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Micro-structural Analysis, Optimization and RSM-based Modeling for Impact and Flexural Strengths of Polymer Bio-composite Reinforced Epoxy Natural Fiber Materials

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Abstract: *The use of composite materials with natural fiber reinforcement (CMNFR) has experienced rapid development in the automotive industry to apply synthetic materials that are expensive and not environmentally friendly. Bio-composite is a composite consisting of a polymer matrix and natural fiber reinforcement. Natural fiber materials are used as a substitute for conventional non-renewable reinforcing materials. This study uses the hand-lay process to fabricate composites using woven kenaf and jute from natural fiber. Response Surface Methodology is used to obtain the optimal response value based on micro-structural analysis. The factors used were NaOH treatment (4, 6, 8 wt%), post-curing temperature (80, 100,), and fiber type (KF, JF, KF-JF). The study's results found a combination of optimization of CMNFR manufacturing parameters using RSM, namely, A1B1C2 with a mean material strength of 0.241 J/ mm2 (impact strength) and a combination of A1B3C1 parameters with a mean material strength of 94.8650 MPa (flexural strength). It was also found that the presence of contaminants and voids harmed the mechanical properties.*

KEYWORDS: *Polymer bio-composite material; micro-structural analysis; natural fiber material; mechanical strengths; response surface-methodology.*

1. INTRODUCTION

The automotive industry's development is growing rapidly, so many researchers are developing their research in the automotive field related to the development of composite materials (Okoronkwo et al. 2022). The use of synthetic materials in the production process on a large scale is detrimental to the environment and requires high production costs (Alzebdeh, Nassar, and Arunachalam 2019). The

negative environmental and economic impacts of using synthetic fibers encourage the need for alternative materials with lower production costs, environmental friendliness, and good mechanical properties (Okoronkwo et al. 2021).

Natural fiber is an environmentally friendly material used as a reinforcement to make biocomposites in various industrial applications

(Yashas Gowda et al. 2018). The development of natural fiber-reinforced composite materials as an alternative to synthetic materials provides the advantages of lower production costs, safety, environmental friendliness, and good mechanical properties (Alzebdeh, Nassar, and Arunachalam 2019). According to the reference, there are shortcomings in natural fibers. Namely, they cannot be applied to the requirements of high material strength and water absorption and cannot withstand high temperatures (Sanjay et al. 2019).

One source of natural fiber is kenaf fiber (KF) (Sathyaseelan, Sellamuthu, and Palanimuthu 2021). Kenaf or *Hibiscus cannabinus* is an annual herbaceous plant from the Malvaceae family, which is very important for making ropes, sacks, and carpets because of its fiber content. From this point of view, KF can be chosen as fiber reinforcement because it is cheap, has high availability, has less production time, is easy to process, and is highly preferred by users for biocomposite fabrication (Prakash and Viswanthan 2019). KF has been the most widely used natural fiber as thermoset and thermoplastic reinforcement for more than 20 years, offering the most sustainable option to reduce the use of synthetic fibers (Saba et al. 2016). Likewise, hemp fiber appears to be cheaper and easier to obtain than natural fibers as compared to allowing a high fill rate, thereby saving costs in composite manufacturing (Dilfi et al. 2018). Jute fiber (JF) is one of the most do. Previous research has been conducted on kenaf fiber-reinforced composites (Khan et al. 2020, 2020; Prakash and Viswanthan 2019). There is a study on the mechanical characterization of hybrid epoxy-based composites reinforced with vetiver fiber and kenaf fiber. Five types of composite laminates have been developed using the hand lay-up process, varying the percentage of vetiver and kenaf fibers. The results showed that the properties of the epoxy composite were improved by hybridization with vetiver and kenaf fibers. The improved mechanical properties of the fiber-reinforced composites were found in the percentage composition of the kenaf fiber (Prakash and Viswanthan 2019). Then, there is research on fabricating and characterizing Echinoidea spike particles and kenaf natural fiber-reinforced *Azadirachta indica* blended epoxy multi-hybrid biocomposite. The results showed that the addition of surface-treated sea urchin particles and kenaf fibers improved the mechanical properties of the composite. Similarly, thermal effects exposed that the addition of sea urchin bioceramic filler increased the thermal stability of neem epoxy biocomposite. Scanning electron microscopy (SEM) showed uniform dispersion of sea urchin filler and improved adhesion of kenaf fiber with epoxy (Prakash and Viswanthan 2019). Research on

