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DETERMINATION OF FIBRE-MATRIX INTEGRITY USING STRAIN RATE SENSITIVITY APPROACH

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Abstract

The integrity of the bonded fiber-matrix interface in a fiber reinforced polymer composite was investigated using the interfacial energetics approach. Plantain fibres were prepared and then treated with nine liquids to make their surfaces hydrophobic. Fiber reinforced composites were then molded, contact angles on fibres and composites were measured, composite tensile strength was determined, and fibre pullout tests were conducted. With fibre contact angles of 73.0^o and 71.3^o, respectively, mercerization (NaOH) and Methyl Ethyl Ketone Peroxide (MEKP) made the fibre more hydrophobic. MEKP and NaOH treated fibre had the lowest surface energies, the highest works of adhesion, and thus better fibre-matrix bonding when compared to other treatments (increased fibre-matrix integrity). The strain-rate sensitivity index, m, obtained ranges from 0.2264 for phosphoric acid-treated composite fiber to 0.2385 for MEKP treatment fiber, with an overall average value of 0.23410.0035 and a value of 0.2321 for untreated fiber. MEKP and NaOH treated fibers were found to be the most hydrophobic, with the highest m values, making them ideal for treating fibers for composite formation. Increased work of adhesion increased tensile energy in the pullout tests,

indicating that stronger bonding will ensure the integrity of fibre-matrix composite stability. The highest free energies of adhesion, MEKP and NaOH, also exhibit the largest pullout forces, meaning that the connection between fiber and matrix was stronger in these treatments, assuring greater fiber-matrix integrity in the composite. The conclusions of this study are important in constructions built of fiber reinforced composite materials, such as aircraft and automobile bodywork.

Introduction

There has been a lot of focus on the behavior of composite materials under stress and composite structure design methodologies using fibre-reinforced polymer matrix composites since the discovery and public declaration of carbon fibre 5 decades ago. Surprisingly few studies have attempted to explain why composite materials do not cause structures to collapse. It is necessary to do a comprehensive quantitative examination of the relationship between design and processing, as well as dependability and durability. Understanding what composite integrity means is the missing link (Beaumont and Soutis, 2016). Concerns about the suitability of engineering composites for long-term use are also included (Sinebe et al, 2019; Achebe et al, 2021).The fibre-matrix interface is a fundamental contributor to non-catastrophic behavior in composites (Smolej, et al. 2009; Vimal et al, 2015; Sinebe et al, 2020).

If the fibre-matrix interface is insufficiently bonded (which normally demands a fibre coating), cracks that reach the contact are redirected around the fibre rather than through it (Begum and Islam, 2013; Agus et al, 2015; Peter and Costas 2016). Fibre withdrawal indicates this damage-tolerant nature (NRC, 1998; Roslam et al, 2015; Sinebe et al, 2020). The amount of de-bonding mechanism and its relationship to interface qualities are key concerns for composite integrity (i.e. structural).

The specification of optimal fibre surface treatment and the qualities of any inter-phase between the matrix and the fibre or coating are other important considerations. As a result, the interface's integrity and the type of the binding are crucial (Beaumont and Soutis, 2016; Frank and Douglas, 2018; Sinebe et al, 2019).As a result, a relationship between the mechanical properties of fiber reinforced polymer composites and their surface energetics properties is required to understand the bonding mechanism and mechanical property variation between surface energy and composite mechanical strength. This research investigates the integrity of the fibre in the matrix under strain, as well as whether the bond between the matrix and the fibre is strong enough to withstand failure. We'll look at a method for calculating strain rate that considers the relationship between stress and fibre adhesive energy with the matrix in this study.

Materials and Methods

Plantain fibres were extracted from plantain pseudostem with a manual scraper (*Sinebe et al, 2019; Ray et al, 2013; Tingju et al, 2013*), prepared and treated with the following liquids: acetylation (*Dhanalakshmi et al., 2012; Bledzki et al, 2008*), acetone (*Samal, (2012*), glycerol, hydrogen peroxide, MEKP, mercerization (NaOH) (*Siregar et al., (2010*), methanol (*Aleksandra et al., 2011*), potassium permanganate (*Dhanalakshmi et al., 2012*) and phosphoric acid, to make the fibre surfaces hydrophobic.

