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The Physical and Morphological Properties of Kenaf Fibres/ Epoxy in Coating Treatment Process

Muhammad Mustakim Mohd Ghaztar[1], Nik Noor Idayu Nik Ibrahim[2], Sarani Zakaria[3], Ahmad Zafir Romli[2*]

[1]Faculty of Applied Sciences, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia

[2]Centre of Polymer Composite Research and Technology (PoCResT), Institute of Science, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia.

[3]School of Applied Physics, Faculty of Science and Technology, Universiti Kebangsaan Malaysia (UKM), 43600 UKM Bangi, Selangor, Malaysia.

**Corresponding author email: ahmad349@uitm.edu.my*

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ABSTRACT

Natural fibre is known useful in many applications however, the existing fibre treatment method able to reduce its overall properties. In this study, a new, simple and cost-effective fibre coating treatment method was developed which was able to improve the physical and morphological properties that open a new path for natural based materials to be used in a more robust application. The physical and morphological properties of various coated kenaf fibres were analysed to comprehend the cutting behaviour of coated fibres after subjected to the pulverisation process. The kenaf fibres were individually immersed in 1:4, 1:5 and 1:6 epoxy to acetone coating solutions prior cured, and pulverised consecutively using 5 mm, 1 mm, 0.5 mm and 0.25 mm mesh sizes aperture. The morphological characteristic was analysed using polarised optical and scanning electron microscope. The result showed that 1:6 coating ratio solution able to effectively coat the fibres' aspect ratio that forming individual coated fibre which in long length pulverised fibres. Moreover, the low viscous 1:6 solution able to penetrate inside fibre structure that supported by density and fibre cross-section analysis compare to the other solutions. In future, this analysis is

crucial to give insight on the coated fibres behaviour after subjected to the mechanical means of cutting process that later relates to the reinforcing mechanism in the composite samples.

Keywords*: kenaf fibre, pulverisation process, epoxy coating treatment, post-cutting behaviour*

INTRODUCTION

Natural fibre is the components that derives from plant-based materials that often used as a filler in the composite system since it offers numerous advantages. The abundant availability (renewable resource), easy to grow, economical as it low in price also tooling cost. It also recyclable which make it favoured choice to the users as it provides 'green' characteristics to the products produced. However, its usage is limited since the natural fibres having drawbacks that related to its variability in physical characteristics, structure deterioration, easily to degrade and low in mechanical properties.

Overall, these drawbacks are caused by the pulverising process during the materials preparation process. Usually, the fibres are pulverised into shorter length to be suited in various fabrication techniques. But the process inflicted several adverse effects to the fibres that been cut. The effect can be the fibre microstructural damages as reported by Romli and Ghaztar [1]. The research highlighted seven (7) types of damages that inflicted to the fibres after pulverisation process that influenced the composites end properties [2]. Moreover, the composites properties are expected to perform differently when using other cutting technique [3, 4].

The fibre damage might happen due to the few reasons; firstly, because of the fibres that are fed into the pulverise machine are in various position. Therefore, the fibres are being cut at any angle thus, creating various fibre damages and shapes [5]. Secondly, due to the chemical composition in the fibre. It influences the probability of the fibres to be repeatedly pulverise by the cutting knives as reported by Ghaztar and Romli [6]. It happened especially for chemically treated fibre. Therefore, to overcome the said problems, a new fibre treatment was conducted in this research that known as coating treatment.

Basically, the coating treatment is also recognised as resin treatment or flexible resin. By far, few researches were conducted that use the same treatment method [7-12]. Overall, most of the researches focus on the physical, mechanical and thermal properties improvement of the natural fibres used. But less attention given on the aspect of coating behaviour onto the fibres during pulverisation process. Especially for fibres that use pulverisation process to reduce its size prior to be use in the composite system. The study is important since to analyse the condition of the coating after subjected to the pulverise process. This study hypothesis is, coating solution ratio or viscosity plays a role in protecting the fibres from deterioration during pulverisation process.

Thus, the focus of this study is to analyse the optimum coating ratios that efficiently coat the individual kenaf fibres using physical and morphological methods. The optimum coating ratios is expected to give increment in fibres density due to the increment in coating uptake. It also protects the fibres from pre-damage as well. The fibres also become stiffer since the coating solution penetrate the fibres' structure and coat the fibres' surfaces simultaneously. In conclusion, this study is crucial to better understanding the behaviour of coated fibres especially before and after pulverisation process and fabricated in the composite system in future.

