



Research article

Characterization and optimization of the properties of untreated high land bamboo fibres

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ABSTRACT

In this experimental study, untreated Ethiopian high land bamboo fibres were characterized and detected the optimal properties. In the investigation, SEM was applied to examine the surface texture of the fibres. The chemical bonds of the molecules (functional groups) were identified by Fourier transform infrared spectra (FTIR). The thermal properties of the fibre were explored with a thermogravimetric analyser, and the results were confirmed by differential thermo-gravimetric analysis (DTG). Mechanical properties were improved using the experimental design principle. The design is based on the RSM methodology three-factor three-level to present mathematical models. At various plant ages, the culm wall thickness in the radial direction and soaking duration of the single fibre is extracted using a roller crusher machine. The operating parameters and optimal mechanical properties were validated using confirmation tests. Breaking force 796.5cN, tenacity 46.8cNtex-1, work done 456cNmm, and modulus 1814cNtex-1 were the optimal mechanical characteristics achieved at the operating parameters 2.0 years age, a calm thickness layer coded values of 0.6 along the radial direction, and 3.8 days soaking time when compared to mechanical properties 1–3 years plant age, calm thickness layers of from primary (-1) to secondary layers (1) along with the radial direction, and 3–9 days soaking time.

1. Introduction

Researchers and technologists have increasingly become focused on natural fibres due to the benefits that these fibres offer over traditional support materials, and the advancement of distinctive fibres in recent years [1]. These natural fibres are good in density and cost with high specific characteristics. Natural fibres are the cellulose fibre that is made up of micro-fibrils embedded in the matrix of crystalline lignin and hemicellulose [2]. These fibres are extracted up of a sequence of fibrils that run the length of the fibre in a straight line. For strength and stiffness; the fibres rely on hydrogen bonding and other couplings. Vary according to the type of fibre utilized; natural fibres have different chemical compositions; Cellulose, hemicellulose, pectin, lignin, and other impurities make up fibres. Each ingredient's features add to the fibre's overall properties [3]. Hemicellulose is accountable for the fibre's microbial degradation, wettability, and depolymerisation since it has the lowest

resistance to biodegradation, moisture absorption, and thermal deterioration, whereas lignin is thermo-stable [4].

Bamboo is a common feature biological plant with great mechanical properties [5]. Bamboo stalks were employed extensively in the design of a building, packing board, furniture board, and wall panelling within the car, as well as the energy production and textile industries, due to these prevalent properties and other variables such as low cost, abundant natural resources, and environmental benefits [6]. Bamboo fibre reinforcement is applicable in different engineering applications. Some of these are biofuel production, paper industry, pharmaceutical industry, biomedical, automotive, aerospace, aircraft, solar panels, wind blade, and turbine blade [7]. The main factors influencing the material properties of bamboo fibres include fibre aspect ratio, fibre orientation, plant age, plant height, species type, culm wall layer thickness, and extraction methods [8]. Such methods of production have advantages and disadvantages over the actions and applications of fibre and have a great effect

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Figure 1. Ethiopian highland bamboo plant.

on the mechanical properties of bamboo fibre. Fibre extraction methods are generally classified as chemical and mechanical [9]. Alkali or acid retting is employed in chemical operations to eliminate amorphous regions and lower the lignin concentration of the elementary fibres [10]. Bamboo fibre obtained through mechanical processes had good quality and yields, but bamboo fibre obtained through the chemical-mechanical process has good quality but poor yield, and bamboo fibre obtained through the steam explosion process has a high specific strength [11]. The fibres at the primary layer resembled brittle, whereas fibres towards the secondary layer resembled ductile fracture. The phenomena were related to changes in the tensile characteristics of bamboo fibres [12].

Ethiopia possesses Africa's most plentiful lifted bamboo resources, and bookkeeping for a considerable amount of total bamboo resources. Ethiopia has around 1.3×10^6 ha of bamboo with two main bamboo varieties, high land bamboo, and low land bamboo [13]. The highland bamboo plants are ordinarily in Ethiopia's southern, southwestern, central, and northwestern highlands and the run of the highland bamboo was assessed at 300,000 ha [14].

This study explored that, a standard RSM design (BBD) was considered to study the factors that affect the properties of high-land bamboo fibres and explore the best mechanical characteristics to use for different applications. In the experimental study, bamboo fibre (high land

Table 1. Coded bamboo culm wall layers.

Culm Layers	Primary Layer	Middle Lamella	Secondary Layer
Code	-1	0	1

Table 2. The range and level of independent factors.

Parameters	Symbol	Units	Range and level		
			-1	0	1
plant age	A	Year	1	2	3
Soaking duration	B	Day	3	6	9
Culm layer	C	Coded	-1	0	1

Table 3. Experimental design values from design expert 11.

Run	A	B	C	Breaking Force	Tenacity	Work done	Modulus
	Year	Day	Coded	cN	cNtex ⁻¹	CNmm	cNmm ⁻¹
1	1	3	0	412	24.27	117	1325
2	2	3	1	1066	62.7	738.4	2239
3	2	6	0	743	43.71	327.4	1952
4	3	9	0	479.4	28.2	94.33	1842
5	2	9	1	685.5	40.32	180.5	2426
6	2	3	-1	532.2	31.31	140.4	2162
7	3	3	0	581.6	34.19	233.2	2176
8	1	6	1	657.9	38.7	292.7	1775
9	1	6	-1	407	23.98	103.2	1683
10	2	9	-1	520.7	30.63	118.6	1185
11	1	9	0	451	26.53	141.9	929.1
12	3	6	-1	582.5	34.26	217	2137
13	3	6	1	758.2	44.61	372	2213

bamboo) was obtained through water retting at different ages (1, 2, and 3 years), culm wall layer (primary layer, middle layer, secondary layer), and soaking durations (3, 6 and 9 days) was used to induce the optimized one based on mechanical properties those responses tested with the universal testing machine (TENSOLAB) based on the ISO testing standard. To analyze the material behavior of fibres, synchronous parameter

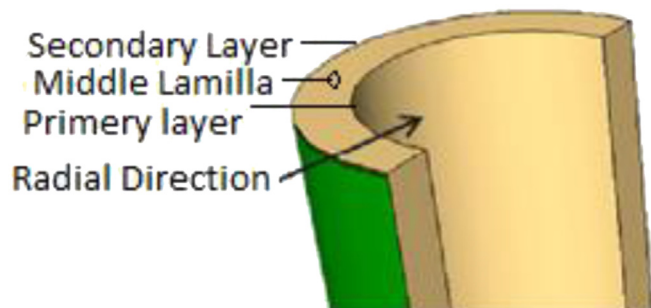


Figure 2. Bamboo culm wall layers cross Section.

