EnvironmentAsia
The international journal by the Thai Society of Higher Education Institutes on Environment

EnvironmentAsia 12(3) (2019) 129-139
DOI 10.14453/ea.2019.50
ISSN 1906-1714; ONLINE ISSN: 2586-8861

The Effect of King Pineapple Leaf Fiber (*Agave cantala* Roxb) Fumigated Toward the Fiber Wettability and the Matrix Epoxy Interlocking Ability

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Received: November 19, 2018; Revised: February 1, 2019; Accepted: June 8, 2019

Abstract

The objective of this research is to determine the epoxy matrix ability in wetting a king pineapple leaf fiber (*Agave cantala* Roxb) and the epoxy matrix bonding ability to a pineapple leaf fibers king (KPLF) as a result of the fiber fumigation treated. The droplet contact angle testing is the way to find out the fiber wettability. Measuring the contact angle is a simple method to determine the interface compatibility of two interlocked surfaces. The contact angle between the epoxy matrixes and the KPLF measurement was performed with a Mitutoyo digital microscope and analyzed by the Image Pro software. KPLF without and with fumigation treatment was observed by SEM to determine the ability of KPLF interlock with the epoxy matrix. The measurement result shows the effect of KPLF fumigation toward the fiber wettability. The epoxy matrix droplets on the KPLF surface has a contact angle of less than 30º, which means that the epoxy matrix has a good ability in wetting the KPLF fiber surface and would affect to the interlock ability between the KPLF and epoxy matrix. The interface shear stress (IFSS) matrix epoxy with KPLF increased by 282.8 % for the fiber fumigated for 15 hours.

***Keywords**:* King pineapple leaf fiber, Fumigation, *Contact angl*, Shear stress

1. Introduction

Natural fiber has already given many advantages in the past few years. Fiber can be used as a composite filler.The advantage of using natural fibers as composite material reinforcement compared to synthetic fiber is that natural fiber are cheap, has a low density, easily separated, widely available, renewable, biodegradable and environmentally friendly (Mu *et al*., 2009; Li *et al*., 2007).As a consequenze, there is a more motivation to explore new natural fiber that can be applied in the fields of automotive and structural construction to replace synthetic fibers.

The problem that arises with the development of composite materials technology is how to utilize the materials that is broadly available. The resources are enormous to anticipate the material crisis, mainly plastic in which the availability is depending on the oil resources which is non-renewable (Rowell, 1999). One the problem solving is to utilize the natural fiber as a mixture of plastic polymer to produce a composite material that can be used extensively for engineering applications, either structural or non structural (Joseph, 1999). Natural fiber that is hydrophilic has a compatibility weakness with the polymer matrix which is hydrophobic. This weakness can be overcome by providing a chemical treatment include alkali treatment that can eliminate most lignin, hemicellulose, wax or oil to make the fiber surface become rough.This fiber surface roughing would increase the fiber compatibility when combined with an epoxy matrix (Ahad *et al.*, 2009; Carvalho *et al.*, 2010). The interlocking between the fiber with the matrix also would influenced by the void which is formed between the fiber with the matrix.In addition, some other factors that affecting the composites strength is the fiber form, fiber position and fiber length (Drzal, 2003).

The matrix ability, either the thermoset orthe thermoplastic group, in wetting the fiber surface optimally is one of the main key to determine the composite material performance (Drzal, 2003; Rider, 1998). The matrix ability to wet the fiber surface is mentioned as wettability. Besides wettability, adhesion and interlocking mechanism between the fibers with the matrix will directly influence the interfacial shear strength. The wettability or unable wetted liquid on a solid surface is measured using a simple contact angle system. The contact angle between the matrix and the fiber surface at an angle of less than 90 ° is grouped as a wetted ability group, while for the contact angle greater than 90° is grouped as a not able to wetted group. If the liquid matrix does not form an epoxy droplet to the fiber surface, it would be called as no wettability, because there is no relationship between the contact angle and the fiber surface with the matrix. The smaller the contact angle the better the wettability, so that the matrix as a medium of fiber adhesive must have the ability of optimally coating the fiber surface area and the contact angle generated by the optimum ability to wet the fiber is not more than 30° (Dron, 1994; Tammar *et al.*, 2004).