METHODOLOGY

Materials

The raw kenaf natural fibre was supplied by Terra Techno Engineering Sdn. Bhd. The received fibre was in continuous form (more than 30 cm in length). The pristine fibre density is 1.4276 ± 0.0111 gcm^{-3}. Kenaf fibre surfaces after being cut can be seen in Figure 1. The epoxy resin (CP362A) and hardener (CP362B) were manufactured by Oriental Option Sdn. Bhd. The physical and chemical characteristics of epoxy resin and hardener are tabulated in Table 1. Acetone was supplied by Classic Chemicals Sdn. Bhd. with the density or minimum essay (GC) is 95% with the molecular weight of 58.08 g/mol.

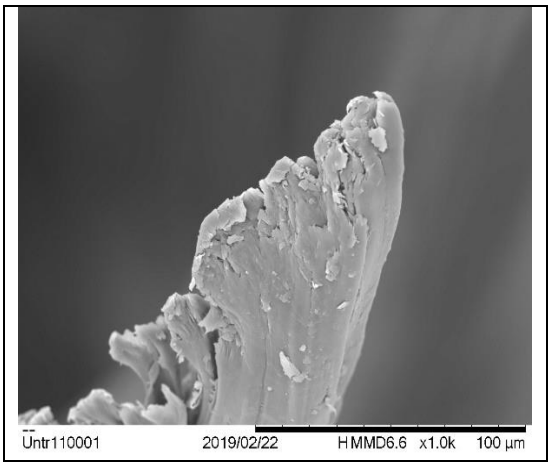


Figure 1: Scanning Electron Micrograph of Pristine Kenaf Fibre (Source by author)

Table 1: The Physical Characteristics of CP 362 Epoxy Resin

Part	Code	Chemical Type	Viscosity (cps)	Colour	Gel Time (25°C)	Post Cure (25°C)	Final Viscosity (cps)
Epoxy	CP 362A	Epoxy DGEBA	13 000	Transparent	35 minutes	9.5 hours	8500
Hardener	CP 362B	Modified Polyamine	400	Transparent			

Source: All Purpose Epoxy [13].

Fibre Coating Process

Received raw kenaf fibre was conditioned at ambient temperature for 48 hours prior to the coating process. In the meantime, the coating solutions of epoxy: acetone at 1:4, 1:5 and 1:6 ratios of coating solutions were prepared. Then, after 48 hours, the conditioned-kenaf fibre was immersed in the prepared coating solutions at room temperature for five minutes.

The fibre coating process was conducted in three stages. In the first stage, three (3) batches of kenaf fibre bundle were dipped into the coating solutions between one to 20 minutes. For the second and third stages, another

three (3) batches of kenaf fibre were immersed into the coating solutions for each 20 to 40 minutes and 40 to 60 minutes, respectively.

All the fibre batches were weighted before the coating process and the weight was recorded. These steps were carried out for the three different coating solutions. This process was conducted in a control condition due to the high volatility of acetone. After being immersed for five minutes, the fibre was taken out, tossed and the fibre that initially in bundle were separated into single fibre. Afterward, the fibre was cured in an air circulated oven at 80 °C for 24 hours. After 24 hours, each fibre batch from each stage were weighted and recorded. The weight difference of kenaf batches before and after the coating process was calculated and presented as the percentage of weight uptake.

$$\text{Weight Difference (\%)} = \frac{Wi - Wo}{Wo} \, x \, 100\% \qquad \text{Eq. 1}$$

Where, Wo is the fibres weight before coating and Wi, is the fibres weight after coating treatment.

Pulverisation Process

After 24 hours of the curing process, the fibre was cut using a pulveriser machine (Fritsch Power Cutting Mill PULVERISETTE 15) passing thru 5.0 mm, 1 mm, 0.5 mm and 0.25 mm pulverise mesh sizes, consecutively. The fibre was added bit by bit into the cutting chamber for an effective pulverising process. The pulverised fibre collected inside the receiving bowl then weighted by batches. The different in weight before and after the pulverisation process was calculated and presented as percentage weight decrement (refer Equation 1).

