MECHANICAL TESTING OF NATURAL FIBRE REINFORCED POLYESTER RESIN COMPOSITES AND MODE 1 FRACTURE TOUGHNESS TESTING OF RESIN BLOCKS

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SYNOPSIS

Present government policies and industrial interest are targeted towards friendliness to the environment. In this study, unidirectional fibre (sisal, kenaf, abaca and flax) reinforced polyester resin composites were processed by hand laying, atmospheric casting of resin and then compressing between plates. The fibres were used as obtained from the suppliers. The samples were tested in tension. The composites reinforced with flax and kenaf were noted to have higher strength and modulus compared to the samples reinforced with abaca and sisal. The resin binds the fibres together and helps to distribute load to the fibres. Hence, single-edge notched bending (SENB) samples were constructed from resin blocks and tested following the mode 1 kind of fracture failure. It was observed that the concentration of catalyst affected the fracture load and toughness. Increase in the concentration, increased the values of the said parameters. The samples were prepared within the resin manufacturers' recommendation (ie 1 – 2 % catalyst).

1. INTRODUCTION

In recent years, industries are getting more and more interested in protecting the environment. The European Parliament directive requires companies to achieve materials recycling greater than 80% in particular in the automotive sector [4]. The research on natural fibre based composite materials fits well into this ecological image. The advantages of natural fibres over the synthetic ones includes, low density, relative cheapness, availability and biodegradability. Commonly used natural fibres are the bast and leaf qualities, such as hemp, jute, kenaf, sisal etc. Coir (coconut) stands apart from other leaf and bast fibres. It contains more lignin than most other natural fibres. Since lignin resists biodegradation, high lignin content also imparts longevity to outdoor applications

The market for natural fibres in thermoplastic composites has experienced exceptional growth in recent years as some new applications and fresh participants emerge. In response to the need for an accurate assessment of the market opportunity for fibres in this new class of composites, Kline & Company, published *Opportunities for Natural Fibres in Plastic Composites* [18], Dale Brosius [6] reported about natural fibres slowly taking root and Anne Ross [1] indicated that Basalt fibres were an alternative to glass. Also two Fife scientists (Dr David Hepworth and Dr Eric Whale) in the United Kingdom have developed material made from carrots to replace glass fibre fishing rods[7].

As in composites reinforced with synthetic fibres, the mechanical properties of the composite depend on the properties of the fibres, matrix and the interface between them. Some studies have been conducted on the composition and morphology of fibres and observed that the cellulose content and micro-fibril angle tend to control the mechanical properties of cellulosic fibres [14 & 10].

Hence, to produce composites with good properties, it is necessary to improve the interface between the matrix and the lignocellulosic material. There are various methods to improve the interfacial adhesion, such as esterification, silane treatment, graft copolymerisation, use of compatibilisers, plasma treatment etc. This implies that the composite can be referred to as '*enhanced natural fibre composites*'. Karmaker and Schneider [19] investigated the performance of short jute fibre reinforced polypropylene composite and observed that the addition of just 3% coupling agent (maleic anhydride grafted polypropylene) immensely improved the mechanical properties.

Natural fibres have a complex structure and not to be taken in a straightforward manner as monofilament fibres. Figures 1 – 4 are SEM images of abaca, flax, sisal and kenaf fibres.

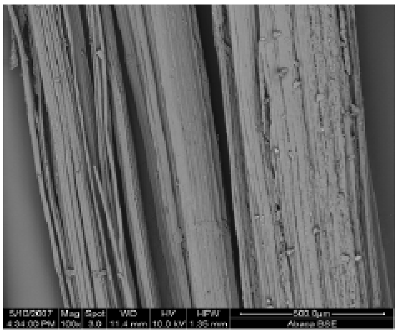


Figure 1: SEM image of abaca fibres.

Figure 3: SEM image of sisal fibres.

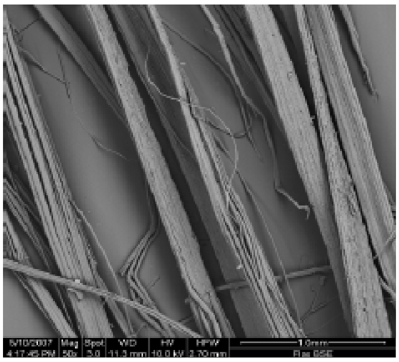


Figure 2: SEM image of flax fibres.