The fiber interface interlocking affects the fiber composite strength. The interface on a composite is a surface that is formed together from the fiber and the matrix which form an intermediary bond needed for the load transfer. Some of the main factors that affecting the bond between the fibers and matrices are: (i) the physical and chemical adhesion, (ii) the mechanical components interlocking and (iii) friction (Tammar *et al*; Yang *et al.*, 2003). A good interface can transfer the load from the matrix to the fiber perfectly so that the composite strength could increase (Yang *et al.*, 2003). One of the important things to be noted to get a composite strength or toughness is the interface interlocking capability between the fiber and matrix (Yang *et al.*, 2003; Reddy and Nang, 2005).

The method for determining the fiber interface strength with the matrix is by doing a pull-out test. The pull-out single fiber test is expected to provide the information about the direct interaction on the fiber interface with the matrixand would an indicator of the fiber micromechanica stress transfer where the better the stress transfer ability to transfer stress produces higher interface shear strength (Drzal, 2003; Rider, 1998, Khalil *et al.*, 2001). The single fiber pull out test has become one of the representative methods and become an important experimental technology in the composite fiber mechanical behavior study (Drazal, 2003; Yang *et al.*, 2003; Silva *et al.*, 2011). The interface nature plays an important role in analyzing the mechanical behavior of composites with natural fiber reinforcement. Natural fiber characteristics normally investigated experimentally by examining some of the parameters such as the physical, chemical and mechanical nature. The natural fiber resources is quite a lot in Indonesia and one of them is the king pineapple leaf fiber (KPLF).

The king pineapple leaf fiber has long been used by people in Tana Toraja and North Toraja, South Sulawesi Province and is usually used for the straps and woven for clothing, especially for corpses wrapping. Culturely in Tana Toraja and North Toraja where straps and clothing made from fumigated king pineapple leaf fibers is used to strengthen and make it durable (Palungan *et al*., 2015).

Pineapple leaves are not utilized after harvest. It is hoped that this research will become an alternative use of pineapple leaves after harvest. Given the enormous potential of the king pineapple leaf fiber as a fiber source then it is necessary to increase its role, not only as a traditional product, but also improve the function into a natural fiber composite reinforcing materials (Palungan *et al*., 2016). KPLF fiber has a length that is greater than the others so that it can be used in making various composites.

Based on the above description generally natural fiber surface treatment using alkali and other chemicals and as an alternative treatment is using afiber surface fumigation treatment implementing the coconut shell smoke. The smoke as a result from coconut shell burning contains carbonyl compounds, phenols, acetic acid and other chemical compounds (Ruiter, 1979).Under a fumigation process a water withdrawal and various chemical compound deposition from the smoke occurs so that it can change the fiber chemical, physical nature because the carbonyl compounds serve to contribute to the fiber surface discoloration become brown yellow to dark brown color. The phenolic compounds functions as an antioxidant and antimicrobial properties so that the fiber fumigated would be durable (Ruiter, 1979; Girard, 1992). The fumigation process can also reduce the fiber lignin and hemicellulose due to the acetic acid that breaks the lignin chain and also penetrates the hemicellulose chains so that the fiber surface becomes rough (Girard, 1992; Palungan *et al*., 2017; McDonough and Shaw, 2003), because lignin and hemicellulose are amorphous while cellulose is a chemical element that is crystalline (Suryanto *et al*., 2014; Palungan *et al*., 2017; Marsyahyo, 2009; Mahato, 2009).

Thus the KPLF fumigation process is predicted to play a role to change the fiber surface topography and would increase the mechanical strength.So a research on fumigated natural fiber cellulose should be done especially king pineapple leaf king.The research result should be supported by a series of test to find out the effect of fumigation on the fiber wettability, the KPLF interface bonding with the epoxy matrix also the compatibllity with epoxy matrix and finnaly test the fiber composite shear strength (IFSS) under a pull out test.

2. Material and Method

2.1 Fiber materials

The materials used in theis research is king pineapple leaf fiber obtained from Tana Toraja in South Sulawesi, Indonesia. The 11 months old king pineapple leave was chosen for the research sample. These leaves were cut from the stamps with a length of 1 m. These cuted leaves was then immerse in fresh water on the batch for three weeks. After immersion, decay occurs on the leaves. Fibers are removed from the rotted leaves mechanically and the KPLF length is in a range of 0.9 - 1m. Furthermore KPLF was cleaned from dirt by washing it with distilled water. The KPLF was then dried up on the open air for 1 hour and finally was grouped and classify for the fumigation treatment.