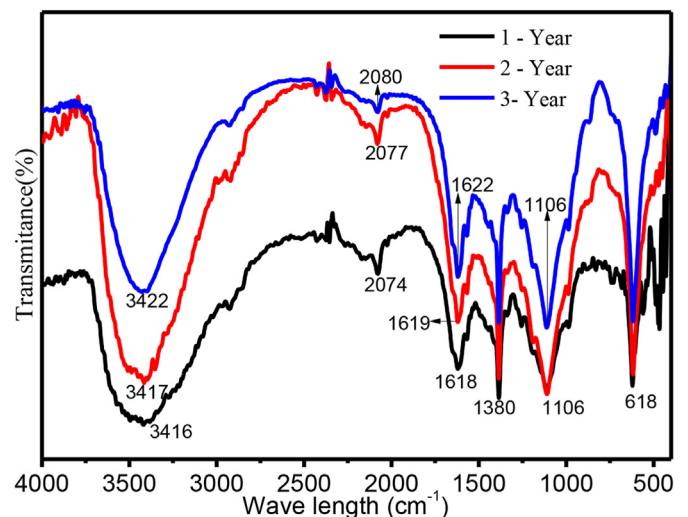


Figure 3. FTIR analysis of untreated high land bamboo fibre at different age.

Table 4. Characteristic bands for a functional group of untreated high land bamboo fibre at various ages [23, 30, 31, 32, 33, 34].

Wave number (cm ⁻¹)	Functional group	Source
3422–3416	O–H stretching	Alcohol (cellulose; hemi-cellulose; lignin), phenol (bond H), carboxylic acid
2080–2074	C–H	Alkanes
1622–1618	C=O	hemicelluloses and lignin
1380	C–H	cellulose and hemicellulose
1106	C–O	Alcohol (cellulose; hemicellulose; lignin), ether, carboxylic acid, ester
618	O–H out of plane bending	Lignin component

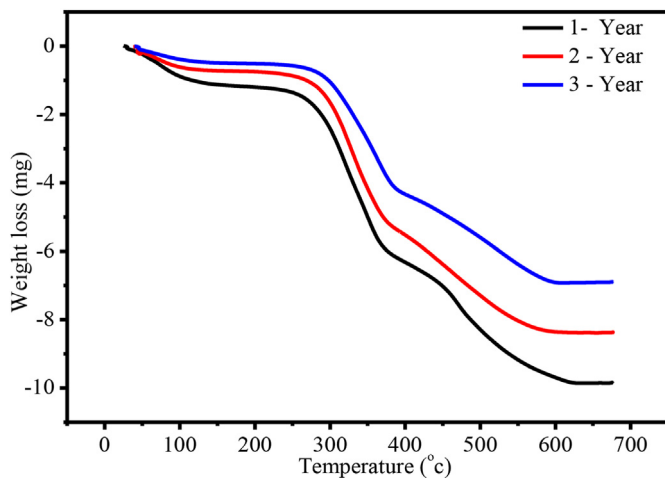


Figure 4. TGA analysis of untreated high land bamboo fibre with various age.

optimization was used. RSM was used to construct the statistical equation for modeling and analysis with three parameters, including fibre age, culm wall layer (primary layer, middle layer, secondary layer), and soaking durations (3, 6, and 9 days). The statistical approach and the accuracy of the parametric optimization were obtained using Design Expert Software 11.1.0. An arrangement of ideal parametric mechanical behavior of fibres was moreover assessed.

2. Materials and methods

2.1. Raw materials preparation

In the experiment, high land bamboo in Ethiopia was employed as the bamboo species; which is common across the country southern, south-western, central, and north-western highlands of Ethiopia, as illustrated in Figure 1.

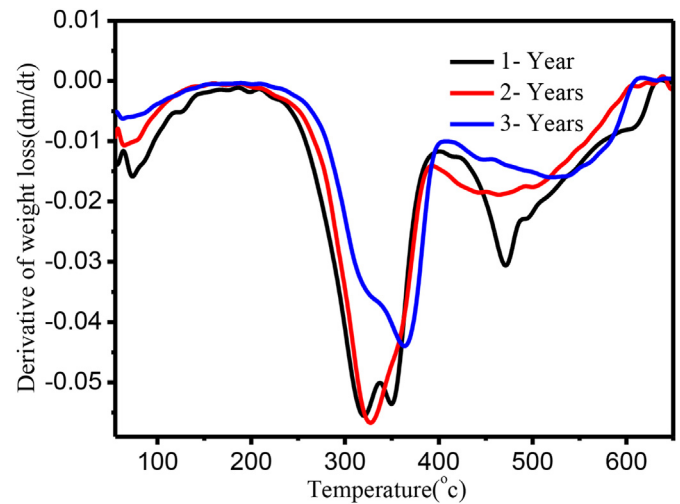


Figure 5. Differential thermal gravimetric analysis of untreated High land bamboo fibre.

They were cut in the central stem, which was around 25–30 cm in length and 4.5 thicknesses, and cleft with a slicer to remove residual parts, cutting the sampling into a strip, and soaking in H₂O with soaking duration (3 days, 6 days, and 9 days). Radially, from the 4.5mm culm layer thickness, a 1.5mm thickness bamboo strip from each layer (Figure 2) is prepared by dividing the culm wall layers equally. Before each test, the fibres were dried in an oven at 105°C for ½ hour.

