EFFECTS OF HYDROTHERMAL AGEING ON THE MICROBOND INTERFACIAL SHEAR STRENGTH OF NaOH TREATED SISAL FIBRE REINFORCED POLYESTER COMPOSITES

Chizyuka G. Chizyuka, Kanyanga B. Shamitiba and Munakaampe M. Grain

University of Zambia
School of Engineering
Department of Mechanical Engineering
P O Box 32379
Lusaka, Zambia

Corresponding email: chizyukagc@yahoo.com

Abstract

Engineering structural failures can be related to the fracture of one or more of the constituent materials that form a composite. Such failures often occur unpredictably and suddenly. It is, therefore, necessary to minimise undesired failures and their consequences when designing and analysing modern-day structures. To suitably design structural components, not only do the fundamental mechanical properties of the components' material constituents need to be known, but also the effects of the service environment on these properties. Moisture is one critical environmental factor that can be destructive to properties of composite materials. Composite constituent components, most especially in natural fibre- reinforced composites constantly absorb or desorb moisture due to varying temperature and relative humidity levels in their service environment. In this regard, this paper presents results of a study on the influence of hydrothermal effects on the composite material microbond interfacial shear strength as a function of ageing period under different thermal loading states. Results from the microbond test coupled with Scanning Electron Microscope (SEM) fractographic analyses indicate that debonding is an established fracture mechanism in this hydrothermally aged NaOH treated sisal fibre-polyester composite systems.

Keywords: *Microbond Interfacial Shear Strength, Natural Fibre-Reinforced Composite, Service Environment, Hydrothermal Effects, Fractographic Analyses, Debonding.*

INTRODUCTION

Micro Fracture

The magnitude of the interfacial shear strength existing in between the embedded reinforcing fibre and the surrounding matrix material in a reinforced composite is usually determined by measuring the force needed to pull a single fibre axially out of the solid matrix. To make such pull-out measurements, however, the length of embedded fibre must be small enough so that the fibre does not break before it pulls free. The small dimensions involved in the method facilitate uniform exposure of the interfacial region and also provided much quicker exposure time to the hydrothermal ageing environment. The main assumption of this technique is that there exists a uniform distribution of interfacial shear

stress along the embedded part of the fibre. One great advantage of this technique is that it requires small quantities of resin and fibre, avoiding excessive waste of materials (Oikonomou, 2010).

This method involves embedding part of the fibre in a pool of liquid resin, and allowing the resin to harden, either by chemical reaction or cooling. The force needed to pull the fibre out of the resin is then determined, usually with a tensile tester. Most researchers have assumed that the measured force is equal to a shearing force that is applied to the entire interface and distributed uniformly. Based on this assumption, the shear strength τ_{Int} of the bond is calculated from (Miller B. *et al*, 1987):

$$\tau_{Int} = {F_P}\Big/{\pi D_f l_e} \qquad (1)$$

where F_p = pull-out force; D_f = fibre diameter; and l_e = embedded length.

A serious limitation inherent in this procedure arises when very thin fibres are used, as is often the case when the reinforcing elements of the composites are to be carbon, glass or aramid fibres. If the required pull-out force exceeds the breaking strength of the fibre, the fibre will break instead of pulling out. This restriction can be expressed in terms of a critical embedment length, l_c which can be obtained by defining it in terms of the pull-out requirement (Equation (2)) (Miller *et al*., 1987):

$$l_c = F_P \pi D_f \tau_{Int} \qquad (2)$$

Figure 1, depicts a typical microbond single fibre pull-out test plot of interfacial shear stress as a function of relative displacement at the interface existing between the fibre and the matrix. Region I relates to a still intact interface, whereas Region II describes the linear stress reduction in the zone with ‘‘imperfect interface’’, and Region III (with constant slip, which depends on the consistency in the fibre diameter) describes a fully debonded interface which transfers friction load only according to Zhandarov and Mader (2005). In order to characterise interfacial strength in fibre reinforced composites using micromechanical methods, Zhandarov and Mader (2005) paid more emphasis on single fibre pull-out and microbond techniques for both stress-based and energy-based approaches respectively over fragmentation test and Broutman test. This is because the former techniques allow to relate the load transfer ability of the interface to adhesion parameters at the molecular level.

Miller *et al.* (1987) analysed and further used the approach which requires depositing a droplet of the epoxy resin on both silane treated, and untreated fibres (aramid and carbon fibres) and supporting the cured droplet appropriately during single fibre pull-out analysis for the fibre/matrix interfacial bond strength fracture. From the results obtained it was observed that the microbond method makes it possible to successfully investigate composite microbond failure under conditions where the resin is not available in large quantity.

Thomason and Schoolenberg (1994) investigated interfacial strength existing between the reinforcing glass fibre and the polypropylene matrix composite system and its influence on the mechanical properties of the overall (bulk) composite. This study compared the effect of fibre surface coating on overall mechanical performance of resulting composite system.

Results point out that silane coupling agent by itself produced a marginal effect on the interfacial strength of glass fibre/polypropylene system which only significantly improved when combined with other components of the coating which include binder, lubricant, antistatic agent, and wetting agent.

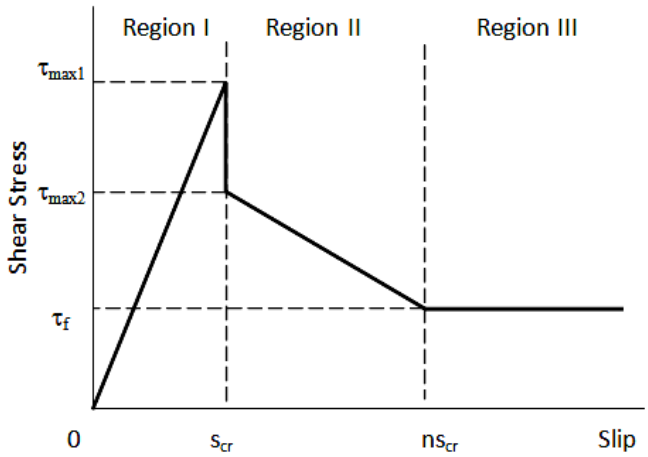


Figure 1: Typical microbond debonding plot with two-stage debonding (imperfect interface). (*Source*: Zhandarov and Mader, 2005)

Gaur and Miller (1990) used the microbond method in the direct determination of interfacial shear strengths in their quest to assess the influence of the environmental conditions on the interfacial adhesive bonding in the aramid/epoxy and glass fibre/epoxy micro composite systems. Sizable drops of up to 70% in the average shear strength and changes in the shear strength normal distribution were observed after exposing the micro composite assemblies to steam or hot water for short periods of time minimum 2 hours, with glass fibre/epoxy system recording the most drastic changes compared to aramid/epoxy composite system. Full restoration of shear strength upon vacuum drying of the aramid/epoxy micro assembly was observed while the glass fibre/epoxy system recorded marginal recovery in its interfacial shear strength.