2.2 Fumigation treatment

KPLF fumigation treatment was carried out in the fumigation box where the smoke obtained for this fumigation from burning coconut shells in a container where the container is connected by pipes to drain the smoke continuously into the fumigation box. KPLF fumigation process was performed for 5, 10, 15, and 20 h and and the fumigation temperature in the chamber is abouy 45°C (Palungan *et al*., 2015). Each KPLF sample without and with fumigation treatment is notated in table 1.

2.3 Epoxy resin material

Epoxy resin used in this research is diglycidyl ether of bisphenol-A (DGEBA) which is the reaction between bisphenol-A with epichorohydrin mixed with methyl ethyl katon peroxide catalyst (MEKPO) in a liquid form and translucent.

2.4 Wettability measurement

A flexible natural single fiber material was laid on a U-shaped profilejig. Liquid epoxy resin which is mixed with the a 1% catalyst is inserted into the syringe tube and then dripped on the KPLF surface, which illustrating that the contact angle between the KPLF fiber surface and epoxy matrix formed by the normal force of the solid surfaces with the liquid surface. The contact angle formed by the solid surface and tangent toward the liquid epoxy surface and the angle between the line of solid-liquid and liquid-vapor as shown in Figure 1. The contact angle measurements performed using a digital microscope with a magnification of 500 times which is connected directly to a computer and animage photo was recorded. This epoxy droplet and KPLF photo was then transferred or copied using the Image Pro software. The smaller the contact angle (θ), the better the fiber wettability, wherein the matrix as a fiber adhesive medium must have the ability to coat the fiber surface area which results an optimal wettability which is not more than 30 ° (Rider, 1998; Dron, 1994). Contact angle (θ) represents the difference between the solid-vapor surface energy with the solid-liquid which presented in the Young equilibrium equations (Tammar *et al*., 2004) where, solid-vapor tension surface layer (γsv), the solid-liquid surface stress (γsl), and the liquid-vapor surface tension (γlv) is presented as follows:

$$\cos\theta = \frac{\gamma sv - \gamma sl}{\gamma lv} \quad (1)$$

2.5. Pull out testing

The pull-out test was conducted to determine the adhesion capability between KPLF and epoxy matrix by planting the fiber into the epoxy matrix. The fiber-planted in the matrix was then drawn by implementing a specified load until the fiber is pulled out from the dry epoxy matrix or the fiber is broken off. In simple terms, Figure 2a shows the pull-out test procedure that is planted in the single fiber matrix with a planting depth lo and subjected to an axial tensile load of P. P load are expected to revoke the fiber embedded in the matrix which is assumed that the shear stress along the fiber surface is uniform and form an equilibrium force. Shear stress (IFSS) between the fiber and matrix as seen Figure 2b could be calculated using the equation:

$$\tau = \frac{P}{\pi . d . l_o} \quad (2)$$

Where P is the maximum load, d is the fiber diameter and l_o is the length of the fiber embedded in an epoxy matrix.

3. Results and Discussion

From the contact angle measurements of each epoxy droplet with KPLFunder several variations fumigation treatment time, there is no contact angle result found greater than 40^o The longer the fumigation time the smaller the contact angle (θ) between the epoxy matrix with KPLF compared whith the KPLF without fumigation as seen in Figure 3.

On Figure 3 it is clearly seen that the KPLF without and with the fumigation treatment has a different color, it is due to the deposition of chemical compounds contained in the burning coconut shells smoke which is carbonyl. Carbonyl has the contribution of KPLF surface discoloration. Other chemical compounds would change the KPLF chemical and physical properties (Ruiter, 1979). KPLF fumigated also affects the contact angle between the surface SDNR epoxy matrix changes. This happens because the epoxy matrix has a good wettability so that the epoxy liquid droplets spread evenly covering the KPLF surface contour.

The longer the fumigation time the smaller the contact angle (θ) between the matrix epoxy and KPLF.The proper and optimal contact angle (θ) between the matrix and fiber is not more than 300 (Dron, 1994; Tammar *et al*., 2004). Thus the KPLF fumigated would result from good effect, because an interface interlock occurs due to the adhesion bond that resulting a contact angle and Cos θ between the epoxy matrix with KPLF as big as 23.230 and 0.92 in F20H as shown in Figure 4.

