Predictive Energy Requirement Models in Bio-fiber Comminution Process

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Abstract

Comminution of bio-fibers is necessary for their effective application in development of reinforced composite and feed-stock; however, comminution is an energy intensive process and minimization of specific energy required in the size reduction of bio-fibers is an optimization task in design of various particulate based materials. The objective of this study was to model specific energy requirement in bio-fiber comminution process. The banana and coir bio-fibers used in the study were respectively extracted from post-harvest banana stem and outer shell of a coconut. Characterization of treated bio-fibers was done using Fourier Transform Infrared Spectroscopy and comminution process was carried out in a laboratory-scale knife mill. Electrical energy was recorded using a plug-in power energy meter while Kick's, Rittinger's and Bond's energy laws provide insight into the relationship between particle size reduction and energy consumption during comminution process. The Rosin Rammler-Bennett (RRB) and Gaudin-Schuhmann (GS) models were fitted with MATLAB® R2014a. The Artificial Neural Networks (ANN) and Response Surface Method (RSM) was utilised in predicting specific energy requirements as controlled by fiber treatment conditions, milling speed, initial fiber length and drying temperature. The predictive accuracy of the RSM and ANN models were adjudged adequate in terms of the process independent variables. A global optimal condition was found at 2% NaOH concentration, 29 mm fiber length, 1981 rpm mill speed and 41 °C drying temperature. At these conditions, specific energy was 1.430 kJ/kg for banana fiber and 1.109 kJ/kg for coir fiber having desirability of unity.

Keywords Central composite design · Optimization · RSM · ANN · Comminution energy

1 Introduction

Mechanical strength of reinforced composite product is dependent on stress transfer. It is guided by bond between the fibers and the matrix, and provides a progressively proficient impact to material brittleness/toughness [1]. A significant limitation in achieving efficient adhesion of bio-fiber strands to hydrophobic polymer matrix is the inherent hydrophilic characteristics of fibers and poor fibre dispersion, however treated particulate bio-fibers has shown improved intermingling, surface roughness, high degree of dispersion, uniformation and compatibility

with matrix [2]. Bio-fiber comminution can help to improve flowability and absorbability of fibers within the matrix. Therefore to enhance packing density/distribution of bio-fibers during composite product development, bio-fibers need be grounded into granules [3]. While fine granulating of fibers can decrease segregation of fiber components and improve compressibility of the bio-fibers, it can involve significant expenses as far as energy consumption is concerned. Various sorts of comminution equipments are handy for size reduction process and efficient use of energy is expected at every application [4].

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Scholars have explored the process of biomass size reduction for various purposes. Liu et al. [4] assessed the specific energy requirement and physical properties of a biomass intended for bioconversion, the study found dominant fracture mechanism across biomass lumen and middle lamella for hammer milling and knife milling respectively. Miao et al. [3] examined the specific energy requirement for mechanical size reduction of selected biomaterials for biomass-based bioenergy using knife and hammer mills. Other aspects of grinding process such as energy efficiency [5], energy use pattern during mineral processing [6], strategies for improvement of milling performances [7] and effect of comminution device on quality of particles [8] are well documented.

Literature has also shown various equipments applicable in comminution process to include hammer mills, single-roller mills, vibrating disk attrition mills, double-roller mills, knife mills, eccentric vibrating mills, ball mills, needle mills and shredders [9, 10]. Generally specific energy requirement in comminution processes is controlled by factors such as mill speed, volume of mill chamber, size reduction mechanism, moisture content, drying temperature, fiber treatment conditions, expected particle size and shape distribution [3, 11]. To identify optimal design arrangements, designers have considered laboratory scale milling experiments to establish particle size distribution functions, size parameters and independent variables which could provide insight into optimal operating conditions [12].

In the past, Le-Moigne et al. [13] used two-parameter Weibull approach to assess fiber particle size distribution and shape parameters of natural fibres after compounding in composites; the particle sizes distribution of natural rubber was investigated by Potivara and Phisalaphong [14] using laser diffraction technique; Zhang et al. [15] also used particle size analyzer to examine the effect of particle size distribution on properties of bio-composites, authors found that surface roughness, specific surface area and size of natural fibre particles has significant influence on mechanical interlocking, interfacial diffusion and mechanical properties of the composites. Despite the useful results so far reported, these post production assessment of particle size distribution however limits the designer's locus of control over critical material and process factors influencing mechanical properties of reinforced composites. This is because, in-debt knowledge of fibre distribution prior to compounding in composites is essential for initial characterization, setting of levels for fibre volume fraction, determination of optimal stirring time and particle size of natural fibres.

Particle size and particle size distribution are therefore considered as major factors which determines quality, performance, physical and mechanical properties of

particulate fiber reinforced composites and other solid agglomerates [16]. They assume significant functions in the flow capacity, mass thickness, compressibility, mechanical quality and strength of densified products. Nagarajan et al. [17] determined particle size distribution of biocarbon particle based on the mass retained in the sieves. El-Sayed and Mostafa [18] deduced the particle-size distribution of biomass using mechanical sieving technique. Although significant energy is generally needed to grind fiber into smaller particle-size, the fiber particle-size distribution needs to be studied and firmly controlled as it influences the quality and load support capabilities of reinforced composites.

In any case, comminution remains a key step in particulate fiber development process requiring proper machine and material based factor optimization [7]. Process modelling is often necessary during decision making and improvement of comminution processes aimed at maximizing performance and minimizing the energy consumption. The use of banana and coir fiber particulates as reinforcement for polymer composites is widely reported [19–21]. However, there is yet an absence of thorough data on particle size distribution and specific energy requirement models during comminution of banana and coir fibers intended for reinforced composite production, a gap that the present study intends to fill.

2 Materials and Methods

2.1 Materials

The banana and coir fibers were respectively extracted from post-harvest banana stem and outer shell of a coconut sourced from local farms in Akegbe-Ugwu, Nkanu-West, Enugu State, Nigeria. The banana stem and outer shell of a coconut were rated for 7 days; fiber extraction was done manually [22]. The extracted banana and coir fibers were chemically modified using NaOH at different concentration levels ranging from 2% to 12%. The treated fibers were further dried using a laboratory scale oven at varying temperature from 40 to 60 °C for one hour prior to comminution.

