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Characterization of tensile properties of the hybrid composite of epoxy resin reinforced with oxidized poly(acrylonitrile)

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Abstract

Oxidized poly(acrylonitrile) fibers (OPF) upon thermal treatment of poly(acrylonitrile) has been achieved and has been used as raw material to produce carbon fibers. The influence of fibers on the mechanical properties of the composite of polymer matrix reinforced by fabric were analyzed in this study by using three types of advanced fibers. For this purpose, 13 composites of epoxy matrix reinforced by fabrics of carbon fiber, Kevlar and Glass fiber with OPF were prepared by manual padding of 4 layers with different arrangements. For the preparation of composite epoxy resin Bisphenol F and polyamine as a hardener were used with resin to fiber ratio of 60:40. The tensile properties and the fractured surface of the composite samples were studied. Results of the study showed that by increasing the ratio of OPF to carbon, to Kevlar and to Glass fabric, the tensile strength decreases but for the samples in which OPF is more than 50% the fracture strain is increased. The results of cross-sectional fracture showed that composite made with a carbon fiber fabric, Kevlar and Glass fabric with OPF have lateral, explosive and edge delamination failure mode occurs on the other hand by increasing the OPF content to composite transverse failure mode happens.

Keywords: Oxidized poly(acrylonitrile) fibers, Tensile properties, Epoxy composite, Failure modes)

I. Introduction

OPF are made by thermal stabilization of PAN fibers (Schwartz 2002, Rahaman, Ismail et al. 2007). The brand name PANOX® by R. K. Carbon Fiber® UK was the first carbon fiber used for practical purposes (Johnson 2006). The thermal treatment is carried out in the atmospheric pressure within the temperature range of 180-300°C and the fiber color changes from white to brown and ultimately black due to the reaction of ring. Thermal stabilization consists of dehydrogenation reaction, ring opening and oxidation reaction in such a way that ring opening takes place at low temperatures and oxidation takes place at elevated temperatures (Arbab and Zeinolebadi 2013, Xue, Liu et al. 2013).

Previous research on the chemical composition of PAN fibers showed that the fiber contains 85% acrylic and 15% co-monomers such as Itaconic acid and Methyl acrylate. Addition of Methyl acrylate increases the rate of reaction of thermal stabilization (Xue, Liu et al. 2013). By adding co-monomer such as Sodium methallyl sulfonate and sodium 2-methyl-2-acrylamidopropane sulfonate cause the increase in tensile strength of the fibers (Edie 1998). Studies show that during thermal stabilization, the tensile strength increases (Wangxi, Jie et al. 2003). Improved orientation in crystalline region of OPF reduces the amorphous structure, reduces voids and improves the strength properties of OPF. Stretching the fibers during thermal stabilization is widely used to improve the orientation of crystalline region but up to certain extent. Studies also show that use of strong magnetic fields is the more appropriate method to improve the crystal orientation (He and Xia 2014).

Another research on mechanical properties of PAN fibers showed that by increasing the fixation time, the tensile strength and elongation follows the decreasing trend but at first the elastic modulus increases and then decreases. This reduction in mechanical properties is due to conversion of nitrile groups into nitro during thermal stabilization. Another reason for the decrease in mechanical properties is due to excessive oxidation in the temperature range of thermal stabilization (Karacan and Erdoğan 2012). Also, the increase in rate of heating up to 2°C/min subsequently increase the strength rapidly however, at high heating rates a more gentle slope occurs (Hou, Sun et al. 2008).

Studies show that ring opening reaction, dehydrogenation, crosslinking and oxidation occurs in an atmosphere containing oxygen. But nitrogen just contributes to ring opening and dehydrogenation reactions only. It has also been observed that by increasing the concentration of oxygen up to 20% volume in the atmosphere of oxygen and nitrogen the strength increased whereas, further increase in concentration of oxygen reduced the strength (Sun, Hou et al. 2009).

The most important characteristics of OPF are high thermal resistance, the inevitable dissolution, high melting point, high electrical resistance, biological safety and the ability to absorb moisture (Horrocks and Anand 2000, Johnson 2006). Such properties make it feasible to produce fire resistant fabrics, surface coatings, fire-resistant layer for batteries and for the synthesis of carbon fibers (Smith Jr 1990, McCarthy 2005, Ogle, Steagall et al. 2006).