Craven, *et al.* (2000) similarly employed the microbond testing technique to evaluate the interface that exists in a silk fibre/epoxy composite system. The tests yielded average interfacial shear strengths of about 15 ± 2 MPa. Practical limitations observed in using this test method on this composite include the low load bearing capacity of silk fibres (due to their small average diameter which ranges between 3.5-12µm), as well as the inherent variability in their cross-sectional geometry and tensile.

Interface strength in glass fibre/polypropylene system was determined by Yang and Thomason (2010) using both the fibre pull-out and microbond test methods. A good correlation between the two methods was obtained. Data from microbond test could be divided into two groups according to whether or not there was constant interfacial friction after debonding. Microscopy observation on tested microbond samples which had exhibited decreasing interfacial friction after debonding revealed considerable residual resin around the debonded area of the samples. Further investigation indicated that this unexpected difference in the amount of residual resin was caused by the variation in mechanical properties of the matrix due to thermal degradation during sample fabrication.

RESEARCH MATERIALS

The following is an overview of the materials used in making composite test specimens in this research.

Sisal Fibres

Sisal, obtained from cultivated plants, remains one of the most extensively used natural fibres. However, its use over the years has steadily diminished with the introduction polymeric yarns such as polypropylene to be used for the same purpose in rope and carpet manufacture. Amid the variety of natural fibres being exploited for reinforcement, sisal fibre still remains a worthwhile material in that it forms high impact strength composites compared to other natural fibres such as banana and coconut fibres despite having moderate flexural and tensile strengths (Bledzki, *et al.* 2002, Samuel, *et al.* 2012).

The annual worldwide sisal fibre production in 2015 stood at nearly 300,000 tonnes (FAO 2015); of which Tanzania and Brazil being the two major producers. Sisal fibre is an extract from sisal leaves (*Agave sisalana*) though a process known as decortication. The length of sisal fibre lies between 1.0 to 1.5 m, with diameters of 100 – 300 μm (Li, *et al.* 2000). The interface that exisits between the two composite components plays the role of transfering load within the composite; a good interface is indispensible in taking advantage of both composite components (Kim and Mai (1998); Geo and Cotterell (1988)). As such, it is essential to treat the surface of the fibres in order to give them improved fibre/matrix interfacial bond which subsequently leads to better strength and toughness for the composite. The effects of suface treatment on the mechanical properties of natural fibre reinforced composites have been extensively studied by Valadez-Gonzalez, *et al.* (1999); Sreekala and Thomas (2003); Thais, *et al.* (2003); Rong, *et al.* (2001); and Luyt and Malunka (2005).

The sisal yarn used in this research was obtained from James Lever Ltd of Bolton, England. Table 1 lists some of its properties.

Table 1: Properties of Sisal Fibres

Properties	**Values (SI)**
Specific Gravity	1.35
Young's Modulus (Average)	≈29 GPa
Coefficient of Thermal Expansion	16.8μm/m^{o}C – Longitudinal 70.8μm/m^{o}C – Transverse
Operating Temperature	Up to 135^{o}C

(*Source*: James Lever Ltd of Bolton, England.)

Polyester Resin

The matrix material selected for the sisal fibre reinforced composite was the General Purpose Polyester Laminating Resin supplied by ABL (Stevens) Resin and Glass Co. of

Cheshire, England. Table 2 lists some mechanical properties of the cured unreinforced polyester resin material.

Polyester is a synthetic polymer made of purified terephthalic acid (PTA) or its dimethyl ester dimethyl terephthalate (DMT) and monoethylene glycol (MEG). With 18% market share of all plastic materials produced, it ranges third after polyethlene (33.5%) and polypropylene (19.5%). Polyester is a category of polymer which contain the ester functional group in their main chain. Although there are many polyesters, the term "polyester" as a specific material most commonly refers to polyethylene terephthalate (PET) (Pai and Chandra 2013).

Depending on the chemical structure polyester can be a thermoplastic or thermoset, however the most common polyesters are thermoplastics (Rosato, *et al.* 2004). Polyester resins can be formulated with a variety of properties ranging from hard and brittle to soft and flexible. Its advantages are low viscosity, fast cure time, and low cost. (Seni, 2007)

EXPERIMENTAL PROCEDURE

Microbond Test Specimen Preparation

Generally mechanical properties of fibre-reinforced composites are evaluated based on a variety of standard tests performed on the bulk composite specimens. These tests provide useful data about the general performance of the composite when loaded. Unfortunately, fibre-matrix interfacial properties within the composite cannot be established using these established standard testing methods. Therefore, it is necessary to understand the interaction along the interface between the composite reinforcing fibre and the surrounding polymer matrix of various fibre reinforced composite systems.

Table 2: Mechanical properties of clear-cast (unreinforced) polyester resins

Tensile Strength (MPa)	Tensile Modulus (GPa)	Elong-ation (%)	Flexural Strength (MPa)	Flexural Modulus (GPa)	Compressive Strength (MPa)	Heat Deflection Temperature (^{o}C)
75	3.38	3.3	130	3.59	120	90

***(Source*: ABL (Stevens) Resin and Glass Co.)**

Making of microbond pull-out specimens basically required the placement of a liquid polyester resin microdroplet concentrically around a portion of sodium hydroxide treated sisal fibre. The placement of the droplet was done with the help of tweezers. The fibres were initially subjected to a 5% diluted NaOH treatment for duration of one (1) hour in order to improve its surface in readiness for bonding. A small amount of resin was first prepared in an open polypropylene container by mixing the polyester resin with an organic peroxide catalyst (Methyl Ethyl Ketone Peroxide). The quantity of catalyst used was 2% of the resin by weight. Both the polyester resin and the catalyst were obtained from the same supplier.