2.2 Characterization of the Treated Banana and Coir Fibers

Buck scientific M530 USA FTIR device furnished with deuterated triglycine sulphate and potassium-bromide beam splitters was used for characterization of the bio-fibers. Approximately 1.0 g of samples and 0.5 ml of Nujol was added; they were mixed properly and placed on a salt pellet. Gram-A1 software was then used to obtain the FTIR

spectra at 4000–600 cm^{-1} frequency zone and at 4 cm^{-1} resolution.

2.3 Grinding of Fibers

The bio-fiber comminution process was carried out in a laboratory-scale knife mill (Model No.: AJ000213/PI/HJ2443) having a 3 cm rotor energised by 220 W electric engine. The knife mill is equipped with 6.7 cm long and 0.1 cm width single bevel edge blade. Knife mill blade tip angle was set at 45°. Williams et al. [23] noted that the knife mill is a constant throughput machine that utilizes large shear powers to crack and reduce the bio-material into particulates by means of steady attrition. The initial fiber length was varied from 10 to 30 mm and mill speed was varied from 1400 to 2100 rpm.

2.4 Sieve Analysis

Lab weighing balance (Model: MS303S/01, Mettler Toledo, Switzerland) was used to take readings of weight of fibers prior to sieve analysis. Particulate fibers were separated into different sizes using sieve sets stacked in a laboratory sieve shakers. The sieves were stacked in such a way that those having tiny openings are placed at the bottom while the sieve having large openings is at the top. Five grams of fiber granule was then loaded on the top sieve and the stack shaken for 5 min. The fiber retained on each sieve was recovered and weighed. The weight of the empty sieves had been determined prior to sieve analysis. The actual weight of the sample is obtained by subtracting the weight of the empty sieve from the weight of the sieve containing the fiber.

2.5 Size Reduction and Energetic Comminution Laws

Electrical energy was recorded using a single phase plug-in power energy meter (Model No: SK410 UK). The instrument is equipped with large liquid crystal display (LCD) which provides simultaneous measurement of the voltage, current, frequency, time and electrical power consumption. The operating/output voltage is 230 V-AC while frequency display and max operating current are 50 Hz and 16 A respectively. Energy consumption (E) and specific energy (SE) in comminution of bio-fibers was found from Eq. (1) and (2);

$$E = \frac{\text{Electrical energy (net energy)}}{\text{Weight of sample}} \quad (1)$$

$$SE = \frac{\text{Energy consumed in kJ/kg}}{\text{Reduction ratio} \left(\frac{L_1}{L_2} \right)} \quad (2)$$

Three energy laws were used to study how particle size reduction relates with energy consumption during comminution process. The Kick's energy (E_K) required to break up a solid lump of fibers into smaller particle sizes was found using Eq. (3):

$$E_K = C_K \ln \left(\frac{L_1}{L_2} \right) \quad (3)$$

where C_K is the Kick's constant, L_1 is the initial fiber size, while L_2 represents the final fiber size.

ShabinaKhanam [24] suggests that Kicks law can be applied to coarse crushing where the feed size is quite large and reduction ratio is low. The Rittinger's energy (E_R) required to break up a solid lump of fiber into smaller particles sizes was found using Eq. (4):

$$E_R = C_R \left(\frac{1}{L_2} - \frac{1}{L_1} \right) \quad (4)$$

where C_R represents the Rittinger's constant, while L_1 and L_2 are the initial and final sizes of fiber respectively [25]. The Bond's energy (E_B) required to reduce bio-fiber into smaller particles sizes was found using Eq. (5):

$$E_B = 10 \times W_i \left(\frac{1}{\sqrt{L_{2,80}}} - \frac{1}{\sqrt{L_{1,80}}} \right) \quad (5)$$

where W_i is the Work Index, $L_{2,80}$ represents the final particle size at 80% cumulative undersize mass fraction, while $L_{1,80}$ correspond to the particle size at 80% cumulative undersize mass fraction.

2.6 Artificial Neural Network Training Scheme

Artificial Neural Network (ANN) was utilized to predict specific energy required in bio-fiber comminution process because it is efficient in learning and modelling non-linear pattern of variables in such a complex system. ANN model was created using the *nntool* command in MATLAB® R2014a. The networks has four (4) input neurons representing the design independent variables (NaOH concentration, fiber length, mill speed and drying temperature for fiber), one hidden layer of ten neurons and an one neuron output layer which represents the specific energy. The network layer transfer derivative function was set to *PURELIN* neuron model. The Neural Network model was then trained using the inputs and targets obtained from statistically designed experiments, the training scheme followed Levenberg-Marquardt back-propagation

algorithm (*TRAINLM*) and the adaptive leaning function was set to gradient descent with momentum bias learning (*LEARNGDM*). The experimental results were divided into three groups, 16(53.3%) was used to train the network, 5(16.7%) was used for validation while 9(30%) was used for network testing.

In order to lessen the variations of target prediction, training cycle was repeated using varied number hidden layer neurons; this trial-and-error process was necessary to set up the reasonable number of neurons needed in the hidden layer. The network performance was assessed using Coefficient of Determination (R^2) and Mean Square Error (*MSE*).

2.7 Central Composite Design (CCD)

ANN could not capture the relationship between input and output and could not deal with uncertainties; therefore a statistical central composite design in Design-Expert® software (version 12) was applied to establish relationships between independent variables (NaOH concentration, fiber length, mill speed and drying temperature for fiber) and specific energy requirement in the comminution process. Basically the process involves execution of statistically designed experiments, estimation of model coefficients and response prediction followed by model adequacy evaluation. The response Y_i can be related to the independent variables as a second-order quadratic model of Eq. (6):

$$Y_i = C_0 + \sum_{i=1}^n C_i A_i + \sum_{i=0}^n C_{ii} A_i^2 + \sum_{i=1}^n \sum_{j=i+1}^n C_{ij} A_i A_j + \varepsilon \quad (6)$$

where Y_i is the predicted specific energy, C_0 is the constant coefficient, C_i the linear coefficients, C_{ii} is the quadratic coefficients, C_{ij} is the interactive coefficients, A_i and B_j are the coded values of the variables, n is the number of independent test variables and ε is the random error [26]. The independent variables were varied according to CCD as shown in Table 1 which yields 30 experimental runs as presented in Table 5.

