and the fibre. As there had been no gaps occurring at fibre-matrix interface, less water molecules would be trapped and diffused in the interfacial region between fibre and matrix.

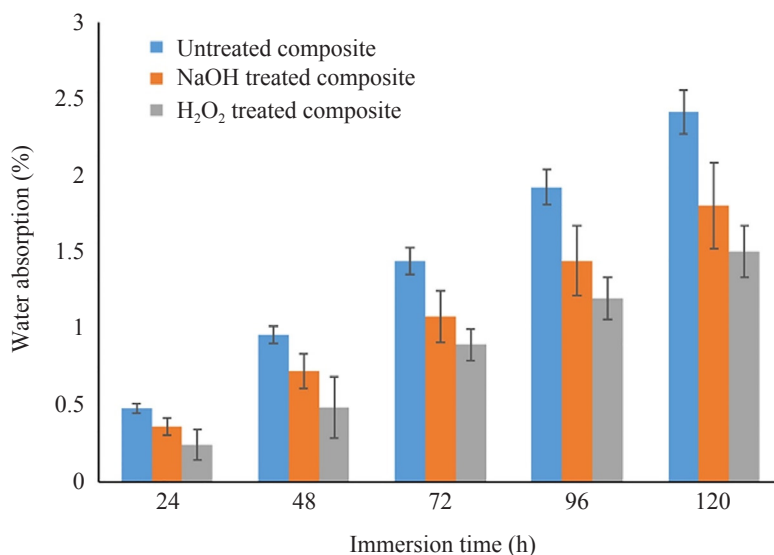


Figure 9: Water absorption of untreated, NaOH treated and H₂O₂ treated composites.

4. CONCLUSION

As a conclusion, the composite fabricated from corn husk fibre mat via VARI technique was able to produce a composite with fibre/matrix mass ratio of 20/80. Based on FTIR analysis, the hemicellulose and lignin have been removed from the corn husk fibre during alkali and bleaching treatment. It had been shown that the bleaching treatment showed better performance in removing hemicellulose and lignin compared with alkali treatment. The composite treated with H₂O₂ bleaching treatment shows the highest tensile strength and modulus among all the composites.

Both NaOH and H₂O₂ treated composites show higher tensile strength and modulus than the untreated CH composites due to the improvement of fibre-matrix interaction adhesion in the composite. The rough surface of NaOH treated CH fibre can be observed in the SEM micrograph. Furthermore, the SEM micrograph also showed micro fibrils in the H₂O₂ treated CH fibre. The SEM micrographs of both treated composites also displayed a better interfacial adhesion. This observation indicates that the better interfacial adhesion was due to the rough fibre surface and fibrillated fibre. All composites absorbed small amount of water after immersing in water for short period of time. The water absorption result shows that the NaOH and H₂O₂ treated composites have lower water absorption compared to the untreated composite. The H₂O₂ treated composites show the lowest water absorption compared with NaOH treatment and untreated composites as they only displayed no more than 1.5% of water absorption after 120 h of immersion time. Overall, the composite made from corn husk fibre mat treated with H₂O₂ bleaching treatment exhibited a lower water absorption, tensile strength and modulus approximately 28 MPa and 2605MPa, respectively. However, this composite is yet to achieve a mechanical strength like composites made from flax fibre and jute fibre. However, the epoxy/corn husk fibre composite still has the potential use in non-structural applications, such as car interior panel and seat back cover.

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