From three independent variables, culm wall layers along the radian direction are coded numerically as shown in Table 1.

3. Methods

3.1. Extraction

Statistically designed experiments on bamboo fibre extraction were used to a certain optimum condition for fibre extraction. The following combinations were considered in the optimization process; Distended water, plant age (1–3 years), and soaking duration (3–9days). Then, they were placed in the roller-mill machine for three passes to derive the fibres. The roller crusher machine (PHOENIX) was utilized to extricate untreated chunks into small fibres. After fibre extraction, the extracted fibres were dried to constant mass at 105 °C in an air-forced dryer.

3.2. Experimental design

The BBD was used to determine the major impacts of the input factors on the responses, besides the model equation for each dependent variable [15]. When compared to traditional factorial design methods, Box–Beh nken designs can significantly reduce the number of experimental sets while maintaining the precision of the optimization [16]. All variables

Table 5. Thermal stability data of untreated high land bamboo fibre with various ages.

Stages	1 Year			2 Year			3 Year		
	T	Weight loss		T	Weight loss		T	Weight loss	
	°c	mg	%	°c	mg	%	°c	mg	%
Dehydration	≤140	1.1	11	≤132	0.7	7	≤127	0.4	4
1 st Degradation	140–228	0.2	2	132–232	0.1	1.0	127–233	0.1	1.0
2 nd Degradation	228–382	4.8	48	232–383	4.5	45	233–388	3.7	37
3 rd Degradation	382–623	3.9	39	383–600	3.1	31	388–600	2.7	27

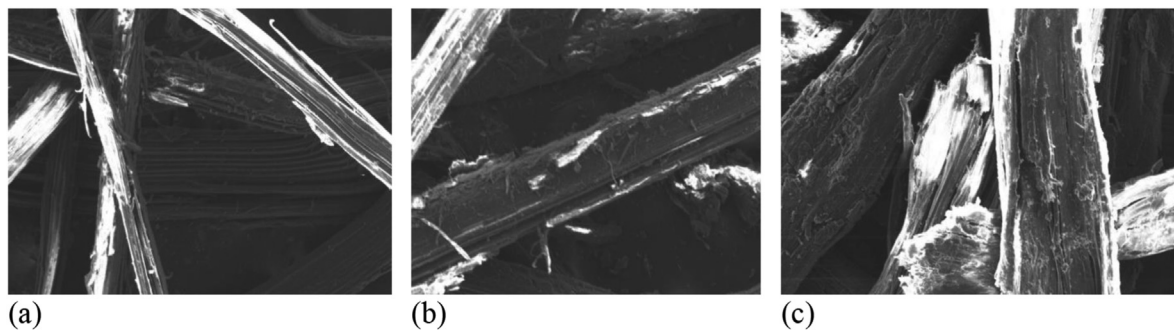


Figure 6. SEM image of (a) 1 year age (b) 2 years age (c) 3 years age untreated high land bamboo fibre micromorphology at 200µm magnification.

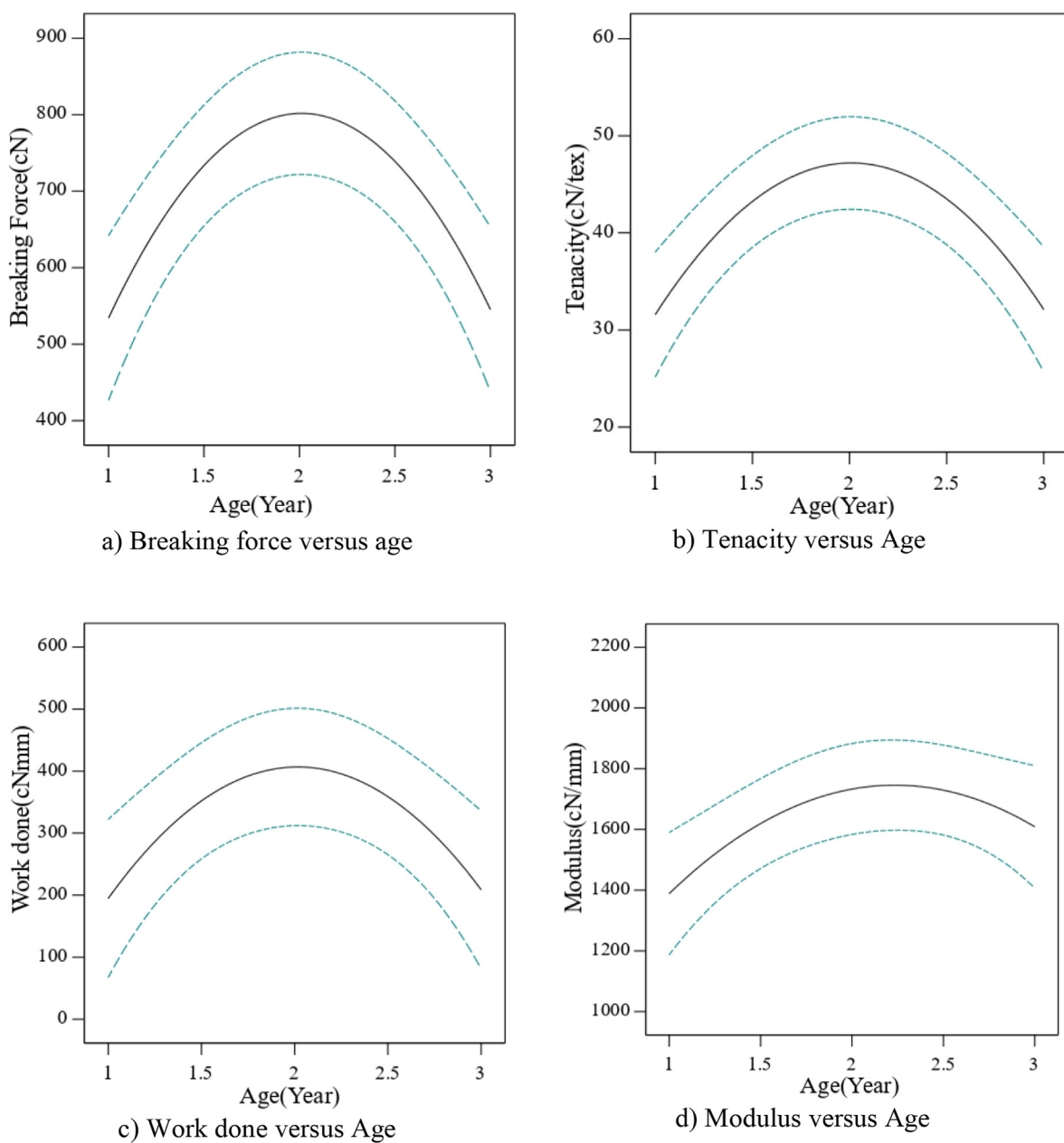


Figure 7. Age effects on the responses (a–d). a) Breaking force versus age. b) Tenacity versus Age. c) Work done versus Age. d) Modulus versus Age.