2.8 Model Error Functions

The predictive accuracy of the proposed models were compared using relevant error functions which includes Residual Sum Square (RSS), Model Predictive Error (MPE)%, Mean Average Error (MAE), Standard Error of Prediction (SEP), Root Mean Square Error (RMSE) and coefficient of determination (R^2) (Eqs. (7)–(11)).

$$RSS = \sum_{i=1}^n (y_{i,exp} - y_{i,pred})^2 \quad (7)$$

$$RMSE = \sqrt{\frac{1}{n} \sum_{i=1}^n (y_{i,exp} - y_{i,pred})^2} \quad (8)$$

$$(MPE)\% = \frac{100}{n} \sum_{i=1}^n \left| \frac{y_{i,exp} - y_{i,pred}}{y_{i,pred}} \right| \quad (9)$$

$$MAE = \sum_{i=1}^n \frac{(y_{i,exp} - y_{i,pred})}{N} \quad (10)$$

$$SEP = \frac{RMSE}{y_{exp,av}} \quad (11)$$

where n represents the total number of observations, y_m represents the average experimental value, $y_{exp,i}$ is the experimental response at observation i while and $y_{pred,i}$ is the predicted response at any observation i .

3 Results and Discussions

3.1 Characterization of Banana and Coir Fiber

Characterization of banana and coir fibers was carried out using FTIR as given in Figs. 1 and 2. The banana fiber FTIR spectrum was recorded for pectin ($C_6H_{10}O_7$), lignin ($C_{31}H_{34}O_{11}$), hemicelluloses ($C_5H_{10}O_5$), cellulose ($C_6H_{10}O_5$)_n and impurities. The C–H and O–H present in pectin are indicated by the peaks at 658.458, 787.969 cm^{-1} , and 889.442 cm^{-1} [27]. Its intensity reduction shows the

Table 1 Comminution process independent variables and their levels

S/N	Independent variables		Designation	Levels				
				−α	−1	0	1	+α
1	NaOH conc.	%	A	2	4	7	10	12
2	Fiber length	mm	B	10	13	20	27	30
3	Mill speed	rpm	C	1400	1517	1750	1983	2100
4	Drying temperature	°C	D	40	43	50	57	60

Fig. 1 FTIR spectrum of absorption for 2% NaOH treated banana fibers

Fig. 2 FTIR spectrum of absorption for 2% NaOH treated coir fibers

removal of pectin from the particulate fibers. The C–H, C=O and O–H present in hemicellulose are indicated by the peaks at 1336.034 cm^{-1} , 1434.002 cm^{-1} , 1624.095 cm^{-1} and 1864.072 cm^{-1} [27]. Its intensity reduction shows the removal of hemicellulose from the particulate fibers. The C≡C, C–H, C=O and O–H present in lignin are indicated by the peaks at 2068.257 cm^{-1} , 2204.97 cm^{-1} , 2458.769 cm^{-1} , 2677.955 cm^{-1} , 2815.165 cm^{-1} and 2912.621 cm^{-1} [27]. Its intensity reduction shows the removal of lignin from

the particulate fibers. The O–H present in cellulose are indicated by the peaks at 3197.795 cm^{-1} , 3292.478 cm^{-1} , 3698.372 cm^{-1} , and 3832.872 cm^{-1} [27]. Its intensity reduction shows the removal of celluloses from the particulate fiber. The N–H group shows the presence of impurities in the fiber. Thus FTIR analysis confirms the pectin, lignin, hemicellulose and cellulose removal. Table 2 shows the FTIR spectrum data table for 2% NaOH treated banana particulates.

Table 2 FTIR spectra data table for 2% NaOH treated banana fibers

S/N	Wavelength (n, cm ⁻¹)	Compound class	Range	Assignment	Intensity
1	658.458	Alcohols and phenols	650–770	O–H bend (out of plane)	Weak
2	787.969	Alkenes	780–850	=C–H and =CH ₂ (out-of-plane bending)	Weak to very weak
3	889.442	Alkenes	880–995	=C–H and =CH ₂	Very weak
4	1336.034	Alcohols and phenols	1330–1430	O–H bending (in-plane)	Weak to very weak
5	1434.002	Aldehydes and ketone	1400–1450	α-CH ₂ bending	Very weak to medium
6	1624.095	Amides	1630–1695	C=O (amide I band)	Medium to weak
7	1864.072	Carbonyls	1800–2100	C=O stretching	Weak to very weak
8	2068.257	Carbonyls	1800–2100	C=O stretching	Very weak
9	2204.97	Alkynes	2100–2250	C≡C (symmetry reduces intensity)	Very weak to medium
10	2458.769	Aldehydes	2300–2500	C–H (aldehyde)	Medium to weak
11	2677.955	Aldehydes	2500–2765	C–H (aldehyde)	Weak
12	2815.165	Carboxylic acids	2500–3300	O–H stretching	Weak
13	2912.621	Alkanes	2850–3000	CH ₃ , CH ₂ , and CH 2 or 3 bands	Weak
14	3197.795	Carboxylic acids	2500–3300	O–H stretching	Weak
15	3292.478	Carboxylic acids	2500–3300	O–H stretching	Weak
16	3411.347	Amines	3400–3500	N–H (1°-amines), 2 bands	Very weak to strong
17	3698.372	Alcohols and hydroxy	3645–3900	Primary alcohol, O–H stretch	Strong
18	3832.872	Alcohols and hydroxy	3645–3900	Primary alcohol, O–H stretch	Strong to weak

