Biocomposite Optimization with NaOH-modified Bagasse Fiber, Polybutylene Succinate, and Poly(Lactic Acid) using RSM Approach

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Alkali-treated bagasse fiber was used as a process variable for optimization of the properties of polybutylene succinate/poly(lactic acid)-based biocomposites using Box-Behnken design (BBD) and response surface methodology (RSM). The optimum conditions for three factors, *i.e.*, NaOH-treated bagasse fiber (0.55 to 1.65 g), polybutylene succinate (1.1 to 2.3 g), and poly(lactic acid) (2.2 to 3.4 g) on the bending strength of biocomposite were investigated. The optimum combination was 0.91 g of NaOH-treated bagasse fiber, 1.14 g of polybutylene succinate, and 3.10 g of poly(lactic acid). The bending strength for NaOH-treated bagasse fiber/polybutylene succinate/ poly(lactic acid) composite was 27.0 MPa, which was 26.0% higher than native bagasse fiber-based composite. The composites were also characterized by thermogravimetric analysis, mechanical testing, Fourier transform infrared, scanning electron microscopy, water absorption, and contact angle tests. Results demonstrated that the bending strength, impact strength, and tensile strength of alkali treated bagasse fiber-based biocomposite increased by 26.0%, 15.5%, and 23.3%, separately, compared with native bagasse-based composite after sequential homogenization, compounding, and hot pressing. The hydrophobicity for alkali-treated bagasse fiber/PBS/PLA was also improved. Thus, NaOH-treated biomass materials/biodegradable polymer was judged to be suitable for preparing green composite materials.

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Keywords: NaOH modified bagasse fiber; Polybutylene succinate; Poly(lactic acid); Novel biocomposite; Box-Behnken design

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INTRODUCTION

Over the past century, excessive development of traditional petroleum-based plastics in different industries has caused worldwide environmental problems due to extensively increasing generation and disposal of post-consumer plastic wastes (Getme and Patel 2020; Mochane *et al.* 2021). By 2025, global plastic production is expected to reach 500 million tons, 60% of which will enter the environment as plastic waste (Aliotta *et al.* 2022). In recent years, great efforts have been made to develop novel green composite materials (Mochane *et al.* 2021; Qi *et al.* 2022; Kong *et al.* 2023). Bio-based polymers are

considered a suitable replacement of petroleum-based polymers (Zhang 2021; Ketata *et al.* 2022; Nithikarnjanatharn and Samsalee 2022).

Among many biodegradable polymeric materials, poly(lactic acid) (PLA) is an attractive material for manufacturing sustainable composites for its degradability, ease of processability, thermo-plasticity, good biocompatibility, safety, and non-toxicity (Albuquerque *et al.* 2021; Yusoff *et al.* 2021; Inseemeesak *et al.* 2022; Wang *et al.* 2022). However, the brittleness of PLA remains a problem and limits its range of applications (Vorawongsagul *et al.* 2021; Aliotta *et al.* 2022). To improve the toughness of PLA, biodegradable ductile polymers have been blended with PLA for toughening the composites. Polybutylene succinate (PBS) was considered as a commercially available polymer with superior biodegradability, high flexibility, and processability (Chang *et al.* 2022; Yue *et al.* 2022). It is one of the best options for recombination with PLA (Sasimowski *et al.* 2021). Vorawongsagul *et al.* (2021) blended PLA with 20 wt% of PBS to fabricate composites with excellent mechanical properties and compatibility

Lignocellulosic biomass is considered one of the most potential and abundant natural materials for reinforcing various composites to produce eco-friendly and cost comparable materials (Jing *et al.* 2020; Chen *et al.* 2021; Mochane *et al.* 2021). The well-known lignocellulosic fibers used as a substitute in polymers include bamboo, flax, hemp, jute, sisal, pineapple, cotton, oil palm, corn straw fiber, rice straw fiber, wheat straw fiber, bagasse, wood fiber, and so on (Aliotta *et al.* 2022; Ketata *et al.* 2022). It is well known from published literature that the compatibility and interfacial interactions between hydrophilic lignocellulosic biomass and hydrophobic polymer materials can be weak (Mochane *et al.* 2021; Fang *et al.* 2022). Pretreatment processes (including chemical, physical, and physicochemical methods) are necessary for lignocellulosic biomass to enhance the interface, improve the compatibility of lignocellulosic fiber and polymer materials, and improve the mechanical properties of biocomposites (Chougan *et al.* 2020, 2022; Zhao *et al.* 2022). It has been reported that among different pretreatment process methods, NaOH treatment is a promising approach to remove hydrophobic extractives along with some lignin (Ge *et al.* 2020; Huerta-Cardoso *et al.* 2020). Additionally, according to published results, silanization is a crucial treatment to promote the compatibility and covalent bonding between hydrophilic fibers and hydrophobic polymer matrix as well as enhance composite strength (Bahrami *et al.* 2021; Bahrami and Bagheri 2022).

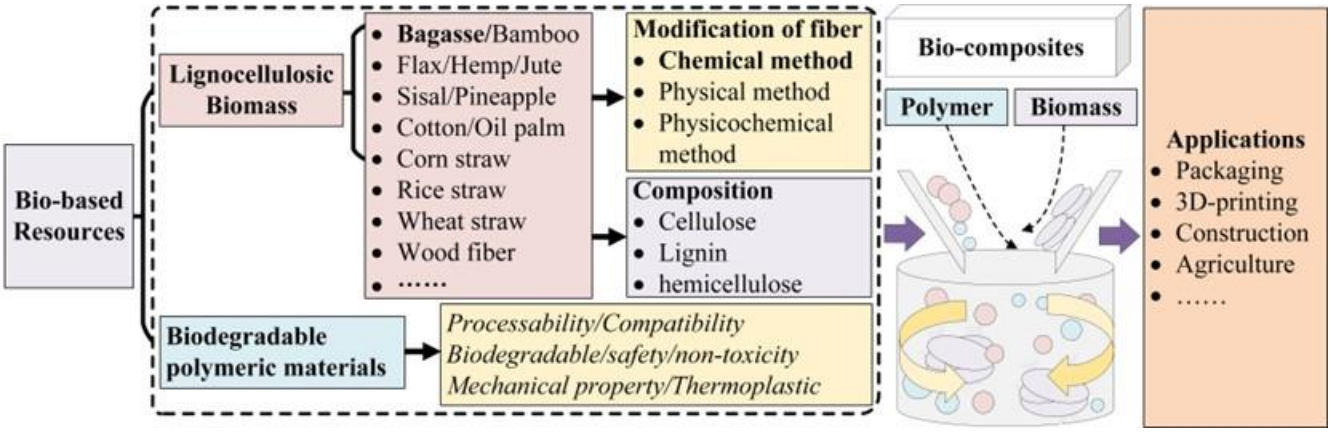

Fig. 1. Typical process options for preparation of green composite materials

Sugarcane bagasse is an important agro-industrial residue generated from sugarcane juice extraction. The world production of sugarcane bagasse is about 75,000 (10^3 Ton) per year (Mochane *et al.* 2021). Bagasse is considered as one of the largest biomass fiber resources due to the large amount of sugarcane production. Bagasse fiber (BF) is renewable, available, cheap, biodegradable, and completely or partially recyclable (Ilyas *et al.* 2021).