the effect of cellulose nanofibers and nano clays on the mechanical, morphological, thermal and dynamic mechanical performance of kenaf/epoxy composites is presented (Okoronkwo et al. 2019). The results obtained showed that the integration of rigid composite natural fiber as fillers improves the mechanical and thermal properties, storage and loss modulus, while a considerable reduction in Tan δ was achieved compared to kenaf/epoxy composites and the results obtained showed that OMMT and composites natural fiber-based kenaf/epoxy composites can be an efficient alternative for construction applications (Khan et al. 2020).

Jute seems less expensive and easily available compared to the opposite natural fibers, allowing high filling levels to save composite manufacturing costs (Dilfi et al. 2018). Jute fiber (JF) is one of the most main dominating bast fibers that are used as reinforcing materials for CMNFR (Sarker et al. 2019).

In previous studies (Devireddy and Biswas 2017; Dilfi et al. 2018; Sarker et al. 2019), there has been research on the effect of surface modification of jute fiber on the mechanical properties and durability of jute fiber reinforced epoxy composites. This study investigated the influence of various surface modifications on the properties and durability of jute fiber reinforced epoxy composites. The jute fibers have been surface modified to improve their compatibility with the hydrophobic epoxy matrix. The surface modifications examined include alkali treatment, silane treatment, and the combined effect of alkali and silane treatment. The results showed that the combined improvements provided better mechanical and thermal properties of the composite compared to the untreated composite due to the strong interfacial adhesion of the fiber matrix. The water absorption of the chemically treated fiber composite was lower than that of the untreated fiber composite. The mechanical and thermomechanical properties of composites were significantly reduced after exposure to moisture. This may be due to fiber degradation and delamination at the interface when the composite sample is immersed in water (Dilfi et al. 2018). Then, the ultrahigh performance of nanoengineered graphene-based natural jute fiber composites is researched. The results obtained showed that the nanoengineered graphene-based natural jute fiber acts as a new fiber structure (NFA) that significantly improves mechanical properties and performance (Sarker et al. 2019). Then, the physical and mechanical behavior of unidirectional banana/jute fiber-reinforced epoxy-based hybrid composites is researched (Ezeokpube et al. 2021). In this study, the present investigation deals with the fabrication and mechanical properties of unidirectional banana/jute hybrid fiber-reinforced composites and

compares them with single natural fiber-reinforced composites. The results achieved with hybrid composites have promising results compared to individual fiber composites (Devireddy and Biswas 2017).

The optimization of CMNFR was successfully carried out by previous researchers (Devireddy and Biswas 2017; Dilfi et al. 2018; Khan et al. 2020, 2020; Prakash and Viswanthan 2019; Sarker et al. 2019), but all of them focused on the analysis of mechanical strength only. This has an impact on not being optimal in determining the strength of the CMNFR material. A good optimization technique is needed to obtain optimal CMNFR parameters through an experimental process. This study will comprehensively explain the application of the experimental method with the Taguchi-Response Surface Methodology (TM-RSM) hybrid approach. Research on experimental methods with the TM-RSM hybrid approach in the field of natural fiber-reinforced composites is still relatively new among researchers in Indonesia. In this study, RSM and Taguchi, in a practical sense, should be an alternative, and even complement each other's shortcomings and when these two methods are integrated, the optimization concept in RSM will coexist with the Robust concept in Taguchi. This study can be an additional reference on optimizing CMNFR manufacturing with the TM-RSM hybrid approach for researchers, practitioners, and companies in the automotive industry.

