Journal of Materials Science Research; Vol. 5, No. 3; 2016
ISSN 1927-0585 E-ISSN 1927-0593
Published by Canadian Center of Science and Education

# Mathematical Modelling of the Interfacial Adhesion of Date Palm/Epoxy

A. Shalwan[1], S. Oraby[1] & A. Alaskari[1]

[1] Manufacturing Engineering Technology Department, College of Technological Studies, PAAET, Kuwait City, Kuwait

Correspondence: S. Oraby, Manufacturing Engineering Technology, College of Technological Studies, PAAET, Kuwait. Tel: 965-9954-9019. E-mail: se.oraby@paaet.edu.kw

Received: January 22, 2016 Accepted: March 3, 2016 Online Published: April 20, 2016

doi: 10.5539/jmsr.v5n3p29 URL: http://dx.doi.org/10.5539/jmsr.v5n3p29

**Abstract**

In recent years, high interests has emerged to use natural fibres as alternative reinforcements synthetic due to its unique benefits regarding renewability, recyclability, degradability, lightweight, and low cost. Recent investigations revealed that the mechanical performance of fibre reinforced polymer composites (FRPCs) is predicated mainly on the interfacial adhesion of fibre with the matrices. In the current work, an empirical approach was exploited to develop mathematical models using linear regression routines available in SPSS IBM program. Such models are established to determine the functional interrelations between, each of the fibres diameters and the percentage of chemical treatment, as independent or response variables, and the interfacial bonding between the DPF and Epoxy resin. Both single fibre tensile testing (SFTT) and single fibre fragmentation testing (SFFT) are considered to study the interfacial adhesion of fibre with matrix and to reflect the real loading conditions. Such testing procedures are carried out for Date Palm Fibres (DPFs) and Date palm fibre reinforced Epoxy composites (DPFEs) with different fibre diameter (0.3-0.7 mm) under different NaOH concentrations (0-9 wt.%). Experimental testing results indicated that the optimum interfacial adhesion and strength of the fibre can be achieved with small fibre diameter when 6 wt. % NaOH concentrations is employed. The use of higher NaOH concentration generally leads to deterioration in the fibre strength. Developed models, on one hand, proved to have the capability to qualitatively and quantitatively grasp the true relationships and, on the other hand, to emphasize the high potential to utilize natural fibres as a replacement of synthetic fibres with affirmation taking into consideration the role of diameter size and chemical treatment of fibres to reach the optimum mechanical behaviour of NFRPCs.

**Keywords:** mathematical modelling, natural fibres, interfacial adhesion, fibre diameter, chemical treatment, polymer composites, date palm fibre, epoxy

## 1. Introduction

From both economically and ecologically point of views, natural fibres have become an earnest opportunity being a practical candidate alternative for synthetic reinforcements of polymeric composites. This is mainly due to their advantages compared to synthetic fibres since they are of lower cost, lighter weight, more tendency for renewable resources, more abundant, non-toxic and non- harm to skin and eyes (Fontaras & Samaras, 2010; Holmberg, Andersson, & Erdemir, 2012; Shalwan & Yousif, 2014). Additionally, from mechanically features points of view, natural fibres have been recently imposed as competitor and alternative to synthetic fibres due to their attractive properties such as less damage to processing equipment, high strength-to-weight ratio and good relative mechanical properties (Aldousiri, Alajmi, & Shalwan, 2013; Shalwan & Yousif, 2014; Yousif, Shalwan, Chin, & Ming, 2012). However, there are still several restrictions which unfavourably impact the use of such natural fibres in composites such as cross-section irregularity along the length of fibre, low moisture resistance and lack of good interfacial adhesion between the fibre and polymer matrix (Aldousiri et al., 2013; Azwa, Yousif, Manalo, & Karunasena, 2013; Chai, Bickerton, Bhattacharyya, & Das, 2012; Hejazi, Sheikhzadeh, Abtahi, & Zadhoush, 2012). It has been found (Herrera-Franco & Valadez-González, 2004; Shalwan & Yousif, 2013; Shinji, 2008; Venkateshwaran, Elayaperumal, & Sathiya, 2012; Wambua, Ivens, & Verpoest, 2003) that fibre-matrix interface and the ability to transfer stress from the matrix to fibre is the core stone aspect to determine the mechanical efficiency of fibre/polymer composite. Many researchers, for instance (Prasad & Sain, 2003; Satyanarayana, Sukumaran, Mukherjee, Pavithran, & Pillai, 1990), have reported that the natural fibre-matrix interface adhesion highly depends on the diameter size and the polarity (hydrophilic) of fibre. Many works (Prasad & Sain, 2003; Satyanarayana et al., 1990) have confirmed that a smaller diameter fibre had introduced enhancement regarding interfacial adhesion of fibre/polymeric composites. This can be mainly attributed to the widespread of the defects, lumens, and impurities in the microstructure and on the surface of fibre as well as the irregular cross-section area whenever larger diameter are used (Andersons, Spārniņš, Joffe, & Wallström, 2005; Duval, Bourmaud, Augier, & Baley, 2011; Hu, Ton-That, Perrin-Sarazin, & Denault, 2010; Placet, Trivaudey, Cisse, Gucheret-Retel, & Boubakar, 2012; Yousif & El-Tayeb, 2007). The NaOH treatment of fibres indicated a positive effect on the fibre-matrix interface adhesion as was generally able to lower the polarity between fibre and matrix (Alawar, Hamed, & Al-Kaabi, 2009; Saha et al., 2010). Furthermore, NaOH treatment assisted in the enhancement of the surface morphology of fibre and in the elimination of possible impurities. However, many researchers (Shinoj, Visvanathan, Panigrahi, & Kochubabu, 2011; Yousif & Ku, 2012) have reported that the employing of high concentration of NaOH treatment could lead to tragic effects on the fibre-matrix interface adhesion.