The overall objective of developing hybrid composite using two fibers is to exploit the benefits and minimize the disadvantages of them. By replacing the ductile fibers instead of crispy or brittle fibers in the composite, the fracture strain of the polymer matrix can be improved. Metallic fibers cause high stiffness and high strain failure but their high-density barrier the practical use. On the other hand, polymer fibers are of low density, and also ductile but causes low stiffness and up to certain extent temperature resistant too (Swolfs, Gorbatikh et al. 2014). OPF has good resistance to elevated temperatures and fracture strain but has lower strength. According to the above explanation about the advantages of two types of fibers together, the aim of this study is to investigate the feasibility of hybrid polymer composites containing OPF with carbon fiber, Kevlar and Glass fiber as well as to study the hybrid impact on tensile properties, elastic modulus and strain failure of the composite.

II. Materials

The type of resin used for the manufacturing of composites was Epoxy Bisphenol F from Aditya Birla® Chemicals Ltd., Thailand and Polyamine hardener from Sanho® Chemical Co. Ltd., Taiwan and these were used as received. The Glass fabric of density of 200g/cm^2 having plain weave (90.0) received from Camelyaf® Turkey was used. Carbon fabric of density of 200g/cm^2 having twill weave (2x2) received from G. Angelona® Italy was used. Kevlar fabric of density 175 g/cm^2 having diagonal weave received from Colan® Australia and OPF having linear density of 1.16dtex having round cross section received from company Courtaulds® UK were used for composite preparation. Due to unavailability of Glass fabric of density 200 g/cm^2 having 2x2 twill weave for the preparation of composite at the time, Glass fabric of same density but with plain weave was used which gave almost the same arrangement and good compressive strength (Gasser, Boisse et al. 2000). Thus the strength properties of composite were developed independent of the type of texture.

III. Preparation of Composite

The 4 layers composites with different arrangements having dimensions of 30x25cm^2 were made by hand lay-up method as shown in Fig. 1. The ratio of resin to hardener was set to 100:15 and time for mixing them together was 2 minutes. Curing of composite samples was carried out for 24 hours at room temperature. Due to high uptake of the resin by OPF the ratio of resin to fiber was set at 60 to 40 weights % respectively.

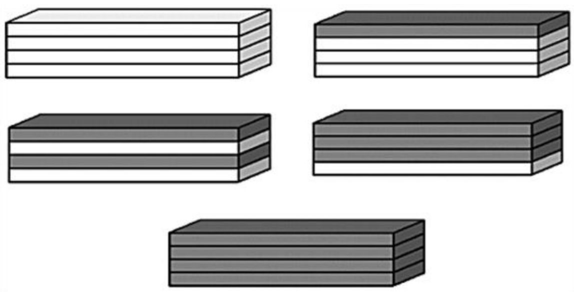

Fig. 1: Arrangements of various layers: clear layers represent fabric of Glass fiber or Carbon fiber or Kevlar fiber and dark layers represent OPF

For the manufacturing of composite, weight of the fibers with known dimensions was measured with the help of digital balance with an accuracy of 0.01g, and then according to the weight ratio 60 to 40 of matrix to reinforcement, the weight of mixture of resin to hardener was determined. In addition to ensuring compliance with this ratio, weight of composites was measured after the curing process. The accuracy of this method was ±2%. For reducing voids in the composite 20 kg weight was placed on the mold's top surface. Symbols for different percentage weight of matrix epoxy utilized in composite, as well as number of layers of fabric and ratio of reinforcement used to prepare the composite are shown in Table 1.

Table 1: Specifications and symbols of Composites prepared

Symbol	No. of layers of reinforcing fabric	The ratio of OPF in total reinforcement of composite (%)
0P-4C	4	0
1P-3C	3	48
2P-2C	2	76
3P-1C	1	89
0P-4K	4	0
1P-3K	3	53
2P-2K	2	75
3P-1K	1	86
0P-4G	4	0
1P-3G	3	51
2P-2G	2	74
3P-1G	1	89
4P	0	100

P=OPF, C= Carbon fabric, K= Kevlar fabric, G=Glass fabric

Testing of Composite

The tensile testing was carried out on the testing device manufactured by Hounsfield H25KS with force 25KN with the rate of extension of 2mm/min. Each test was performed according to standard method ASTM D3039. Five samples of each composite having dimensions 250x20x2 mm (length x width x thickness) were cut and sanded. The gauge length of tensile machine was selected as 100mm. The piece of the sandpaper used as a tab shown in Fig. 2. The purpose of using polymeric tab not only increases the friction between the clamp and the composite sample but also avoid the rupture in the area of gauge of tensile machine. Also standard on the use of sand paper instead of plastic tab has been mentioned.