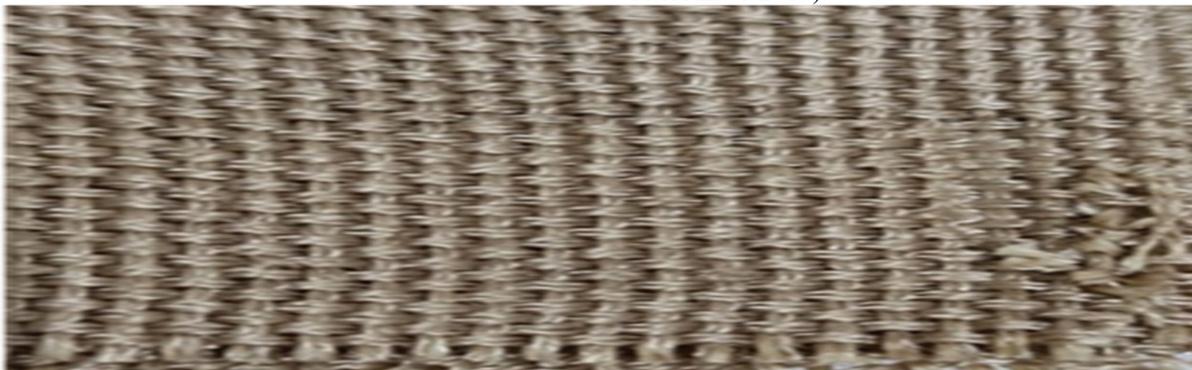## II. MATERIALS AND METHODOLOGY

Materials

As shown in Fig. 1, the kenaf woven fiber is produced by natural fiber craftsmen and obtained from the local market in Imo State, Nigeria. The jute woven fiber, as shown in Fig. 2, used is produced by natural fiber craftsmen but obtained from the local market in Imo State, Nigeria. Composites consist of two main components, namely, reinforcement and matrix. In this investigation, the matrix is Bisphenol-A Epoxy Resin. The characteristics of Bisphenol A Epoxy Resin are presented in Table 1.

Alkaline Treatment

The mechanical properties of the natural fibers are weakened when natural fibers are exposed to the atmosphere as they absorb moisture. This moisture needs to be removed or reduced by alkaline treatment methods (Alzebdeh, Nassar, Arunachalam and Okoronkwo 2019). Natural fibers were cleaned with clean water and soaked in NaOH solution for 2 hours at room temperature (Alzebdeh, Nassar, Arunachalam and Okoronkwo 2019). Alkaline treatment has an effect on the fiber such as removing the waxy layer on the fiber, lignin, oil, and dirt; increasing the surface roughness of the fiber; and resulting in better mechanical strength properties ((Adeniyi et al. 2019), (Okoronkwo et al. 2020). Three levels of NaOH were used for the test samples in this research, namely, 4, 6, and 8 (wt%) concentrations for 4 hours, following methods adapted from (Hamidon et al. 2019). It is not recommended to treat kenaf fiber with a NaOH solution greater than 10%, where this concentration can damage the texture of the fiber (Mahjoub et al. 2014). Finally, the fiber was washed with distilled water to remove any excess NaOH particles until neutral and then dried in air for 24 hours (Guo, Sun, Satyavolu and Okoronkwo 2019).

Fig. 1. Kenaf fiber.

Fig. 2. Jute fiber.

Table 1. Bisphenol-A epoxy resin property.