One efficient technique for the qualitative and quantitative assessment of a set of experimental data, that dealing with many interactive variables, is the development of the empirical modelling together with the response surface methodology (RSM). As initially defined by (Box & Wilson, 1992), RSM usually explores the hidden relationships between several explanatory variables and one or more of the measured response variables (Koshy, Rajendrakumar, & Thottackkad, 2015; Öktem, Erzurumlu, & Kurtaran, 2005). The developed models may be used as a work-floor database creator to be consulted with interpolative and extrapolative possibilities within the design stage. Building mathematical models can be a reliable filter of experimental data of somewhat stochastic nature (Oraby & Hayhurst, 1991).

It is intended in the current work to investigate, verify, and formulate the possible mutual mechanical properties-fibre diameter-chemical treatment interrelationship by using the experimental data. Response surface and contour graphs techniques are developed based on the developed models for better understanding of the functional interrelationship between the affecting and the being affected parameters. The developed models in this study can be used in a software closed loop iterative strategy in which the optimal mechanical properties of fibre reinforcement process are determined in advance.

## 2. Materials Preparation and Experimental Procedures

### *2.1 Materials Preparation*

Raw date palm meshes surrounding the steams of date palm trees were extracted from a farm in Kuwait. The fibres were separated from the meshes manually and washed up using tap water (2% detergent solution) to remove the attached dirt and dust. The extracted fibres were air dried for 48 hrs at room temperature. Using optical microscopy (NK Vision), the cleaned fibres were classified into three groups according to the diameters: set 1 (0.3 ± 0.05 mm), set 2 (0.5 ± 0.05 mm) and set 3 (0.7 ± 0.05 mm). The fibre diameter was considered as the average of three measurements taken at different cross-sections of each fibre shown in Figure 1a. The fibres were then cut off to the desired length of 40 mm. Some of the extracted fibres were treated with concentration (0–9 wt.%) Sodium Hydroxide (NaOH) for 24 hrs at room temperature. Then, the fibres were rinsed with fresh water and then dried at room temperature for 24 hrs. The fibres were classified into 12 sets according the fibre diameter ($D_f$) and concentration of NaOH solution as listed in Table1.

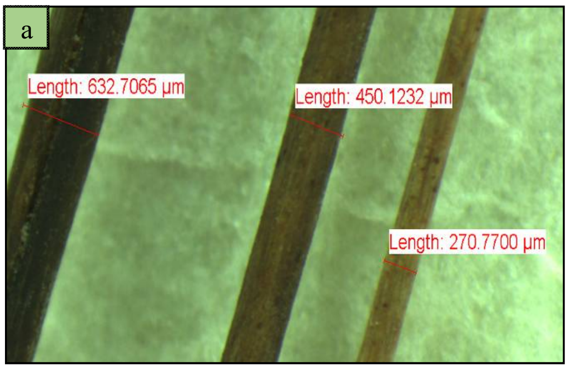


Figure 1.a Diameter measuring using OM


Figure 1.b Fibre treatment using NaOH solution

Table 1. Sample sets according the fibre diameter (Df) and concentration of NaOH solution

| Sample | S1 | S2 | S3 | S4 | S5 | S6 | S7 | S8 | S9 | S10 | S11 | S12 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Df mm | 0.3 | 0.3 | 0.3 | 0.3 | 0.5 | 0.5 | 0.5 | 0.5 | 0.7 | 0.7 | 0.7 | 0.7 |
| NaOH % | 0% | 3% | 6% | 9% | 0% | 3% | 6% | 9% | 0% | 3% | 6% | 9% |

For the matrix selection, epoxy was selected as thermo set resin as it is one of the common, cheap and widely used types in engineering applications. Epoxy resin (R246TX) and Kinetix (H160 medium) hardener, supplied by Australian calibrating services PTY. Ltd., Australia were used for the current work. As recommended by the manufacturer, the mixing epoxy-to-hardener ratio of the matrix was 3:1. The single fibre tensile experiments were conducted according to standard ASTM: D3379-7. The single fibre fragmentation test (SFFT) was carried out according to Kelly and Tyson (Kelly & Tyson, 1965). In preparation of the SFFT samples, a single fibre encapsulated in epoxy resin is placed in a mould in shape of dog bone. Figures 2a & 2b show the specimen geometry, allocation of the fibre, and dimensions of the mould.