The FTIR spectrum of absorption results of 2% NaOH treated coir fiber is given in Fig. 2. FTIR spectrum was recorded for lignin (C₃₁H₃₄O₁₁)_n, hemicelluloses (C₅H₁₀O₅)_n, cellulose (C₆H₁₀O₅)_n and impurities. The C–H and O–H present in lignin are indicated by the peaks at 740.806 cm⁻¹, 895.598 cm⁻¹, 1049.105 cm⁻¹, 1177.988 cm⁻¹, 1324.070 cm⁻¹, 1401.206 cm⁻¹, 1609.606 cm⁻¹, 1673.221 cm⁻¹, and 1869.124 cm⁻¹ [27]. Its intensity reduction shows the removal of lignin from the fibers. The C≡C, C–H, C=O and O–H present in hemicellulose are indicated by the peaks at 2083.797 cm⁻¹, 2188.501 cm⁻¹, 2455.375 cm⁻¹, 2565.896 cm⁻¹, 2721.119 cm⁻¹, 2831.695 cm⁻¹, and 2913.120 cm⁻¹ [27]. Its intensity reduction shows the removal of hemicellulose from the particulate fibers. The O–H present in cellulose are indicated by the peaks at 3014.837 cm⁻¹, 3131.669 cm⁻¹, 3302.217 cm⁻¹, 3518.963 cm⁻¹, 3686.150 cm⁻¹ and 3829.762 cm⁻¹ [27]. Its intensity reduction shows the removal of celluloses from the particulate fibers. The C–N, N–H and S=O group shows the presence of impurities in the fibers. Thus FTIR analysis confirms the lignin, hemicellulose and cellulose removal. Table 3 shows the FTIR spectrum data table for 2% NaOH treated coir fibers.

In general, the maximum absorption wavelength for banana fibers is 3832.872 cm⁻¹. This is higher than that for coir fibers which has its maximum as 3829.762 cm⁻¹. The minimum absorption wavelength for banana particulates

is 658.458 cm⁻¹ which is lower than that for coir particulates (740.806 cm⁻¹). In 2% NaOH treated banana fibers, the median absorption wavelength is 2068.257 cm⁻¹ while in 2% NaOH treated coir fibers, the median absorption wavelength is 2188.501 cm⁻¹. The 2% NaOH treated coir fibers has more absorption wavelength than the 2% NaOH treated banana fibers.

3.2 Modelling by Response Surface Method

The relation between independent variables (NaOH concentration, fiber length, mill speed and drying temperature for fibers) and specific energy requirement in the comminution of banana and coir fibers were established using the response surface method (RSM) of Design Expert® software version 12. The NaOH concentration has the most compelling effect on the specific energy requirement during comminution process. Final equations in terms of actual factors are shown in Eqs. (12) and (13):

$$\begin{aligned}
 BF_Specific\ Energy &= 0.128099 + 0.425351 * A - 0.00531 \\
 &\quad * B - 0.00014 * C + 0.03329 \\
 &\quad * D - 0.00171 * A * B - 1.9E - 05 \\
 &\quad * A * C + 0.002938 * A * D - 0.00029 \\
 &\quad * B * D - 0.00875 * A^2 - 0.000284 \\
 &\quad * B^2 - 0.00025 * D^2
 \end{aligned}
 \tag{12}$$

Table 3 FTIR spectra data table for 2% NaOH treated coir fibers

	Wavelength (n, cm ⁻¹)	Compound class	Range	Assignment	Intensity
1	740.806	Alcohols & Phenols	650–770	O–H bend (out-of-plane)	Very weak
2	895.598	Alkenes	880–995	=C–H and =CH ₂	Very weak to weak
3	1049.105	Sulfoxide	1030–1060	S=O	Weak
4	1177.988	Amines	1000–1250	C–N	Weak
5	1324.070	Sulfone	1300–1350	S=O	Weak
6	1401.206	Aldehydes and Ketone	1400–1450	α-CH ₂ bending	Weak
7	1609.606	Amines	1550–1650	N–H ₂ scissoring (1°-amines)	Weak
8	1673.221	Amides	1630–1695	C=O (amide I band)	Weak
9	1869.124	Carbonyls	1800–2100	C=O stretching	Very weak
10	2083.797	Carbonyls	1800–2100	C=O stretching	Very weak
11	2188.501	Alkynes	2100–2250	C≡C (symmetry reduces intensity)	Very weak to strong
12	2455.375	Carboxylic acids	2500–3300	O–H stretching	Strong to weak
13	2913.120	Alkanes	2850–3000	CH ₃ , CH ₂ and CH 2 or 3 bands	Weak
14	3014.837	Alcohols and hydroxy	3645–3900	Primary alcohol, O–H stretch	Weak
15	3131.669	Alcohols and hydroxy	3645–3900	Primary alcohol, O–H stretch	Weak
16	3302.217	Amines	3300–3350	N–H stretching	Weak
17	3518.963	Alcohol	3400–3700	O–H stretching	Weak to strong
18	3686.150	Alcohol	3400–3700	O–H stretching	Strong to medium
19	3829.762	Alcohols and hydroxy	3645–3900	Primary alcohol, O–H stretch	Strong to weak

$$\begin{aligned}
 CF_Specific\ Energy &= -0.31797 + 0.462449 * A - 0.00491 \\
 &\quad * B - 0.00035 * C + 0.039409 \\
 &\quad * D - 0.0007 * A * B - 0.00008 \\
 &\quad * A * C + 0.0054 * A * D - 1.1E - 05 \\
 &\quad * C * D - 0.0045 * A^2 + 2.33E - 07 \\
 &\quad * C^2 - 0.00022 * D^2
 \end{aligned}
 \quad (13)$$

Equations (12) and (13) reveal how the individual variables affected specific energy requirement during bio-fiber comminution process. A comparable pattern was noticed for both banana and coir fibers. The negative coefficient values indicate factors that negatively affect specific energy requirement (i.e. more energy required), whereas positive coefficient values mean that factors minimise specific energy requirement in the tested range. Higher values of initial fiber length (B) and mill speed (C) contributed to high energy demand during bio-fiber comminution process. NaOH concentration (A) and drying temperature (D) main effects has positive coefficient values which suggests 2% NaOH treatment concentration and 40 °C dry comminution process. Pierre et al. [28] traced high energy consumption during size reduction processes to high moisture contained within biomass which acts as a plasticising agent. Similarly, Williams et al. [23] showed that very dry biomass readily breaks down during comminution.