Fig. 2: Tensile test specimens with tab of sandpaper

For each series of tensile test, the average value and standard deviation for each property was determined with the help of following formulae;

$$\bar{x} = \left(\sum_{i=1}^{n} x_i\right) \Big/ n \qquad (1)$$

$$s_{n-1} = \sqrt{\left(\sum_{i=1}^{n} x_i^2 - n\bar{x}^2\right) \Big/ (n-1)} \qquad (2)$$

Where:

$\bar{x}$	=	sample mean (average);
s_{n-1}	=	sample standard deviation;
n	=	number of specimens; and
x_i	=	measured or derived property.

IV. Results and Discussion

Fig. 3 represents the stress-strain curves of hybrid composite samples reinforced by Carbon fabric, Kevlar fabric and Glass fabric with OPF. The force-displacement graphs of the samples were achieved according to standard tensile test method ASTM D3039.

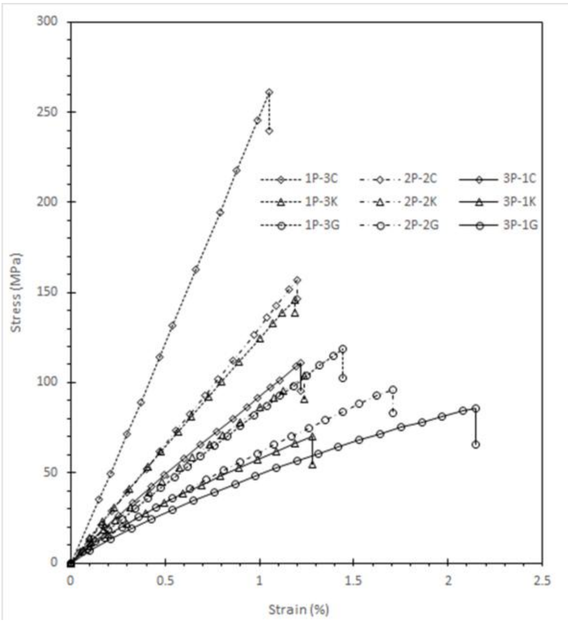

Fig. 3: The stress-strain diagram hybrid composites

Tensile properties of composite (OPF and Carbon fabric)

Fig. 3 shows the results of tensile test data obtained in Table 2, which are the OPF composite with Carbon fabric with different layers. According to the results obtained, the composite layout with four layers of Carbon fabric has tensile strength 566.60±15.80 MPa that is the highest strength whereas composite layout with four layers of OPF has tensile strength 61.08±6.73 MPa and that is the lowest. The tensile property of OPF decreases due to the crosslinking of weak structure of fibers upon thermal treatment (Kalfon-Cohen, Harel et al. 2010). That is why composite of OPF even with four layers layout has the lowest tensile strength. Thus to improve the strength properties of the composite reinforced with Carbon fiber fabric and OPF, the hybrid composite have been layered. By increasing the ratio of weight % of Carbon fiber fabric to OPF, the tensile strength of composite increases and that is in fact because of the higher tensile properties of Carbon fiber.

According to the Table 2, composite with arrangement of 4 layers of Carbon fiber fabric shows the fracture strain of 1.3%. By increasing the weight ratio of Carbon fiber fabric to OPF to 48%, the failure strain follows negative trend and after that shows an increasing trend. The highest strain failure obtained was 1.68% which is of OPF composite with 4 layers arrangement. During stabilization ladder polymer structure is formed within OPF in such a way that the percentage of this structure increases enormously regardless to the length of the fibers. That is why composite reinforced with 4 layers OPF has the highest strain failure. The initial reduction of strain failure of composite up to 48% by weight containing OPF and Carbon fiber, is probably due to incompatibility between these two fibers. The results of the composite show that the presence of OPF in the composite of Carbon fabric, improves the strain failure. Based on these results we can conclude that OPF hybrid with Carbon fiber fabric, the composite becomes less brittle and consequently ductility increases.