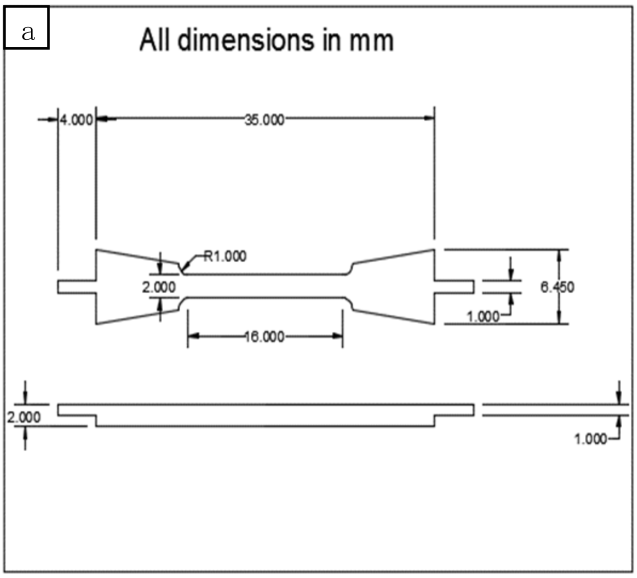


Figure 2.a The specimen geometry


Figure 2.b Allocation and encapsulation of the fibre in mould

*2.2 Experimental Procedures*

Tensile and fragmentation experiments of date palm fibre and fragmentation samples were conducted using a universal testing machine, Hounsfield Tensometer System, with a crosshead speed of 1.0 mm/min. It should be mentioned here that several tests were performed and, the closest three trends were selected and presented in the current study. The ends (terminals) of the fibres were adhered on a silicon carbide paper then placed into the gripper to avoid any slipping during the experiment. For the fragmentation tests, three samples for each set were tested and the average for each set was determined.

*2.3 Mathematical Modelling Procedures*

Mathematical models are established to relate both the fibre diameter sizes and the degree of chemical treatment of the data palm fibre to the composite mechanical properties using either multiple linear or nonlinear regression routines in the forms (1, 2, 3 or 4). A modelling strategy is followed as to start with the fitting of the simplest form (1) then, if not approved statistically, the more complex one is considered instead. However, in many situations it is found that a linear-nonlinear structure is necessary for a better representation of data.

The simplest structure is the first order linear model (1 and 2) where the response (Y) is related to the independent parameters in their natural values ($\xi$ j) or, in their natural untransformed form ($x_n$) (Box & Wilson, 1992).

$$Y = bo + \sum_{j=1}^{p} b_j \xi_j + \varepsilon_n, \quad (1)$$

$$Y = b_o + b_1(x_1) + b_2(x_2) + ........ + b_n(x_n), \quad (2)$$

where $\varepsilon_n$ is the error absolute value using linear non-transformed model while $b_o$ and $b_j$ are the estimated values of the model coefficients.

When experimental data clearly shows the existence of quadratic effect and/or interaction among the variables, a second order model of the form (3) is recommended (Kowalski, 1977; Myers, Montgomery, Vining, & Robinson, 2012).

$$Y = b_o + b_1(x_1) + b_2(x_2) + b_{11}(x_1^2) + b_{22}(x_2^2) + b_{12}(x_1.x_2), \quad (3)$$

However, when a possible nonlinear trend is evident within the experimental data, a nonlinear model of form (4) is suggested (Kowalski, 1977; Myers, Montgomery, Vining, & Robinson, 2012).

$$Y = ao\ x_1^{a1}\ x_2^{a2} ..........x_n^{an}, \quad (4)$$

In regression procedures, the least squares techniques are applied to select the model with least residuals. Models significance and adequacy are usually examined using some statistical criteria such as the determination factor R2, the t-statistic value and the F-ratio (Box & Wilson, 1992) along with the residual zero-mean uncorrelated hypothesis (Kowalski, 1977; Myers, Montgomery, Vining, & Robinson, 2012).

Surface response representation is introduced in terms of 3-dimensional graphs and contours using the selected adequate and significant model.

In the single fibre testing, regression statistical routine is employed together with the experimental data to reach the most adequate and significant relationship between each of the dependent variable; fibre diameter ($D_f$) and NaOH%, and the independent variables; ultimate tensile strength (UTS) and modulus of elasticity (MOE). For fragmentation testing, max normal stress (MNS) and max shear stress (MSS) are of concern as independent variables.

*2.4 Mechanical Properties of the Date Palm Fibre-Date*

The properties of the date palm fibres were investigated in a previous study by (Shalwan & Yousif, 2014) where the SFTT and SFFT tests were conducted on different diameter of fibre (0.3-0.7 mm) using NaOH in different concentration (0- 9 wt.%). While the aforementioned study dealt only with the plain data from mechanical viewpoint, the current study is allocated to study the effect from statistical, qualitative and quantitative point of views. Table.2 introduces a list of results measurements from SFTT and SFF testing. This includes Ultimate Tensile Stress (UTS), Modulus of Elasticity (MOE) for SFTT testing and Maximum Normal Stress (MNS), Max Shear Stress (MSS) for SFFT test.