A polyester resin was used as the matrix material. Because of the higher potential for accuracy, a cylindrical samples mould was created. Cylindrical forms of the composite material were created. Water and glycerol were used as probe liquids to determine contact angles on the fibres and matrix materials. The average values of interfacial free energies were derived from contact angles using the methods of Neumann (1975) and Fowlkes (1968), and the work of adhesion was computed for each treated material using equation (4) (Chukwuneke et al, 2015; Okpe et al, 2021). The ASTM D638 criteria were used to assess the tensile strengths of untreated and treated fibres using Universal testing equipment. The fibre pull-out from the fibre-matrix bond was performed using a Multifunctional Electric Fabric Strength Machine on untreated, treated NaOH, and MEKP plantain fibres to determine the force and elongation at pullout, as described by Lei et al, (2014). (Machine Model- YG026D).

Strain Rate Model: Backofen, et al. (1964) described and obeyed the power-law connection by describing and reporting the behavior of stress-strain rate on metals at low temperatures.

$$\sigma = [k\acute{\epsilon}^{m}]_{\epsilon,\,T} \quad (1)$$

Where

σ is flow stress, ἑ is strain rate, m is strain rate sensitivity index (0 < m < 1) as a function of parameters forming, strain rate and temperature and is associated with microstructural characteristics, and k is a dynamic modulus.

It is critical to specify the extent to which the load would have decreased over time in a structural member that is under load. At constant strain,, and temperature, T, this is assessed by a quantity, m, called the strain rate sensitivity index, which is given in eq. (2). Mohammadzadeh et al., 2014) (Hart, 1967; Gobble and Wolff, 1993; Mohammadzadeh et al., 1967):

$$m = [\partial \ln(\sigma)]/[\partial \ln(\dot{\epsilon})]_{\epsilon,T} \quad (2)$$

Where

σ is stress, è is strain rate and assuming the conditions approximate a steady state process. Stress-relaxation testing can be used to calculate the strain-rate sensitivity index in theory.

In determining strain rate, the relationship between stress and fibre adhesive energy with the matrix was taken into account. The strain rate of the composite's deformation has been defined as the ratio of work of adhesion to stress on the composite (Smolej et al, 2009):

$$\text{Strain rate} = \frac{\text{Work of Adhesion}}{\text{Tensile Strength}} \quad (3)$$

Thus, to determine the strain rate, the adhesion work (ΔF^{adh}) between fibre (f) and matrix (m) is determined using the expression by (*Chukwuneke et al, 2017; Aboelkasim et al, 2015; Zhenkun et al, 2014*):

$$\Delta F^{adh} = \gamma_{mf} - \gamma_{mv} - \gamma_{fv} \quad (4)$$

Where

γ is the interfacial surface energy in mJ/m^2 and the tensile strength *σ* is in N/mm^2.

Response Surface Methodology

The experiment was designed using the central composite design (CCD) of RSM of Design-Expert program (ver.10). The experimental design for this study will be a two-level, two-factor full factorial design with 13 experiments. The surface energy

derived from contact angle measurement was chosen as the response.

Results and Discussion

Figure 1 shows the average contact angle values, surface energies calculated from contact angle data, and change in free energy of adhesion calculated from surface energies data for untreated and treated fibre using nine (9) different treatment liquids for each test probe liquid (water and glycerol).