Because of the adequate mechanical strength and elasticity modulus, bagasse fiber has been considered a promising filling material for reinforcement of different polymers to produce composites (Bartos *et al.* 2020). The BF contains 32 to 55% of cellulose, 11 to 25% of lignin, and 17 to 25% of hemicellulose (Chen *et al.* 2022). On the other hand, the inherent hydrophilicity and strong polarity of lignocellulosic materials usually causes poor interfacial bonding and its incompatibility with hydrophobic polymer matrix that results in poor physical and mechanical performances of composite materials. The effects of chemical pretreatment on the property and structure of biomass fibers have been studied and reported.

The increase in strength and stiffness after alkali treatment was observed by researchers. Pretreatment modifies the composition, structure, and properties of bagasse fibers, which is expected to result in increasing the fiber strength compared to untreated ones (Bartos *et al.* 2020, 2021). Alkali-treated BF reinforced post-consumer high-density polyethylene (HDPE) biocomposites were fabricated and investigated by Chen *et al.* (2022). The tensile strength of 5 wt% alkali-treated BF/HDPE composite was 20.8 MPa, which increased 12% compared to untreated BF-based matrix (18.5 MPa). The obtained biocomposite exhibits improved performances as well as wide application aspects (Dixit *et al.* 2021).

At present, BF is used for reinforcing non-biodegradable polymer matrix (such as high-density polyethylene, polypropylene, polyurethane, *etc.*) for producing composites (Qiu *et al.* 2021; Chen *et al.* 2022). No relevant studies have been reported on the preparation of biodegradable materials from bagasse fiber, PBS, and PLA. In this work, response surface methodology (RSM) was used to carry out an optimization of different independent factors for achieving the optimum output response. Some authors have explored the suitability of RSM Box-Behnken design (BBD) to obtain the most favorable values of independent variables in their published literatures (Tharazi *et al.* 2017; Pei *et al.* 2022). According to the authors' applications, RSM-BBD was chosen to curtail the number of experiments and optimize the process parameters.

In this study, NaOH-treated bagasse fiber was used as a process variable for optimization of the properties of PBS/PLA-based biocomposite using RSM-BBD. A total of 17 sets of experiments based on different mass fractions of NaOH-treated bagasse fiber, PBS, and PLA, were performed. The impact of independent variables on the response (bending strength of biocomposites) and the optimum combination for biocomposite was observed. Moreover, the optimized novel biocomposite was also characterized using thermogravimetric analysis, Fourier transform infrared spectroscopy (FTIR), morphological property, contact angle, and water absorption analyses, as well as mechanical property testing to assess the suitability for preparing green composite materials.

EXPERIMENTAL

Materials

Poly(lactic acid) (4032D) and polybutylene succinate (TH803S), both size 100-mesh, were purchased from ShunJie Plastic Technology Co., Ltd., Guangdong Province, China. Bagasse fiber that could pass through a 100-mesh screen was obtained from Guangxi Province, China. The average particle size of bagasse fiber was 0.154 to 0.125 mm. Bagasse fiber was placed in an oven for 12 h at a temperature of 80 °C for further experiments. KH-560 (liquid, Shanyi Plastic Chemical Co., Ltd., Guangdong Province, China) was used as the silane coupling agent.

NaOH Treatment of Bagasse Fiber

In this study, bagasse fiber was subjected to NaOH treatment. A total of 10 g sieved bagasse fiber was treated with 100 mL NaOH (3%, 6%, 9%, 12%, w/v%) for 1 day at room temperature. During the pretreatment process of bagasse fiber, the sample was stirred every 6 to 8 hours. NaOH-treated biomass residue was filtered and washed with deionized water to reach pH 7. NaOH modified bagasse fiber was dried in an oven at 80 °C for further analysis.

$$\text{Bagasse fiber biomass} - \text{OH} + \text{NaOH} \rightarrow$$
$$(\text{Bagasse fiber biomass} - \text{O}^-)\text{Na}^+ + \text{H}_2\text{O} + \text{Impurities} \quad (1)$$

Preparation of Biocomposite based on NaOH-treated Bagasse Fiber/PBS/PLA

Biocomposites based on different compositions of NaOH-treated bagasse fiber, polybutylene succinate, and poly(lactic acid) were prepared using a hot-pressing method (Fig. 2). Initially, treated bagasse fiber was modified with KH-560 solution (silane coupling agent) and oven-dried at 80 °C. Further, alkali-treated bagasse fiber, polybutylene succinate, and poly(lactic acid) were homogenized for 20 to 30 min followed by compounding using a single screw extruder. Subsequently, the compounded components were loaded into a standard hot-press mold for hot pressing (160 °C, 10 MPa, 20 min). After hot pressing process, samples were cooled to ambient temperature. The specific ratio for composites (Table 3) was recommended by RSM-BBD.

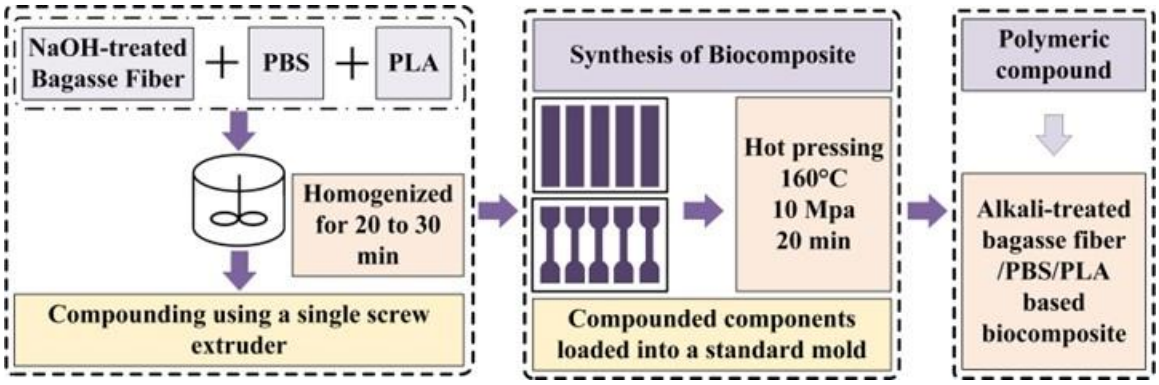

Fig. 2. Preparation process of novel biocomposite based on NaOH-treated bagasse fiber /polybutylene succinate/poly(lactic acid)