Figure 5 is the single fiber KPLF pull out test result and it is clearly seen that the effect fumigation on shear strength between

Table 1. King pineapple leaf fiber sample group name

Fiber sample group	Information
KPLF	King pineapple leaf fiber
WF	Without fumigation
F5H	Five hours fumigation
F10H	Ten hours fumigation
F15H	Fifteen hours fumigation
F20H	Twenty hours fumigation

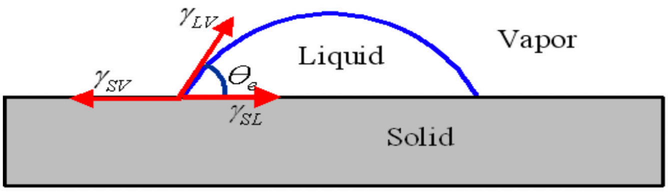


Figure 1. The contact angle illustration (Dron, 1994)

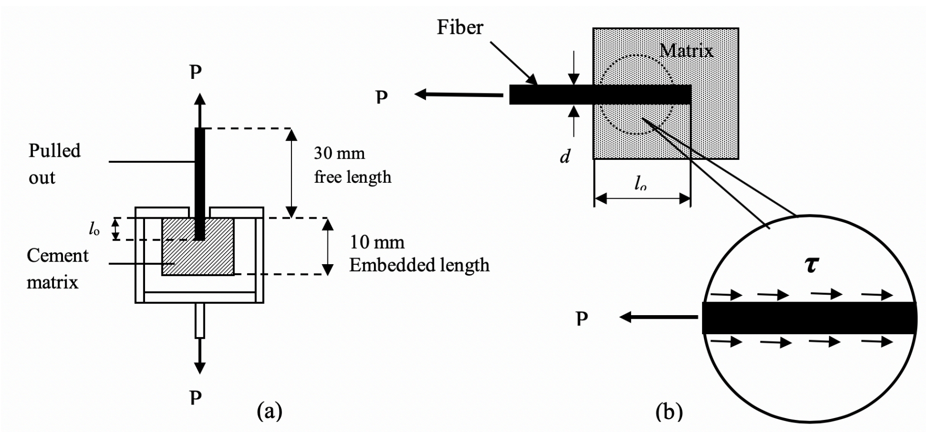


Figure 2. Pull-out test mechanism (a).Single fiber pull-out specimen (b).Interfacial strain and shear balance between the fiber surface and epoxy matrix (Palungan, 2016)

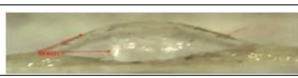
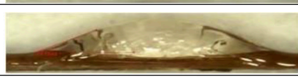
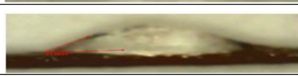
No	KPLF Sample	Figure	Contact angle	
			θ (°)	Cos θ
1	WF		36.45	0.80
2	F 5H		32.72	0.84
3	F10H		30.52	0.86
4	F15H		29.36	0.87
5	F20H		23.22	0.92

Figure 3. Matrix epoxy droplet on the KPLF surface forming a contact angle

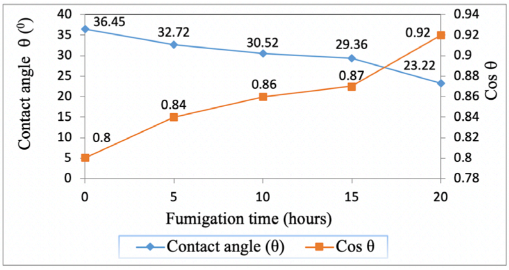


Figure 4. KPLF fumigation time toward the contact angle size and Cos qepoxy matrix droplet

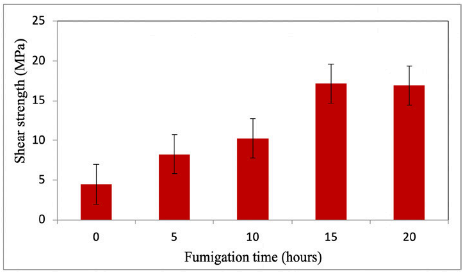


Figure 5. Shear strenght vs Fumigation time

KPLF and epoxy matrix when compared with KPLF without fumigation. The optimum shear strength was reach on the 15 hours fiber fumigation and relatively decreased until the 20 hours fiber fumigation as seen as follows:

The fiber surface conditions associated with the interlocking epoxy matrix interface surface with KPLF was influenced by the fumigation time as shown in Figure 6. In Figure 6a, the KPLF condition without fumigation which is planted on the epoxy matrix seen has a large gap as indicated by arrow 1. In Figure 6b KPLF with F5H planted on an epoxy matrix showing the interlocking condition with a lesser gap (arrows 1), when compared with KPLF without fumigation. Figure 6c shows KPLF with F10H plant in an epoxy matrix that showing the interlocking with a smaller gap (arrow 1) and appear to have an irregular surface that could lead a strong mechanical adhesion between KPLFand epoxy matrix.

Figure 6d shows KPLF with F15H embedded in a matrix of epoxy that shows a strong mechanically engagement for their bonding adhesion so that the gap does not appear anymore (arrow 1), as well as in the Figure 6e with F20H show the KPLF that plant in the matrix epoxy and the interlocking is relatively similar with the F15H with a strong mechanical interlock, (arrow 1), but it seems that the KPLF surface is cracking (arrow 2), this is happening because of the strong engagement between KPLF and epoxy matrix.

Based on the KPLF interlocking condition without fumigation treatment compared with the KPLF fumigated seems a significant differences engagement. This is happening because of the surface fumigation treatment effect toward the KPLF surface. Fumigation treatment may reduce the lignin and hemicellulose due to acetic acid contained in the coconut shells make. Acetic acid can break the lignin chain and also penetrates the hemicellulose chains so that the fiber surface becomes rough (Ruiter, 1979; Girard, 1992; Palungan *et al.*, 2017). It can trigger the matrix epoxy to wet the KPLF surface, resulting in a smaller contact angle between the matrix epoxy with KPLF as shown in Figure 4. Also in the fumigation process, water was withdrawn from KPLF resulted in KPLF water level decreased (Palungan *et al.*, 2015). So that the KPLF surface was perfectly wetted by the matrix epoxy and occurs a strong mechanical bond because their bonding adhesion resulted from an engagement between KPLF the matrix epoxy which is compatible.

Figure 7 (a, b, c, d, and e), is the KPLF without and with fumigation treated cross-sectional SEM result. The KPLF surface without fumigation (WF) looks smooth and without pores as seen in Figure 8a. After the fumigation treatment, the KPLF surface appears open pores are indicated by the yellow arrow, as seen in Figure 7(b, c, d, and e). This is happening because during the smoking process takes place water in the KPLF was evaporated. The pores seen on the KPLF surface originally was occupied by the water molecules and then filled with the chemicals contained in the smoke (Michailof *et al.*, 2008; Arsyad *et al.*, 2015; Swastawati and Frothea, 2008).The KPLF physical properties would be better as a result of the KPLF moisture content reduced (Palungan *et al.*, 2017). This would cause a denser KPLF as seen in Figure 7(d-e).

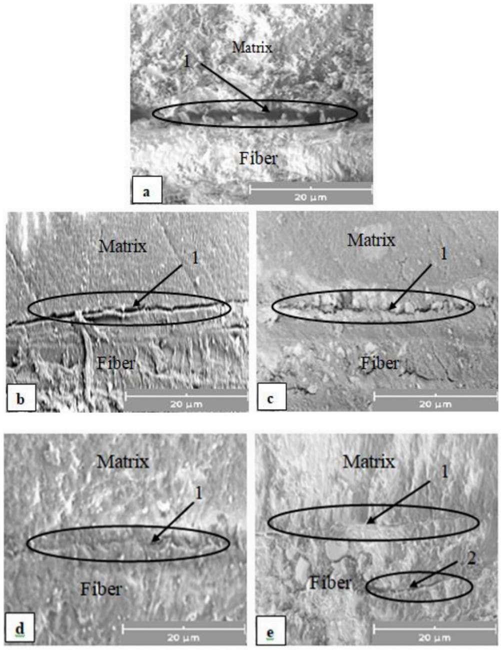


Figure 6. SEM for the KPLF interface interlocking with epoxy matrix(a). WT, (b).F5H, (c).F10H, (d).F15H, (e).F20H