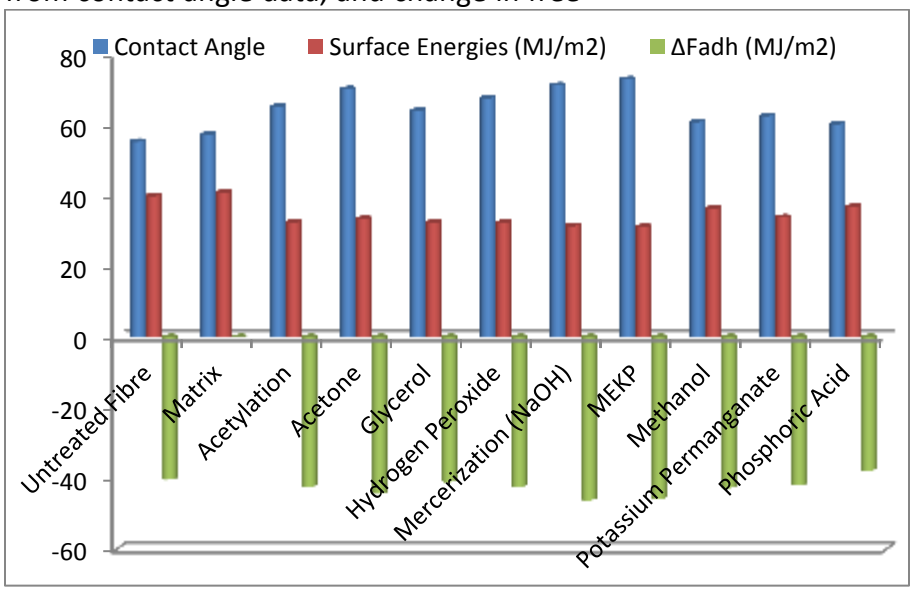

Figure 1: Average Contact Angle, Surface Energies and Change in Free Energy of Adhesion for Untreated and Treated Fibre

Figure 1 shows that in all cases, treated fibres have a higher measured contact angle than untreated fibres, indicating that they are badly wetted. As a result, fibre treatment tends to enhance the hydrophobicity of the fibre, whereas the untreated surface tends to increase the hydrophilicity. The validity of hydrophobic behavior is supported by the treatment applied to the fiber surface, which improved the contact angle.

The fibre treated with Methyl ethyl ketone peroxide (MEKP) had the highest fibre contact angle, followed by NaOH, indicating that these treatments are superior to others, whereas phosphoric acid-treated fibre has a poor fibre contact angle but is higher by about 8% than untreated fibre contact angles. The difference between the lowest and highest fibre treatments is roughly 17%.

Untreated fibres had a larger average surface free energy of 40.02mJ/m^2 than treated fibres, implying that treated fibre has the ability to reduce surface free energy. MEKP has a surface free energy of 31.24 mJ/m^2, while Phosphoric Acid has a surface free energy of 36.93 mJ/m^2. When compared to untreated fibre, MEKP, NaOH, and Phosphoric Acid treatments lower surface energy by around 22%, 21%, and 7.7%, respectively.

This demonstrates that the fibres treated with MEKP and NaOH have the highest surface energy bonding. MEKP, on the other hand, has the greatest potential as a surface energy reduction instrument (because to its proclivity for rendering the

surface hydrophobic), despite the fact that it is not widely utilized and has a potential comparable to NaOH.

The adhesion energies in Figure 1 are entirely negative, indicating attractive net van der Waals forces, which suggest the strength of the fiber-matrix bond. It is lower for untreated fibre than for all treated fibres, indicating that treating the fibres enhances the connection between the fibre and matrix, possibly increasing the composite's potency.

Treatments of fibres increase attractive van der Waals forces, which diminish the surface area at the phase boundary, resulting in a decrease in hemicellulose and lignocellulose characteristics. Treatment liquid molecules decrease free energy at the surface as a result of interactions with composite particles in the neighboring phase, resulting in a reduction in surface energy. These findings support the use of sodium hydroxide (NaOH) to make fibre surfaces hydrophobic. It also demonstrates that MEKP can be used as a fibre surface treatment.

Table 1: Average UTS, ΔF^{adh}, Strain Rate Sensitivity Index and Strain Rate of Treated and Untreated Fibres

Untreated, Treated and Unreinforced Polyester	Samples	σ - UTS (N/mm^2)	Ave	ΔF^{adh} (mJ/m^2)	Ave	Strain rate έ (10^{-9} s^{-1})	Ave	Strain rate Sensitivity Index, m	Ave
	Untreated		171.98		-40.242		0.2348		0.2321
	Acetone		190.41		-42.227		0.2225		0.2362
	Acetylation		186.63		-44.223		0.2367		0.2359
	Glycerol		177.07		-40.887		0.2329		0.2334
	Hydrogen Peroxide		185.25		-42.246		0.2288		0.2352
	Mercerization		194.43		-45.726		0.2359		0.2377
	MEKP		198.49		-46.044		0.2326		0.2385
	Methanol		179.29		-42.605		0.2384		0.2342
	Potassium Permanganate		165.51		-41.899		0.2540		0.2312
	Phosphoric Acid		148.84		-37.671		0.2540		0.2264
	Unreinforced Polyester		148.18		-		-		-

Table 1 demonstrates that the MEKP and NaOH treated fibres in the fibre-matrix composite have the highest ultimate tensile strength and free energy of adhesion, demonstrating that fibre treatment improves fibre strength. The adhesive bonding results in an increase in the composite's tensile strength. The tensile strength of Phosphoric Acid-treated fibre is low, which corresponds to the lowest free energy of adhesion.