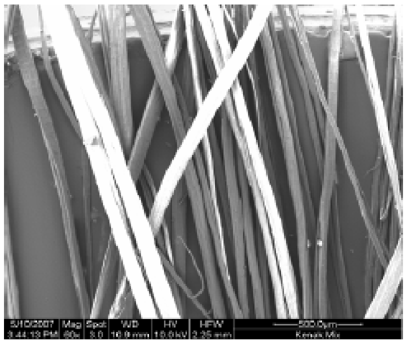


Figure 4: SEM image of kenaf fibres.

Fracture toughness is an indication of the amount of stress required to propagate a pre-existing flaw in a material or structure. It is a very important material property since the occurrence of flaws is not completely avoidable in the processing, fabrication or service of a material/component. Studies on green fibre composites have indicated that the properties can be improved by fibre surface treatment and hybridization and that composites with fibres showing a high micro-fibrillar angle, indicated higher composite fracture toughness compared to those with small angles [3]. Pavithran et al [15] evaluated the enhancement in properties of coir-glass intermingled hybrid composites and observed significant improvement in strength and reduction in moisture absorption.

Van de Weyenberg et al [9] investigated the influence of flax fibre treatment on the properties of composites reinforced with the fibres. It was noted that the processing of the fibres has influence on the performance. The better the degree of fibre retting, the better the composite properties. The objective of the treatment is to enhance the adhesion between the fibre and the matrix. These investigators reported that treating flax fibres with alkali and dilute epoxy, before using as reinforcement in composites gives very good mechanical properties. The epoxy was diluted with acetone. In a similar study by Kulkarni et al [2] on coir fibres, the modulus was the same for both retted and unretted fibres, but deviations existed at the plastic zones.

A number of studies on the application of green fibres in composites like pineapple, sisal, coconut coir, jute, palm, cotton, bamboo etc as the reinforcements in composites have been reported in the literature. Sapuan, S M et al [17] have tested and reported in a short communication the mechanical properties of woven banana fibre reinforced epoxy composites for use in household utilities in Malaysia. In another related study Dandekar C and Mallick P K [5] have reported that the tensile properties of natural fibre composites can be improved by combining with either geotextile composite or carbon fibre composites in a sandwich construction.

The properties of natural fibres are mainly determined by the chemical and physical composition, such as the structure of fibres, cellulose content, angle of fibrils, cross-section and by the degree of polymerisation. The fibres are usually treated before being manufactured into composites, with the aim of improving the interface properties between the fibres and the matrix. **In this paper**, a preliminary study on the mechanical properties of natural fibre (untreated) reinforced styrene polyester resin and the flexural (three point bending) properties of the resin at different concentrations of the catalyst using single-edged notched bending (SENB) samples is reported.

2. MATERIAL PROPERTIES

2.1. Fibres

The fibres used in this study are abaca, flax, sisal and kenaf. They have been tested at various humidity levels and the results of the mechanical properties published in reference [13]. The fibres were not mixed ie each category (eg abaca) was obtained from a specific source. In this way, growth environmental effect maybe thought to be reduced.

2.2. Cured resin

The properties of the resin (Crystic 703PA) cured at ambient temperature 20^0C for 24 hours, as reported by the manufacturers ‘Scott Bader’ is as shown in table 1.

Table 1: Properties of cured resin.

s/n	Property	Value
1	Tensile Strength (MPa)	49
2	Tensile Modulus (MPa)	2758
3	Elongation at Break (%)	2.1

3. MOULD PREPARATION

3.1. Tensile test samples

The mould was constructed from a block of aluminium. The mould is rigid. It was properly cleaned and coated with a mould release tape. It is capable of producing five samples of composites in a single batch. The sample size (in mm) is 20 x 3 x 170, appropriate to an ASTM D638-02A specimen, except for the necking section that was eliminated because the fibres run in a single direction making an orthotropic material.

3.2. Single-Edged Notched Bending (*SENB*) samples

This mould was milled out of two halves of aluminium plates each measuring (in mm) 15 x 160 x 240 using a numerically controlled machine. It was washed, cleaned with acetone and coated with the mould release spray before use.