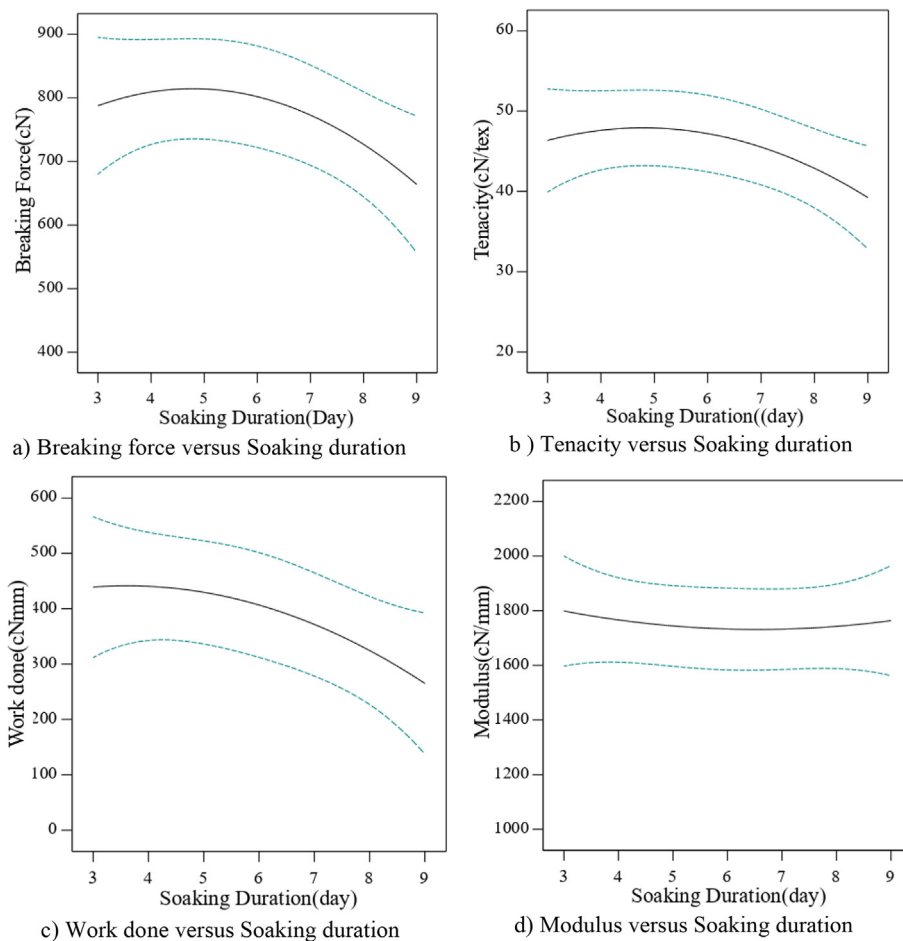


Figure 8. Soaking duration effect on the responses (a–d). a) Breaking force versus Soaking duration. b) Tenacity versus Soaking duration. c) Work done versus Soaking duration. d) Modulus versus Soaking duration.

(plant age, coded culm layers, and soaking time) can be coded as, low (−1) medium (0), and high (+1). The extent and extent of each independent factor based on BBD were presented in Table 2.

Design Expert[®] 11 was utilized within the plan lattice and examination of the test information. From the design of the experiment, 17 runs of experiments with 5 central points per block were carried out, as shown in Table 3.

$$y = \beta_0 + \beta_1A + \beta_2B + \beta_3C + \beta_{12}AB + \beta_{11}A^2 + \beta_{22}B^2 + \beta_{33}C^2 + \beta_{13}AC + \beta_{23}BC + \beta_{123}ABC \quad (1)$$

Where β_0 is the constant term, β_i , β_{ij} , and β_{ijk} are the coefficients of the various order terms.

Eq. (1) verified that the interaction of input factors (plant age(A), soaking time(B), and culm thickness layer(C)) and responses 'y' (breaking force, tenacity, work done, and modulus) were fit to the 3-factor polynomial model:

3.3. Characterization of high land bamboo fibres

Thermal stability of 10mg sample weight of untreated fibre was examined using TGA (BJHENVEN HCT-1) with temperatures ranging from 20 to 700°C at a rate of 20°C per minute in an air atmosphere. FT-IR (JASCO MODEL FT-IR 6660) was used to detect functional groups and other components in untreated high-land bamboo fibres. Scanning

electron microscopy (JCM 6000 PLUS) was used to illustrate the morphological alterations of fibres with a random orientation at various ages using various magnifications and a 10kV acceleration voltage 13. Mechanical properties (breaking force, tenacity, work done, and modulus) of samples were analysed based on design expert software suggestions. The test was carried out under the ISO13934–1:2013 testing standard [17]. The available load cells of the machine were 20N, 100N, 500N, 1000N, and 5000 N. The machine's speed and force measurement accuracy are 0.01% and 0.03%, respectively, under stable conditions. The load cell used was 20N with a force maximum resolution of 0.0002cN. The gauge length of the measurement is 50mm and the testing speed range is 0.001–1000 mm/min. The samples were aligned and an axial tensile load was then applied.