Phosphoric Acid and potassium permanganate had lower ultimate tensile strength than the untreated sample, indicating that the treatment drastically diminishes the composite's strength. This could be explained by the fact that the chemical may have chopped up the fiber. The strength of Glycerol and Methanol treated fibres is similar to that of untreated fibres, indicating that the treatment does not significantly boost strength. Meanwhile, MEKP, Mercerization, and Acetone treated fibre has a significantly higher strength than unreinforced polyester, as well as better bonding and adhesion properties than the remainder of the treated fibre. Due to the force of adhesion and bonding effects, bonding improves the composite's strength. The adhesion energies are negative, indicating that the fiber and reinforcement are attracted to one other.

The strain rate for Acetone treatment ranges from $0.2225x10^{-9}$ s^{-1} to $0.2540x10^{-9}$ s^{-1} for Potassium Permanganate and Phosphoric Acid treatments (Table 1). These values represent the pace at which the distances between adjacent parcels of the material in the region of that fibre vary over time. In a load-bearing medium, polymer materials display this time-dependent behavior. This is a viscoelastic phenomenon that is important to the design process in thermoplastics in applications where the material is loaded at a consistent amount of deformation for an extended period of time, such as filaments in tension and seals in compression (Gobble and Wolff, 1993; Sripathi and Padmanabhan, 2016).

The low strain rates ($10^{-9}s^{-1}$) indicate that relative motions between the fibre and matrix, as well as inside the composite, are very low, and that systems treated with potassium permanganate and phosphoric acid are more likely to fail than those treated with acetone. Untreated fiber has a strain rate of the same order of magnitude. This begs the question of what role strain rate plays in fibre/matrix integrity.

The relative motions of fibres in the matrix, on the other hand, are critical. The strain rate for the fibre reinforced polymer matrix was on the scale of 10^{-9}/sec, while that for polypropylene was on the order of 10^{-6}/sec, according to the literature. This result could indicate that the friction between the fibre and the polymer matrix to which it was bound was infinitesimally small, causing no detectable change in their bonding.

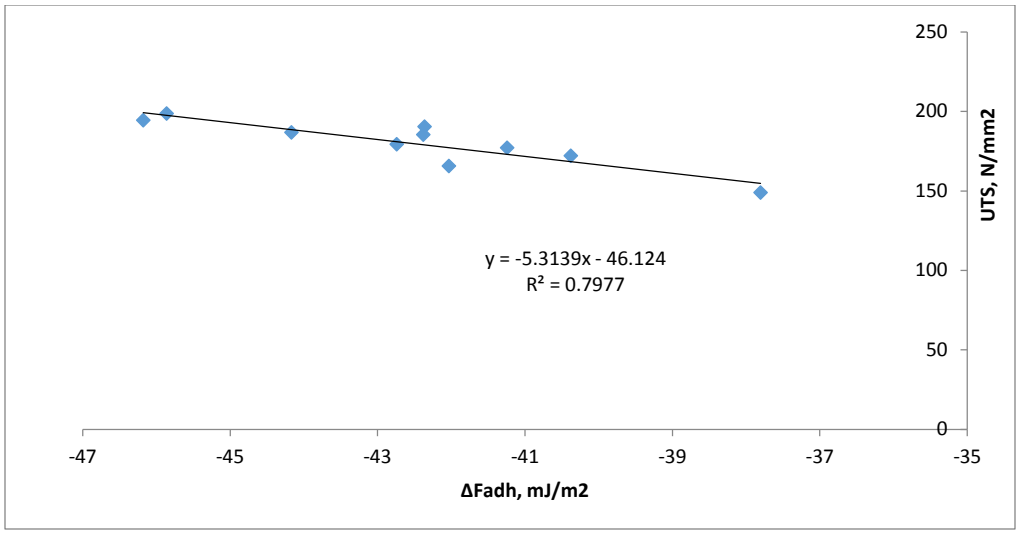

Figure 2: The effect of adhesive bonding on the tensile strength of composite

The strength of the link between the fibre and the matrix was viewed as crucial since it determined whether the fibre would simply pull out or stay in the matrix. The research revealed that, as expected, an increase in work of adhesion led to an increase in tensile energy, as illustrated in Figure 2. The integrity of the fibre-matrix composite will be ensured through stronger bonding.