Once the resin droplets cured at room temperature, these micro composites were then placed onto three porous Petri dishes for further hydrothermal conditioning in 36 litre water baths (Figure 2) at pre-set temperatures of 23^{o}C, 40^{o}C, and 60^{o}C, respectively for a

period of 192 hours as elaborated in Table 3. During this time, periodic microbond tests were conducted on the specimens with a view to assessing the resultant effect of the hydrothermal ageing on the same microbond interfacial shear strength. For good average interfacial shear strength, each test sample consisted of between 21 to 35 test specimens, dried under vacuum in an autoclave before preparation of coupon specimens. Specimen coupons were then prepared by gluing individual fibres onto cardboard paper coupons bearing an open gauge length of 10 mm. The microbond test specimen arrangement is shown in Figure 3(a). Measurements of droplet embedded lengths and fibre diameters were taken with the aid of an Olympus optical microscope and recorded for further use in the analysis.

Isothermal water uptake studies

Isothermal water uptake studies were conducted along-side the accelerated hydrothermal ageing for the determination of the relative rate of absorption of water by the sisal-polyester composite. This was done by immersing standard composite test specimens into distilled water at different pre-set environmental temperatures of 23^{o}C, 40^{o}C and 60^{o}C respectively. This study was conducted in accordance with ASTM D570-98 standard. This test method for the rate of water absorption is significant as a guide to the proportion of water absorbed by the composite material and consequently the relationship between absorbed moisture and mechanical properties as a function of conditioning temperature. Water uptake was monitored on three (3) samples. As prescribed in the standard, each sample consisting of three (3) composite specimens (with dimensions of 76 mm x 25.5 mm x 2.5 mm). This study was done by periodic monitoring of gravimetric changes in the test specimens throughout the observation period.

Testing Procedures

Microbond Pull-out Testing

Before the experiments commenced, the upper part of the test specimen coupon was held and suspended in grips attached to an Instron 5564 tensile testing machine (see Figure 3(b)) which was coupled with a 10 N load cell. The specimen cardboard in Figure 3(a) was then cut along the indicated cutting line on either sides of the fibre to allow the fibre to freely hang with the droplet held below the two knife-edges and take the entire applied load without the cardboard interfering. The gap between the knife-edges was precisely set and kept constant to 0.35 mm through the rig micrometer as shown in Figure 4. This setting left a slight clearance between the fibre surface and tips of the knife-edges. This gap was wide enough to prevent the resin droplet from passing through the gap while being dragged along the fibre length. The cured polyester resin droplet (the matrix) was then pulled down when the test started at a constant crosshead speed of 1 mm/min until failure of the interface occurred. With the help of the testing machine's data acquisition systems, plots of the load-displacement were acquired from which the debonding force was later determined.

Figure 2: Hydrothermal Ageing Water Baths.

Table 3: Experimental hydrothermal ageing conditions for microbond test specimens.

Ageing Solution	Distilled water
Ageing Temperature *T* (^{o}C)	23, 40, 60
Immersion time *t* (hours)	0 (Unconditioned), 48, 96, 144, 192

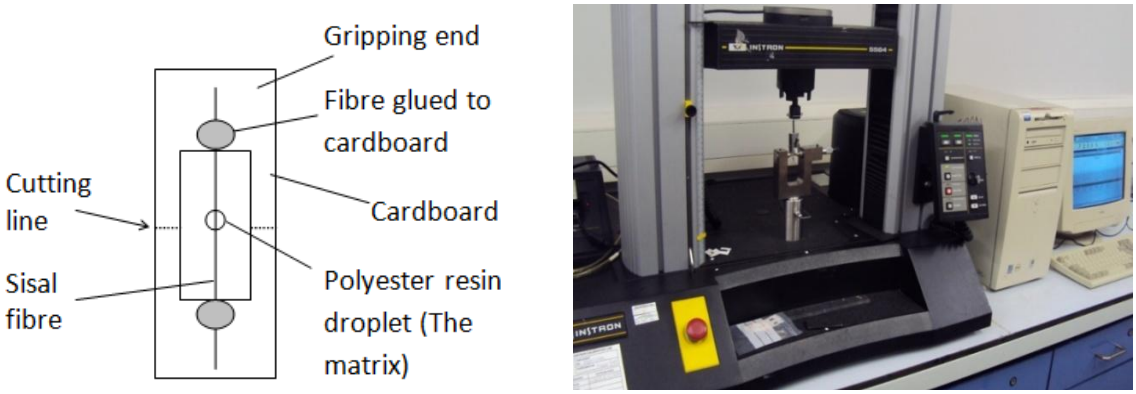


Figure 3: (a) Microbond test specimen arrangement. (b) Microbond Pull-out Testing Rig.

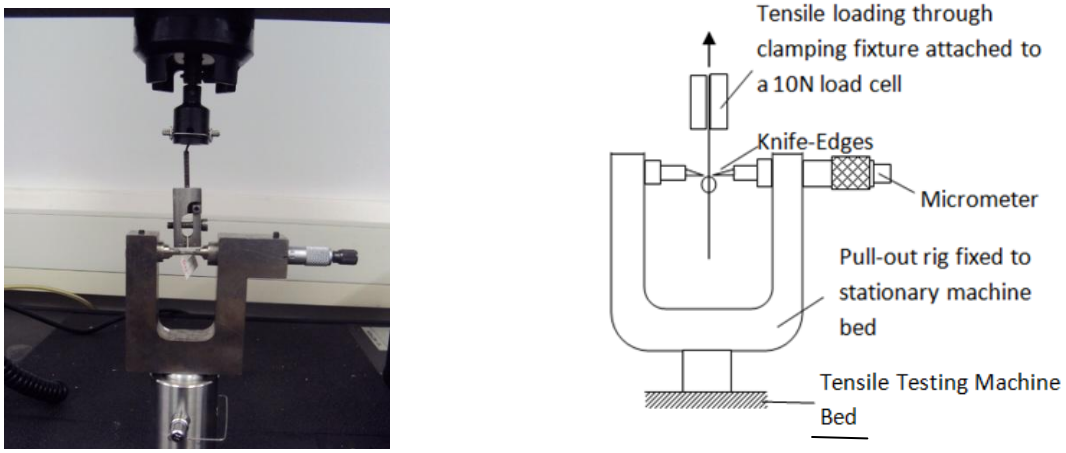


Figure 4: Close up on the microbond test pull-out grip arrangement.