Table 2. Experimental testing parameters and mechanical results of SFTT and SFFT

| **Sample** | | **Group** | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | **S1** | **S2** | **S3** | **S4** | **S5** | **S6** | **S7** | **S8** | **S9** | **S10** | **S11** | **S12** |
| **Df mm** | | 0.3 | 0.3 | 0.3 | 0.3 | 0.5 | 0.5 | 0.5 | 0.5 | 0.7 | 0.7 | 0.7 | 0.7 |
| **NaOH %** | | 0% | 3% | 6% | 9% | 0% | 3% | 6% | 9% | 0% | 3% | 6% | 9% |
| **SFTT** | UTS (MPa) | 115.7 | 151.2 | 145.2 | 133.6 | 106.7 | 129.5 | 118.1 | 110.2 | 63.3 | 123.2 | 86.7 | 78.4 |
| | MOE (GPa) | 2.55 | 2.88 | 2.8 | 2.7 | 2.68 | 3.06 | 2.85 | 2.75 | 1.36 | 2.87 | 1.89 | 1.31 |
| **SFFT** | MNS (MPa) | 51.7 | 52.7 | 67.1 | 57.1 | 51 | 50.7 | 66.7 | 56 | 44.5 | 50.3 | 60.9 | 50.1 |
| | MSS (MPa) | 9.92 | 9.96 | 10.56 | 10.08 | 5.76 | 5.84 | 6.06 | 6 | 3.14 | 4.88 | 4.01 | 3.95 |

## 3. Results and Discussion

### *3.1 Single Fibre Tensile Testing*

#### 3.1.1 Ultimate Tensile Strength

Figure 3 shows a response surface together with its counterpart contour graph for the row experimental data obtained regarding the effect of both the fibre size (diameter, $D_f$) and the chemical treatment concentration (NaOH%) on the Ultimate Tensile Strength (UTS). Increasing NaOH concentration up to 3 wt.% is observed to increase UTS that is reduced for any further increase in concentration. However, the fibre diameter range (0.3 – 0.5 mm) is observed not to have a significant influence on UTS while it tends to have different effect at 0.7 mm. At 3% NaOH, UTS is suddenly increased and then the trend is reversed for any further increase in NaOH concentration. Accordingly, the overall surface trend may be tentatively considered as a two-stage that can be postulated as a linear-nonlinear structure, Equation 5.

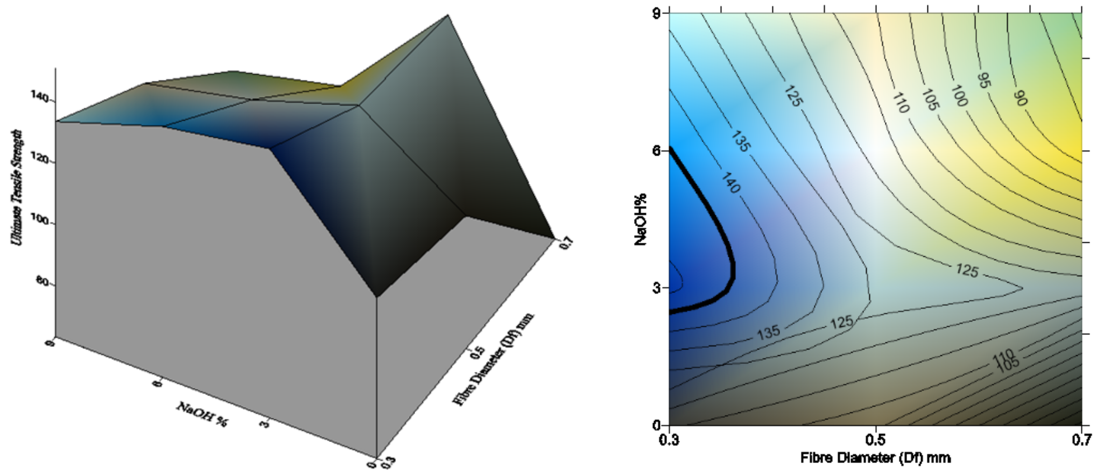


Figure 3.a Response surface Figure 3.b Contour graph

Figure 3. Experimental data for fibre diameter-NaOH%-UTS

Each stage has been processed individually using the corresponding experimental results in association with relevant linear or nonlinear regression procedures. Such procedures have led to the prediction model, Equation 6, with statistical measures indicating the significance and the adequacy of both the two parts of the proposed model, Table.3.

$$Ultimate\_Tensile\_Strength\,(UTS) = \begin{cases} ao + a1.(D_f) + a2.(NaOH\%) \Rightarrow (0 \geq NaOH\% \geq 3) \\ bo.(D_f)^{b1}.(NaOH\%)^{b2} \Rightarrow (3 \geq NaOH\% \geq 9) \end{cases} \quad (5)$$

$$UTS = \begin{cases} 145.48 - 100.5.(D_f) + 13.13.(NaOH\%) \Rightarrow (0 \geq NaOH\% \geq 3) \\ 117.02.(D_f)^{(-0.435)}.(NaOH\%)^{(-0.189)} \Rightarrow (3 \geq NaOH\% \geq 9) \end{cases} \quad (6)$$

Table 3. Statistical criteria for Two-Stages model postulation for UTS-Df-NaOH relationship

| | Correlation Factor | F-Ratio | t-statistics | | |
|---|---|---|---|---|---|
| | R2 | | βo | B1 | B2 |
| Part a: Linear | 91.1 | 15.35 | 9.3 | -3.5 | 4.3 |
| Part b: Nonlinear | 88 | 457 | 8.4 | -5.6 | -3.2 |

Figures 4&5 show the response surface and the contour graph of the developed UTS model, Equation 6,.In the first stage, UTS linearly increases as NaOH increases up to 3% concentration, Figure 4. Within such interval, it is shown that increasing $D_f$ also leads to a decrease in the UTS as shown by the contour graph, Figure 5. In the second stage, it is observed that any increase in either NaOH concentrations over 3% or $D_f$ size leads to a nonlinear decrease in UTS.