Equations (12) and (13) also showed various interaction and second order effects of independent variables. The model adequacy was rationalized using the Analysis of Variance (ANOVA) of specific energy as given in Table 4 for banana and coir fibers.

The ANOVA Table 4 shows model F-value of 3139.99 and *p* value of <0.0001 for BF_Specific Energy, also shown is F-value of 5684.899 and *p* value of <0.0001 for CF_Specific Energy. These values imply that the proposed quadratic model is statistically viable. Values of “Prob F” < 0.05 indicate model terms are significant. In this case A(NaOH Conc), B(Fiber length), C(Mill Speed) and D(Drying temperature) are significant model terms. Some interactions between the parameters as shown in Table 6 are also significant.

The “Pred R-Squared” of 0.9977 and “Adj R-Squared” of 0.9992 were recorded for BF_Specific Energy while “Pred R-Squared” of 0.9990 and “Adj R-Squared” of 0.9995 were recorded for CF_Specific Energy. These values of Pred R-Squared and Adj R-Squared are in reasonable agreement; Adj R-Squared measures the signal-to-noise ratio and compares the range of predicted values at design points to the average prediction error, a ratio greater than 4 is desirable. The ratio of 180.272 for BF_Specific Energy and ratio of 251.913 for CF_Specific Energy indicates an adequate signal. These models were therefore used to navigate the design space.

Table 4 Analysis of variance table for reduced response surface quadratic model

Source	BF_Specific energy				CF_Specific Energy			
	Sum of squares	Mean square	F-value	p value Prob > F	Sum of Squares	Mean Square	F-value	p value Prob > F
Model	75.016	6.820	3139.99	<0.0001	142.013	12.910	5684.899	<0.0001
A-NaOH conc.	68.306	68.306	31,450.21	<0.0001	124.729	124.729	54,923.050	<0.0001
B-Fiber length	0.795	0.795	366.014	<0.0001	0.201	0.201	88.473	<0.0001
C-Mill Speed	0.211	0.211	97.123	<0.0001	1.233	1.233	542.968	<0.0001
D-Drying temperature	1.321	1.321	608.096	<0.0001	3.168	3.168	1395.113	<0.0001
AB	0.117	0.117	54.012	<0.0001	0.020	0.020	8.631	0.0088
AC	0.018	0.018	8.083	0.0108	0.314	0.314	138.091	<0.0001
AD	0.345	0.345	158.921	<0.0001	1.166	1.166	513.612	<0.0001
BD	0.014	0.014	6.357	0.0213				
CD					0.024	0.024	10.579	0.0044
A ²	0.776	0.776	357.155	<0.0001	0.207	0.207	91.023	<0.0001
B ²	0.013	0.013	6.021	0.0246				
C ²					0.023	0.023	10.137	0.0051
D ²	0.017	0.017	7.945	0.0114	0.013	0.013	5.769	0.0273
Residual	0.039	0.002			0.041	0.002		
Lack of Fit	0.039	0.003			0.041	0.003		
Std. Dev.=0.047, Mean=3.665, C.V. %=1.271, PRESS=0.174, R-Squared=0.999, Adj R-Squared=0.999, Pred R-Squared=0.998, Adeq Precision=180.272					Std. Dev.=0.048, Mean=3.989, C.V. %=1.195, PRESS=0.149 R-Squared=0.999, Adj R-Squared=0.999, Pred R-Squared=0.999, Adeq Precision=251.913			

df: model = 11, residual = 18, total = 29, lack of fit = 13, pure error = 5

Fig. 3 (a) Actual value of specific energy for BF versus predicted. (b) Regression plot for validation

3.3 Modelling by Artificial Neural Networks (ANN)

The Artificial Neural Networks was trained for 5 iterations. The Mean Square Error of the network for BF_Specific Energy prediction was 0.12, having a Coefficient of Determination (R^2) of 0.98 while the network for CF_Specific Energy prediction has Mean Square Error of 0.0043 and

Coefficient of Determination (R^2) of 0.997 (Figs. 3 and 4). For both banana and coir fibers, the training data suggests a good fit and the validation results showed high Coefficient of Determination (R^2) values that are close to unity.

Figures 3 and 4 represents the predicted values of the specific energy by the proposed model and corresponding actual specific energy. It was observed that the predicted

Fig. 4 (a) Actual value of specific energy for CF versus predicted. (b) Regression plot for validation

Table 5 Comparing the predictions of RSM and ANN with experimental data

Exp. no.	Independent variables				BF_Specific energy (kJ/kg)			CF_Specific energy (kJ/kg)		
	A	B	C	D	Actual	RSM	ANN	Actual	RSM	ANN
1	7	20	1750	30	3.17	3.19	4.00	3.14	3.17	3.15
2	7	20	1750	50	3.75	3.76	3.75	3.96	3.98	3.96
3	2	20	1750	50	1.64	1.63	1.61	1.32	1.29	1.32
4	2	10	2100	40	1.59	1.58	1.59	1.17	1.22	1.17
5	12	30	1400	40	4.92	4.94	4.92	5.98	6.01	5.98
6	2	30	1400	40	1.51	1.52	1.51	1.24	1.19	1.17
7	12	10	2100	40	5.21	5.21	5.21	5.68	5.62	5.68
8	2	30	1400	60	1.62	1.63	1.62	1.42	1.45	0.85
9	7	40	1750	50	3.5	3.46	3.50	3.83	3.78	3.83
10	12	30	1400	60	5.55	5.65	5.55	7.45	7.36	7.60
11	7	20	2450	50	3.59	3.57	3.72	3.63	3.64	3.62
12	7	20	1750	50	3.75	3.76	3.75	3.96	3.98	3.96
13	7	20	1750	50	3.75	3.76	3.75	3.96	3.98	3.96
14	2	30	2100	60	1.46	1.51	1.09	1.15	1.2	1.15
15	2	10	1400	40	1.68	1.7	2.68	1.31	1.31	1.31
16	12	30	2100	40	4.67	4.69	4.67	5.37	5.36	5.37
17	7	20	1750	50	3.75	3.76	3.75	3.96	3.98	3.96
18	2	30	2100	40	1.43	1.4	2.04	1.11	1.09	1.11
19	2	10	1400	60	1.92	1.93	1.92	1.57	1.58	1.57
20	12	10	2100	60	5.96	6.03	5.96	6.83	6.81	6.83
21	12	10	1400	60	6.29	6.29	6.29	7.59	7.62	8.05
22	7	10	1750	50	4.02	4	4.18	4.1	4.08	3.69
23	12	30	2100	60	5.45	5.39	5.45	6.51	6.55	6.51
24	7	20	1750	50	3.75	3.76	3.75	3.96	3.98	3.96
25	7	20	1050	50	4.02	3.95	3.60	4.53	4.55	4.53
26	7	20	1750	50	3.75	3.76	3.75	3.96	3.98	3.96
27	7	20	1750	70	4.2	4.13	4.20	4.62	4.62	4.62
28	12	10	1400	40	5.49	5.47	5.49	6.31	6.28	6.39
29	17	20	1750	50	6.75	6.71	6.75	8.65	8.69	8.65
30	2	10	2100	60	1.82	1.81	1.82	1.41	1.33	1.41