Table 2: Tensile test results of composite with different arrangements of OPF and Carbon fabric

Symbol of sample	Tensile strength (MPa)	Elastic modulus (GPa)	Failure Strain (%)
0P-4C	566.6±15.8	42.45±0.47	1.3±0.03
1P-3C	260.91±12.3	25±0.6	1.05±0.01
2P-2C	156.84±8.09	12.69±0.4	1.19±0.04
3P-1C	111.07±2.7	8.86±0.2	1.22±0.05
4P	61.08±6.73	4.87±0.06	1.68±0.04

Modulus of Elasticity of the composite made with 4 layers of carbon fiber fabric is 42.45±48GPa and that of 4 layers of OPF is 4.87±0.06GPa and that are the highest and lowest modulus among all other composites made with various layers. Modulus of elasticity of OPF behaves in the similar way as the tensile strength of the fiber (Kalfon-Cohen, Harel et al. 2010). That is why by increasing the weight ratio of Carbon fiber fabric to OPF in the composite, the modulus of elasticity follows the increasing trend.

Tensile properties of composite (OPF and Kevlar fabric)

Table 3 displays the results of tensile test of 4 layers composites reinforced by Kevlar fiber fabric and OPF in different arrangements that are plotted in Fig. 3. As it can be seen that data related to tensile strength and elastic modulus follows the trend likewise the trend followed by the composite of 4 layers reinforced by Carbon fiber fabric and OPF. Based on the observations, the 4 layers composite reinforced by Kevlar fiber fabric has tensile strength of 322.44±10.01MPa and 4 layers composite reinforced by OPF has 61.07±6.73MPa which are the highest and lowest tensile strength respectively. By increasing the weight % of Kevlar fiber fabric to OPF in the composite follows the increasing trend of tensile strength.

Table 3: Tensile test results of composite with different arrangements of OPF and Kevlar fabric

Symbol	Tensile strength (MPa)	Elastic modulus (GPa)	Failure Strain (%)
0P-4K	322.44±10.01	19.6±0.9	1.75±0.06
1P-3K	138.59±8.51	11.86±0.56	1.22±0.03
2P-2K	103.03±8.32	8.74±0.36	1.23±0.06
3P-1K	70.69±3.63	6.41±0.27	1.28±0.03
4P	61.08±6.73	4.87±0.06	1.68±0.04

The elastic modulus of 4 layers composite reinforced with Kevlar fiber fabric is 19.60±0.90GPa and that of composite reinforced with OPF has 4.87±0.06GPa and that are the highest and lowest elastic modulus respectively among all other composites. By increasing the weight % of Kevlar fiber fabric to OPF in the composite, the elastic modulus increases due to the presence of Kevlar fabric and its effect on elastic modulus of the composite. The results interprets that the presence of Kevlar fiber fabric in the composite reinforced by OPF, the mechanical properties are improved.

According to the data in Table 3, the 4 layers composite reinforced with Kevlar fiber fabric has the highest strain failure. By increasing the weight % of Kevlar fiber fabric to OPF in the composite to 53%, failure strain decreases but further increase in weight percentage reduces the failure strain. Initial reduction in failure strain is due to the incongruity between Kevlar fiber fabric and OPF. However, by increasing the weight ratio of OPF to Kevlar fiber fabric further, the failure strain is optimized.

Tensile properties of composite (OPF and Glass fabric)

Table 4 shows the results of tensile test of 4 layers composite reinforced by OPF and Glass fiber fabric with different arrangements. Just like Kevlar fiber fabric and Carbon fiber fabric the Glass fiber fabric also causes the increase in tensile strength and elastic modulus of 4 layers composite reinforced by OPF with different arrangements. It is observed that, the 4 layers composite reinforced by Glass fiber fabric has the tensile strength of 215.91±9.65MPa and 4 layers composite reinforced by OPF has 61.07±6.73MPa which are the highest and the lowest tensile strengths respectively. By increasing the weight % ratio of Glass fiber fabric to OPF in the composite the tensile strength increases.