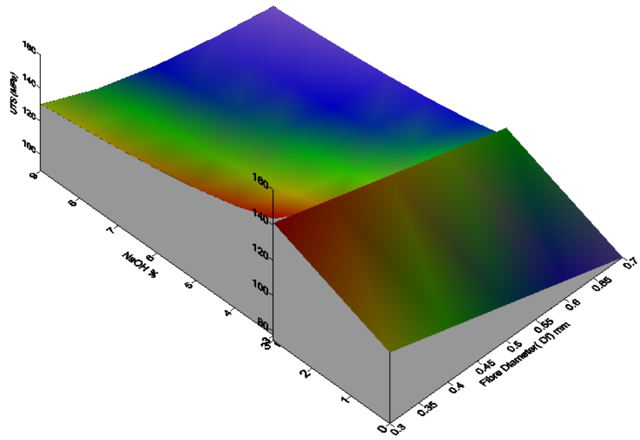

Figure 4. Three-dimensional surface for fibre diameter-NaOH%-UTS relationship

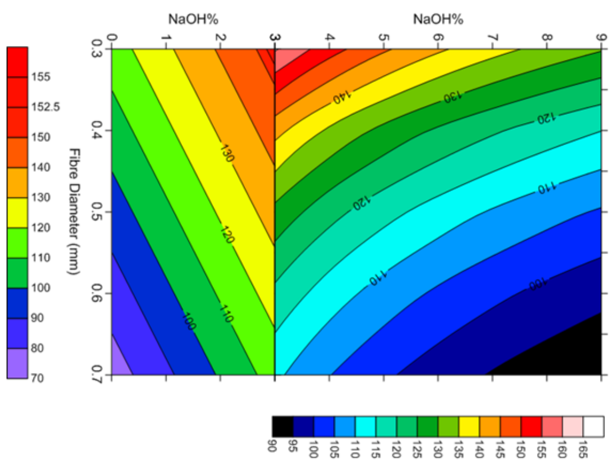


Figure 5. Contour graph for fibre diameter-NaOH%-UTS relationship

3.1.2 Modulus of Elasticity

Figure 6 shows a plot of the experimental results regarding the effect of chemical treatment and fibre size on the model of elasticity. Plots reveal high discrepancy where a definite conclusion cannot be virtually grasped. Applying all possible model structures and fitting techniques has led to the best possible two-stage model Equation 7. As found out in the last section, operational domain can be divided also into two parts according the concentration of NaOH employed during chemical treatment of the fibre. Table.4 lists the statistical criteria of the developed model indicating somewhat low measures that reflecs the data deterministic nature. As shown in the response surface and contour graphs, Figure 7, Modulus Of Elasticity (MOE) increases by increasing NaOH concentration up to 3% after which, it tends to decrease. Also, a fibre with larger diameter size usually leads to a reduction in the MOE linearly with greater extent within an operational domain of NaOH% $\leq$ 3.

$$MOE = \begin{cases} 2.95-1.5.(D_f)+0.247.(NaOH\%) \Rightarrow (0 \geq NaOH\% \geq 3) \\ 4.214\text{-}1.925.(D_f)\text{-}0.114.(NaOH\%) \Rightarrow (3 \geq NaOH\% \geq 9) \end{cases} \quad (7)$$

*3.2 Single Fibre Fragmentation Testing*

3.2.1 Max Normal Stress MNS

Experimental results for the relationship between the Max Normal Stress (MNS) and both NaOH concentration and fibre diameter $D_f$ are plotted in Figure 8. Strong attitude and trends are revealed that up to 6% NaOH, max normal stress increases at all fibre diameter employed. Using a greater concentration of NaOH chemical treatment leads to a reduction in MNS. On the other hand, while a slight increase in MNS is observed as $D_f$ increases from 0.3 to 0.5, its attitude is reversed at larger diameter size of 0.7 mm. To formulate the MNS-NaOH-$D_f$ relationship, the same modelling procedures that followed in the last sections are applied as a two-stage postulation approach but with different boundary conditions, Figure 9. Fitting procedures led to the establishment of the two-stage model structure expressed by Equation 8. As indicated by fitting and statistical measures listed in Table 5, the developed model has an excellent adequacy and significance. As NaOH concentration increases, maximum normal stress increases nonlinearly reaching its maximum value at 6% NaOH concentration. Further increase in NaOH concentration seems to nonlinearly deteriorate the fibre max normal stress, Figure 10. Besides, it is observed that a larger size fibre diameter usually leads to lower maximum normal stress regardless the chemical treatment employed.