Increases in free energy of adhesion (i.e. bonding energy) lead to increased tensile strength of the composite and thus fibre/matrix composite stability, as seen in Figure 2.

Table 1 shows that the strain rate sensitivity index varies from 0.2264 for Phosphorus Acid treated fiber to 0.2385 for MEKP treated fibre. The sensitivity index for untreated fibre is 0.2321. MEKP and NaOH treated fibres have the highest m-values and are hence the most desirable for composite fiber treatment. According to Gobble and Wolff (1993), m for Polyvinyl Chloride (PVC) ranged from 0.0260 to 0.687 and for high-density polyethene from 0.1291 to 0.1316. (HDPE).

According to Smolej et al. (2009), the m-values, which were calculated using actual stress, true strain curves, and the jump-test method, ranged from 0.35 to 0.70, depending on the forming conditions. Majidi et al. (2017) also showed that the m-value is not constant and is significantly dependent on the strain rate, strain intensity, and testing method used. The fact that m is higher for superior treatment liquids than for untreated fibres shows that the strain rate sensitivity index is a better attribute to examine when considering fibre-matrix integrity than plane strain rate (Nwodo, et. al. 1988; Fuguet, 2005).

In many situations, the m values calculated in this study are bigger. Using the upper limits, the value reported in this study for the polyester composite is 10.44 percent greater than the value computed for polypropylene using data from Nwodo et al. (1988). The conclusion of this study is likewise 44.8 percent higher than that of Gobble and Wolff (1993) for high-density polyethene (HDPE). Variations in material type and surface treatment could explain the variances.

Figure 3 depicts the link between tensile strength and the strain-rate sensitivity index, m.

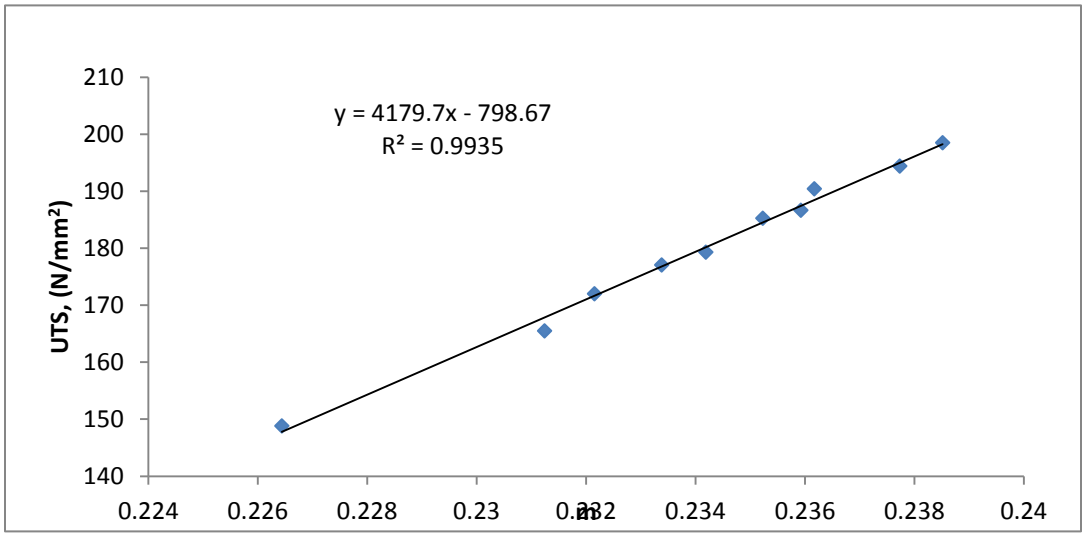

Figure 3: The Relationship between Stress and Strain-rate Sensitivity Index

That linear relationship is expressed mathematically as: = 4179.7m – 798.67. The stress and strain-rate sensitivity index are significantly associated, as evidenced by R^2 = 0.9935. The utility of m is demonstrated in this graph. Increasing m via increasing bond strength leads in a harder composite and, as a result, increased fibre-matrix stability and fiber/matrix integrity.