TEST RESULTS

Microbond Pull-Out Test

Figure 5 shows one of the recorded plots of microbond shearing force against corresponding droplet displacement. From this plot, it is evident that the recorded force rises initially from the starting frictional force level to a peak of 0.00165 kN.

This peak defines the maximum adhesive bonding force existing at the polyester matrix/sisal fibre interface. This phase is then followed by an abrupt drop back to some lower frictional force level as the resin droplet is pulled down the fibre. This drop is indicative of the failure of the interface that binds the droplet to the sisal fibre.
For sisal fibres, this level varies from one fibre to another and is dependent upon the variations in the fibre diameter along its entire length. This variation in fibre diameter also defines the profile type of the frictional resistance the pull out test will exhibit post debonding. It can further be observed from hydrothermally treated specimens that in cases where a low microbond shearing force is recorded as a result of deterioration of interfacial, relatively higher frictional forces have been recorded. This to a much lesser extent tends to continue providing resistance to continued fibre pull-out.

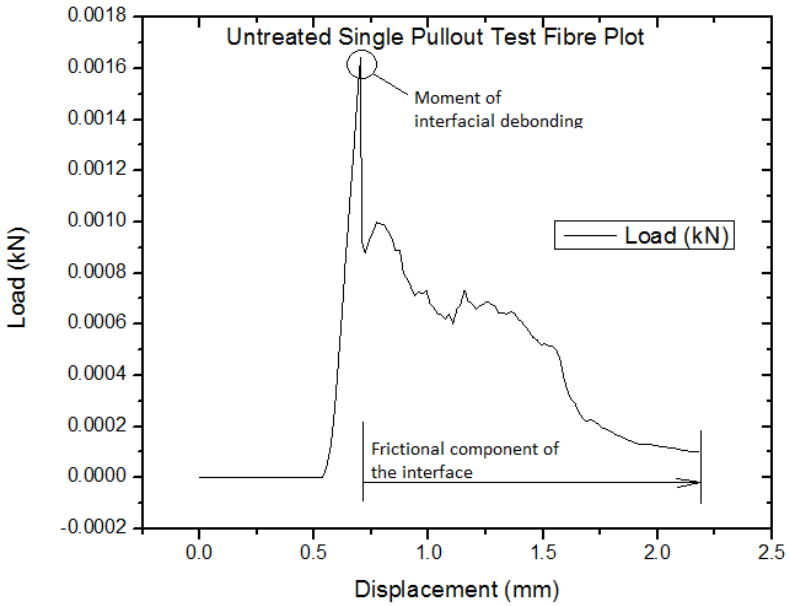


Figure 5: Single fibre pull-out test plot of a sisal/polyester micro composite.

With the fibre diameters and embedded lengths of each test specimen already measured prior to conducting of each test, the shear debonding loads determined from the microbond pullout tests are then plotted against their corresponding interfacial embedded area for that particular fibre/matrix microbond system. A collection of results from several microbond pull-out tests are shown in the sample scatter plot in Figure 6, for each particular ageing temperatures and exposure duration. All scatter plots result in well-defined linear relationships from which average interfacial shear bonding strengths are determined from the respective slopes of the trend lines.

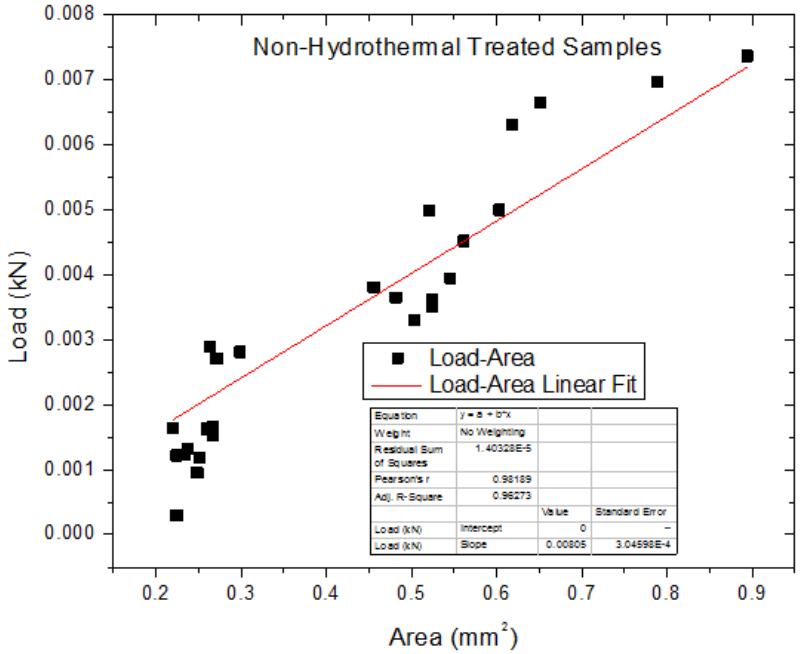


Figure 6: Microbond debonding load vs embedded scatter plot for untreated polyester resin.

Figure 7 shows resultant plots of interfacial shear strength variations for single fibre microbond tests after specimens were hydrothermally treated at temperatures of 23°C, 40°C and 60°C for a maximum period of 192 hours.

The drops in the shear strength appear to be sudden in the beginning of observation with steepest declines being more pronounced in samples treated in water at 60°C temperature. This is in agreement with results presented by Chizyuka and Kanyanga (2013) while investigating the effect of environmental effects on the same composite system macro fracture.