4. Results and discussion

4.1. Chemical and physical properties of high land bamboo fibres

As illustrated in Figure 3, FTIR graph of untreated Ethiopian high land bamboo fibre at different ages. The results of all of the samples displayed typical cellulose ware spectra, which was in line with previous studies [18, 19, 20]. All of the samples exhibited virtually comparable FTIR spectra, demonstrating that no new functional groups in the cellulose molecules were added during aging from 1 year to 3 years. However, as age increases, the broad absorption band in the region decreases. The

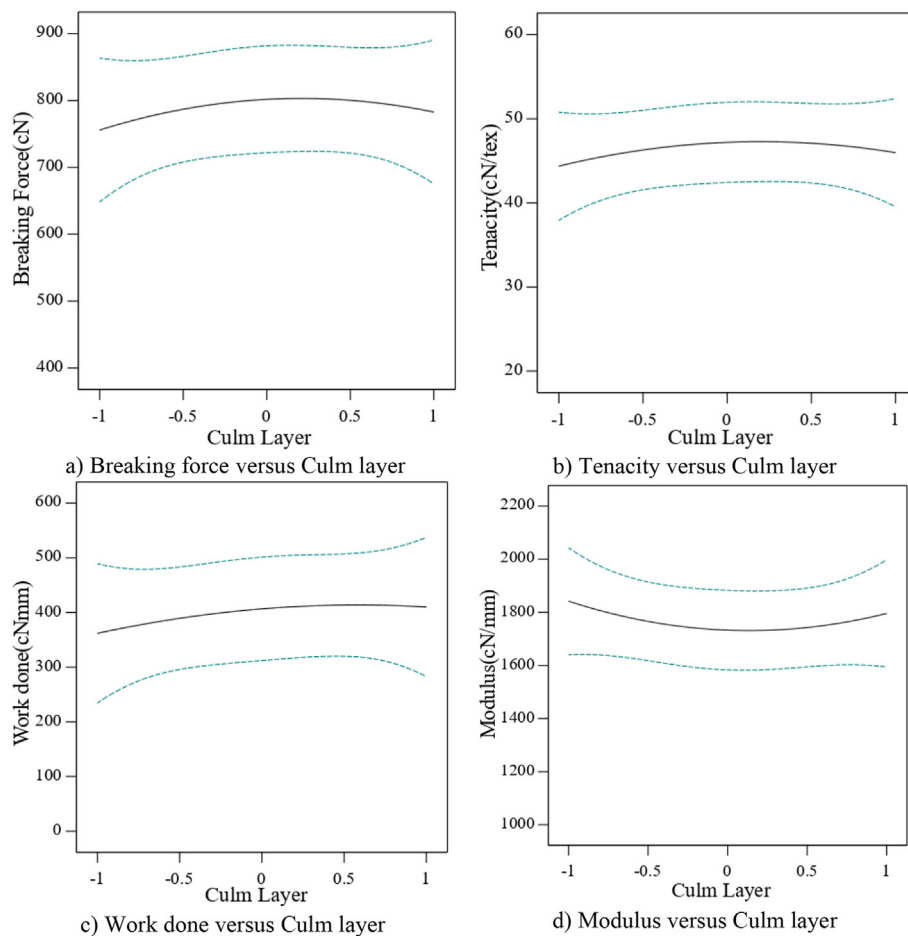


Figure 9. Culm layer effect on the responses (a–d). a) Breaking force versus Culm layer. b) Tenacity versus Culm layer. c) Work done versus Culm layer. d) Modulus versus Culm layer.

functional group and chemical compounds of untreated high-land bamboo fibres at different ages extracted from Figure 3 was shown in Table 4.

Figure 4 illustrated the variations in thermal degradation for high-land bamboo fibre, as characterized by maximum temperature and weight reduction from 1 to 3 years of aging. The dehydration stage is the first step in the decomposition process. This phase was ascribed to moisture in the fibre and some extractive evaporation. As a result, shown in this zone, young age (1 year) bamboo loses the most weight (11%), while elderly age (3 years) bamboo loses the least (4%) and the middle 2-years bamboo fibre has weight losses of 7%. According to this scenario, young bamboo fibres release more moisture and bind water. The findings were in line with earlier research [19, 21]. The 1st degradation was the hemicellulose degradation phase. It degrades and the majority of hemicellulose weight loss occurred. In contrast to hemicellulose, cellulose comprises a longer chain of polysaccharides that is more thermally stable and its degradation is at a higher temperature range. Due to its complicated structure and presence of aromatic compounds, lignin was the most difficult of the three components to degrade. As a result, lignin decomposition is slow and difficult throughout the full temperature range up to 700 °C [22, 23, 24]. As seen in Table 5, the main composition removal of the fibres caused the weight loss in the first, second, and third degradation stages [25]. At all stages of degradation, young bamboo (1-year-old) loses more weight than mature bamboo (2 and 3 years old). As a result, young bamboo fibre degraded more major contents than mature bamboo fibres.

Maximum temperature (T_{max}) is the decomposition temperature that corresponds to the maximum weight loss and decomposition rate, as well as a key indicator of the materials' thermal stability [26]. The maximum decomposition temperature of untreated high land bamboo fibres aging from 1 - year to 3 - years, was in the 2nd degradation zone with 228–388°C. In this degradation phase, mostly cellulose decomposition was carried out and maximum weight loss was done. This experimental investigation concluded that among the 1-year to 3 years aging of untreated high land bamboo fibres, 3 years of bamboo fibre has optimal thermal stability properties. Figure 5 shows how DTG analysis confirms this maximum temperature and weight loss debate.

Figure 6 exhibited the surface morphology of randomly arranged untreated high-land bamboo fibres with various fibre ages. The increasing content of non-cellulose major components of the fibres is responsible for the change in fibre diameter with age. In previous studies, the same conduct was also observed [19, 27, 28]. Figure 5 explored that, the morphological roughness of the surface increased with age. Because they allow excellent surface connection between polymeric matrix, these rough surfaces have significant advantages in the production of composite materials [29].