The bonding between fibre and matrix must be strong enough to withstand delamination, pull-out, or separation of fibre from the matrix during use for the composite to keep its integrity and remain a strong composite. Single fibre micromechanical testing investigations, one type of which is realized by applying an external load to a single fibre; fibre pullout test, micro bond test, and so on, can be used to get crucial parameters (Lei et al., 2014). Untreated, NaOH, MEKP, and Phosphoric Acid treated fibres were all considered in this study, and the results of the experiment are given in Figure 4.

Figure 4 shows the maximum pullout force, which is an average of results from three studies, together with the related free energy of adhesion, or bonding energy, for treated and untreated fibres.

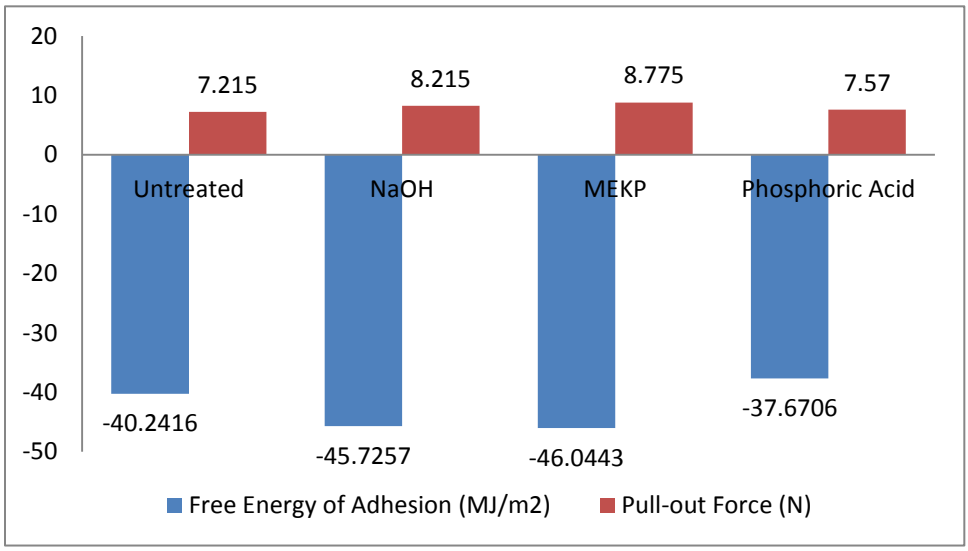

Fig. 3: Pullout Force Compared with Free Energy of Adhesion

Figure 4 shows the trend of these results in terms of adhesion free energy. It's worth noting that fibre surface treatment increases the pullout force at the same time as the free energy of adhesion, over and beyond untreated fibre values. MEKP and NaOH, which have the highest adhesion free energies, also have the highest withdrawal forces. Both high forces indicate that the connection between the fiber and the matrix was stronger in these treatments, implying that the composite will have better fibre-matrix integrity.

Figure 5 depicts the effect of contact angle and interfacial energy on the produced composite's surface free energy of adhesion for untreated, NaOH, and MEKP treated fibres. As the contact angle and interfacial energy increased, so did the surface free energy of adhesion. This suggests that the surface free energy of adhesion grows with contact angle and interfacial energy on the variability of surface properties, and that the surface energy is at its best when contact angle and interfacial energy are at their highest values. The interrelationships between the surface qualities of the fibres are shown in this diagram. The surface angle is a metric that is used to describe the surface. The change in the free energy of adhesion increases as the contact angle increases due to an increase in surface energy, as seen in Figure 5.

The significant of the model is shown in Table 2(a) by the model F-value of 6.01 for surface free energy of adhesion untreated fibre, Table 2(b) by the model F-value of 7.39 for surface free energy of adhesion NaOH treated fibre, and Table 2(c) by the model F-value of 19.56 for surface free energy of adhesion MEKP treated fibre. Due to noise, there is only a 0.06 percent to 0.07 percent chance that this big figure will occur. Model terms are significant if the "ProbF" value is less than 0.0500. It was also indicated that the factors and their interactions have a statistically significant effect on the surface free energy of adhesion of the developed composite with p-values less than 0.0500. In this case (Table 2a): A, B, A^2, B^2; Table 2b: A, B, AB, A^2, B^2; Table 2c: A, B, AB, A^2, B^2 are significant model terms. Values greater than 0.1000

indicate the model terms are not significant.