4. PREPARATION OF STYRENE-POLYESTER RESIN WITH CATALYST

The materials used were:

- **Catalyst:** 'Methyl Ethyl Ketone Peroxide' from Scott Bader with the card number of 'M' UN3105

- **Resin:** Crystic 703PA polyester resin with low viscosity and controlled exotherm characteristics. Polyester resins are the most widely used resin systems, particularly in the marine industry. By far the majority of dinghies, yachts and workboats built in composites make use of this resin system. The styrene also performs the vital function of enabling the resin to cure from a liquid to a solid by 'cross-linking' the molecular chains of the polyester, without the evolution of any by-products [8].

4.1. Mixture

It is a good practise to pass the resin through a vacuum unit for degassing before use, especially for the making of composite structures. The quantity of the catalyst is between 1% -- 2% of the resin, as recommended by the manufacturers. This mixture of the resin and catalyst was stirred thoroughly before casting atmospherically into the mould with fibres laid in the mould, for the tensile testing samples. The blocks for the SENB samples were produced by a vacuum infusion process after the mixture.

5. THE REINFORCEMENT FIBRES

The properties of natural fibres are highly variable. They are affected by the environment and conditions of growth. It is therefore difficult to get the same or very close results even after repeated testing. The fibres used for this study were obtained from Wigglesworth Limited and Toyota auto body (Japan). Abaca, flax and sisal were obtained from the former and kenaf the latter. The fibres were not mixed; in this way it may be assumed that the fibres were grown under similar environmental conditions.

6. SAMPLE PREPARATION

6.1. Tensile test specimens

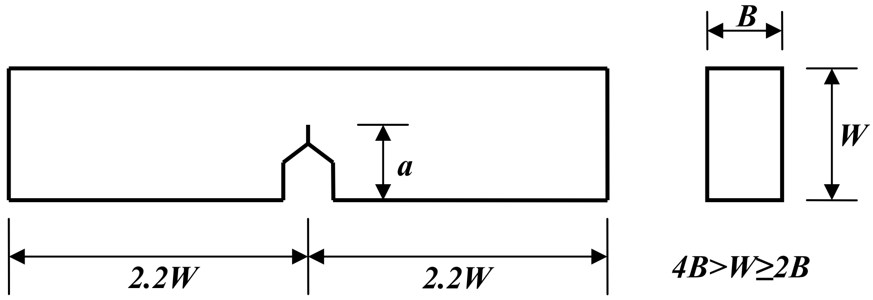
The natural fibres were processed into unidirectional bio-composites with fibre volume fractions of approximately 36%, determined by mass measurement and the use of fibre and resin densities. Aluminium tabs were bonded to the ends of the composite strips with araldite. The testing equipment grips the samples at these positions during testing. The laminates were being allowed to cure for 24 hours at 20°C, after casting the resin onto the fibres in the mould. A representative illustration of the test sample made with abaca fibre is shown in figure 5.

Figure 5: Natural fibre composite ready for testing.

6.2. Single Edge Notched Bend (SENB) Specimens

The samples for fracture toughness test were prepared from blocks of resin manufactured by vacuum infusion. The resin in the mould was left to cure for 24 hours, at ambient temperature of about 20^0C. The mould was positioned vertically, the entrance at the bottom and exit at the top. The aim is to aid air evacuation as the resin settles gradually in the mould. The resin has a very low viscosity suitable for infusion, but was kept sitting on a bath of very hot water to further reduce the viscosity albeit shortening the curing time.

The block of solid resin from the mould was used to produce the standard 'Single Edge Notched Bend, (SENB)' specimens for testing. The dimensions of the specimens used for this study are as shown in figure 6. To manufacture this, the resin block was cut to near the size with a saw and then milled to smoothness. The notch was created with a 60^0 slotting tool and a sharp line obtained by the tap of a very sharp blade.



W = 40 mm B = 16 mm a = 17 mm

Figure 6: Standard ***SENB*** specimen configuration [11].

Considering, the brittle nature of the resin and the avoidance of unstable fatigue crack growth, it was ensured that no crack was generated by tapping with the sharp blade.

7. TENSILE TESTING OF COMPOSITES

A tensile test also known as tension test is probably the most fundamental type of mechanical test performed on materials. It provides knowledge of how the material will react to forces being applied in tension and also indicates the capacity of the material to resist breaking in tension. As the properties of natural fibres are variable it is therefore difficult to get the same result from repeated testing. Hence, in this study four fibre reinforced composites were tested for each type of fibre (eg sisal) and the results analysed.