Experimental Design using RSM-BBD

In this study, RSM-BBD was selected to acquire the optimized parameters for the fabrication of NaOH-treated bagasse fiber/polybutylene succinate/poly(lactic acid) biocomposite. The RSM has many benefits compared with traditional testing methods. It requires less time to perform as well as provides optimized response variables. The proportion of NaOH-treated bagasse fiber, polybutylene succinate, and poly(lactic acid) were chosen as process variables. The coded and actual values for variables (selected based on single-factor experiments) are listed below (Table 1). A total of 17 experiments needed to be conducted in this model (Table 3) and were derived from Design-Expert software (Stat-Ease, version-13, Minneapolis, MN, USA). Actual response values (the bending strength of biocomposite) obtained from experiments were used to perform regression analysis to optimize the independent variables as well as determine the coefficients of second-order polynomial equation. The general mathematical regression model is represented in Eq. 2,

$$Y = \alpha_0 + \sum_{n=1}^{k} \alpha_n x_n + \sum_{n=1}^{k} \alpha_{nn} x_n^2 + \sum_{1 \le n \le m}^{k} \alpha_{nm} x_n x_m + \varepsilon \quad (2)$$

where x_n shows the process factors, Y represents the response (bending strength of composite); ε, α_0, α_n, α_{nn}, and α_{nm} indicate random error, offset coefficient, linear coefficient, quadratic coefficient, and interaction coefficient in this study, respectively.

Table 1. Encoding Levels and the Corresponding Actual Values for Variables of RSM-BBD

Variables of RSM-BBD	Encoding Levels and the Corresponding Actual Values		
	-1	0	1
A- NaOH-treated Bagasse Fiber (g)	0.55	1.1	1.65
B- Polybutylene Succinate (g)	1.1	1.7	2.3
C- Poly(Lactic Acid) (g)	2.2	2.8	3.4

Analytical Methods

Composition analyses of native and alkali-treated bagasse fiber

The compositional analysis of native and alkali-treated bagasse fiber was performed in this study. The National Renewable Energy Laboratory (NREL) method was used to test the composition changing of cellulose, hemicellulose, and lignin for bagasse fiber (Sluiter *et al.* 2008).

Thermogravimetric analysis

The thermal stability for biocomposites prepared in this study were investigated using a thermogravimetric analyzer (TGA 8000, PerkinElmer, Inc., Waltham, MA, USA). Composite samples were heated up to 600 °C from room temperature according to ASTM E1131 (2020) standard. Moreover, the heating rate was set at 10 °C/min under N_2 atmosphere (20 mL/min).

FTIR analysis

The IR spectra of biocomposite materials were generated using the spectrum FTIR (IS50, Thermo Fisher Scientific, Waltham, MA, USA) in an attenuated total refection mode with 4 cm^{-1} resolution and 32 scans in the range of 500 to 4000 cm^{-1} (Kong *et al.* 2023).

Morphological property analysis

The surface morphological properties of the PBS/PLA composite, native bagasse fiber/PBS/PLA composite, and NaOH-treated bagasse fiber/ PBS/PLA composite were analyzed using field emission scanning electron microscopy (Kong *et al.* 2023). The samples were gold sputtered before being subjected to SEM analysis (SU5000, Hitachi High Technology Co., Tokyo, Japan).

Contact angle analysis

The hydrophobic property for PBS/PLA composite, native bagasse fiber/PBS/PLA composite, and NaOH-treated bagasse fiber/PBS/PLA composite were investigated using contact angle measurement test (Dixit *et al.* 2021). The contact angles for all prepared samples were evaluate by drop shape analyzer using a sessile drop method at room temperature (JY-PHa Contact Angle tester, Shenzhen Lanxing Yu Electronic Technology Co., Ltd., Shenzhen, China).

Water absorption analysis

The water absorption analysis for composites was carried out referring to ASTM D570 (2022) standard. Samples with a dimension of 10 mm × 10 mm were used in this test. Water absorption test was conducted for 24 h with immersion of biocomposites in distilled water. Results of the water absorption were calculated based on weight change using the following equation,

$$\text{Water absorption percentage } (\%) = \left(\frac{W_2 - W_1}{W_1}\right) \times 100 \quad (3)$$

where W_1 represents the weight (g) of dry sample and W_2 stands for the final weight (g) of sample immersed in water after 24 h.

Mechanical properties of biocomposite based on NaOH-treated bagasse fiber/PBS/PLA

Bending strength for all biocomposites were analyzed according to ASTM D790 (2017) standard using a universal testing machine (KRWDW-100E, Jinan Kerui Testing Machine Manufacturing Co., Ltd., Shandong, China). The dimensions of the samples were 127 mm × 12.7 mm × 3.2 mm. The strain rate was 0.01 mm per minute. The tensile strength for samples (Dumbbell-type sample, 80 mm length × 4 mm width × 1 mm thickness) were tested referring to ASTM D638 (2022) standard by using a PC Auto Tensile Tester (DLS-07, Jinan Sum spring Experimental Instrument Co., Ltd., Shandong, China). Impact testing machine was used to measure the impact strength of biocomposites (Digital display impact testing machine ST-5.5D, Xiamen Ester Instrument Co., Ltd., Fujian, China). According to ISO 180 (2019), the dimension of the samples for impact test was 80 mm × 10 mm × 4 mm.

RESULTS AND DISCUSSION

Compositional Analyses for Native and Alkali-treated Bagasse Fiber

The compositional analyses for native and alkali-treated bagasse fiber are depicted in Table 2. After alkali treatment, the content (on dry basis) of hemicellulose, acid soluble lignin, and acid insoluble lignin declined from 22.15% to 13.99%, 2.51% to 1.77%, and 13.72% to 9.09%, respectively. A possible reason for this result was that NaOH treatment dissolved a portion of the lignocellulose components (hemicellulose and lignin) and reduced the content of obstacles at the same time (Yi *et al.* 2015; Chougan *et al.* 2020, 2022). Results in Table 2 also express the increase of desirable cellulose content in bagasse fiber after alkali modification. After NaOH pretreatment, the cellulose content of bagasse fiber increased from 38.4% to 43.6%. By contrast, the removal of cellulose from bagasse fiber occurs when the concentration of NaOH exceeds 9%. Therefore, modification of bagasse fiber with 9% of NaOH can achieve optimal cellulose recovery, which explores better for reinforcement of biocomposites. In the following experiment, 9% NaOH was selected to modify the biomass fiber. Researchers have reported similar results in their previous studies (Bartos *et al.* 2020; Dixit *et al.* 2021). When the fibers were subjected to alkalization, the NaOH hydrates formed in the dilute solution readily penetrated the amorphous regions between the crystallites and formed new hydrogen bonds, inducing intracrystalline swelling in the cell walls. The absorbed hydrate molecules in swollen regions act as plasticizers and multiply the mobility of the cellulose chains. As some of the cementing material is removed during alkalization, the structure becomes less dense and the internal constraint was relieved (Cui *et al.* 2014, Dixit and Yadav 2019; Bahrami *et al.* 2021). Furthermore, it was reported that alkali pretreatment can destroy the complex structure of lignocellulose materials, which is helpful for increasing the contact area between lignocellulose fiber and other materials as well as benefits for subsequent preparation process of composites (Mochane *et al.* 2021; Pei *et al.* 2022).