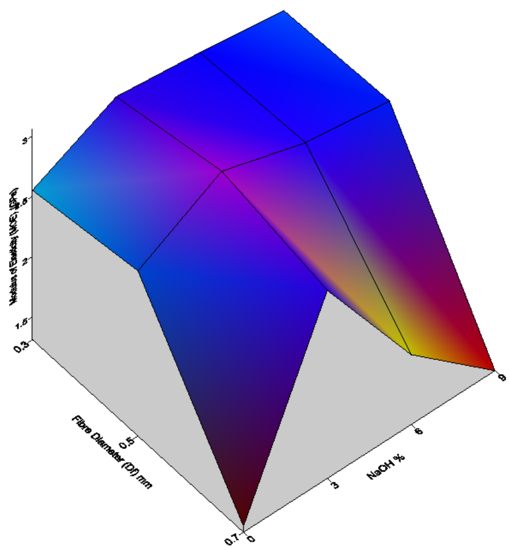


Figure 6.a Response surface

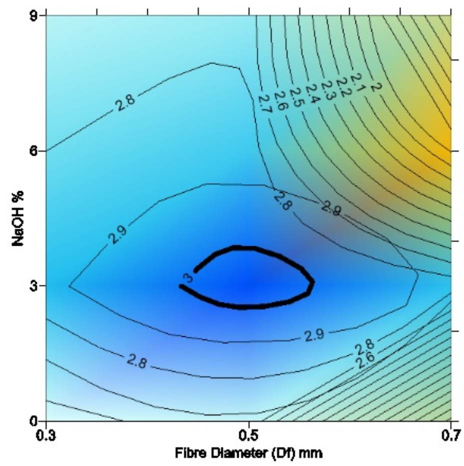


Figure 6.b Contour graph

Figure 6. Experimental data for fibre diameter-NaOH%-MOE

Table 4. Statistical criteria for Two-Stages model postulation for MOE-Df-NaOHrelationship

| | Correlation Factor $R^2$ | F-Ratio | t-statistics | | |
|---|---|---|---|---|---|
| | | | βo | B1 | B2 |
| Part a: Linear | 62 | 2.5 | 4.4 | -1.22 | 1.85 |
| Part b: Linear | 60 | 4.47 | 7.4 | -2.2 | -2.0 |

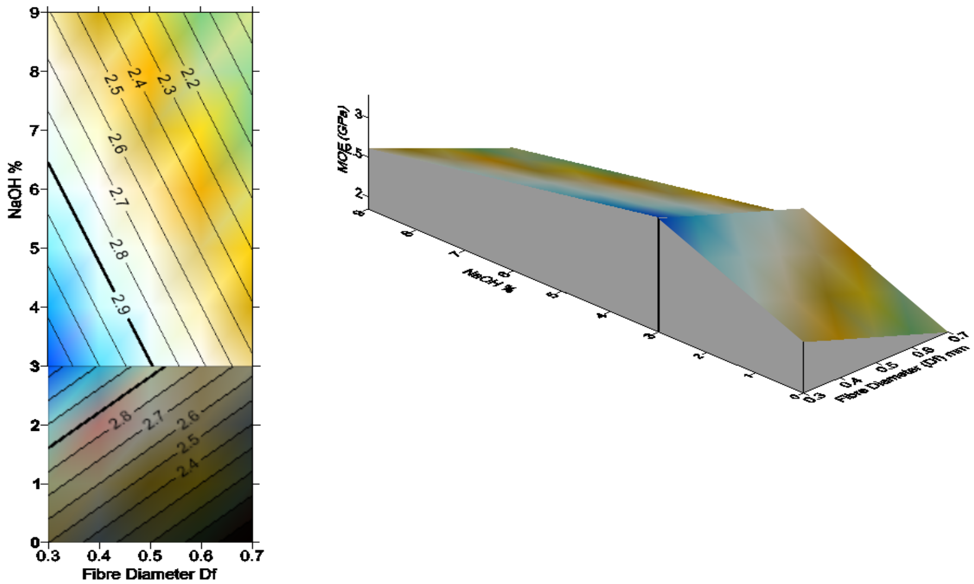


Figure 7. Response surface and Contour Graph for fibre diameter-NaOH%-MOE

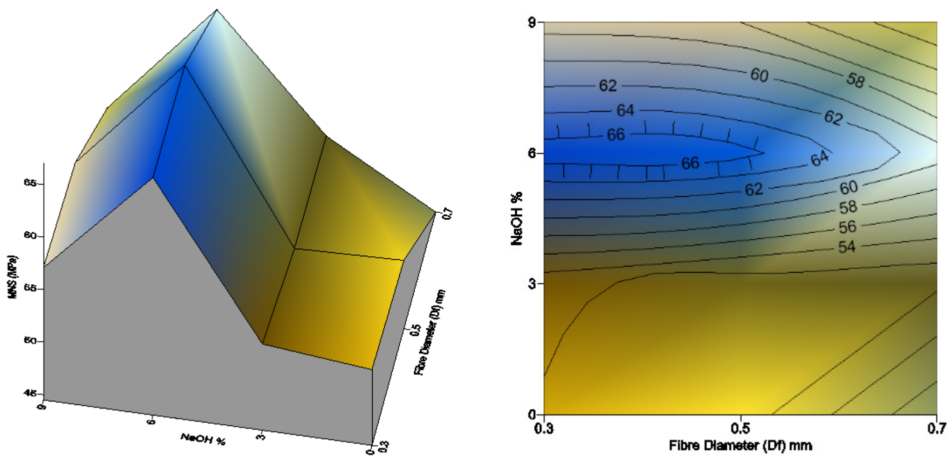


Figure 8. Experimental Data for Max Normal Stress-NaOh%-Df

$$Max\_Normal\_Stress = \begin{cases} 54.529\, e^{(-0.274 D_f)} + e^{(0.474 NaOH\%)} \Rightarrow (0 \geq NaOH\% \geq 6) \\ 129.127 (D_f)^{(-0.118)} (NaOH\%)^{(-0.434)} \Rightarrow (6 \geq NaOH\% \geq 9) \end{cases} \quad (8)$$