Properties	Unit	Value
Viscosity	MPa	13.000 ± 2.000
Epoxy number	%	22.7 ± 0.6
Epoxy equivalent	g/equiv	189 ± 5
Epoxy value	Equiv/100 g	0.53 ± 0.01
Total chlorine content	%	<0.2
Hydrolyzable chlorine content	%	<0.05
Color according to the Gardner scale	-	<1
Density at 25°C	g/cm3	1.17 ± 0.01
Refractive index at 25°C	-	1.572 ± 0.003
Volatile content at 3 h, 140°C	%	<0.2
Vapor pressure at 80°C	mbar	<0.1
Flashpoint, according to DIN 51,584	°C	>250

Post-curing

After the manufacturing process, the composite plate received post-curing treatment. The post-curing process removes the water contained in the fibers. Post-curing is also performed to improve the modulus and strength of both polymers and composites as well as reduce residual stress. In this investigation, the temperature for post-curing treatment is 80°C, 100°C, and 120°C for 2 hours, determining the temperature value used as a parameter in this study was adopted from previous research by Devireddy and Biswas (2017).

Table 2: CMNFR fabrication parameters

Control factor	Level 1	Level 2	Level 3
A	4%	6%	8%
B	80°C	100°C	120°C
C	JF	KF	KF-JF

Response surface methodology

RSM is the combination of statistics and mathematical procedures that utilize system modeling and problem analysis to create a response of interest (Okoronkwo et al. 2022). This response is affected by several variables and the target value (Campana et al. 2018). RSM can analyze a set of designed experimental parameters to get an optimal response (Alzebdeh, Nassar,

Arunachalam and Okoronkwo 2019). This research analyzed the effect of CMNFR fabrication parameters on bending and flexural strength using RSM. In general,

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{p=1}^{k} \beta_{ii} x_i^2 + \sum \sum_{i<j}^{\infty} \beta_{ij} x_i x_j + \varepsilon.$$ (1.0)

Mechanical testing of materials

The impact strength was determined according to the American Society for Testing and Materials (ASTM) D4812–99, with a sample size of 63.50 × 12.70 × 3.2 mm (Annual Book of ASTM D 4812– 99). The flexural strength was determined according to the American

second-order polynomial functions are commonly used in various studies. The second-order RSM models are Society for Testing and Materials (ASTM) D790 with a specimen size of 125 × 12.70 × 3.2 mm (Annual Book of ASTM D 790–03). Experimental outcomes of the 27 experiments measured by impact testing and flexural testing are given in Table 3. The factor is written as a coded factor value.

Table 3: Mechanical strength of CMNFR

| Trial number | Coded value of factor | | | Impact Strength (J/mm^2) | Flexural Strength (MPa) |
	A	B	C		
1	1	1	2	2.6×10^{-2}	6.5×10^1
2	1	1	2	2.4×10^{-2}	3.4×10^1
3	1	1	2	2.3×10^{-2}	3.9×10^1
4	1	2	1	1.2×10^{-2}	6.7×10^1
5	1	2	1	1.2×10^{-2}	7.1×10^1
6	1	2	1	9.6×10^{-3}	7.3×10^1
7	1	3	3	1.4×10^{-2}	7.1×10^1
8	1	3	3	1.4×10^{-2}	7.5×10^1
9	1	3	3	1.4×10^{-2}	7.4×10^1
10	2	1	1	8.9×10^{-3}	5.7×10^1
11	2	1	1	1.1×10^{-2}	6.3×10^1
12	2	1	1	9.9×10^{-3}	5.3×10^1
13	2	2	3	1.1×10^{-2}	6.8×10^1
14	2	2	3	1.2×10^{-2}	6.8×10^1
15	2	2	3	1.3×10^{-2}	7.0×10^1
16	2	3	2	1.7×10^{-2}	9.1×10^1
17	2	3	2	1.9×10^{-2}	9.4×10^1
18	2	3	2	1.6×10^{-2}	9.1×10^1
19	3	1	3	1.3×10^{-2}	7.2×10^1
20	3	1	3	1.2×10^{-2}	7.3×10^1
21	3	1	3	1.4×10^{-2}	7.2×10^1
22	3	2	2	1.9×10^{-2}	7.2×10^1
23	3	2	2	1.8×10^{-2}	7.3×10^1
24	3	2	2	1.7×10^{-2}	7.0×10^1
25	3	3	1	5.8×10^{-3}	6.8×10^1
26	3	3	1	8.2×10^{-3}	6.4×10^1
27	3	3	1	5.6×10^{-3}	6.6×10^1