Table 2. Composition Analyses of Native and Alkali-Treated Bagasse Fiber[a]

Biomass Fiber Resource	Cellulose (%)	Hemicellulose (%)	Acid Soluble	Acid Insoluble
Native bagasse fiber	38.39 ± 1.75	22.15 ± 0.95	2.51 ± 0.03	13.72 ± 0.24
3% NaOH-treated Bagasse fiber	40.89 ± 1.20	17.02 ± 0.82	2.50 ± 0.04	12.18 ± 0.31
6% NaOH-treated Bagasse fiber	41.31 ± 0.89	15.38 ± 0.73	2.48 ± 0.08	10.98 ± 0.24
9% NaOH-treated Bagasse fiber	43.62 ± 0.98	13.99 ± 0.77	1.77 ± 0.02	9.66 ± 0.18
12% NaOH-treated Bagasse fiber	42.29 ± 1.02	14.31 ± 0.69	1.80 ± 0.05	9.09 ± 0.23

a: Composition change of cellulose, hemicellulose, and lignin for bagasse fiber were measured according to the National Renewable Energy Laboratory (NREL) method.

RSM and ANOVA Analysis

Suitability check of the RSM-BBD model

After conducting 17 sets of experiments provided by RSM-BBD (Table 3), the experimental data were analyzed using analysis of variance (ANOVA) analysis. The predicted values originated from RSM-BBD model. The actual values represent response data obtained from experiments.

Table 3. Predicted and Actual Values for the Bending Strength of Biocomposites using RSM-BBD

Actual Values for Variables Provided by RSM-BBD					Predicted and Actual Value of Response	
Standard Order	Run	NaOH-treated Bagasse Fiber (g)	Polybutylene Succinate (g)	Poly(Lactic Acid) (g)	Predicted (MPa)	Actual (MPa)
1	11	0.48	0.54	2.90	26.62	26.92
2	2	1.44	0.54	2.90	16.34	16.27
3	16	0.48	1.50	2.90	22.88	22.95
4	12	1.44	1.50	2.90	24.50	24.20
5	15	0.48	1.02	2.40	23.86	23.96
6	9	1.44	1.02	2.40	15.51	15.98
7	10	0.48	1.02	3.40	23.86	23.39
8	4	1.44	1.02	3.40	23.53	23.43
9	3	0.96	0.54	2.40	19.99	19.59
10	8	0.96	1.50	2.40	23.42	23.25
11	14	0.96	0.54	3.40	25.21	25.38
12	7	0.96	1.50	3.40	26.21	26.61
13	6	0.96	1.02	2.90	26.91	25.26
14	17	0.96	1.02	2.90	26.91	24.08
15	5	0.96	1.02	2.90	26.91	31.08
16	1	0.96	1.02	2.90	26.91	27.91
17	13	0.96	1.02	2.90	26.91	26.24

Figure 3 exhibits the actual and predicted values for the bending strength of NaOH-treated bagasse fiber/polybutylene succinate/poly(lactic acid) biocomposites. Figure 3 reveals that variable data points were scattered around a straight line (actual value *vs.* predicted value), which suggests the suitability and adequacy of the suggested mathematical model.

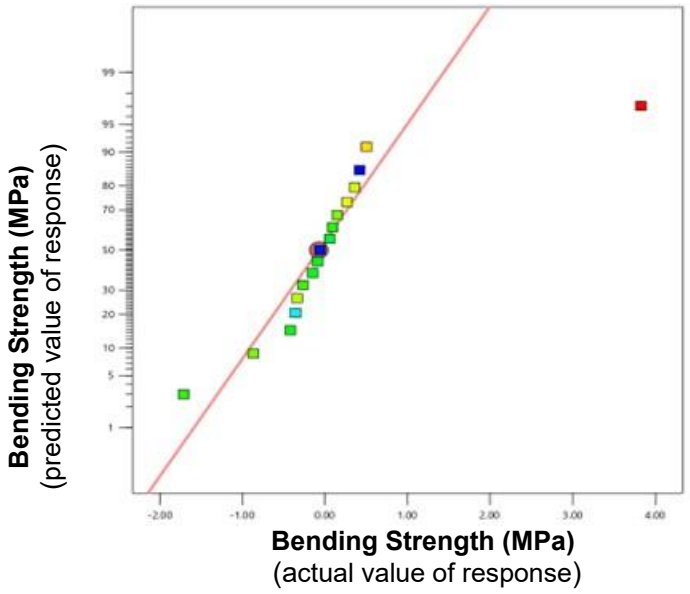

Fig. 3. Predicted and actual values for the mathematical model

Impact of independent factors on the response

The suggested polynomial equation obtained from multiple regression analysis for the bending strength of the biocomposites was depicted as follows (Eq. 4). Linear terms (A, B, and C), interaction terms (AB, BC, and AC), and quadratic terms (A^2, B^2, and C^2) are listed in the RSM-BBD model to show the role of variables on response value:

$$Y = 26.91 - 2.17A + 1.11B + 2.00C + 2.98AB + 2.01AC - 0.61BC - 3.17A^2 - 1.16B^2 - 2.05C^2 \quad (4)$$

According to the results of Table 4, p-value (probability of error, P = 0.0204, less than 0.05) and Model F-value (F = 5.2) indicated that the ANOVA analyzed model was reliable. The p and F values for linear terms (A, B, and C), interaction terms (AB, BC, and AC), and quadratic terms (A^2, B^2, and C^2) can also be used to check the suitability of the RSM-BBD model. The significant p-values for A, C, AB, and A^2 were 0.0219, 0.0301, 0.0248, and 0.017, respectively. The regression correlation coefficient R^2 value was 0.86. The adequate precision value was 7.11. The most influential factor for response was the additive of NaOH-treated bagasse fiber (F = 8.6), followed by poly(lactic acid) (F = 7.35). Those outcomes represented the reliability of RSM-BBD model.