The experiments were performed under ambient conditions. Tensile testing on the composites were performed with a '*ZWICK REL Model 2061 Universal Testing Machine'* in accordance with ASTM D 638-02a – *Standard for tensile properties of plastics*. ZWICK Rel 2061 testing machine is equipped with specially designed jaws to grip (figure 7) the sample. The samples during testing were gripped on the aluminium tabs bonded to the specimens by araldite mixed with hardener. The load applied is a continually increasing uni-axial load at the rate of 1 mm/minute until such time as failure occurs. The gauge length was fixed at 50 mm for all the samples tested by an extensometer.

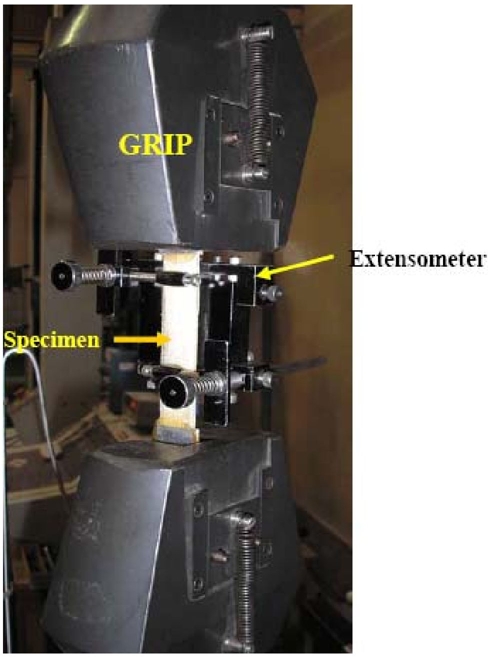


Figure 7: Composite sample gripped under tensile testing.

8. TEST RESULTS AND DISCUSSION

Figure 8 shows representative stress – strain graphs obtained from the tensile testing data of the bio-composites. Van de Weyenberg et al [9] prepared samples of fibre volume fraction 40% , while Wambua, P at al [14] used samples of 30% volume fraction. In this study the fibre volume fraction of the composite is approximately 36%.The relationships between the stresses and strains are almost linear up to the peak stresses, when the materials fail. As seen there is little or no plastic deformation, hence the constitutive model can be represented by the linear elastic model instead of the relatively robust Ramberg-Osgood strain hardening model.

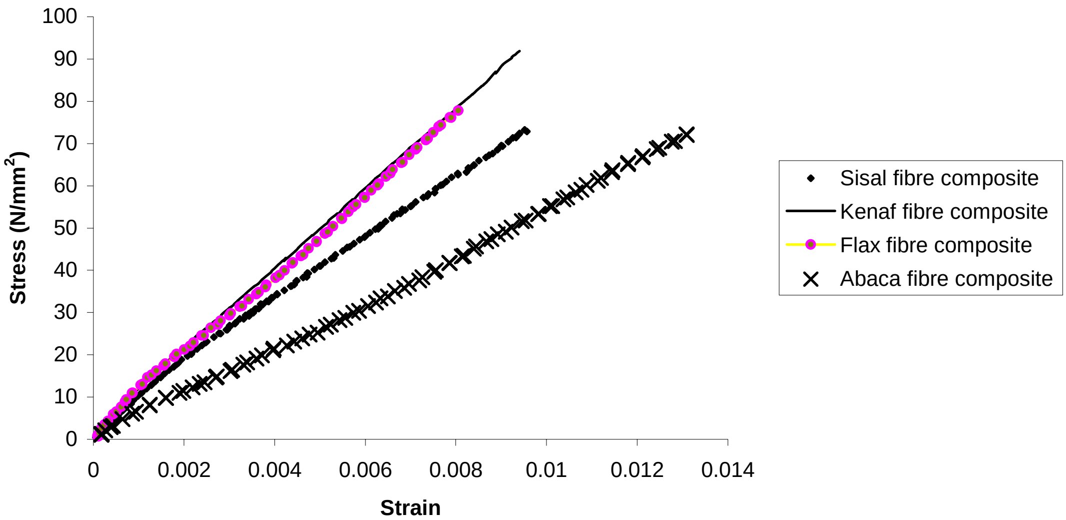


Figure 8: Stress – strain plots of the green composites.