4.2. Mechanical properties of high land bamboo fibres

Modulus was not affected by plant's age as much as breaking force, tenacity, and work done. Figure 7 illustrates that these mechanical

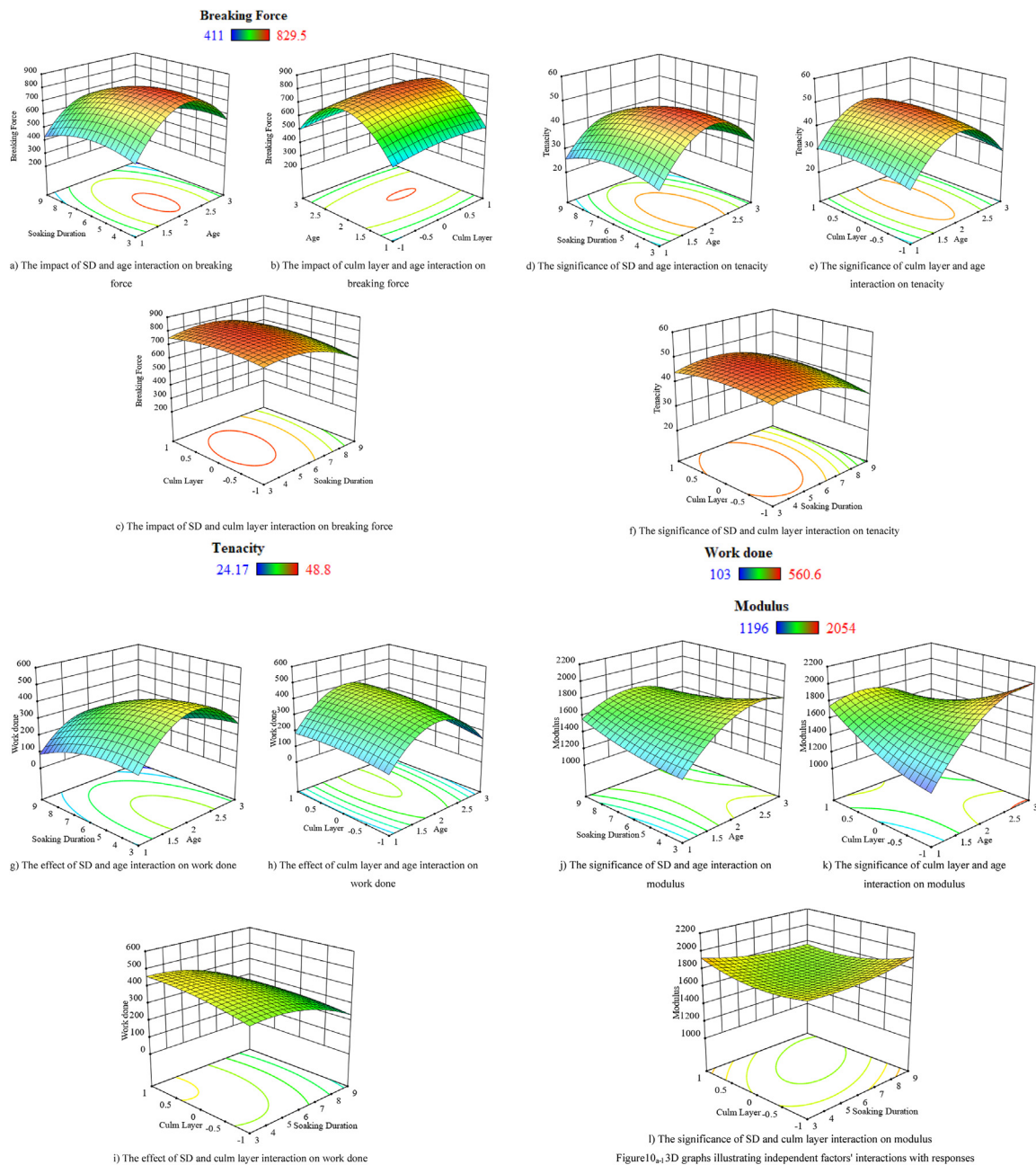


Figure 10. 3D graphs illustrating independent factors' interactions with responses. a) The impact of SD and age interaction on breaking force. b) The impact of culm layer and age interaction on breaking force. c) The impact of SD and culm layer interaction on breaking force. d) The significance of SD and age interaction on tenacity. e) The significance of culm layer and age interaction on tenacity. f) The significance of SD and culm layer interaction on tenacity. g) The effect of SD and age interaction on work done. h) The effect of culm layer and age interaction on work done. i) The effect of SD and culm layer interaction on work done. j) The significance of SD and age interaction on modulus. k) The significance of culm layer and age interaction on modulus. l) The significance of SD and culm layer interaction on modulus.

properties rise with age until two years, after which they decreased. This observation is consistent with the findings of a prior Moso bamboo study (*Phyllostachys hetero-cycla* var. *pubescens*) [35]. And also in modulus up to nearly 2-years-old, the modulus was increased and then slightly lowered, in contrast with previous studies, on the Lv and Ma (*Dendrocalamopsis oldhami* and *Dendrocalamus latiflorus* Munro) bamboo [36]. Breaking force, tenacity, and work done decreased when the soaking time was increased, however, there was no significant variation in modulus as shown in Figure 8. These observations are comparable to

previous studies [37]. Figure 9 shows that the mechanical properties except for modulus increased a touch up to around 0.6 coded values along the radian direction and then go almost constantly. Somewhat, the modulus reduced from the primary to the middle lamella before increasing slightly, which contradicts previous research, which found that the culm layers had a significant impact on mechanical properties [12].

The main properties of high-land bamboo fibre are affected by the input parameters of this experimental study. The graphic demonstrates

Table 6. Coefficients and constant values of responses.