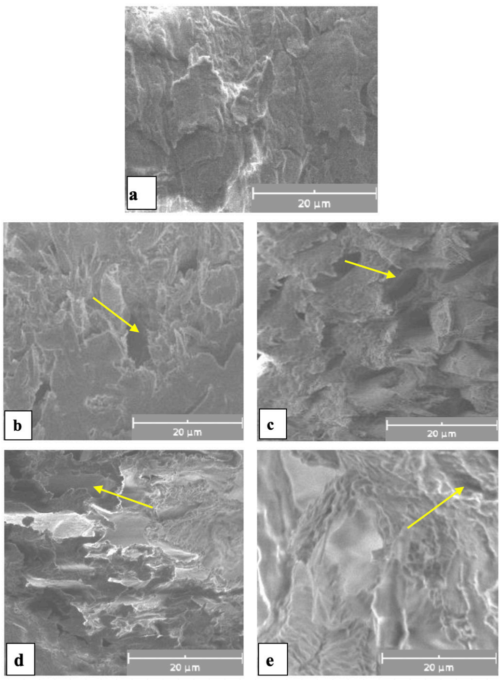


Figure 7. KPLF cross-section SEM result, (a).WT, (b).F5H, (c).F10H, (d).F15H, (e).F20H

The pores formed at the KPLF surface can facilitate epoxy matrix adhesion with KPLF resulting in a strong bond.

In Figure 8(a, b, c, d and e) respectively, are the pull out test results of the single fiber up to detached. It could be seen that there are many diferent matrix epoxy cross sections. Figure 8a is the KPLF without fumigation and Figure 8(b-e) is the KPLF with fumigation under different fumigation times. The KPLF detached from epoxy matrix, where on Figure 8a the fiber surface looks smooth, while on Figure 8(b-e) the KPLF detached surface has an irregular surface as indicated (yellow arrow) as follows.

The matrix surface condition, which is associated with the KPLF interface interlock with the matrix epoxy could be seen in Figure 8a.When associated with Figure 6a it is seen that there is a less good interlock because the KPLF surface was not coated with the matrix epoxy so that there is a large enough gap, indicated by arrow 1. So that the KPLF embedded in an epoxy matrix, could easily be uprooted as seen on the pull out test Figure 8a.

Figure 8b shows KPLF with F5H taken off from an epoxy matrix and appears a better interlock. It is indicated from the uneven epoxy matrix peripheral cross section compared to fibers without fumigation. Figure 8c shows the KPLF with F10H, where the KPLF was unrooted from the epoxy matrix and show a better interlock because. It is indicated from the irregular fiber peripheral surface as seen in Figure 6c. Figure 8dshows KPLF with F15H, unrooted from the epoxy matrix and seems very strong and has the same condition when associated with Figure 6d.

Due to the strong interlock where the epoxy matrix was also uprooted and the epoxy periphery surface of the epoxy was completely damaged. Figure 8e shows the KPLF with F20H was uprooted from the epoxy matrix and appear relatively similar with the KPLF with F15H condition, but there appears a fracture as shown in Figure 6e and affecting to be lowering the KPLF mechanical properties.

Based on the single KPLF pull out test results as shown in Figure 5, showing an increase in shear strength between KPLF and epoxy matrix until F15H and relative decline in F20H. This phenomenon occurs due to the fumigation treatment that affecting the epoxy matrix wetted changes as an adhesive medium that has the ability to coat the fiber surface area optimally to produce a contact angle of not more than 30° (Dron, 1994; Tammar *et al.*, 2004). The interlock between the matrix epoxy and KPLF would be very well so that the contact angle become smaller Figure 5, and this causes the KPLF interface adhesion bond with the matrix epoxy become better and stronger because the KPLF surface is rougher. So that the fiber hydrophobicity increased and the fiber compatibility with the matrix becomes better and the interfacial shear strength (IFSS) increased (Zhou *et al.*, 2011).

The strength of the bond adhesion is because of the epoxy matrix penetration into the KPLF surface pores, resulting from a mechanical interlock between KPLF and epoxy matrix which is compatible as indicated by the yellow arrow in Figure 8(b-e). Which is indicated by optimum shear strength until F15H as seen in Figure 5, and the relative shear strength decline starts from F20H.

