

Process Optimization of Pineapple Leaf Fibre-Based Kraft Paper Integrated with Gum Arabic

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Abstract

The ever-increasing environmental concern on pollution from polymer derived packaging materials has led to research on new, sustainable and environmentally friendly materials. Among the new ventures are non-wood fibres and agro-waste. Pineapple Leaf Fibre (PALF), a byproduct of pineapple cultivation, proffers a promising and sustainable alternative for kraft paper production. This study explores the optimal processing conditions for the integration of Gum Arabic to strike a good balance between the tensile strength and flexibility of a PALF-based kraft paper. Experiments were conducted to evaluate the effects of chemical treatments on the pulp yield, chemical composition and mechanical properties of the kraft paper produced. The tensile strength values of the PALF-based kraft paper recorded has a mean $\pm$ standard deviation of $16.24\text{N/mm}^2 \pm 5.46\text{N/mm}^2$. This result outperformed papers produced from non-wood fibres and matches the tensile strength values of PALF composites reported in literature. Through systematic experimentation, it was found that the multi-response optimal process parameter combination for a PALF-based kraft paper is cooking temperature at 130°C , moderate alkali concentration at 4% and 50 % Gum Arabic concentration with a cooking time of 90 minutes and a beating time of 12 minutes. These findings accentuate the viability of PALF as a raw material for kraft paper, offering a promising sustainable solution for the packaging industry and potential commercial applications.

Keywords: Pineapple Leaf Fibre; Gum Arabic; Kraft Paper; Mechanical Properties; Sustainable Packaging

1. Introduction

Packaging Kraft or Kraft Paper is a common type of paper known for its strength and used for a variety of purposes, including packaging, cushioning, and safeguarding items during storage and transportation. Typically, kraft papers are made using recycled paper or wood pulp [1], [2]. Natural fibres have emerged as a preferred alternative to synthetic fibres in various applications, including packaging, textiles, construction, and automotive sectors. These fibres are classified based on their origins; plant, animal, or mineral and are recognized for their environmental benefits and performance characteristics. Fibres such as jute, flax, sisal, hemp, and pineapple leaf fibre (PALF) are gaining traction due to their renewability, biodegradability, low cost, and favorable mechanical properties [3], [4]. These natural fibres exhibit high pulp yield and mechanical properties suitable for kraft paper production [5], [6]. PALF stands out as a potential substitute for traditional wood pulp because

of its high cellulose content, contributing to its tensile strength, lightweight nature, and versatility in packaging applications [1], [7], [8], [9].

The surge in global industrialization has significantly boosted paper consumption, particularly in packaging, which accounts for over 41% of worldwide paper usage [10], [11]. According to the Food and Agriculture Organization of the United Nations' 2013–2018 capacities survey, it is projected that global paper consumption will reach 500 million tons by 2025, growing at a rate of 1.6% annually [11], [12]. This extensive use of paper has led to rapid deforestation, causing a substantial depletion of natural resources, particularly hardwood. Consequently, continuous research efforts have been dedicated to finding alternative pulp sources suitable for paper manufacturing, especially in packaging. Various non-wood substitutes have been explored as potential options for paper production, with pineapple leaf fibre (PALF) emerging as one such

substitute [2], [12]. PALF offers not only a green alternative but also addresses the environmental challenges associated with waste disposal and pollution from pineapple leaf by-products [13], [14], [15].

The Food and Agriculture Organization (FAO) reports that the total production of pineapples was approximately 27.4 million tons in 2017 and is projected to reach 31 million tons in 2028. Approximately 76.4 million tons of byproducts are produced worldwide [9]. During the post-harvest and processing of pineapples, the leaves are primarily disposed of as waste, burned in open fields, or composted with only a fraction of the annual PALF production being utilized as a feedstock for energy production, thus exacerbating the environmental impact [16], [17]. Unfortunately, the decomposition and burning of organic wastes can produce greenhouse gases like methane as well as air pollutants including carbon monoxide (CO), particulate matter (PM) and polycyclic aromatic hydrocarbons (PAHs) which do not benefit the environment or the economy. Addressing this environmental challenge, ongoing research endeavors aim to transform these generated wastes into value-added products, seeking to mitigate their adverse environmental consequences [9].

Teo et al., explored the utilization of pineapple leaf agro-waste into eco-friendly paper as an alternative to the conventional wood fibres and to reduce waste [18]. The abundance of pineapple leaves, with a yield of up to 1.5 kg per plant, makes pineapple leaf (PAL) an attractive option for pulp production [13]. Moreover, PALF, when used as a viable feedstock for producing sustainable materials, including kraft paper or packaging material, exhibits favorable mechanical properties due to its high cellulose content above (66.2%) and low lignin content (4.2%), providing resistance against potential abrasion and tear [14]. PALF's potential as a non-wood alternative for pulp production holds significance in addressing the growing global demand for paper and the need for sustainable alternatives to conventional packaging materials [19]. By using PALF as a raw material, the production of kraft paper can be both environmentally friendly and resource-efficient, contributing to a circular economy. Researchers have explored PALF as a raw material for pulping under various conditions, demonstrating its potential as a substitute for pulp production [14], [15].