Table 4. ANOVA Analysis for the Bending Strength of Biocomposites using RSM-BBD

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	204.47	9	22.72	5.2	0.0204	Significant
A-Alkali pretreated BF	37.58	1	37.58	8.6	0.0219	
B-PBS	9.79	1	9.79	2.24	0.1781	
C-PLA	32.12	1	32.12	7.35	0.0301	
AB	35.4	1	35.4	8.1	0.0248	
AC	16.08	1	16.08	3.68	0.0966	
BC	1.48	1	1.48	0.34	0.5793	
A^2	42.4	1	42.4	9.7	0.017	
B^2	5.62	1	5.62	1.29	0.2939	
C^2	17.71	1	17.71	4.05	0.084	
Residual	30.59	7	4.37			
Lack of Fit	1.02	3	0.34	0.046	0.9851	Not significant
Pure Error	29.57	4	7.39			
Cor Total	235.06	16				
Std. Dev.	2.09		R-Squared	0.8699		
Mean	23.91		Adj R-Squared	0.7026		
C.V. %	8.74		Pred R-Squared	0.7341		
PRESS	62.5		Adeq Precision	7.111		

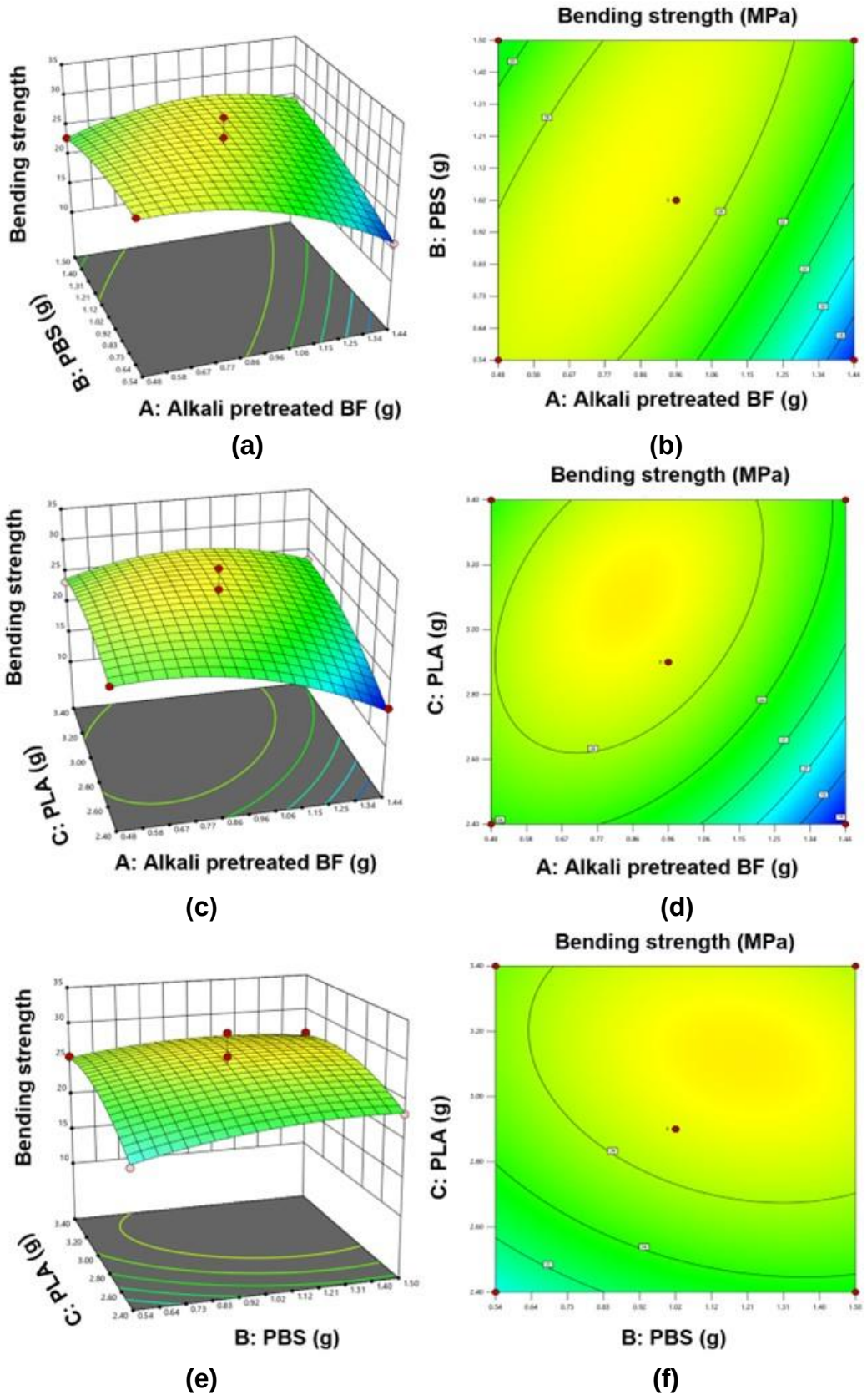

Fig. 4. 3D surface and contour plots for the bending strength of biocomposites using RSM-BBD

The 3D surface plots in Fig. 4 represent the dependability of bending strength for biocomposites with different process factors. Figures 4a and 4b reveal that the bending strength of biocomposites continuously decreased with increasing concentration of NaOH-treated bagasse fiber (from 0.55 to 1.65 g). This result demonstrated the benchmark impact of alkali-treated bagasse fiber on bending strength of biocomposite. Similar behavior was observed in Figs. 4c and 4d from interaction pattern of NaOH-treated bagasse fiber and poly(lactic acid) for the bending strength of composite. Polybutylene succinate (from 1.1 to 2.3 g) had an insignificant effect on bending strength of biocomposite in this experiment ($P = 0.17$). Figures 4c to 4f showed that the bending strength of composites increased with increasing the amount of poly(lactic acid) (from 2.2 to 3.4 g), which was in agreement with previous studies (Bartos *et al.* 2020; Chang *et al.* 2022; Inseemeesak *et al.* 2022). According to the results and discussions above, it could be concluded that the usage of NaOH-treated bagasse fiber and poly(lactic acid) influences the bending strength of biocomposite significantly. This behavior assures the optimization of composite components for manufacturing biocomposite with better mechanical properties.

Optimization of the RSM-BBD model for the bending strength of biocomposite

The optimized run was performed with 0.91 g of NaOH-treated bagasse fiber, 1.14 g of polybutylene succinate, and 3.10 g of poly(lactic acid). Predicted response at optimum concentrations provided by RSM-BBD was 27.6 MPa for bending strength. The experimental results under the optimized parameters yielded 27.0 MPa for bending strength (Figure 9). The small error for responses demonstrated that desirable bending strength could be obtained from reliable RSM-BBD model under optimized conditions for variables.