4. Conclusion

Based on the previous description of the contact angle measurement, engagement between epoxy matrix and KPLF surface and the pull out test due to the KPLF fumigation treatment effect, it can be concluded that:

1. The longer the KPLF fumigation treatment time the smaller the contact angle (θ) between the epoxy matrix and KPLF due to the good adhesion bond. From the wettabilitytest it is found the smallest contact angle (θ) of 23.22° and a Cos θ of 0.92 on F20H.

2. The interface engagementability or the compatibility between KPLF and the epoxy matrix become better which is indicated from the interfacial shear strength increased along with the fumigation duration time. The optimum interfacial shear strength was reachdon the 15 hours fumigation and relative decline up to the 20 hours fumigation time.

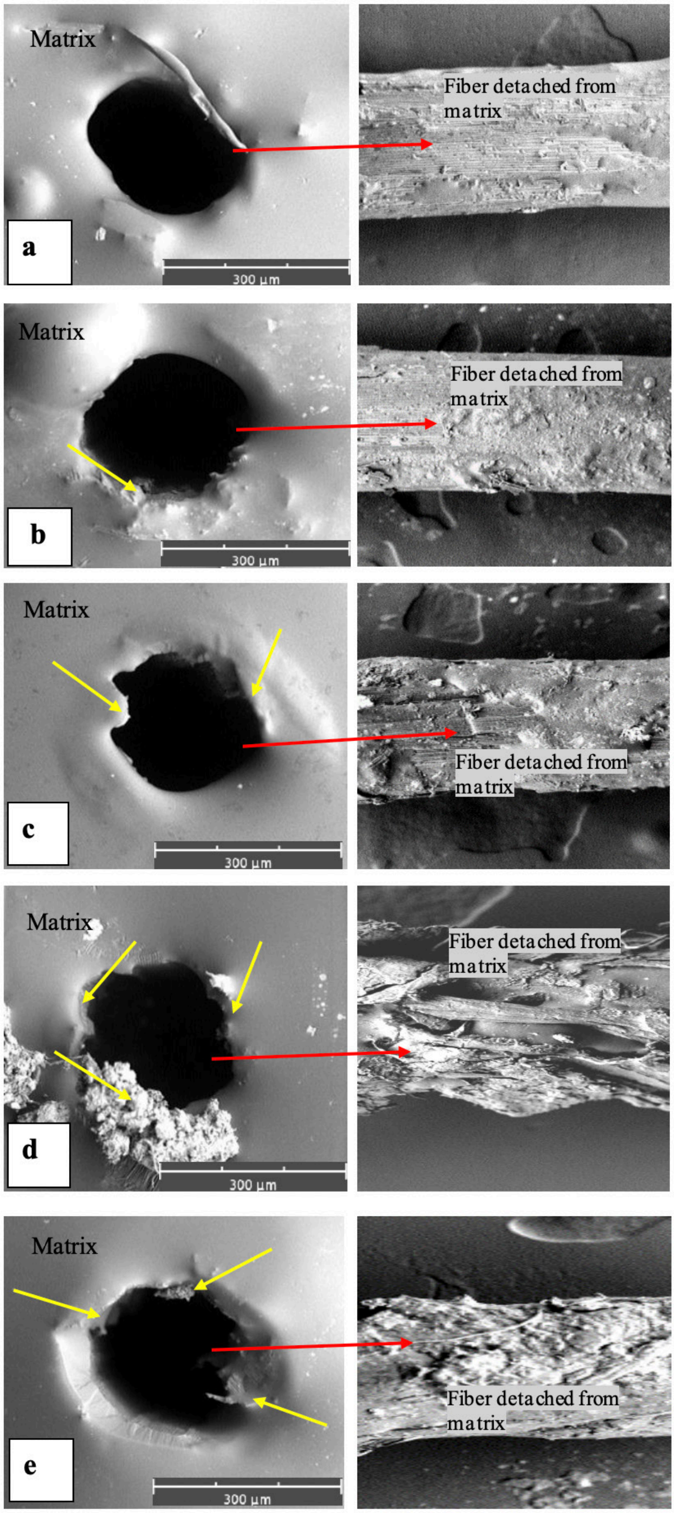


Figure 8.KSEM pull-out tests: (a). WF, (b). F5H, (c). F10H, (d). F15H, (e). F20H

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