III. RESULT AND DISCUSSION

Manufacturing of composite materials with natural fiber reinforcement is carried out in several steps. The first step is multi-regression analysis to determine the relationship of each parameter setting, a variable, with

the impact and flexural strength values. The second step is to test the optimal value by analyzing the maximum confidence level (less than 5 %). The third step is the analysis of the effect of each factor on parameter settings. The forth step is determining the optimal parameters for manufacturing composite

materials with natural fiber reinforcement. The last step is a micro-structural analysis of the fracture that occurs in the test samples.

Analysis of variance in the RSM

ANOVA was used to evaluate the regression model significance of the parameters (NaOH concentration and post-curing temperature) and individual model coefficients. Table 4 presents the ANOVA analysis of the experimental results for impact and flexural strength. The experimental design was evaluated at a 95 % confidence level. The value of the contribution is considered in identifying the levelof significance of the variable. The results of the analysis show that the most influential parameter influencing the impact strength value is the concentration of NaOH (A) 11.54 %; post-curing temperature (B) 6.54 %; and fiber type (C) 8.86 %. Thus, the concentration of NaOH has a higher contribution to the process of making CMNFR. Likewise, the most significant parameter affecting the flexural strength value is A (4.99 %); B (35.37 %); C (4.79%). Table 3 presents the validation results from the experiment according to the optimal combination of parameters. The confidence intervals for impact strength and flexural strength were calculated to be 0.014 and 68.65. The results of the response validation test give a 95% CI value.

Table 4: Analysis of variance (ANOVA) table for ramie fibre epoxy Composite strength properties