Optimized Biocomposite Characterization

Thermogravimetric analysis

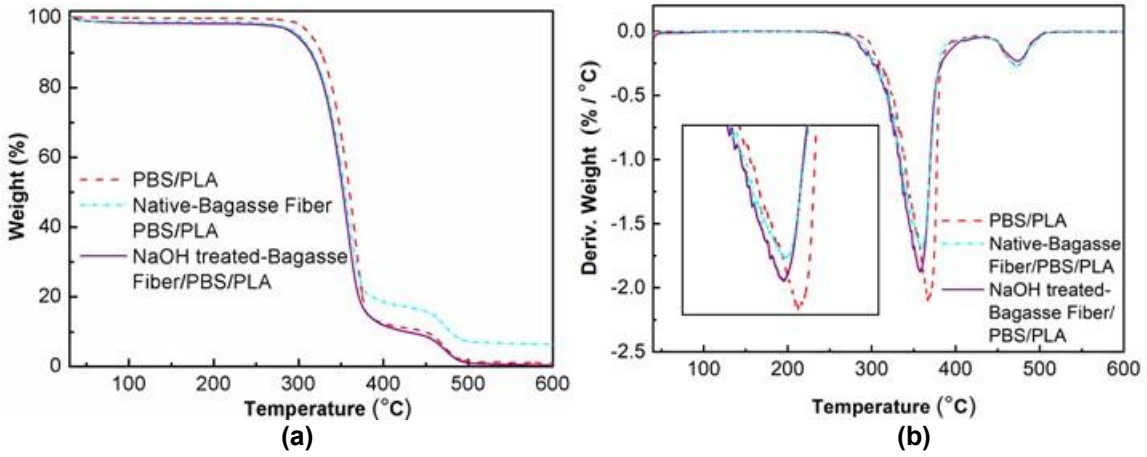

Fig. 5. TG and DTG analyses for composites (a: TG of composites; b: DTG of composites)

The TG and DTG curves for PBS/PLA composite, native bagasse fiber/PBS/PLA composite, and NaOH-treated bagasse fiber/PBS/PLA composite were analyzed (Fig. 5). The weight loss process for composite samples could be mainly divided into 3 parts. There was a slight decline in mass (0.5 to 2.4%) from 90 °C to 260 °C, which was probably due to the volatilization of small molecules and evaporation of moisture in samples (Pei *et al.* 2022). The weight loss for all curves declined drastically between 270 and 490 °C. During

this process, the mass loss was mainly caused by the decomposition of bagasse fiber, polybutylene succinate, and poly(lactic acid). Generally, natural fiber, PLA (from 260 to 390 °C), and PBS (from 310 to 430 °C) are pyrolyzed in sequence (Ketata *et al.* 2022). The second stage of degradation starts at around 296 °C for PBS/PLA composite with 1.13% weight loss, 262 °C for native bagasse fiber/PBS/PLA composite with 2.42% weight loss, 273 °C for NaOH-treated bagasse fiber/PBS/PLA composite with 1.76% weight loss.

The thermal degradation ended at around 493 °C for PBS/PLA composite with 1.54% residual, 504 °C for native bagasse fiber/PBS/PLA composite with 6.76 % residual, 496 °C for NaOH-treated bagasse fiber/PBS/PLA composite with 1.22% residual. The decomposition of lignin in bagasse fiber and pyrolysis residues occurred in this stage. The inorganic content of non-wood materials was high. He (2019) has reported that non-wood materials contains about 10% inorganic content. Therefore, when the temperature exceeds 600 degrees, untreated bagasse fiber resulted in about 7% residual mass. According to the result of initial thermal-degradation temperature (273 °C), amount of pyrolysis residue (1.22%), and pyrolysis speed (Fig. 5b), it could be concluded that the thermal stability of NaOH-treated bagasse fiber/PBS/PLA composite was slightly higher than native bagasse fiber/PBS/PLA composite. It was reported that the alkaline treatment plays a crucial role in governing the ultimate properties of fibers and biocomposites (Bahrami *et al.* 2021). NaOH-treatment could remove part of hemicellulose (thermo-decomposed temperature from 230 to 310 °C) and lignin components (thermo-decomposed temperature from 300 to 400 °C) in bagasse fiber, which results in better thermal thermogravimetric stability. Bahrami *et al.* (2021) have published similar outcomes that alkalization in NaOH solution removed the hemicellulose and pectin constituents in the structure of native fibers partially while increased the processing temperature of biocomposites to above 20°C. The most pronounced influence of alkalization for biomass fibers can benefit their processability at elevated temperatures, especially in composite manufacturing. Dixit *et al.* (2021) also reported similar outcomes in their article.

FTIR analysis

The FTIR spectra for PBS/PLA composite, native bagasse fiber/PBS/PLA composite, and NaOH-treated bagasse fiber/PBS/PLA composites are shown in Fig. 6. Characteristic FTIR peaks at 3331 cm^{-1} corresponded to the vibration of -OH group in the lignocellulose component of bagasse fiber. For native bagasse fiber-based composite and NaOH-treated bagasse fiber-based composite, the intensity of the -OH group decreased after NaOH pretreatment of bagasse fiber. This result was probably because of dissolving a portion of lignin and hemicellulose in bagasse fiber, which corresponded to the compositional analysis result in Table 2. The peak near 2928 cm^{-1} and 1357 to 1444 cm^{-1} represented the stretching vibration and bending vibration of C-H in lignocellulosic components of bagasse fiber, main chain of PLA, and PBS. The absorption band at 1744 cm^{-1}, 1175 cm^{-1}, and 1081 cm^{-1}corresponded to C=O groups, stretching vibration of C-O, and Al-O, respectively, which could be found in biocomposites for all samples. The absorbance intensity of C=O for native bagasse fiber-based composite was higher than NaOH-treated bagasse fiber-based composite, which could be attributed to the reduction of fiber components in alkali-treated composites. The absorption peak near 1270 to 1000 cm^{-1} (aromatic oxide) confirmed the presence of lignin in crop fiber-based biocomposites. The intensity for native bagasse fiber-based composite in this area was higher than NaOH-treated bagasse fiber-based composite, which could be contributed to the decreasing of

lignin components that were washed away by NaOH treatment. This result was associated with the suitability of treated bagasse fiber in polymer matrix with removing part of lignocellulosic components, increasing the contact area, better adhesion, and accessibility (Bartos *et al.* 2020; Chougan *et al.* 2020).