The 4 layers composite reinforced by Glass fiber fabric has elastic modulus 12.80GPa and the same reinforced with OPF has elastic modulus 4.87GPa that are highest and the lowest elastic moduli respectively. By increasing the weight % of Glass fiber fabric to OPF in the composite the elastic modulus increases.

Table 4: Tensile test results of composite with different arrangements of OPF and Kevlar fabric

Symbol	Tensile strength (MPa)	Elastic modulus (GPa)	Failure Strain (%)
0P-4G	215.91±9.65	12.8±0.3	1.76±0.04
1P-3G	118.93±10.73	7.96±0.5	1.34±0.05
2P-2G	96.36±12.62	6.7±0.25	1.7±0.02
3P-1G	85.75±7.65	5.3±0.15	2.12±0.07
4P	61.08±6.73	4.87±0.06	1.68±0.04

According to the data, the failure strain of the 4 layers composite reinforced by Glass fiber fabric is much higher than the 4 layers composite reinforced by OPF. The results shows that by increasing the weight % of OPF to Glass fiber fabric to 51% the strain failure decreases however, further increase in weight % of the same increases the strain also. The reduced strain is because of difference in the type of fabric and the fiber and that causes the lack of uniformity in the composite. This lack of uniformity in other arrangement of composites is less which ultimately increase the strain failure. Highest rate of strain failure is obtained in the composite having weight ratio of 89%. According to the results of fracture strain, it can be concluded that hybrid effect of OPF with Glass fiber fabric increases the ductility of the composite.

Comparison of the tensile properties of the composites

The results show that among the three different groups of composite selected in this study having same weight ratio % and same arrangement, the composite containing OPF and the Carbon fiber fabric has the highest values of tensile strength and elastic modulus. This tensile property decreases with the increase in weight % ratio of OPF in the composite. Above 80% of weight ratio of OPF the tensile strength and elastic modulus is almost similar to the composite of Kevlar fabric containing more than 80% of OPF by weight as shown in Fig. 4 and Fig. 5.

According to the results presented in Fig. 6, composite reinforced with OPF and Glass fiber fabric and the composite reinforced with OPF and Carbon fiber fabric have highest and lowest failure strain % respectively.

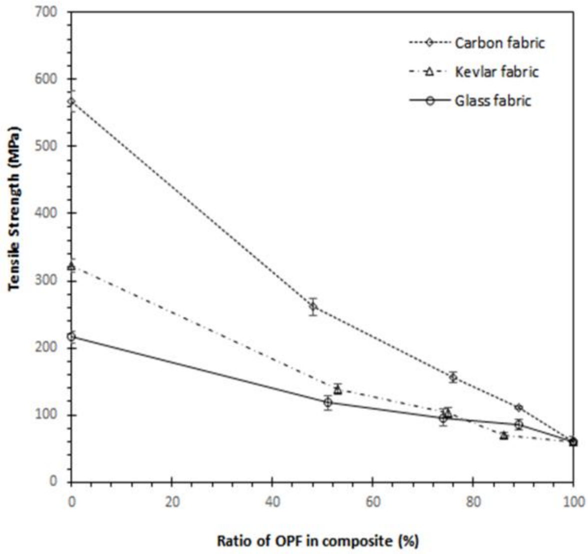

Fig. 4: Changes in tensile strength based on the percentage of reinforcing fibers in composite

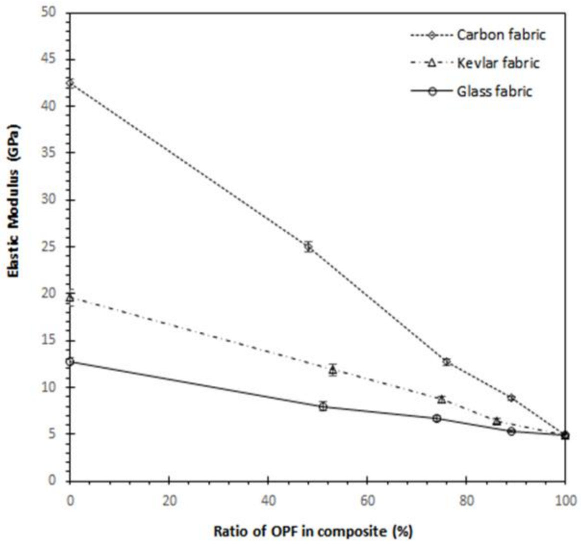

Fig. 5: Changes in elastic modulus based on the percentage of reinforcing fibers in composite