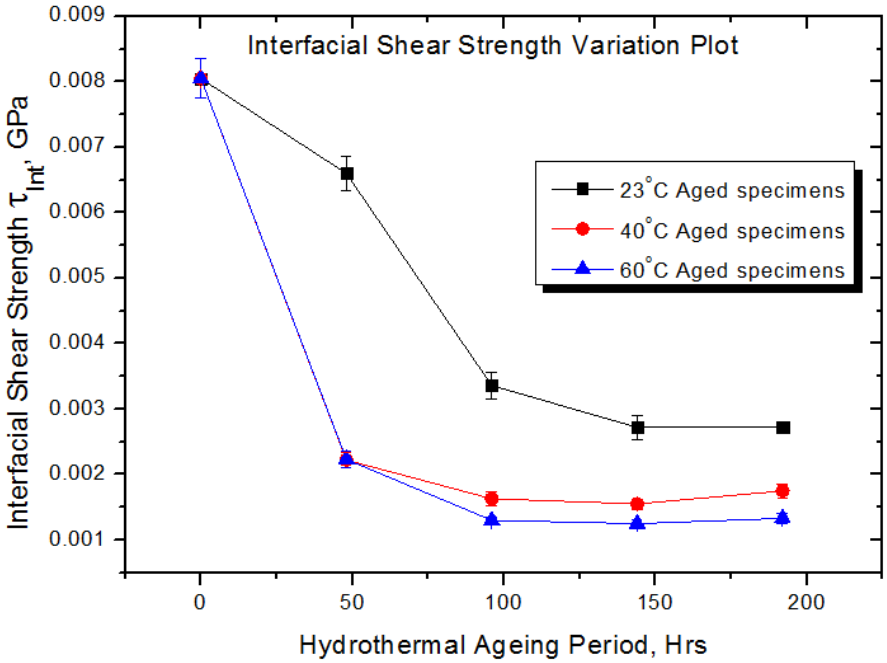


Figure 7: Resultant microbond shear stress variation plot for hydrothermally aged single fibre micro composite specimens over a maximum period of 8 days (192 hours).

For each hydrothermal treatment, microbond pulled-out specimen fibres were further examined on a NeoScope JCM 5000 Scanning Electron Microscope (SEM) for presence of residual polyester matrix material after debonding. Analysing the debonded surfaces of the sisal fibres allowed for understanding the appropriate failure mode on this composite system.

Fractographic images presented in Figure 8 and Figure 9 indicate little to no traces of the residual matrix material were present on the sisal fibre at the original location of the polyester resin droplet along the fibre. The presence of residual matrix appears to be dependent on the duration and temperature of the applied hydrothermal ageing. This supports the conclusion that possible complete interfacial bond failure occurred during the pull-out tests in some specimens as a result of the hydrothermal ageing process. This variation in the amount of residual matrix material on the fibres could as well have been attributed to changes in mechanical properties of the matrix due to the influence of the applied ageing treatment. Gautier, *et al.* (1999) in their findings associated osmotic cracking in the matrix, as well as at the interface and subsequent interfacial debonding to the overall composite failure when subjected to hydrothermal treatment.

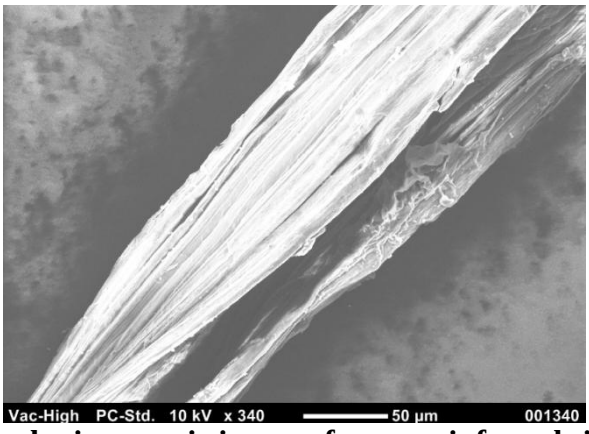
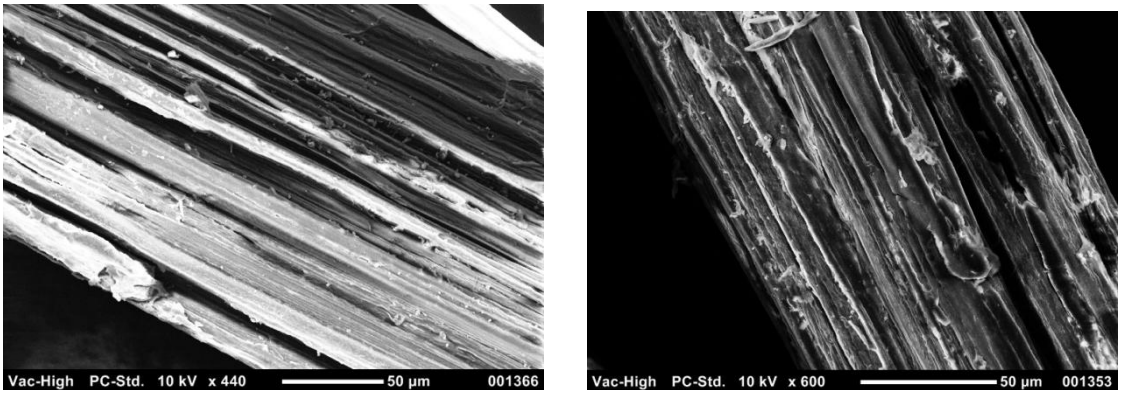


Figure 8: Scanned microscopic image of an unreinforced single sisal fibre.



(a) (b)

Figure 9: Scanning Microscopic image of the surface of a sisal fibre following interfacial debonding of hydrothermally treated sisal/polyester microbond composite system at temperature of (a) 23^{o}C for a period of two days; (b) 60^{o}C for a period of four days.

Preliminary tests on non-hydrothermally treated microbond specimens yielded average interfacial shear strengths of about 8.05E-4±3.04E-4 MPa (for details, refer to Figure 6).

Rapid drops in the interfacial strengths were observed in all of the three ageing conditions, but with steeper declines experienced in test samples which were subjected to 40°C and 60°C of hydrothermal ageing treatment, as evident in Figures 7, inclusive. The strengths drop and settle to average values of about 2.7 MPa for the 23°C treated test specimens, whereas for the 40°C and 60°C treated samples their strengths settle down to values of about 1.5 and 1.2 MPa, respectively. This can be attributed to the deterioration of the interfacial bond resulting from the hydrothermal exposure.