specific energy requirement is comparable to experimental data. Table 5 shows the central composite design matrix for banana and coir fibers.

3.4 Comparison of Response Surface Method and Artificial Neural Network models

The predictive veracity of the proposed Response Surface Method and Artificial Neural Network models were checked using Root Mean Squared Error (RMSE), Model Predictive Error (MPE)%, Mean Average Error (MAE) and Standard Error of Prediction (SEP) as shown in Table 6. The minimum RMSE, MPE, MAE and SEP are the best. It is noticed that RSM values were lower for RMSE, MPE, MAE and SEP. This indicates that RSM model is more precise in predicting specific energy requirement for bio-fiber comminution process; although this finding is contrary to those reported elsewhere [29], it may be attributed to the finite experimental data used in network training. In general terms, the Artificial Neural Networks needs an exceptionally huge number of data points for initial training of networks [30]. In line with the preceding observations, RSM was adopted in further optimization program.

3.5 Numerical Optimization Using Response Surface Method

The numerical optimization program was implemented to minimize specific energy requirement in bio-fiber comminution process using Design-Expert® software version 12. This was achieved by setting the needed criteria for each independent and dependent variables. On graphical user interface, the “Goal” field for variables is set to one of the five decisions: “none”, “maximum”, “minimum”, “target”, or “in range”. The objectives are combined into a global desirability function (D) (Eq. (14)) which reflects the desirable ranges for each response (d_i) from 0 to 1 with n number of measures. The shape of the desirability for each goal varies according to its weight (wt_i) field.

$$D = \left(\prod_{i=1}^n d_i \right)^{\frac{1}{n}} \quad (14)$$

According to StatEase [31], the numerical optimization algorithm (Fig. 5) requires that goal seeking must begin

from a random starting point and then progress towards the steepest descent to a minimum. Notwithstanding the design points, a lot of arbitrary points are verified to get an attractive solution. The algorithm defined the constraints as an array of alternate functions which creates an arrangement of m requirements that can be tackled as an unconstrained optimization problem by adding a penalty function term to the objective function as shown in Fig. 5, where X (a vector of x_i for $i = 1 \dots n$) serve as control factors (A: NaOH conc., B: fiber length, C: mill speed, D: drying temperature); y_j , U_j , L_j (for $j = 1 \dots m$) serve as constraints; $y(X)$ serve as the response (Specific energy in kJ/kg) to be optimized; p serve as the non zero penalty coefficient for $j = 1 \dots m$.

The penalty boundary begins at one and advances per iteration by a factor of one hundred for maximum of fifteen iterations. Cut-off points of the design space are kept up by assessing the $y(X)$ till $+10^{10}$ at the design boundaries. The inquiry around the first convergence mark is resumed-over utilizing a bigger penalty coefficient. Convergence is accomplished when the objective function variation is less than a 10^{-6} ratio. After the 100th trial, a global optimal condition was found at 2% NaOH concentration, 29 mm fiber length, 1981 rpm mill speed and 41 °C drying temperature. At these conditions, specific energy was 1.430 kJ/kg for banana fiber and 1.109 kJ/kg for coir fiber having desirability of unity (Fig. 6).

Corresponding confirmation experiments conducted at the global optimal conditions gave a specific energy requirement of 1.261 kJ/kg for banana fiber and 1.185 kJ/kg for coir fiber, these values are in reasonable accord with the statistical results of 1.430 kJ/kg for banana fiber and 1.109 kJ/kg for coir fiber, showing that they are reasonable conditions. In this way, it is suggested that bio-fiber comminution ought to be carried out at the given optimal conditions so as to minimize energy consumption.

3.6 Particle Size Distribution (PSD)

Particle size distributions (PSD) were dictated by mechanically sieving the comminuted bio-fiber as per BS EN 15149-2:2010 [32]. The fiber particulate samples were sieved into 250, 355, 500, 710, 1400 and 3350 μm sieve sizes. Sieving was conducted on a laboratory sieve shaker (Model BA 200N, CISA Cedaceria Industrial Spain). Each stage of sieving was carried out for an oscillation time of

Table 6 Comparison between statistical indices for RSM and ANN models

Source	RSM				ANN			
	RMSE	MPE	MAE	SEP	RMSE	MPE	MAE	SEP
BF_Specific energy (kJ/kg)	0.036	0.019	0.0003	0.0098	0.284	1.594	0.064	0.077
CF_Specific energy (kJ/kg)	0.037	0.061	7.40×10^{-18}	0.0093	0.157	2.506	0.012	0.039

Fig. 5 The algorithms used to obtain the optimal results of bio-fiber comminution process

Desirability = 1.000
Solution 1 out of 100

Fig. 6 Ramps view of optimized conditions for obtaining a minimum specific energy requirement in bio-fiber comminution process

5 min. The MATLAB® R2014a Curve Fitting Toolbox™ functions were used in fitting particle size distributions to the Rosin Rammler-Bennett (RRB) and Gaudin-Schuhmann (GS) models of Eqs. (15) and (16):

Rosin Rammler-Bennett (RRB) model:

$$W_R = 1 - e^{\left(\frac{-S}{S_R}\right)^{u_R}} \quad (15)$$

Gaudin-Schuhmann (GS) model:

$$W_G = \left(\frac{S}{S_G}\right)^{u_G} \quad (16)$$

where W_R and W_G are the cumulative weight fraction of fiber particulates retained on the sieve, S is the reduced fiber size assumed equivalent to the diagonal sieve opening (μm), S_R and u_R are the respective size and distribution parameter of Rosin Rammler-Bennett model, while S_G and u_G are the respective size and distribution parameter of Gaudin-Schuhmann model. The particle size distribution of the 2% NaOH treated fibers is shown in Figs. 7 and 8.