Composite Strength properties	Source	Sum Sq.	d. f.	Mean Sq.	F	Prob>F
Impact strength	X1	0.00005	2	0.00003	0.03	0.9732
	X2	0.00558	2	0.00279	2.8	0.0824
	Error	0.02193	22	0.001		
	Total	0.02756	26			
Flexural strength	X1	434.89	2	217.444	2.03	0.1551
	X2	1563.56	2	781.778	7.3	0.0037
	Error	2355.56	22	107.071		
	Total	4354	26			
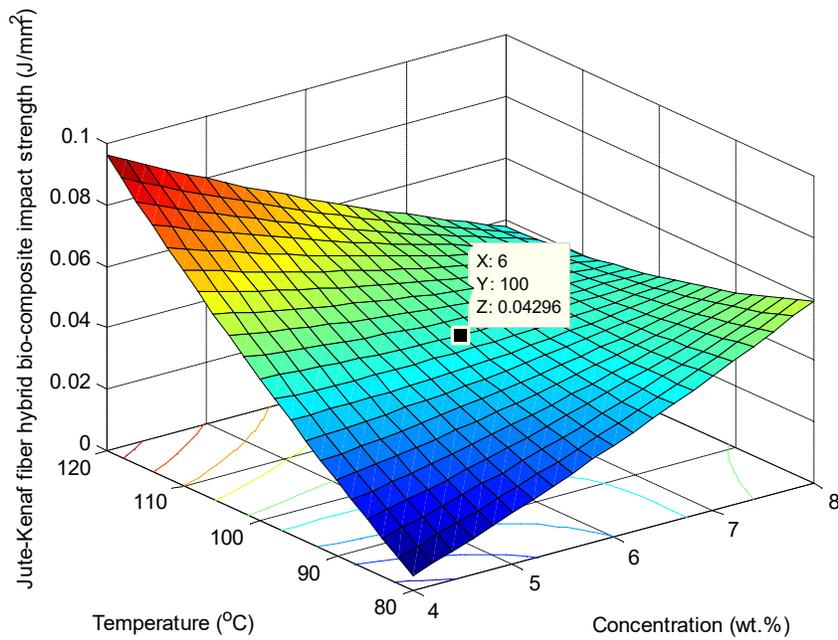
(a)
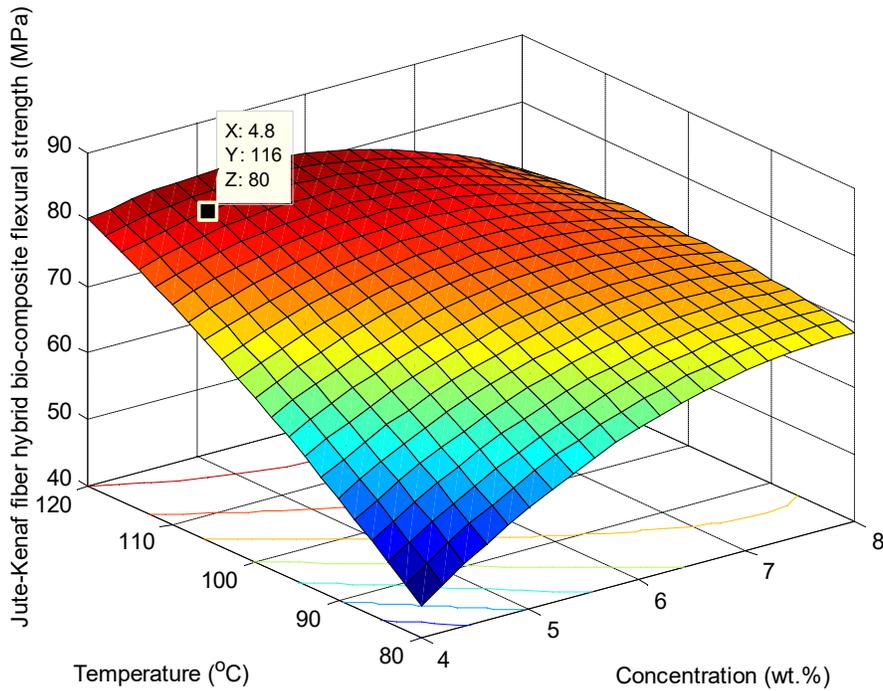
(b)

Fig. 10. 3D RSM plot of the strength of natural fiber composite materials: (a) NaOH vs. post-curing temperature for impact strength; (b) NaOH vs. post-curing temperature for flexural strength.

RSM-based modeling for impact and flexural strength

The level of influence can be determined from the contribution factor (% contribution) to the mathematical model of the maximum value of material strength. A larger contribution value indicates a greater influence of the factor. The second-order regression model can be seen in equations (4) and (5). This regression model is a function of three manufacturing parameter variables (A, B, and C).

Table 5: Individual composite strength linear regression analysis objective functions

Composite Strength properties	Regression models	R^2 %
Impact strength	$Y = -0.34 + 0.062*(c_1) + 0.003*(c_2) - 0.0007*(c_1 c_2) + 0.0006*(c_1^2) + 1.06e - 005*(c_2^2)$	95.94
Flexural strength	$Y = -222.03 + 41.24*(c_1) + 2.89*(c_2) - 0.21*(c_1 c_2) - 1.54*(c_1^2) - 0.006*(c_2^2)$	85.16

where Y is predicted impact strength, predicted flexural strength.