Figures 10 to 12 show composite plots of the relationships between moisture absorption and microbond interfacial strength with increasing exposure times for single sisal fibre polyester micro bond composite specimens hydrothermally treated at 23°C, 40°C and 60°C, respectively.

Isothermal Water Uptake

Effects of isothermal water uptake are represented by plots of the average moisture gain and microbond interfacial shear strength versus hydrothermal ageing period, shown if Figures 10 -12, inclusive, on the same graph. It is evident from the curves in these figures that the higher the temperature of hydrothermal ageing environment, the faster the composite reaches its state of equilibrium for moisture absorption. This is subsequently followed by rapid deterioration of the fibre/matrix interfacial bond.

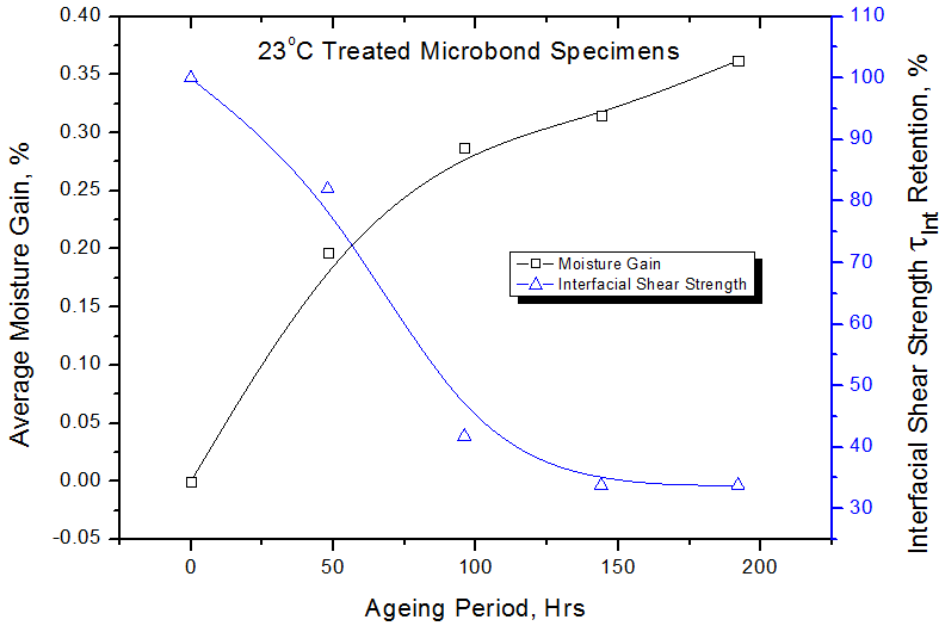


Figure 10: Composite plot showing relationship between water absorption and microbond interfacial strength with increasing exposure times for single sisal fibre - polyester micro composite specimens hydrothermally treated at 23°C.

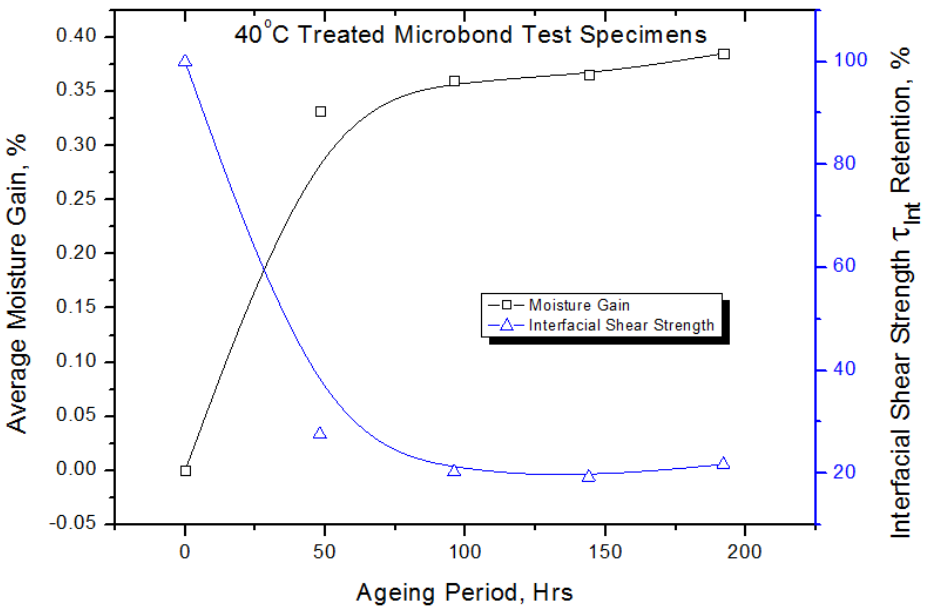


Figure 11: Composite plot showing the relationship between water absorption and microbond interfacial strength with increasing exposure times for single sisal fibre - polyester micro composite specimens hydrothermally treated at 40°C.