Density Test

The bulk density of all fibres was determined using pycnometer bottle based on ISO [14]. The density test was conducted at two (2) fibre parts of the un-pulverised coated kenaf fibre stand. The first part was at both ends

(25% from fibres ends) and the second part at fibres middle area of fibres. The average length of the cut fibres is 3.83 ± 1.44 mm. The parts taken from the coated fibres were illustrated in Figure 2.

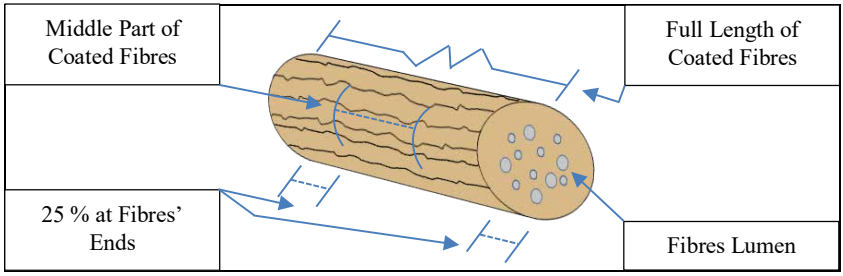


Figure 2: The Illustration of Physical Features of Kenaf Fibres and the Parts Taken for Density Test (Source by Author)

Another density test was conducted for pulverised fibre passing thru 0.25 mm mesh size aperture. To begin, the weight of the overall bottle (including lid), and 1 to 3 grams of fibres were dry weighed separately. Then, the density of the oil, the weight of overall bottle with and without the fibres in the immerse oil were measured. The coated fibres density can be calculated as in Equation 2.

$$\text{Bulk Fibre Density (g/cm}^3\text{)} = \frac{Wd}{Woil - Wim}\ x\ \rho oil \qquad \text{Eq. 2}$$

Where, Wd is the dry weight of the fibres. Woil is the weight of oil without fibres whereas, Wim is the weight of oil and fibres inside the overall bottle. The ρoil is the density of the oil.

Characterisation Analysis (Polarised Optical microscope and Scanning Electron Microscope)

Coated fibres that were pulverised through 5 mm mesh size was collected and scanned using Epson L220 scanner. The images then, further magnified to observe the individual fibre length and coating behaviour. The 0.25 mm mesh size pulverised fibres were magnified using optical microscope Olympus BX51 as referred to ASTM F728-81 (1997) [15]. The morphology of coated fibre was observed by using Scanning Electron

Microscope (SEM) (Hitachi TM3030 Plus, USA) at 5.0 kV acceleration voltage as in ASTM E766-14 (2014) [16].

RESULT AND DISCUSSIONS

Fibre Coating and Pulverisation Process Analysis

Figure 3 shows the graph of percentage of fibres weight for every batch of coating process. In general, all fibre ratios experience weight increment after coating process. The fibre weight increment might be occurred due to the coating that absorb and attached into the fibre structure after curing process. It is also recorded that all coating ratios exhibited increment in coating uptake for every stage. This might be contributed by the increment in 3-Dimensional (3D) molecular formation inside the coating dilution as the time increased [17].

Since the coating dilution was using acetone diluent to thin the epoxy matrix, the short-branched molecules of epoxy scattered away from each other and cannot form 3D network. Hence, during the coating process, the epoxy coating that deposited and penetrated thru the fibres surfaces [18] during the first stage was less compared to the second and third stages.