Table 3: Analysis of Variance and Coefficients for the Prediction Models

(a) Untreated Fibre

Responses	Sources of Variance	Sum of Squares	DF	Mean Squares	F-value	p-value Prob > F	Decision
Surface Free Energy of Adhesion	Model	95.16	5	19.03	6.01	0.0180	Significant
	A-Interfacial Energy	*34.42*	*1*	*34.42*	*10.86*	*0.0132*	
	B-Contact Angle	*14.37*	*1*	*14.37*	*4.53*	*0.0707*	
	AB	*2.295E-004*	*1*	*2.295E-004*	*7.242E-005*	*0.9934*	
	A^2	*28.89*	*1*	*28.89*	*9.12*	*0.0194*	
	B^2	*23.49*	*1*	*23.49*	*7.41*	*0.0297*	
	Residual	22.18	7	3.17			
	Lack of Fit	*5.03*	*3*	*1.68*			
	Pure Error	*17.15*	*4*	*4.29*			
	Cor Total	117.35	12				

(b) NaOH Treated Fibre

Surface Free Energy of Adhesion	Model	98.58	5	19.72	7.39	0.0103	significant
	A-Interfacial Energy	*19.46*	*1*	*19.46*	*7.29*	*0.0306*	
	B-Contact Angle	*3.22*	*1*	*3.22*	*1.21*	*0.0383*	
	AB	*25.53*	*1*	*25.53*	*9.56*	*0.0175*	
	A^2	*9.13*	*1*	*9.13*	*3.42*	*0.1069*	
	B^2	*45.72*	*1*	*45.72*	*17.13*	*0.0044*	
	Residual	18.68	7	2.67			
	Lack of Fit	*5.53*	*3*	*1.84*			
	Pure Error	*13.16*	*4*	*3.29*			
	Cor Total	117.27	12				

(c) MEKP Treated Fibre

Surface Free Energy of Adhesion	Model	236.98	5	47.40	19.56	0.0006	significant
	A-Interfacial Energy	*0.64*	*1*	*0.64*	*0.27*	*0.0624*	
	B-Contact Angle	*0.70*	*1*	*0.70*	*0.29*	*0.0619*	
	AB	*37.33*	*1*	*37.33*	*15.41*	*0.0057*	
	A^2	*3.51*	*1*	*3.51*	*1.45*	*0.0376*	
	B^2	*184.77*	*1*	*184.77*	*76.26*	*< 0.0001*	
	Residual	16.96	7	2.42			
	Lack of Fit	*6.71*	*3*	*2.24*			
	Pure Error	*10.25*	*4*	*2.56*			
	Cor Total	253.94	12				

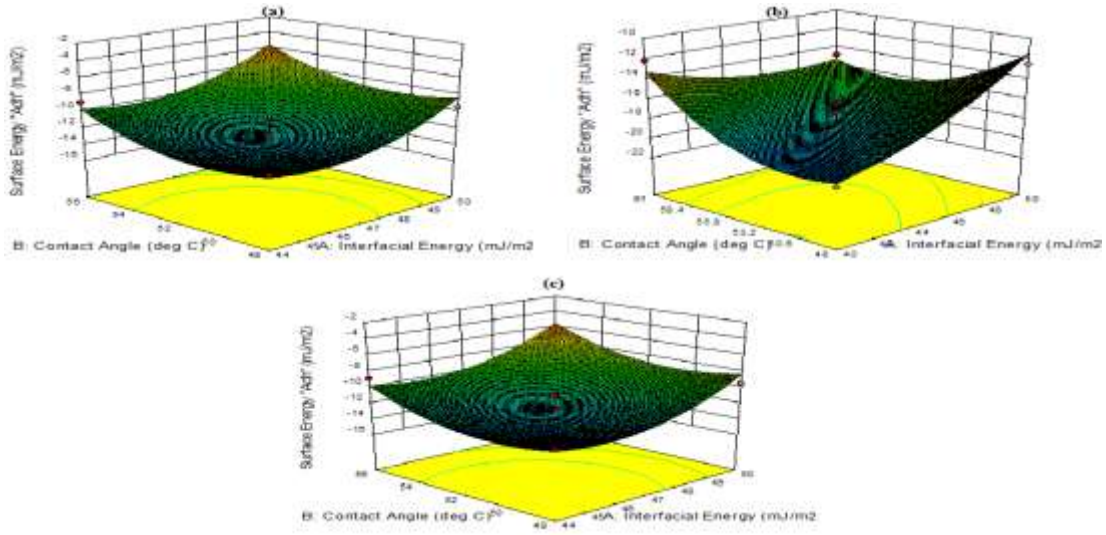

Figure 5: 3-D Response Surface Plots (a) Untreated Fibre (b) NaOH Treated Fibre (c) MEKP Treated Fibre