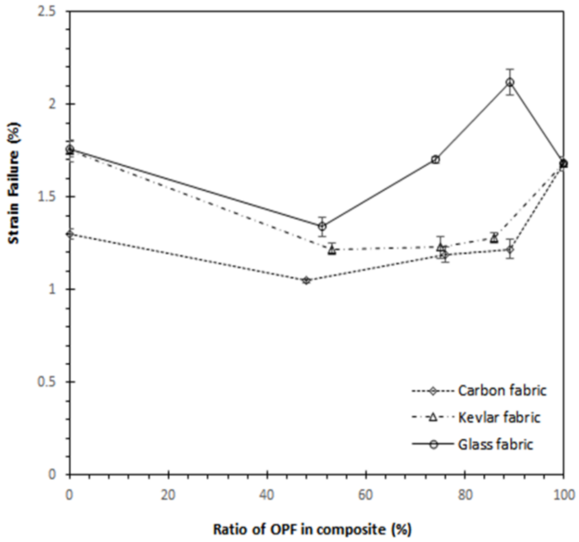

Fig. 6: Changes in strain failure based on the percentage of reinforcing fibers in composite

Types of failure of composite

In the tensile test, modes of failure especially failure area, failure type and the failure location are more concerned parameters. Different types of failure, failure locations and failure areas are presented in Table 5 (Paiva, Mayer et al. 2006, Materials 2008). Based on the Table 5, the results of failure modes of composite reinforced with Carbon fiber fabric, Kevlar fiber fabric and Glass fiber fabric with OPF are summarized and tabulated in Table 6, Table 7 and Table 8.

Table 5: Failure modes in tensile test

Failure type	Failure area	Failure location
Angled	Inside grip/tab	Bottom
Edge Delamination	At grip/tab	Top
Grip/tab	<1w from grip/tab	Left
Lateral	Gage	Right
Multi-mode	Multiple areas	Middle
Long splitting	Various	Various
Explosive	unknown	Unknown
Other		

Table 6: Failure modes of tensile test of composite containing OPF and Carbon fiber fabric

Symbol	Failure type	Failure area	Failure location
0P-4C	Lateral	Multiple areas	Various
1P-3C	Lateral	Gage	Top
2P-2C	Multi-mode	Gage	Middle
3P-1C	Angled	Gage	Middle
4P	Angled	Multiple areas	Various

Table 7: Failure modes of tensile test of composite containing OPF and Kevlar fiber fabric

Symbol	Failure type	Failure area	Failure location
0P-4K	Explosive	Gage	Top
1P-3K	Explosive	Gage	Middle
2P-2K	Explosive	Gage	Middle
3P-1K	Angled	Gage	Middle
4P	Angled	Multiple areas	Various

Table 8 :P Failure modes of tensile test of composite containing OPF and Glass fiber fabric

Symbol	Failure type	Failure area	Failure location
0P-4G	Explosive	Gage	Top
1P-3G	Edge Delamination	Gage	Various
2P-2G	Edge Delamination	Gage	Middle
3P-1G	Multi-mode	Gage	Middle
4P	Angled	Multiple areas	Various

In Fig. 7, the results of the fracture surfaces of composite reinforced with OPF and Carbon, Kevlar and Glass fiber fabric after the tensile test, show the angled, lateral, explosive and delamination states respectively. According to the research conducted on the composites reinforced with Carbon fibers show lateral fracture which interprets the brittle fracture. However, by increasing the weight % ratio of OPF in the composite, lateral failure mode is changed to angled fracture as shown in the Fig.

7. This means that the presence of OPF in the composite cause the ductility of the composite and the results of fracture strain prove it likewise.