This research gets a response strength of the material in both tests, which gave an R2 of 95.94% (impact) and 85.16% (flexural). This shows that the R2 value is close to 100%, and therefore, the two regression models formed above can be used to predict the material strength of certain design parameters. After forming the second-order regression model and the ANOVA generated from the response data, the predicted optimum parameter values can be derived using the surface plot curve and the desirability function. The effect of interaction variables on the strength of the material is illustrated using a three-dimensional (3D) plot that fits the second-order model (Equations 4 and 5). The results of observations from Table 8 show that the factors that influence the value of material strength (impact) are factors A, B, C, and flexural strength are factors A and B. The interaction factor A*B (impact and flexural strength) is considered to be the most influencing parameter material strength value. The P-value of a factor less than 0.05 indicates that the factor is deemed to have a significant effect. The analysis can be illustrated using a 3D plot, as shown in Fig. 10.

Micro-structural analysis

Microstructure observations were carried out to determine the micro-condition of the composite structure. These observations usually involve the presence of contaminants and voids. Microstructure observations were carried out on each specimen. It was found that there were significant differences in microstructure in the specimens with a strength value of 0.026 J/mm2 and 0.006 J/mm^2 (impact test). Likewise, the flexural test found significant differences in the specimens with values of 94.2 MPa and 34.5 MPa. Based on these observations, the analysis was carried out through micro-photographs as shown in Fig. 12. The results of the micro-photo analysis on the impact test specimen had contaminants and voids in the specimen with a strength value of 0.006 J/mm^2.

On the other hand, the impact test specimen with a strength value of 0.026 J/mm^2 does not have voids and contaminants. Likewise, the flexural test specimen experienced the same phenomenon in the specimen. Thus, the presence of contaminants and voids has a negative impact on the mechanical properties. This is following previous studies (Gholampour and Ozbakkaloglu 2020; Senthilkumar et al. 2018; Van de Werken et al. 2019; Yu et al. 2019), which stated that contaminants and voids could affect the mechanical properties of the specimen. More contaminants and voids will degrade the mechanical properties of the material. The functions of kenaf and jute fiber will be mixed in a resin matrix. Strength will increase, but toughness will decrease.

The results of the research analysis it is necessary to be careful about air leaks that may occur and the cleanliness of the composite moulding tool so that voids and contaminants can be reduced or eliminated.
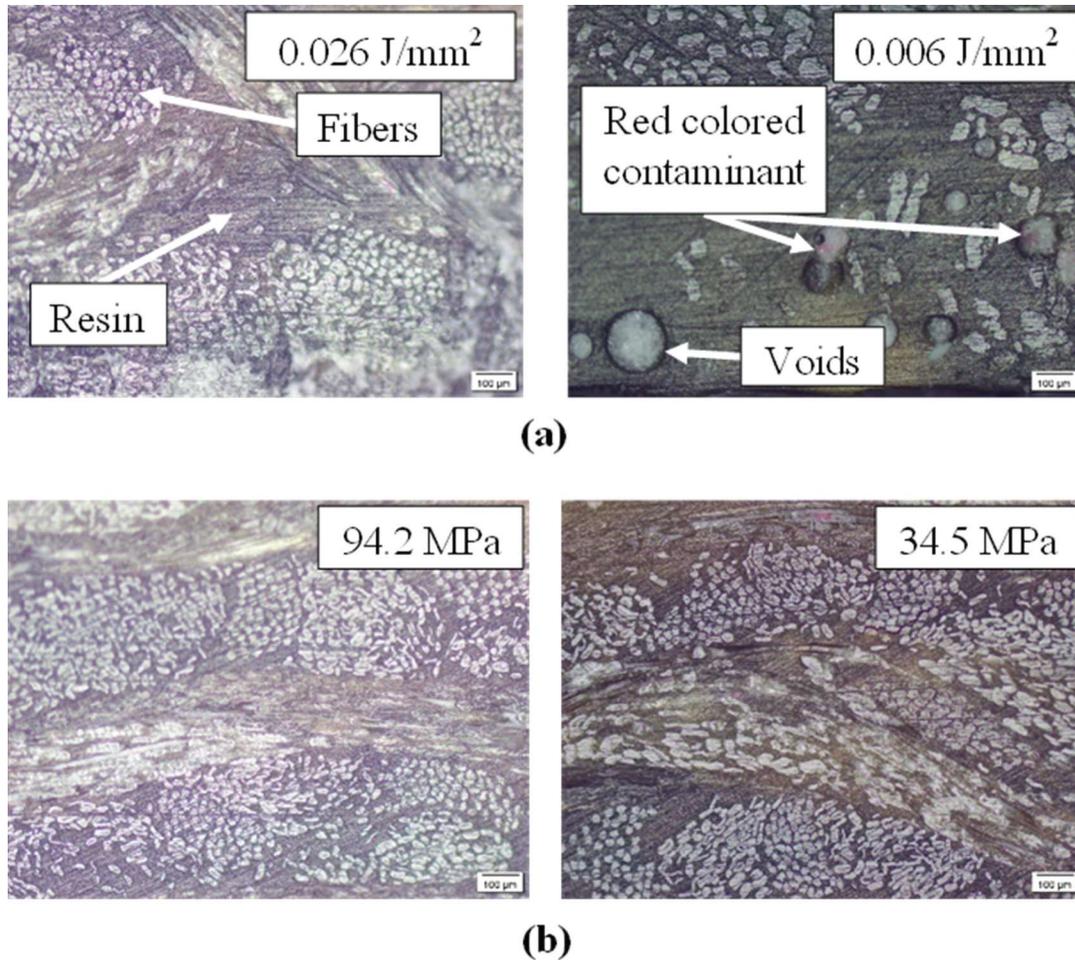
Fig. 12. Micro photos of composite specimens; (a) impact test specimen, (b) flexural test specimen.