Table 5. Statistical criteria for Two-Stages model postulation for MNS-Df-NaOH relationship

| | Correlation Factor $R^2$ | F-Ratio | t-statistics | | |
|---|---|---|---|---|---|
| | | | βo | B1 | B2 |
| Part a: Nonlinear | 96.2 | 2855 | 25 | -3.6 | 36 |
| Part b:Nonlinear | 93 | 1436 | 6.5 | -2.7 | -5.6 |

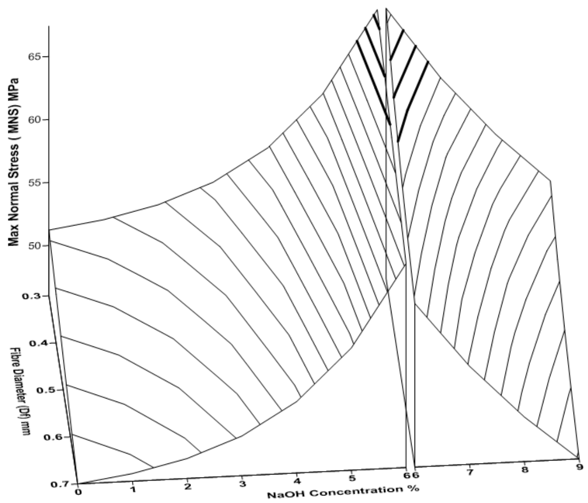


Figure 9. Surface representations for MNS-NaOH%-Df relationship

3.2.2 Max Shear Stress MSS

Figure 11 shows a response surface of the experimental row data representing the true functional relationship between the max shear stress (MSS) and both the NaOH concentration and the fibre size $D_f$. A clear and tight trend is observed where MSS is positively and steadily affected as fibre size increases. On the other side, it is observed that the chemical treatment has almost no effect on the max shear stress. All these observations are reflected in the fitting procedures where a one variable ($D_f$) nonlinear model, Equation 9, is resulted to reveal the relation. The high correlation factor $R^2$ of 98%, together with the very high F-ratio of 1641 and excellent parameters t-statistics of 17 and -19 confirm the adequacy and significance of the developed model.

The estimation capability of the developed model is examined where the predicted values of max shear stress are plotted against the experimental corresponding counterpart as shown in Figure 12a. Except for cases 9 and 10, which is less ± 1% deviation, Figure 12b, an excellent correlation is observed.