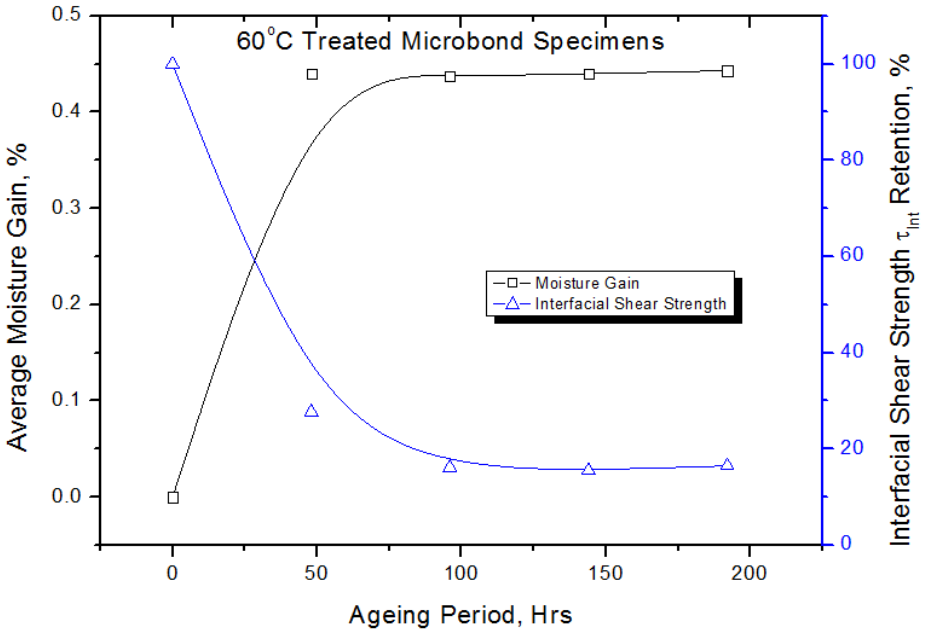


Figure 12: Composite plot showing the relationship between water absorption and microbond interfacial strength with increasing exposure times for single sisal fibre - polyester micro composite specimens hydrothermally treated at 60°C.

Based on the Fickian diffusion model, the isothermal water uptake study yielded diffusion coefficients of 0.00327 $mm^2.sec^{-1}$, 0.01277 $mm^2.sec^{-1}$ and 0.0.03695 $mm^2.sec^{-1}$ for specimens placed in the respective study environments of 23°C, 40°C and 60°C, respectively. Using Arrhenius Life-Stress analysis, the resultant activation energy of diffusion for this NaOH treated fibre reinforced composite system was determined to be 54.04 $kJ.mol^{-1}$.

Using the microbond pull-out test data, a more comprehensive model description of the effects of temperatures and absorbed water on microbond interfacial strength over time was further developed. The model was built with the help of JMP 10, a statistical software's response surface methodology central composite design. Figure 13 shows a much clearer picture of the effect hydrothermal treatment poses on the sisal reinforced polyester composite system based on the model, depicting a 3-dimensional surface plot of the microbond strength as a function of ageing period and hydrothermal ageing temperature. A corresponding model function is shown in equation (2).

$$\tau_{Int} = 0.00179 - 0.00057\alpha_{Temp} - 0.00306\beta_{Time} - 0.00035\alpha_{Temp}\beta_{Time} + 0.00039{\alpha_{Temp}}^2 + 0.00295{\beta_{Time}}^2 \quad (2)$$

where

τ_{Int} - Microbond Interfacial Shear Strength, GPa.

α_{Temp}– Temperature based parameter which is equal to,

$\alpha_{Temp} = \frac{T-40}{20}$,

where T – Ageing temperature, in ^{o}C.

β_{Time}– Duration based parameter which is equal to,

$\beta_{Time} = \frac{d-4}{4}$,

where d – Ageing duration, in days.

Equation (2) describes the variation with R^2 = 0.9898. This coefficient of variation suggests that this empirical model fits the experimental data well.

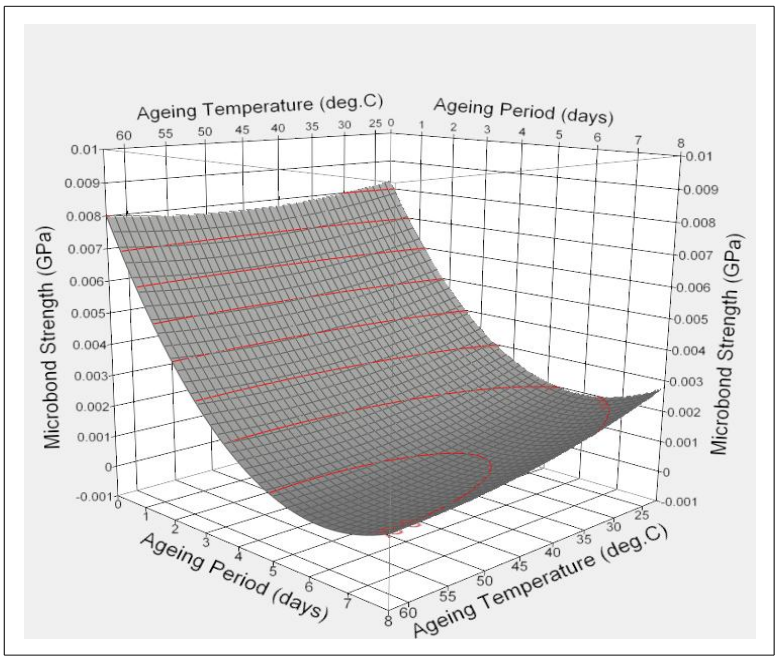


Figure 13: The fitted surface plot describing variations in microbond interfacial strength as a function of hydrothermal ageing temperature and treatment period based on the derived empirical model.

DISCUSSION

The first 144 hours of hydrothermal treatment of the microbond composite specimens yielded steep drops, with increase in the conditioning temperature, in the average interfacial shear strength of the single sisal fibre/polyester microbond composite specimens before levelling up. It was found that at the end of observation period, these ageing environments yielded total interfacial strength reductions averaging 70%, 78.2%, and 83.5% for the 23^{o}C, 40^{o}C and 60^{o}C treatment environments, respectively. This shows a presence of a thermal gradient as seen from these resultant diffusion coefficients of 0.00327 mm^{2}.sec^{-1}, 0.01277 mm^{2}.sec^{-1} and 0.0.03695 mm^{2}.sec^{-1} for the respective environments. This points to the fact that temperature enhanced the rate of water uptake into the composite material, and subsequently led to lowering of the interfacial shear strength values. This to a lesser extent contributed towards reductions, for the same composite system, in the translaminar fracture toughness (K_{TL}) of the bulk composite amounting to 32.2%, 39.5% and 46.6 % for the respective 23^{o}C, 40^{o}C and 60^{o}C ageing environments observed by Chizyuka and Kanyanga (2013). This conclusion was equally drawn by both Bhandakkar *et al* (2014) and Kim and Mai (1998).

These reductions seen in the interfacial shear strength after hydrothermal treatment of the microbond composite test specimens is mainly as a result of the reduction in the interfacial bonding existing between the fibres and the surrounding matrix material.