Coefficients	Responses			
	Breaking Force	Tenacity	Work done	Modulus
β_0	801.8	47.2	406.7	1733
β_1	5.62	0.2663	6.99	110.11
β_2	-61.78	-3.55	-86.98	-17.67
β_3	13.41	0.805	24.11	-23.09
β_{12}	-32.3	-2.06	-32.08	-141.13
β_{13}	-2.35	-0.125	12.2	-295.05
β_{23}	18.97	1.11	-15.21	-62.54
β_{11}	-262.01	-15.35	-204.9	-234.35
β_{22}	-76.09	-4.41	-54.62	48.27
β_{33}	-32.44	-2.03	-20.70	85.95
β_{123}	0.00	0.00	0.00	0.00

how the interplay between plant age, Culm layer, and soaking period parameters affected all mechanical characteristics. Breaking force and tenacity were maximal in the plant age and soaking time ranges of 1.17–2.3 years, and 3.0–5.8 days, respectively. These mechanical properties are at their weakest near 1 year and 3 years, and near 3 days and in the range of 8–9 days. The work done had maximum value in the range between plant age of 1.7–2.6 years and soaking duration of 3–6 days, and the minimum around 1 year and in the range 2.8–3 years, the soaking time in the range between 6 to 9 days. In the modulus, the maximum value is attained at 3–9 days soaking duration and 1.3–2.7 years of plant age. Figure 10 explored the interaction effect for the response surface plot of soaking duration versus culm layer on the mechanical properties is represented. Breaking force and tenacity were slightly increased by increases in soaking duration up to the mean point and increased linearly along with the culm wall layer in the radial direction. The minimum values of these mechanical properties attained near 9 days soaking duration at the primary culm layer along the radial direction. The work done maximum value attained more than the middle lamella along the radian direction of below 3.5 days soaking duration. The minimum values attained near 9 days soaking duration at the primary culm layer along the radial direction. The lowest modulus value occurred near the midpoint of the soaking duration of the middle lamella culm layer along the radial direction and the maximum value was at the lowest duration of the soaking time of the secondary culm layers along the radial direction. The breaking force was maximized at the mean point of the plant age around the middle lamella of the culm layer whereas; the minimum value is attained at the 1 years old of primary layers of bamboo wall thickness. The maximum tenacity is attained around the mean value of plant age along with the culm layer in the radial direction from the primary to the secondary layer, and also the minimum value near 1 year and 3 years in the primary culm layer wall thickness. The maximum characteristics of the fibre that were characterized in this experimental study due to the interaction effect of plant age and culm layer occurred around 2 years of culm layer from the middle lamella to the secondary layers. The

minimum mechanical property was attained below 1.4 years and above 2.7 years of high-land bamboo fibre.

4.3. Statistical analysis

4.3.1. Modelling and validation

The ANOVA for the regression model was significant, with R^2 values of 0.9030, 0.8995, 0.8288, and 0.8522 for breaking force, tenacity, work done, and modulus, respectively. This means that the experimental variables accounted for 90.03 %, 89.95 %, 82.88 %, and 85.22 % variations in breaking force, tenacity, work done, and modulus, respectively. Raise the R - squared value to show that the model closely matches the experimental values and that the models are suitable for data correlation. It should be at best 0.80 to be regarded as satisfactory [38].

The values of interaction between input variables and responses were calculated using P-values. The breaking force F-value of 7.24 indicates that the model is significant. Plant age quadratic terms ($p \leq 0.0002$) were the only significant model terms in this scenario. The F-value of 6.96 for the tenacity model suggests that it was significant, as this value has a 0.9% chance. The plant age quadratic term was the only significant model term ($p \leq 0.0002$). To establish the importance of substantial interactions between input variables and work done, the p-values of significant interactions were used, and the only significant model factors ($p \leq 0.0285$) were soaking time and quadratic plant age values. The modulus model has an F-value of 4.48. This F-value has a because of noise [39].

The quadratic model was used in comprehensive ANOVA, and independent variables defined in Eq. (1) were affected for all of the results. Except modulus, Table 6 shows that the quadratic coefficients of plant age have the highest values among the linear, quadratic, and interaction coefficients of significant independent variables. The coefficient of AC in the modulus equation has the greatest value among the coefficients of the significant independent variables, indicating that the interaction of plant age and culm layer ($p \leq 0.0042$) has the most negative effect on the model equation.

Figure 11 depicts a linear graph of expected as well as actual tensile strength and modulus of bamboo fibres. All scatter points are precisely distributed near or around the line, indicating that the experimental and predicted values are highly correlated.

The responses (breaking force, tenacity, work done, and modulus) on the ranges 411cN–829.5cN, 24.17cNtex⁻¹ to 48.8cNtex⁻¹, 103cNmm to 560.6cNmm, and 1196cNtex⁻¹ to 2054cNtex⁻¹ were set as predicted values of breaking force, elongation, tenacity, work done, and modulus, respectively, with the three factors balanced, a plant age of 2.0 years, a culm layer coded value of 0.6, and a soaking time of 3.8 days were used to improve mechanical properties. The optimum values for breaking force, tenacity, work done, and modulus, according to the BBD results, were 796.5cN, 46.8cNtex⁻¹, 456cNmm, and 1814cNtex⁻¹, respectively. The model's accuracy was confirmed using five experiments run. Using the optimal variables generated by BBD, the testing results (breaking force, tenacity, work done, and modulus) were 800cN, 49.32cNtex⁻¹, 420.15cNmm, and 1783.21cNtex⁻¹, respectively (2 years age, 0.6 culm layer coded value, and actual, and 3.8 soaking days) Figure 12. According to the

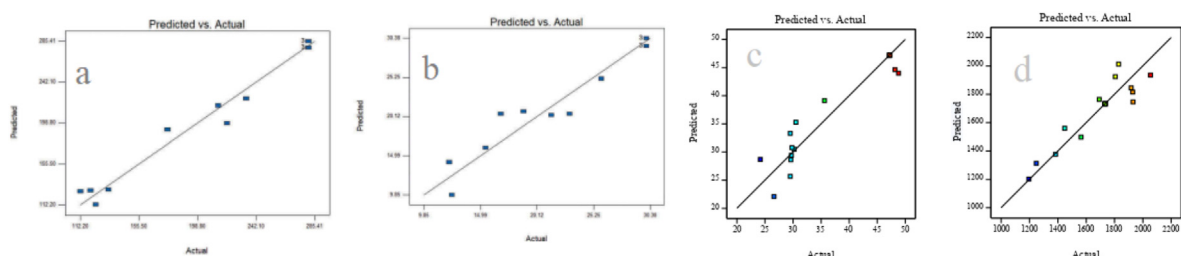


Figure 11. Tensile strength (a, c) and modulus (b, d) of predicted versus actual plot.