$$Max\_Shear\_Stress\ \ (MSS) = 2.76\ D_f^{\ (-1.081)} \tag{9}$$

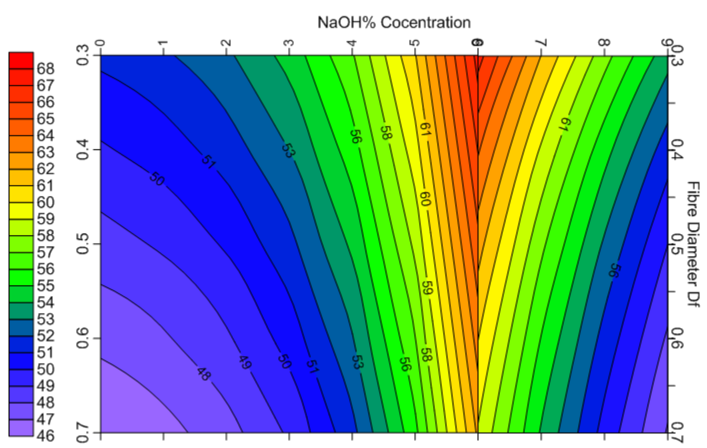


Figure 10. Contour graph representation for MNS-NaOH%-Df relationship

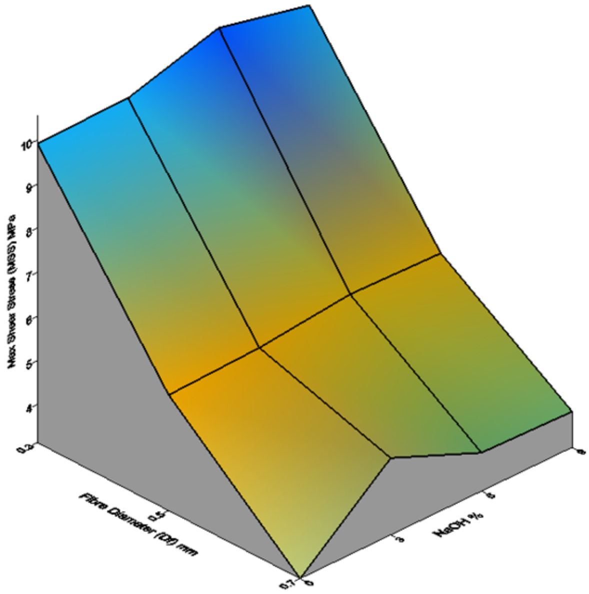


Figure 11. Surface representations for MSS-NaOH%-Df experimental results

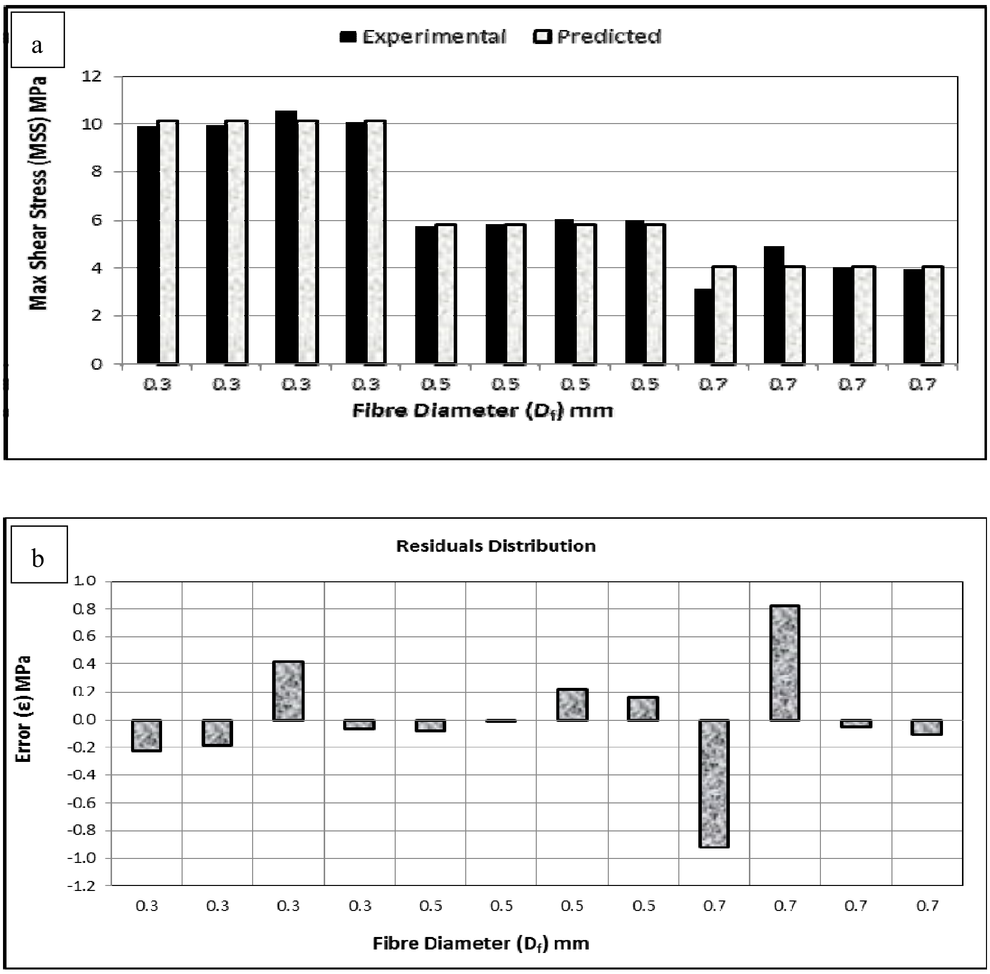


Figure 12.a Experimental vs. Predicted values Figure 12.b Residuals values and distribution

Figure 12. Comparison between experimental and predicted values for MSS

## 4. Conclusions

- Mathematical modelling approach proved to be efficient to reveal the complex interrelationships between the mechanical properties, the geometry and the chemical treatment method of the palm fibre within its reinforcement processes. The best developed model is selected among many iterative statistical fitting procedures including the preferences between linear and nonlinear structures. In most situations, it was necessary to propose the use of a two-stage linear-nonlinear approach. Model adequacy and significance are examined using statistical and ANOVA criterion measures.
- For the qualitatively and quantitatively assessment of the functional true trends of the encountered variables, the developed models have been employed to generate response surfaces maps in terms of 3-dimensional and contouring graphs.
- In the single fibre tensile testing, the linear-nonlinear two-stage shows that, in the first stage, UTS linearly increases as NaOH concentration (up to 3%) increases. Within such a domain, $D_f$ exhibits a negative effect regarding UTS. However, within the second stage, it is observed that both NaOH concentrations, over 3%, and $D_f$ has a negative impact on UTS with relatively higher effect for the latter.
- In the single fibre tensile testing, Modulus Of Elasticity (MOE) is found to be positively affected by NaOH up to a concentration of 3% after which it tends to decrease. Also, a fibre with larger diameter size usually leads to a reduction in the MOE linearly with greater extent within an operational domain of NaOH% $\leq$ 3.
- In the single fibre fragmentation testing, a 6%NaOH concentration showed its max nonlinear positive effect on NSS concentration. Further increase in NaOH concentration seems to nonlinearly deteriorate the fibre max normal stress. Besides, it is observed that larger size fibre diameters usually lead to lower maximum normal stress regardless the chemical treatment employed.
- In the single fibre fragmentation testing, a very solid conclusion is obtained where MSS is positively and steady affected as fibre size increases. However, it is observed that the chemical treatment has almost no effect on the max shear stress.

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