The ultimate strength of the composites depends on several factors, but important among them are the properties of the reinforcement and matrix, volume fraction of the fibres and the alignment of the fibres. The fibres in the composite considered here were hand laid unidirectionally. Also the angle between the fibre axis and the fibrils of the fibre affect the modulus and strength of the composite.

The stress – strain plots depict the failure modes of these natural fibres composites. The tensile failure is associated with the drop in load as pulled in tension. Although in all the stress – strain plots it is almost linear to the point of failure, the fracture behaviours are different after this point. The energy absorbed to failure in the composite was calculated by integrating the load – extension plot from zero to the breaking point (figure 9) of the composite. After testing photographs of the samples were taken; such as the image in figure 10. The crack path is unpredictable; this is thought to be for several reasons including the following:

1. The fibres were hand laid.
2. Effect of the micro-fibrillar angles.
3. Single fibres have different ultimate strengths.

The tested samples revealed cracking of the matrix and splintering of the fibres. The fibres, which are the load bearing component, are generally non uniform in cross-section, coupled with variation in cross sectional shape along the fibre length.

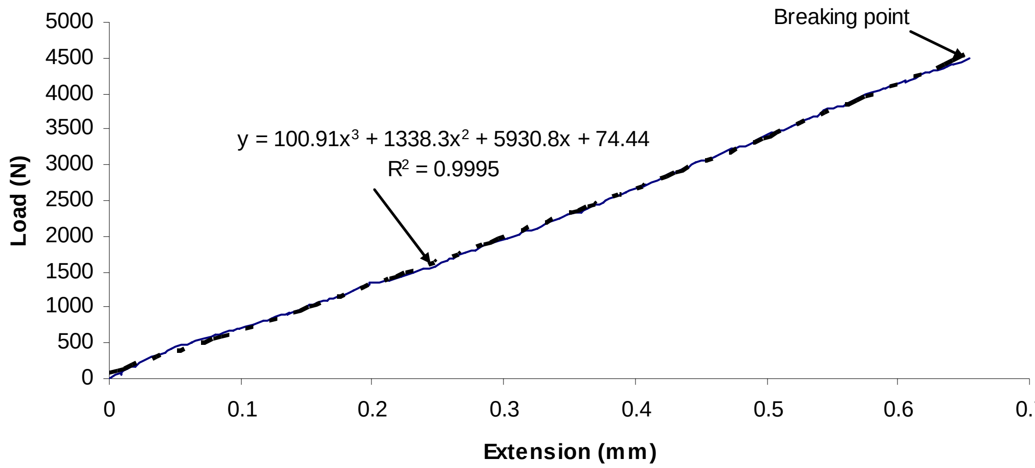


Figure 9: A representative load – extension plot to illustrate breaking load and energy.

Figure 10: Photograph of tested composite samples

Table 2: Composite properties from tensile tests

	Abaca	Flax	Kenaf	Sisal
Strength (MPa)	74 ± 3	85 ± 10	91 ± 3	79 ± 6
Stiffness (GPa)	5.90 ± 0.3	9.42 ± 0.6	10.36 ± 0.5	7.30 ± 0.6
Energy to failure (J)	1.40 ± 0.16	1.18 ± 0.28	1.25 ± 0.11	1.40 ± 0.22

A summary of the properties of the natural fibre composites, showing the mean values from four sets of tests and standard deviation are shown in Table 2 above.

9. FRACTURE TOUGHNESS STUDY OF THE RESIN BLOCKS

Fracture toughness is a quantitative way of expressing a material's resistance to brittle fracture when a crack is present. If a material has a large value of fracture toughness it will probably undergo ductile fracture. Brittle fracture is very characteristic of materials with a low fracture toughness value.

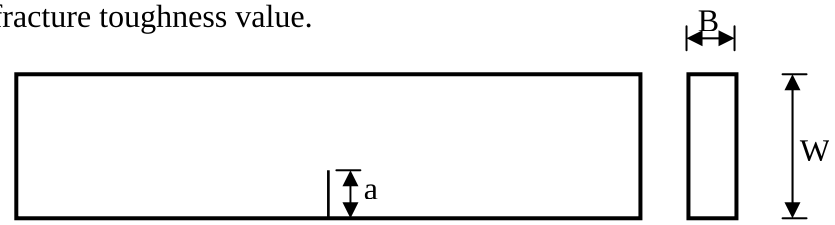


Figure 11: Schematic of the modified SENB sample used by Jun Ma et al [12].