Fig. 7 Particle size distribution of Banana fibers as regressed by the GS and RRB models

Figures 7 and 8 reveal that the RRB model best fit the experimental data for both untreated and the 2% NaOH treated banana and coir fibers. The extent of the fit is shown in Table 7 with higher R^2 , lower RSS and lower RMSE. The RRB model described the particle size distribution of the two fibers with R^2 of 0.9783 for banana and

Fig. 8 Particle size distribution of coir fibers as regressed by the GS and RRB models

0.9851 for coir, RSS of 0.01854 for banana and 0.01212 for coir and RMSE of 0.05559 for banana and 0.04495 for coir.

3.7 Size and Distribution Parameter Analysis

Uniformity Index (I_u) which represents fiber particle spread from small to large size [33] was found using Eq. (17).

$$I_u = 100 \times \left(\frac{S_5}{S_{95}} \right) \quad (17)$$

Coefficient of Uniformity (C_u) and Coefficient of Gradation (C_g) functions were determined using Craig [34] (Eqs. (18) and (19)).

$$C_u = \frac{S_{60}}{S_{10}} \quad (18)$$

$$C_g = \frac{S_{30}^2}{(S_{10} \times S_{60})} \quad (19)$$

Also the Mass Relative Span (RS_m) in Eq. (20) was used as indicator for distribution width

$$RS_m = \frac{S_{90} - S_{10}}{S_{50}} \quad (20)$$

Other statistical parameters for distribution as developed by Folk & Ward [35] are shown in Eqs. (21)–(27).

$$\text{Graphic Mean (GM)} = \frac{S_{16} + S_{50} + S_{84}}{3} \quad (21)$$

$$\text{Standard Geometric Deviation of high region (SGD}_1) = \frac{S_{84}}{S_{50}} \quad (22)$$

$$\text{Standard Geometric Deviation of Low region (SGD}_2) = \frac{S_{50}}{S_{16}} \quad (23)$$

$$\text{Standard Geometric Deviation of the total region (SGD}_{12}) = \sqrt{\left(\frac{S_{84}}{S_{16}} \right)} \quad (24)$$

$$\text{Inclusive Graphic Standard Deviation} = \left[\left(\frac{S_{84} - S_{16}}{4} \right) + \left(\frac{S_{95} - S_5}{6.6} \right) \right] \quad (25)$$

$$\text{Inclusive Graphic Skewness (IGS)} = \left[\left(\frac{S_{16} + S_{84} - 2S_{50}}{2(S_{84} - S_{16})} \right) + \left(\frac{S_5 + S_{95} - 2S_{50}}{2(S_{95} - S_5)} \right) \right] \quad (26)$$

Table 7 The statistical and model parameters for banana and coir fibers particle size distribution

Material		Statistical parameters						Model parameters			
		RRB			GS			RRB		GS	
		R ²	RSS	RMSE	R ²	RSS	RMSE	x _R	n _R	x _G	n _G
Untreated	BF	0.9876	0.01184	0.04442	0.8678	0.1258	0.1448	6.066 × 10 ⁷	2.109 × 10 ⁵	2006	0.2397
	CF	0.9701	0.02185	0.06034	0.9535	0.03398	0.07525	474.5	0.9433	2795	0.3263
Treated with 2% NaOH	BF	0.9783	0.01854	0.05559	0.9028	0.08319	0.1178	3.04 × 10 ⁸	7.13 × 10 ⁵	2588	0.2985
	CF	0.9851	0.01212	0.04495	0.9135	0.07015	0.1081	280.9	0.4276	2807	0.394

Table 8 Size parameters for banana and coir fibers in μm

Size parameters	Banana fiber		Coir fiber	
	Untreated	2% NaOH	Untreated	2% NaOH
S_5	22	18	31	11
S_{10}	20	19	28	39
S_{16}	18	63	25	99
S_{25}	75	129	101	188
S_{30}	107	166	150	236
S_{50}	234	300	360	424
S_{60}	290	360	452	478
S_{75}	390	570	967	1090
S_{84}	495	1488	1448	1683
S_{90}	230	2187	2161	2308
S_{95}	1657	2768	2755	2829

Table 9 Distribution Parameters for banana and coir particulates

Distribution parameters	Banana fiber		Coir fiber	
	Untreated	2% NaOH	Untreated	2% NaOH
I_u	1.328	0.650	1.125	0.389
C_u	14.5	18.947	16.143	12.256
C_g	1.974	4.029	1.778	2.988
RS_m	0.897	7.227	5.925	5.351
GM	249	617	611	735.333
SGD_{12}	5.244	4.860	7.611	4.123
IGSD	366.977	772.917	768.477	822.970
IGS	1,000,725	3,683,338	3,349,652	3,546,456
GK	211,075.8	497,028.7	966,796.7	1,041,736

$$\text{Graphic Kurtosis (GK)} = \frac{(S_{95} - S_5)}{2.44(S_{75} - S_{25})} \quad (27)$$

where S and associated subscript in Eqs. (21)–(27) represents particle size and percentage cumulative weight fraction of fiber retained on the sieve respectively. The Inclusive Graphic Skewness (IGS) of Eq. (26) measures the degree of asymmetry of normal distribution curve. The

Graphic Kurtosis (GK) measures the sorting in the tails of distribution curve and the sorting in central portion [36] and was calculated using Eq. (27).