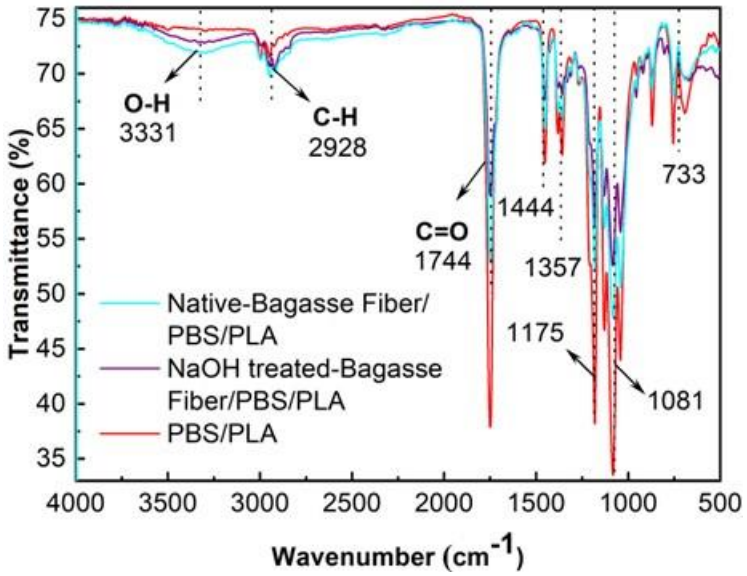

Fig. 6. FTIR spectroscopy analyses for composites

Morphological property analysis

The morphological structure of PBS/PLA composite, native bagasse fiber/ PBS/PLA composite, and NaOH-treated bagasse fiber/ PBS/PLA composite were observed by SEM analysis. Figure 7 represents 500 x (Fig. 7a, Fig. 7b, and Fig. 7c) and 2000x (Fig. 7d, Fig. 7e, and Fig. 7f) magnification micrographs of composite samples. The plain surface of PBS/PLA-based composite was observed in Fig. 7a and 7d. Figure 7b and 7e show strong adhesion and uniform distribution of NaOH-treated biomass in the PBS/PLA polymer matrix. Compared with NaOH-treated bagasse fiber-based composite, the roughness of the surface for native bagasse fiber-based composite increased significantly (Fig. 7c and 9f). Both native bagasse fiber and alkali treated bagasse fiber were modified with KH-560 solution (silane coupling agent) before the preparation of biocomposite. Therefore, the difference of compatibility between native bagasse fiber/PBS/PLA composite and NaOH-treated bagasse fiber/PBS/PLA composite observed in Fig. 7 was likely due to alkali treatment. Based on published results, the silane coupling agent is important in promoting the compatibility between the fibers and the hydrophobic polymer matrix (Bahrami *et al.* 2021; Bahrami and Bagheri 2022). Without the silane coupling agent, the dispersion between fiber and matrix could be particularly poor. Large gaps could occur between the fiber and matrix. It could be seen from Fig. 7 that NaOH-treated bagasse fiber was well dispersed in the PBS/PLA matrix after hot pressing, which indicated good miscibility between treated biomass and polymer matrix composite. NaOH treatment could disrupt the complex structure of cellulose, hemicellulose, and lignin in biomass to improve the effectiveness, compatibility, and assessment of biomass materials with polymer materials (confirmed by higher mechanical strength in Fig. 9). Alkali pretreatment resulted in a rougher surface at the microscopic level, which made it possible for the matrix material to flow and form an interlocking structure with the reinforcing material. Other studies have

reported similar results (Chougan *et al.* 2020; Vorawongsagul *et al.* 2021; Avci *et al.* 2023; Pei *et al.* 2023).

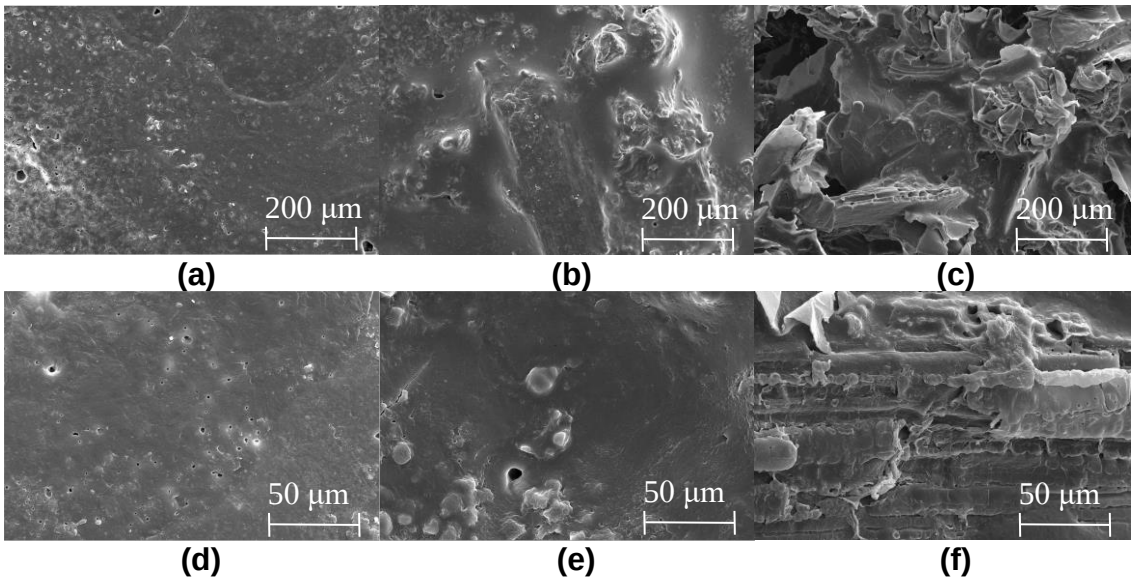

Fig. 7. Morphological property for polymeric composites (a: PBS/PLA composite, 500X; b: native bagasse fiber/PBS/PLA composite, 500X; c: NaOH-treated bagasse fiber/PBS/PLA composite, 500X; d: PBS/PLA composite, 2000X; e: native bagasse fiber/PBS/PLA composite, 2000X; f: NaOH-treated bagasse fiber/PBS/PLA composite, 2000X)

Contact angle analysis

The contact angle test explores the wettability and hydrophobicity for the surface of biocomposites. Contact angles for all prepared samples are shown in Fig. 8.