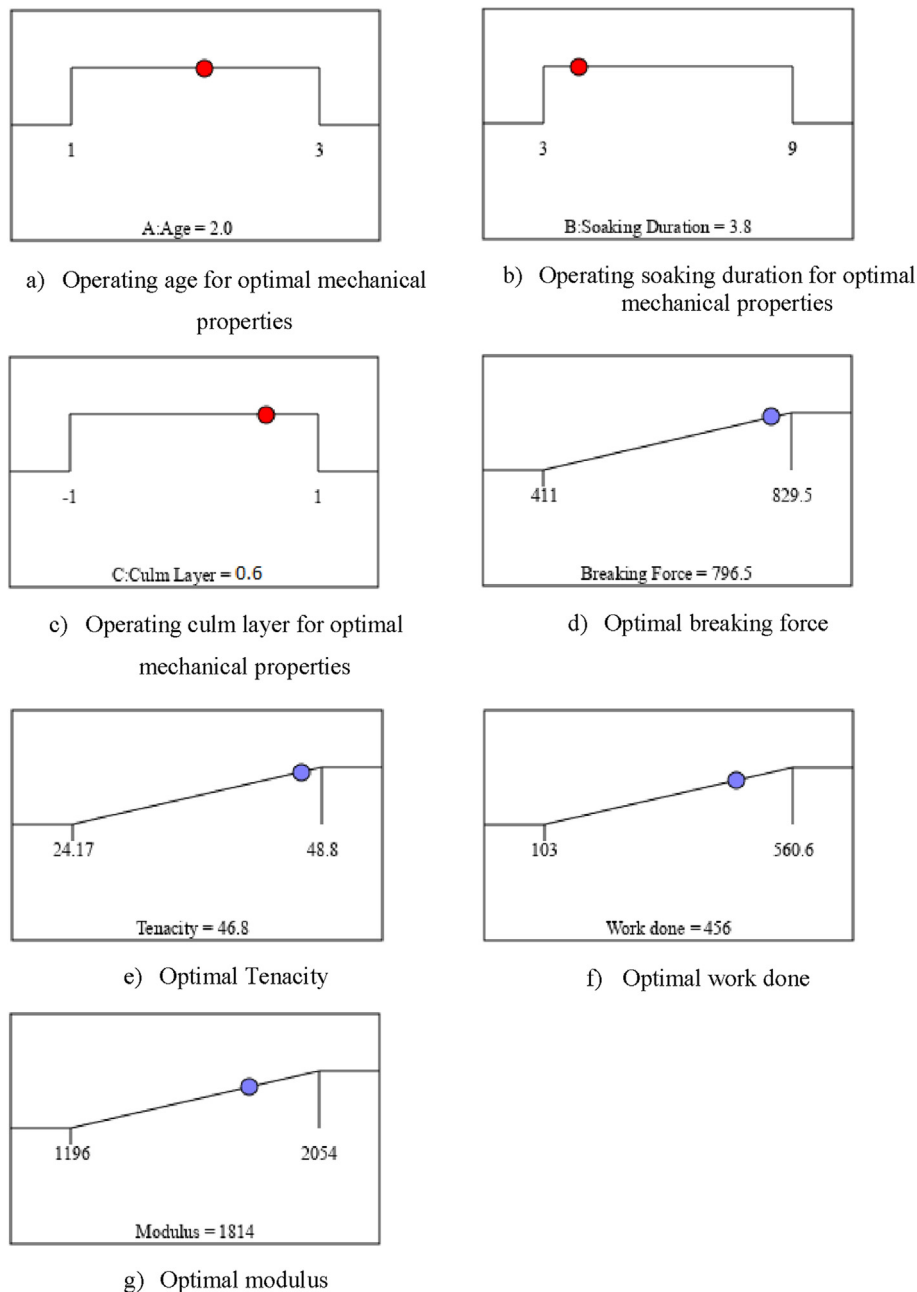


Figure 12. Selections of input factors for optimal mechanical properties for untreated high land bamboo fibre. a) Operating age for optimal mechanical properties. b) Operating soaking duration for optimal mechanical properties. c) Operating culm layer for optimal mechanical properties. d) Optimal breaking force. e) Optimal Tenacity. f) Optimal work done. g) Optimal modulus.

results, the error between the experimental and predicted values was acceptable within the range of 4%, which is consistent with prior studies [35, 40, 41, 42]. The existence of the optimal point was demonstrated by a good match between predicted and experimental data, proving the model's validity.

5. Conclusions

The BBD concept was established as a low-cost approach in this study. The quadratic design model was used. The breaking force, tenacity, work done, and modulus were all measured. Optimal mechanical properties of breaking force, elongation, tenacity, work done, and modulus with numerical values of 796.5.8cN, 46.8cNtex⁻¹, 456cNmm, and 1814cNtex⁻¹

respectively was attained at operation parameters of a plant age 2.01.8 years, culm layer coded value of 0.6, and soaking duration of 3.8 days. During these phases, thermal, physical, and chemical structural identification of bamboo fibre was investigated. Young bamboo fibres lose more weight in the thermal analysis due to their higher moisture content and ability to bind water. The diameter and thermal sustainability of untreated high-land bamboo fibre aged from one to three years were directly proportional. In contrast, the level of moisture was inversely proportional. The operational parameters that significantly improved the characteristics of untreated high-land bamboo fibre were selected for further studies in the composite development. The responses' optimum values are also essential for the application of the fibre for various purposes to design the desired one.

Declarations

Author contribution statement

Dawit Tessema Ebissa: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Tamrat Tesfaye, Dellele Worku: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

David Wood: Analyzed and interpreted the data.

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Data availability statement

The data that has been used is confidential.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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