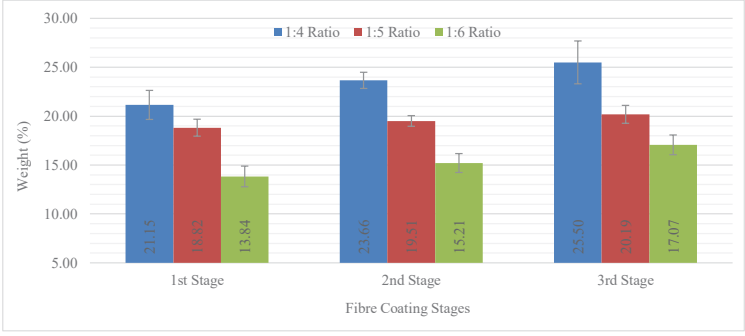


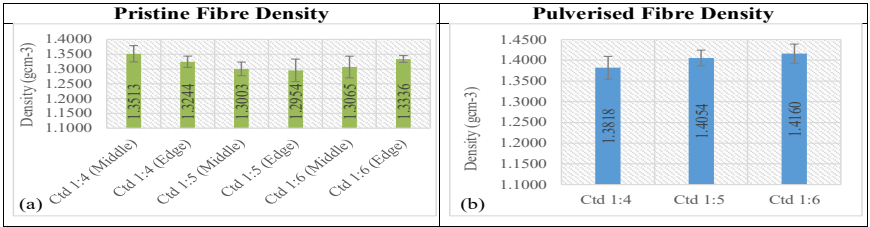
Figure 3: Fibre Weight Increment after Coating Process for Every Batches of 1:4 and 1:5 and 1:6 Coating Ratio Solutions

From the aspect of different coating viscosity, 1:4 dilution recorded the highest weight uptake compared to the 1:5 and 1:6 dilution. This might be due to the influence of the coating ratio or viscosity itself. As the amount of acetone increasing from 1:4, 1:5 and 1:6, the capability of the short chains molecules of epoxy to form 3D network is reducing. Since the viscosity of the 1:6 ratio is the lowest, it showed the lowest coating uptake for every stage of the coating process.

When the viscosity of the epoxy is low, the chains of epoxy are unable to form a linked-network due to the effect of gravitational forces [19]. This resulted in the dripping of coating dilution from the fibre bundle during the curing process leaving only thin coating layer on the fibre surface. Moreover, the diffusion of coating dilution inside the fibre's structure will also reduce. In addition, it was observed that, after pulverisation process, there are reduction in fibre batch weight. It might be due to the turbulence that created by the cutting knives that flew away the low dense fibre out from the pulverise bowl [20].

Fibre Density Analysis

Figure 4 shows the density of coated kenaf fibre before and after the pulverisation process taken at the middle and end parts of the fibres. It is recorded that 1:4 coated fibre having highest density at both fibres part as in Figure 4 (a). It might be happened due to the influence coating uptake combine with higher amount of hemicellulose [21] in the fibre structure. Since, the low amount of acetone unable to completely remove the constituent inside the fibre thus, the 1:4 coated fibre exhibit some of the uncoated or pristine fibre density.



Note: The pristine kenaf fibre density is 1.4276 ± 0.0111 gcm^{-3}.

Figure 4: The (a) Un-Pulverised Fibre Density for coated 1:4, 1:5 and 1:6 Fibres at Middle and Edge Parts. (b) The Pulverised Coated 1:4, 1:5 and 1:6 Fibres Density

In addition, the trend of density for coated 1:5 is decreasing but increasing for coated 1:6 fibres at both fibres' parts. It might be due to the higher hemicellulose removal from the fibres as higher amount of acetone used. The density for 1:6 coated fibres is higher since the fibre might starting to be dominated by the coating. Since high amount of acetone might create bigger lumen size inside the fibre thus it allows higher amount of coating solution to flow and cured inside the fibre especially at its ends.

However, the density of pulverised fibres as in Figure 4 (b) showing increasing density pattern from 1:4 to 1:6 coated fibres. It might be due to the higher amount of coating that coated and absorbed in the fibre structure. The coating was not detached during the pulverisation process that contributed in fibres weight which led to increment in its density.

Fibre Characterisation Analysis

Figure 5 shows the coating behaviour of 1:4, 1:5 and 1:6 pulverised coated kenaf fibres. It can be seen that, the 1:6 coated fibres successfully coat the fibre individually as shown in Figure 5 (c) as the fibre form long fibre form, mangle shape of fibre end [22] and consistent fibre width at all parts of the fibre after pulverisation process. However, the 1:5 coated fibre appeared to have few fibres that attached together that having thicker width as well as the fibre's end forming a granular fracture failure [22]. Whereas, the same situation also happened to 1:4 coated fibres as more fibres are being coated together, thicker in width and having some fibre detached from the coated fibres.