The size-related parameters were determined by interpolation and extrapolation from cumulative weight fraction data. The distribution related parameters were calculated using Eqs. (21)–(27). The size-related parameters S_5 , S_{10} , S_{16} , S_{25} , S_{30} , S_{50} , S_{60} , S_{75} , S_{84} , S_{90} and S_{95} are presented in Table 8.

From Table 8, comminution of 2% NaOH treated fiber results in less finer sizes with median size S_{50} of 300 μm for banana fiber and 424 μm for coir fiber. This is due to the hardness of the fibers caused by chemical modification of fibers' surface. The distribution-related parameters I_u , C_u , C_g , RS_m , GM, SGD_{12} , IGSD, IGS, GK are presented in Table 9.

From Table 9, the uniformity index I_u decreases with fiber treatment for both banana and coir particulates. The graphic skewness (IGS) values for banana and coir particulates are positive. This implies positive distribution. The Mass Relative Span (RS_m) being greater than 1 indicated wide distribution of particle sizes while RS_m less than 1 shows narrow distribution of particle sizes. The graphic mean (GM) increases with chemical treatment for both banana and coir particulates. Also the chemical treatment of the fibers and the resulting hardness, may have contributed to the observed increase in the specific energy requirement as depicted in Table 10.

It is observed from Table 10 that the bond's constant have values in-between kick's constant and Rittinger's constant for both untreated fiber and 2% NaOH treated fiber. This suggests that the Kick's model for comminution energy applies to large particle sizes; Bond's model for comminution energy applies to intermediate particle sizes while Rittinger's model for comminution energy applies to finest particle sizes, therefore the Rittinger's model for comminution energy best describes the energy consumption in comminution of banana and coir particulates.

It can be observed in Table 11 that treated banana and coir fibers have the lowest specific energy amongst the bio materials compared. This could be attributed to the comminution energy minimization strategies adopted in the present study; it could also be as a result of comminution

Table 10 Reduction ratio and constant values of the energy laws for banana and coir fibers

State of banana fiber		Average particle size (μm)	Reduction ratio	Kick's constant (C_K) (kJ/kg)	Rittinger's constant (C_R) (kJ/kg)	Bond's work index (W_i) (kJ/kg)	Specific energy (SE) (kJ/kg)
Untreated	BF	945	1181.74	51.28	18,351.00	237.61	0.31
	CF	953	605.45	58.59	39,934.04	359.94	0.62
Treated with 2% NaOH	BF	946	265.28	69.23	21,943.16	276.94	1.46
	CF	952	362.84	69.74	41,522.22	390.53	1.13

Table 11 Comparison of banana and coir fibers comminution parameters with other bio-materials

bio-material	Initial size of material (mm)	Comminution speed (rpm)	Comminution time (s)	Specific energy (kJ/kg)	Comminution machine	Source
Mango ginger	–	–	3600	5.12–9.53	Hammer mill	Murthy and Manohar [37]
Mango ginger	–	–	3600	5.36	Pin mill	Murthy and Manohar [37]
Coriander Seeds	–	1440	3600	14.9–48.6	Hammer mill	Shashidhar et al. [38]
Coriander Seeds	–	–	3600	34.9	Pin mill	Shashidhar et al. [38]
Miscanthus	123–127	2550–3000	–	50–65	Hammer mill	Moiceanu et al. [39]
Willow	24–47	2550–3000	–	35–50	Hammer mill	Moiceanu et al. [39]
Banana fiber	10–30	1400–2100	30	1.40–6.75	Knife mill	Present study
Coir fibers	10–30	1400–2100	30	1.09–8.65	Knife mill	Present study

Note: The type of the comminution machine could affect the specific energy during fiber comminution process

machine used. However, classical reports have shown that energy consumption during grinding process depends on material, machine and noise factors; these factors ought to be harnessed and controlled adequately for optimum comminution efficiency.

3.8 Implications for Reinforced Composite Product Applications

This research has featured a few significant applications in bio-fiber comminution for reinforced composite product development. The study has shown that lower concentration of NaOH in modification, high mill speed, smaller fiber length and moisture free bio-fiber showed significant drop in specific energy requirements without jeopardizing the granule particle size and shape. Information gathered from particle size distribution parameters is needed by engineers to effectively tailor and manipulate the materials and manufacturing conditions for a better, reliable and more cost-efficient composite product design. Particle size reduction increases the exposure of poorly soluble bio-fibers by increasing surface area and thereby improving wettability and packing density.

The present study is therefore relevant to particulate composite product development considering that mechanical properties of reinforced composite product vary in accordance with the fiber distribution within the polymer matrix; accordingly the examination of particle size is of prime significance in the fabrication and quality assurance of particulate fiber composite materials [40, 41].

4 Conclusion

The comminution process was carried out as a function of NaOH Concentration (A), initial Fiber length (B), Mill Speed (C) and Drying temperature (D). Specific energy

requirement in bio-fiber comminution process decreased at lower levels of NaOH treatment concentration and drying temperature while it increased with increase in initial fiber length and mill speed. The progressive quadratic model explores sufficiently the response surface space considering the high values of Adjusted Coefficient of Determination (R^2) of 0.999 for banana and coir fibers having adequate precision ratio which measure the signal to noise ratio of 180.272 for banana fiber and 251.913 for coir fiber.

The predictive accuracy of RSM and ANN models were compared using relevant error functions. RSM model was found more precise because of its lower MPE, MAE, SEP and RMSE in contrast to the ANN which has higher values; however, both RSM and ANN models can be applied in predicting specific energy requirement for bio-fiber comminution process. The RSM based empirical equation was subsequently solved and a global optimal condition was found. Confirmation experiments at selected optimal levels yielded feasible and comparable results. Also Rosin Rammler-Bennett and Gaudin-Schuhmann models were tested; however, statistical evidence suggests that Rosin Rammler-Bennett models gives best description of the bio-fiber particle size distribution.

Conflict of Interest The author(s) declare that they have no competing interests.

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