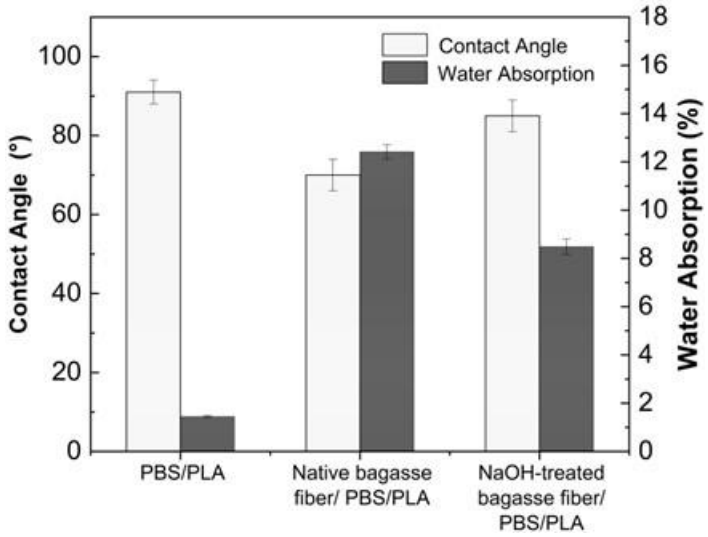

Fig. 8. Contact angle and water absorption properties of biocomposites

The contact angle values for PBS/PLA, native bagasse fiber /PBS/PLA, and NaOH-treated bagasse fiber /PBS/PLA-based composites were 91°, 70°, and 85°, respectively. The contact angle for alkali-treated bagasse fiber/PBS/PLA was higher (increased 15°) as compared to native bagasse fiber/PBS/PLA. This result indicated the enhancement of

waterproofing nature for NaOH-treated bagasse fiber-based composite to a certain extent. Generally, surface roughness and hydrophilic groups influence the surface tension between the biocomposite sample and water (Dixit *et al.* 2021). It was likely that alkali-treated bagasse fiber reinforced PBS/PLA matrix developed a more hydrophobic nature after NaOH affected the surface structure and hydrophilicity (Bartos *et al*. 2020; Pei *et al.* 2023).

Water absorption analysis

Figure 8 also depicts the water absorption result for composite samples. The water absorption result for PBS/PLA, native bagasse fiber/PBS/PLA, and NaOH-treated bagasse fiber/PBS/PLA-based composites were 1.44%, 12.41%, and 8.48%, respectively. The water absorption performance of NaOH-treated bagasse fiber/PBS/PLA composite was better compared to native bagasse fiber-based composite. This result could be attributed to the uniform distribution of NaOH modified bagasse fiber in the polymer matrix, which correlated with better compatibility and water resistance. This result was consistent with the SEM result and contact angle analysis above as well as similar to results in published articles (Bartos *et al*. 2020; Dixit *et al.* 2021; Chen *et al.* 2022).

Mechanical properties of biocomposite

Mechanical properties play an important role in preparing high-quality biocomposite. To examine the mechanical strength of biocomposites, bending strength, impact strength, and tensile strength tests were conducted. Results are listed in Fig. 9. The bending strength for PBS/PLA composite, native bagasse fiber/PBS/PLA composite, and NaOH-treated bagasse fiber/PBS/PLA composites were 43.4, 21.4, and 27.0 MPa, respectively. Moreover, the impact strength for prepared composite samples were 8.76, 4.57, and 5.28 kJ/m^2, respectively. The tensile strength for composites were 40.89, 18.82, and 23.21 MPa, respectively. Biocomposite based on native bagasse fiber shows poor adhesion between biomass and polymer, which resulted in a notable decline for mechanical property (compared with PBS/PLA composite).

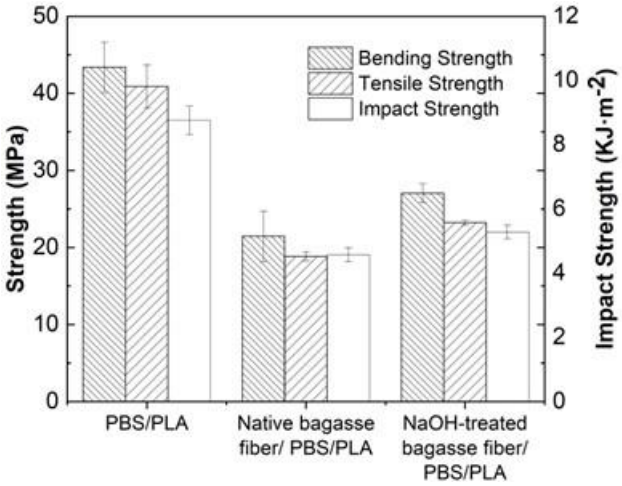

Fig. 9. Mechanical properties of biocomposites

The bending strength for NaOH-treated bagasse fiber reinforced composite increased from 21.4 to 27.0 MPa compared to native bagasse fiber-based composite (26.0% increase). Moreover, the impact strength increased 15.5% (from 4.67 to 5.28 kJ/m^2) after

blending NaOH-treated bagasse fiber with PBS/PLA. A notable increment (23.3%) in tensile strength was observed after incorporation of NaOH-treated bagasse fiber with PBS/PLA in comparison with native bagasse fiber-based composite. Better mechanical properties were associated with the suitability of NaOH modified bagasse fiber in PBS/PLA polymer matrix, which demonstrated its potential for preparing novel composite materials (Huerta-Cardoso *et al.* 2020; Chen *et al.* 2022; Chougan *et al.* 2022).

CONCLUSIONS

1. Incorporation of NaOH-treated bagasse fiber in poly(butylene succinate)/poly(lactic acid) (PBS/PLA) matrix was carried out for manufacturing the optimized biocomposite based on response surface methodology with Box-Behnken design of experiments (RSM-BBD). The process factors mass fractions of alkali-treated bagasse fiber, PBS, and PLA were optimized for the bending strength of composite materials. The optimized values for NaOH-treated bagasse fiber, PBS, and PLA were 0.91 g, 1.14 g, and 3.10 g, respectively. The optimum bending strength provided by RSM-BBD model was 27.6 MPa, which was consistent with the experimental result.
2. The optimized composite material was further characterized by thermogravimetric analysis, Fourier transform infrared (FTIR) analysis, morphological property analysis, water absorption analysis, contact angle analysis, and mechanical property analysis. The thermal stability, accessibility, compatibility, and hydrophobic property of optimized biocomposite were enhanced due to the addition of alkali-treated bagasse fiber in matrix as compared to native bagasse fiber-based composite. Alkali-treated bagasse fiber/PBS/PLA exhibited the lower water absorption percentage of 8.48% weight gain after 24 h immersion than raw bagasse fiber/PBS/PLA. The bending strength, impact strength, and tensile strength also increased 26.0%, 15.5%, and 23.3%, respectively, for alkali-treated bagasse fiber-based composite. These outcomes suggested that alkali modified bagasse fiber reinforced degradable materials presented a promising prospect for producing green polymeric composites.

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