IV. CONCLUSION

This study uses Taguchi and RSM methods to optimize CMNFR manufacturing parameters. The results of this study can be summarized as follows:

(1) The optimum material strength based on the Taguchi method approach shows the optimum impact strength value of 0.025 J/mm^2 and the optimum flexural strength value of 83.948 MPa. The optimal combination is in position A1B1C2 (impact strength); A2B3C3 (flexural strength).

(2)vBased on the RSM and the composite desirability method, the optimal manufacturing parameters of the VARI method, namely, a NaOH concentration of 4%, the post-curing temperature of 80°C, and fiber type of kenaf with an average impact strength of 0.0241 J/mm2, can be achieved at a desirability (dF) of 0.93036.

Likewise, for flexural strength using 4% NaOH concentration parameters, a post-curing temperature of 120°C and jute fiber type with an average flexural strength of 94.8650 MPa can be achieved at a desirability (dF) of 1.0000. Thus, the main factors affecting impact strength and flexural strength, respectively, contributed 93 % and 100 %.

(3) The results of the microstructure analysis found that the presence of voids and contaminants in the specimens had a negative impact on the mechanical properties of the composites, as evidenced by specimens containing contaminants and voids having low mechanical properties. The contaminants and voids will degrade the mechanical properties of the material. The functions of kenaf and jute fiber will be mixed together in a resin matrix. Flexural strength will increase, but toughness impact will decrease.

Future research will focus more on the application of use of this material and the process of designing and

manufacturing automotive accessory components such as mirrors, spoilers, and others.

Highlights

The use of composite materials with natural fiber reinforcement (CMNFR) has experienced rapid development in the automotive industry

This study uses the vacuum-assisted resin infusion (VARI) process to fabricate composites using woven kenaf and jute fiber

Two optimization technique methods are used to obtain the optimal composite material composition

Nomenclature

CMNFR- Composite materials with natural fiber reinforcement

RSM- Response surface methodology

KF- Kenaf fiber

JF- Jute fiber

NaOH- Sodium Hydroxide

Conflict of interest

The authors declare that there is no conflicting interest in the publication of this paper.

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