Table 3: Goodness of Fit and Regression Statistics

(a) Untreated Fibre				(b) NaOH Fibre			
Std. Dev.	**Mean**	**C.V %**	**PRESS**	**Std. Dev.**	**Mean**	**C.V %**	**PRESS**
1.78	-10.49	16.96	62.57	1.63	-16.36	9.98	59.86
R-Squared	**Adj. R-Squared**	**Pred R-Squared**	**Adeq Precision**	**R-Squared**	**Adj. R-Squared**	**Pred R-Squared**	**Adeq Precision**
0.8110	0.6759	0.4768	6.021	0.8407	0.6969	0.4995	7.363

(c) MEKP Fibre							
Std. Dev.	**Mean**	**C.V %**	**PRESS**	**R-Squared**	**Adj. R-Squared**	**Pred R-Squared**	**Adeq Precision**
1.56	-16.01	9.72	63.72	0.9332	0.8855	0.7491	11.867

Table 3(a) shows the Pred. R-Squared value of 0.4768 is in logical harmony with the Adj. R-Squared value of 0.6759 (i.e. the variation is less than 0.2). "Adeq. Precision" measures the signal-to-noise ratio greater than 4 is enviable. The ratio of 6.021 showed an adequate signal. Table 3(b) the Pred. R-Squared value of 0.4995 is in logical harmony with the Adj. R-Squared value of 0.6969. Adeq.

The precision ratio of 7.363 showed an adequate signal. Table 4(c) the Pred. R-Squared value of 0.7491 is in logical agreement among the Adj. R-Squared value of 0.8855 (i.e. the difference is less than 0.2). Adeq. The precision ratio of 11.867 indicates an adequate signal. This model can be used to navigate the design space. The response surface models for untreated, NaOH and MEKP treated fibres are shown in equations (5-7). The models show that the interfacial energy has high interactive effects.

$$SE^{adh} = 746.37 - 20.56A - 11.58B + 0.23A^2 + 0.11B^2 \quad (5)$$

$$SE^{adh} = 44.38 + 0.42A - 3.02B - 0.08AB - 0.06B^2 \quad (6)$$

$$SE^{adh} = -141.50 - 4.63A + 7.99B + 0.06AB + 0.67A^2 - 0.09B^2 \quad (7)$$

Conclusion

From the standpoint of interfacial energetics, the integrity of the bonded fibre-matrix interface was examined.

Methyl ethyl ketone (MEKP) and sodium hydroxide (NaOH) treatments improve fibre and matrix bonding (increased fibre-matrix integrity). The relationship between tensile strength and free energy of adhesion revealed that an increase in free energy of adhesion (bonding energy) leads to an increase in composite tensile strength and thus fibre-matrix composite stability. The low strain rates indicate that the friction between the fibre and the polymer matrix to which it was bound was infinitesimally small and would not result in a discernible change in their bonding.

Industrial uses of fibre-reinforced polymer composite constructions would be more trustworthy if they showed little or no deformation under load over time, making the fibre reinforced matrix a highly good material for industrial applications. Because the strain rate sensitivity index, m, is less than 0.30 in this investigation, it may be assumed that the reinforced plastic is relaxed and so would not be adversely affected by stress build-up at the fibre-matrix interface. Because the values of the strain rate indices studied are comparable to those reported in the literature, this work suggests that using the adhesive energy concept to compute the strain rate is valid. MEKP and NaOH treated fibres are the most hydrophobic; they also have the greatest m value, making them the most desirable for use in composite construction. Increased bond strength results in harder composite and thus improved fibre-matrix stability when m is increased.

The significant pull-out forces found in this study (MEKP and NaOH) show that the connection between fibre and matrix was stronger for these treatments, implying that the composite will have better fibre-matrix integrity. The findings of this study are critical in the design of structures built of fibre reinforced polymer composites. Wherever fibre-reinforced polymer composites are utilized in the manufacturing industry, they should be tested on a regular basis to ensure that the composite maintains its integrity and that no fibre delamination occurs.

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