Composite reinforced with Kevlar fiber fabric shows explosive mode of fracture as shown in Fig. 7. Addition of OPF in the composite changes the explosive mode of fracture to angled fracture. These changes in the fracture mode are in close agreement with the changes in the fracture strain. Although the 4 layers composite reinforced by the Glass fiber fabric shows explosive mode of fracture however, by increasing the weight % ratio of OPF the delamination mode of fracture dominates. With the increase in weight % ratio of Glass fiber fabric in the composite multiple mode of failure are observed including delamination failure and lateral failure and ultimately 4 layers composite reinforced with OPF only shows lateral failure mode. These results are in close agreement with the results of failure strain of the composite reinforced with OPF and Glass fiber fabric as illustrated by the Fig. 6.

V. Conclusion

The results of the present study showed that the presence of Carbon, Kevlar and Glass fiber fabric as a reinforcing material improves the fracture stress and elastic modulus of epoxy composite containing OPF. The highest fracture stress and elastic modulus belongs to the composite reinforced with Carbon fiber fabric and OPF.

The hybrid effect of OPF in the composites reinforced with Carbon, Glass and Kevlar fiber fabrics improves the fracture strain. The highest value of strain failure was achieved with the composite reinforced by OPF and Glass fiber fabric.

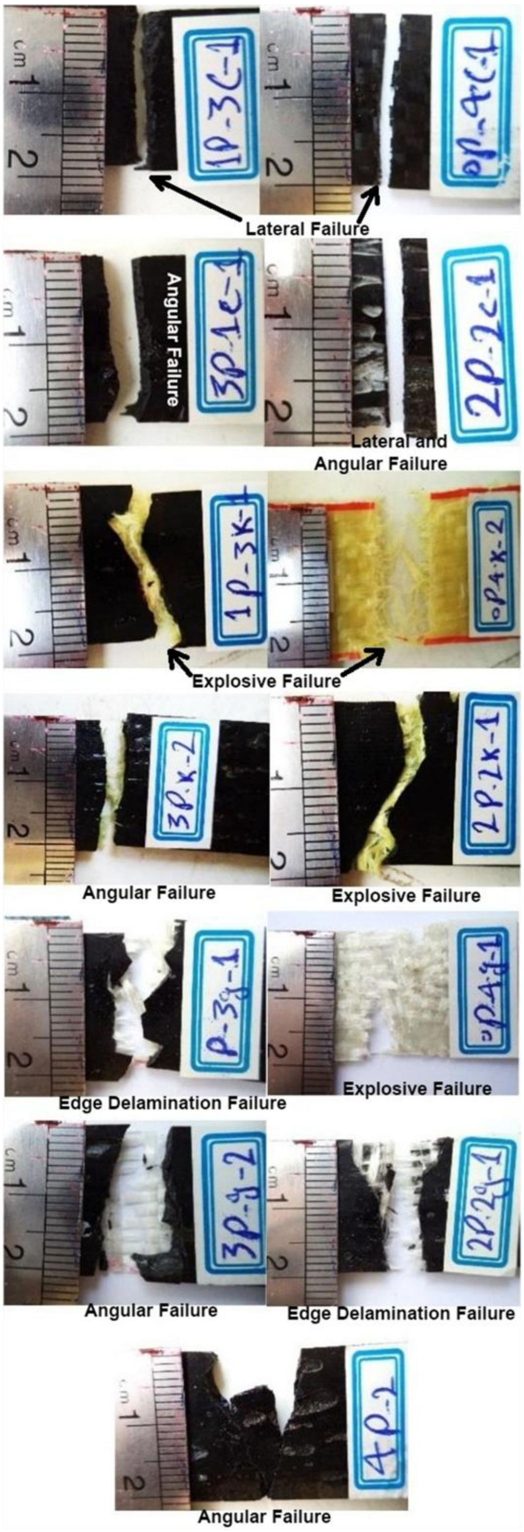

Fig. 7: Surface of fractured segment of various composites

The analysis of fractures surface shows that the composite prepared with Carbon, Kevlar, Glass fabrics and OPF indicate the lateral, explosive, delamination and angular modes of fracture during tensile test respectively. The results of evaluation of fracture surface showed the similar behavior to that of fracture strain. Therefore, it can be concluded that by combining OPF to Carbon, Kevlar and Glass fiber fabric in the preparation of composite, the fracture stress, elastic modulus and fracture strain are optimized.

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