CONCLUSION

The moisture absorption behaviour and the influence of moisture on the interfacial strength of the single sisal fibre/polyester microbond composite test specimens were investigated as a function of ageing period under different thermal loading states.

In addition, SEM imagery of the surface of the fibre extracted from the debonded specimens (both non-aged and hydrothermally aged), revealed the harsh debonding effect of the absorbed moisture on the fibre-matrix interface. This subsequently led to the sisal fibre pullout failure mechanism to easily take place. It can therefore be concluded that this fractographic difference in the two SEM images of the fibre surfaces is one indication that debonding is an established fracture mechanism in hydrothermally aged NaOH treated sisal fibre-polyester composite systems. It is worth noting that the influence of moisture on facture toughness of this polymeric composite system is enhanced with the increase of prevailing ambient temperature coupled with the increase in exposure period.

REFERENCES

Bhandakkar A., Kumar N., Sastry, S.M.L. (2014) “Interlaminar Fracture Toughness of Epoxy Glass Fibre Fly Ash Laminate Composite.” Material Science and Applications 5:231-244.

Bledzki, A. K., Sperber, V. E., & Faruk, O. (2002). Natural and Wood Fibre Reinforcement in Polymers. Rapra Review Report, Rapra Technology Ltd. **13:** 144.

Chand, N., Tiwary, R. K., & Rohatgi, P. K. (1988). "Bibliography Resource Structure Properties of Natural Fibres – An Annotates Bibliography." International Journal of Material Science **23**: 381 – 387.

Chizyuka, C.G and Kanyanga, S.B. (2013). "Effects of Hydrothermal Ageing on the Fracture Damage of Sisal Fibre Reinforced Polyester Composites." The Zambian Engineer **46**(1): 11 - 19.

Craven, J. P., Cripps, R., & Viney, C. (2000). "Evaluating the Silk/Epoxy Interface by means of the Microbond Test." Composites **Part A**(31): 653 – 660.

Gaur, U. and Miller, B. (1990). "Effects of Environmental Exposure on Fibre/Epoxy Interfacial Shear Strength." **11**(4): 217 - 222.

Gautier, L., Mortaigne, B., & Bellenger, V. (1999). "Interface Damage Study of Hydrothermally Aged Glass-Fibre-Reinforced Polyester Composites." Composite Science and Technology **59**(16): 2329 - 2337.

Geo, Y. C. and Cotterell, B. (1988). "Fracture of Fibre-Reinforced Materials." ZAMP(Journal of Applied Mathematics and Physics) **39**: 550 -572.

"Future Fibres". [Online]. Available: http://www.fao.org/economic/futurefibres/fibres/sisal/en/ . [Accessed on 2-11-2015]

Kim, J. K. and Mai, Y. W., Eds. (1998). Engineered Interfaces in Fibre Reinforced Composites. Oxford, Elsevier Science Ltd.

Li, Y., Mai, Y. W., Ye, L. (2000). Microstructure and Mechanical Properties of Sisal Fibre and Adhesion to Vinyl Ester Resin. Proceedings of the 9th European Conference on Composite Materials (ECCM-9), Brighton, UK, IOM Communications Ltd, London.

Luyt, A. S. and Malunka, M. E. (2005). "Composites of Low-Density Polyethylene and Short Sisal Fibres: The Effect of Wax Addition and Peroxid Treatment on the Thermal Properties." Thermochimica Acta **426**: 101 – 107.

Miller, B., Muri, P., & Rebenfeld, L. (1987). "A Microbond Method for Determination of the Shear Strength of a Fibre/Resin Interface." **28**(1): 17 - 32.

Oikonomou, A. 2010. "Composite Micromechanics of Flax Fibres and Poly(L-lactic acid) Microdroplets." (a MPhil Thesis), University of Manchester, Manchester, 2010.

Pai, S and Chandra, R.Y. (2013). "Analysis of Polyester Fibre-Reinforced Concrete Subjected to Elevated Temperatures." International Journal of Civil, Structural, Environmental and Infrastructural Engineering Research and Development **3**(1): 1 - 10.

Rong, M. Z., Zhang, M. Q., Yang, g. C. & Zeng, H. M. (2001). "Treatment on the Mechanical Propertie of Unidirectional Sisal Reinforced Epoxy Composites." Composite Science Technology **61**: 1437 – 1447.

Rosato, D. V., Rosato, D. V., & Rosato, M. V. (2004). Plastic Product Material and Process Selection Handbook. Oxford, Elsevier Science & Technology Books.

Samuel, O.S., Agbo, S., Adekanye,T.A. (2012). "Assessing Mechanical Properies of Natural Fibres Reinforced Composites for Engineering Applications." Journal of Mineral and Minerals Characterisation and Engineering **11**:780 - 784.

Seni, M. I. B (2007). "The Study of Mechanical properties and Microstructure of Thermal-Curing Process on Natural Fiber (Kenaf) Reinforced Thermoset Composite". Melaka: Technical University of Malaysia.

Sreekala, M. S. and Thomas, S. (2003). "Effects of Fibre Surface Modification on Water-Sorption Characteristics of Oil Palm Fibres." Composite Science Technology **63**: 861 – 869.

Thais, H. D., S. M. Sydenstricker, S. M., & Sandro, C. A. (2003). "Pull-out and other Evaluations in Sisal Reinforced Polyester Biocomposites." Polymer Testing **22**: 375 – 380.

Thomason, J. L. and Schoolenberg, G. E. (1994). "An Investigation of Glass Fibre/Polypropylene Interface Strength and its Effect on Composite Properties." Composites **25**(3): 197 - 203.

Valadez-Gonzalez, A., Cervantes, J. M., Olayo, R. & Herrera-Franco, P. J. (1999). "Effects of Fibre Surface Treatment on Fibre-Matrix Bond Strength of Natural Fibre Reinforced Composites." Composites **30**: 300 – 320.

Yang, L. and Thomason, J. L. (2010). "Interface Strength in Glass Fibre–Polypropylene Measured using the Fibre Pull-Out and Microbond Methods." Composites **Part A**(41): 1077 – 1083.

Zhandarov, S. and Mader, E. (2005). "Characterization of fibre/matrix interface strength: applicability of different tests, approaches and parameters." Composite Science and Technology **65**: 149 - 160.