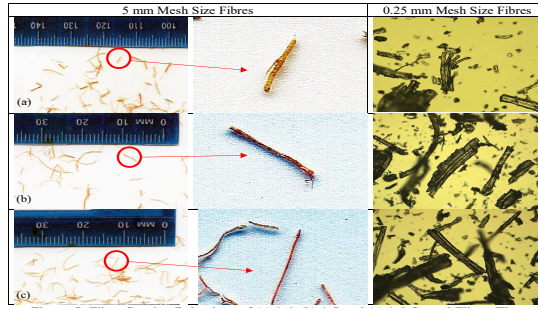


Figure 5: Fibre Coating Behaviour of (a) 1:4, (b) 1:5 and (c) 1:6 Coated Fibres That Pulverised using 5 mm and 0.25 mm Sieve Sizes Captured via Epson L220 Scanner and Polarised Optical Microscope using Bright Field at 20x Magnification (Source by author)

This analysis signifies that, coated 1:4 and 1:5 are unsuccessful to coat the individual fibre that is a requirement to proceed in the composite system where in theory, an area of the fibres that not being coated cannot protect the fibre from deformation. Its physical, mechanical and thermal properties are expected to reduce as some of the fibres bundle's area are not protected by the thin layer of epoxy. In order to further study the surface coating formation and absorption, a scanning electron micrograph for coated fibre is shown in Figure 6.

It signifies that in coating process, it gives variability of coating behaviour especially in different coating ratio or viscosity as the lowest coating solution able to absorb into the fibre's lumen and creating low fibre porosity (refer Figure 6 (d)). In addition, the inconsistency in fibre structure and coating solution exposure to the fibre makes some of the area inside the fibre was not being coated. The coated fibre structure can be compared to the uncoated fibre structure as in Figure 6 (a).

In the aspect of coating, there are some inconsistency since the coating thickness is not identical at all aspect ratio of the fibre (refer Figure 6 (b) and (c)) as it depends on the coating viscosity and its ability to form individual fibre coating. The inconsistency of the coating extends to the formation of porosity inside the coating as it might be due to the presence of the acetone that evaporated (56 °C boiling point) during the drying process [23] and leaving the coating with porosity once it cured.

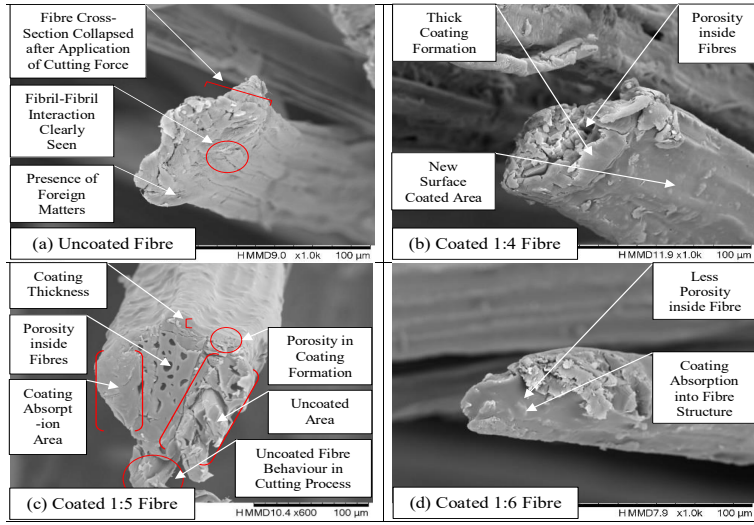


Figure 6: Scanning Electron Micrograph of (a) Uncoated, Coated (b) 1:4, (c) 1:5 and (d) 1:6 Kenaf Fibres (Source by author)

CONCLUSION

The effect of coating process (ratios and coating stages) to the pulverisation process behaviour were studied. The coated fibre behaviour after the pulverisation process were considered. It is expected that successful coating process can reduce the inconsistency in physical and morphological properties. The results show that 1:6 coating ratio solution able to penetrate and coat the fibres individually, produce longer fibre length after pulverisation process. It produces high density of fibres as well. Vice versa for 1:4 and 1:5 coating solution as the fibres create bundle of coated fibres, shorter fibre length, and lower fibre density recorded. In conclusion, by correlating the fibre cutting behaviour towards coating ratios and coating stages, the reinforcing mechanism of pulverised coated fibres inside composite system can be fully comprehended.

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