The traditional single-edge notched bending (SENB) fracture toughness measurement is fine with ductile materials, but with very brittle materials (like thermoset resins) it is difficult to generate the sharp crack. Jun Ma et al [12] used modified SENB samples as shown in figure 11, for their study. In this investigation conventional samples have been employed with a fine line at the end of the notch produced by a sharp blade. Two tests were performed for each percentage of catalyst concentration. The results were similar.

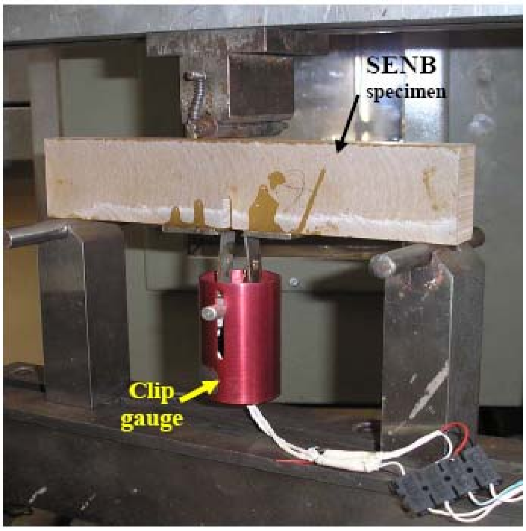


Figure 12: Crack opening displacement experimental set-up.

The displacement at the point of loading was measured by a clip gauge (figure 12) during the tests, and the load– displacement curves were plotted. Figure 13 is a representative graph. It shows that, from the origin to the peak, the relationship of load against displacement is almost linear. The load reaches the peak value at a displacement, referred to as the crack opening displacement and after the peak, the load reduces rapidly to zero. The behavior of the specimen indicates that linear elastic fracture mechanics (LEFM) is suitable for investigating the fracture characteristics of the resin blocks. The crack-tip opening displacement (CTOD) can be considered as the strain-based estimate of fracture toughness of the thermoset resin.

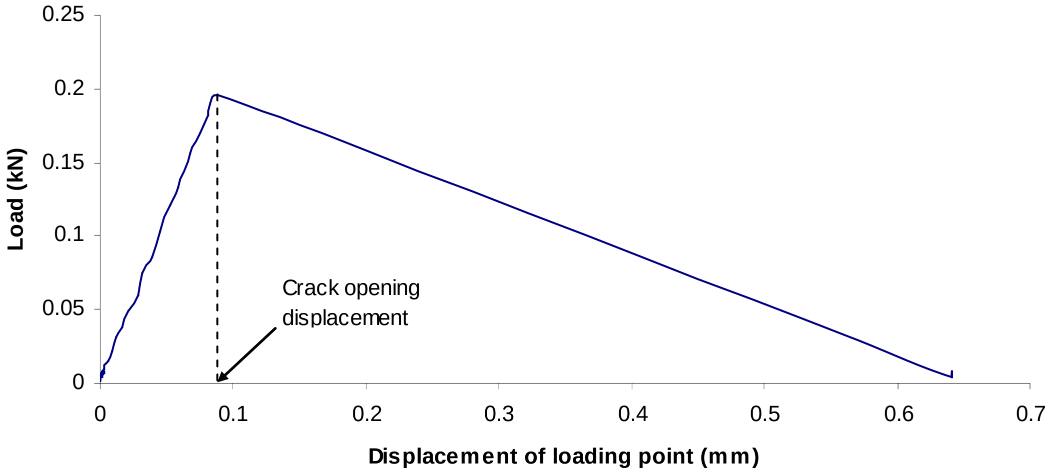


Figure 13: Representative load – displacement plot obtained from single-edge notched bending test

This test has been performed by considering a procedure set by the European Structural Integrity Society – E399 [11]. These are not standards in the usual sense, but rather testing protocols that have been agreed by experts. The rate of loading used was 1mm/min, as too high cross head speeds will introduce dynamic effects into the results.

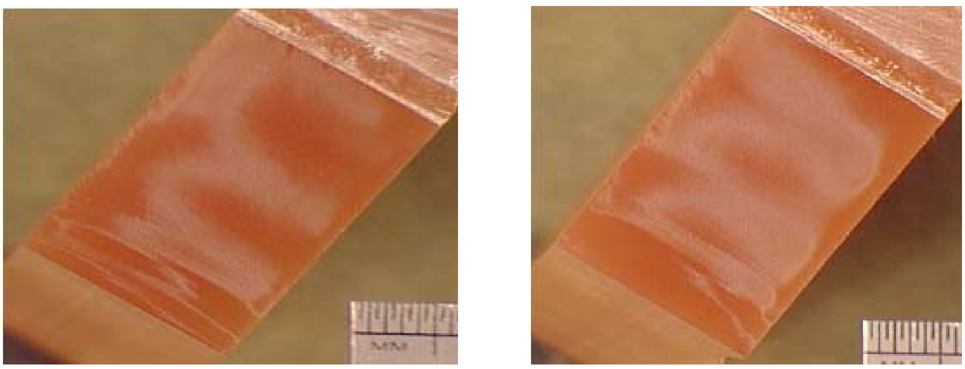


Figure 14: Photograph of fractured faces of resin blocks after SENB testing

It is difficult to describe in detail the fracture surface appearance of composites because their fracture micro-morphologies vary and this depends on the fracture characteristics of the reinforcements and matrix. In addition the fibre-matrix interface, environment and temperature influence the fracture surfaces and path [16]. Typical macroscopic photographs of the fractured surfaces of the resin blocks are displayed in figure 14 and the results of the fracture load and toughness represented as the crack opening displacement are presented in figure 15 and summarized in Table 3. In general, increase in catalyst concentration resulted in increase in the fracture load and toughness.

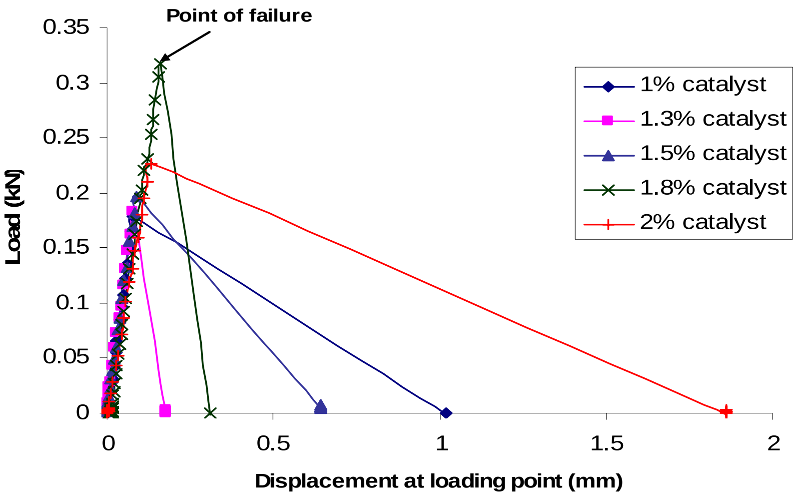


Figure 15: Load – displacement plot of fracture toughness test on resin blocks of various catalyst concentrations.

Table 3: Summary of fracture toughness tests

Percentage of catalyst	Fracture load (kN)	Crack opening displacement (mm)
1% catalyst	0.176 ± 0.004	0.077 ± 0.003
1.3% catalyst	0.183 ± 0.003	0.083 ± 0.002
1.5% catalyst	0.198 ± 0.002	0.090 ± 0.001
1.8% catalyst	0.313 ± 0.003	0.150 ± 0.002
2% catalyst	0.227 ± 0.004	0.133 ± 0.003

10. CONCLUDING REMARKS

Experimental investigations of natural fibre (abaca, sisal, kenaf and flax) reinforced styrene polyester resin composites under tension load was undertaken. In addition, fracture toughness of resin blocks of varied catalyst content was also examined. The composites were manufactured at an estimated fibre volume fraction of 36%. From the test results and graphs plotted, it was possible to locate / calculate the strength, modulus and the energy to failure of the composites. The strength of kenaf and flax were relatively close to each other, but kenaf was higher and abaca was the least strong. The morphology of the fracture path is unpredictable taking cognizance of the varied characteristics of the structure.

Although in this study the fibres were not treated. In the next phase of this investigation, they will be treated and the introduction of nano-technology in the resin considered. Also discussed were the results of fracture toughness tests of styrene polyester resin blocks. Single-edge notched bending (SENB) samples were manufactured with varied catalyst content (within manufacturer’s boundaries). The fracture strength and toughness increased as the percentage of catalyst increased.

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