Despite these promising findings, there remains a substantial knowledge gap regarding the optimization

of processing conditions to maximize the potential of PALF in sustainable packaging applications [20]. Comprehensive research is therefore necessary to fill this void, involving a nuanced exploration of aspects such as comparing the yield and quality of PALF pulp produced through various pulping techniques and assessing the impact of different additives on the papermaking process of PALF pulp. This study focuses on investigating the combined effects of Gum Arabic, beating time and processing conditions on the mechanical properties of a PALF-based kraft paper. By optimizing these processing conditions, a superior quality kraft paper could be developed to meet the increasing demand for eco-friendly packaging solutions in industries such as food, consumer goods, and logistics [21].

PALF's unique fibre structure facilitates strong hydrogen bonding during paper formation, resulting in enhanced mechanical strength [16], [22]. PALF is composed of approximately 70-80% cellulose, making it an ideal candidate for paper and packaging applications [16], [23]. PALF boasts high tensile strength, low density, good thermal stability, and a low water absorption capacity, making it resilient to moisture and enhancing its applicability in packaging [16], [20], [24]. Additionally, the mechanical properties of PALF can be further optimized through treatments such as alkali treatment and bleaching [8], [21]. PALF's versatility extends to its role as a reinforcing agent or filler in paper composites, showcasing its potential to enhance overall material performance [11].

PALF extraction methods are crucial determinants in achieving optimal dry pulp yield and understanding morphological properties [11]. Mechanical or chemical techniques can be used to extract PALF from pineapple leaves [16], which can then be pulped using a variety of techniques such as soda and organosolv [25]. The mechanical-chemical extraction method, employing a 3% NaOH solution concentration, a solid/liquid ratio of 1:15, and 90 minutes of cooking time, has proven to be the most efficient, providing a dry pulp yield exceeding 6% (wt.) [11]. Several methods are employed to extract PALF from pineapple leaves, including mechanical, chemical, semi-mechanical methods and enzymatic extraction methods [17]. The extraction and treatment processes involve collecting and drying pineapple leaves, followed by alkali treatment and bleaching [20], [26], [27].

The treatment of PALF has been a focal point in research efforts to address its inherent challenges and

enhance its suitability for pulp and paper making. Alkali treatment, a common approach, has proven effective in removing lignin and hemicellulose, thereby increasing crystallinity and crystallite size, and ultimately enhancing PALF's mechanical properties [8]. Additionally, bleaching is employed to further reduce the lignin content and increase the brightness of PALF [21]. Notably, soda-anthraquinone (AQ) pulping, followed by elemental chlorine-free (ECF) or totally chlorine-free (TCF) bleaching, has been identified as a method to produce high-quality PALF pulp with favorable yield, strength, and brightness [21]. Chemical modification stands out as a crucial aspect of PALF treatment, with methods such as alkali treatment, acetylation, and grafting with polymer additives proving instrumental in improving fibre compatibility with the kraft pulping process and enhancing mechanical properties [20].

Previous research discussed the potential of pineapple leave fibre for paper production and emphasized the need to optimize processing conditions to achieve high quality pulp [21], [28]. The incorporation of additives such as Gum Arabic and adjustments in beating time during pulp production have been shown to significantly enhance the mechanical properties of kraft paper produced from PALF, ensuring cohesion and strength [16], [25]. Gum Arabic, a natural polymer, improves fibre bonding, thus enhancing the strength and durability of paper, while beating time increases fibre fibrillation, promoting better bonding [17], [24]. In a study by Nnodu et al., high-density polypropylene was blended with PALF, incorporating maleic anhydride-graft-polypropylene (MA-g-PP) as a compatibilizer [20]. The outcome is superior stiffness and high strength in PALF-based polymer composites compared to conventional cellulose-based materials but a balance between the flexibility and tensile strength of the paper was not achieved. There is limited research on the combined effects of processing parameters on the dependent variables such as tensile strength and flexibility. To address this gap, it is crucial to investigate the influence of varying processing conditions on the mechanical properties of a PALF-based kraft paper. By a methodological and experimental approach in varying these parameters and analyzing their corresponding impact the optimal processing conditions for achieving high quality and kraft paper with desirable mechanical properties can be identified.

2. Methods

2.1. Material Preparation

Mature pineapple leaves from the same variety were sourced from agrarian waste in Kanyoni farms, a

community in the Kiambu County of Kenya. The leaves were washed and cut into small pieces, targeting a size of 1 - 2 cm in length [29]. The freshly chopped leaves were then oven-dried at a temperature of 115°C for 23 hours to ensure effective moisture removal. The dried leaves were subsequently dry milled using a hammer mill or a pulverizer with a 3mm sieve at 1500 RPM for 10 minutes. The pulverized leaves were subjected to alkaline treatment using NaOH solutions of varying concentrations.

2.2. Alkaline Treatment and Pulping

The concentration of NaOH is crucial for effective delignification. Higher NaOH concentrations can lead to excessive degradation of cellulose which reduces the mechanical strength of the fibres. Gaba et al. 2021 studied the effects of NaOH concentration on PALF using 1% - 9% alkali charge [8]. Likewise earlier research studied NaOH concentrations on PALF at 2%, 4% and 6%, with the fibre treated at 6% NaOH showing absence of impurities on the surface [30], [31], [32]. Cooking temperature increases the rate of chemical reaction for delignification, but higher temperatures can lead to the degradation of cellulose. Wutisatwongkul et al., studied the effects of varying cooking temperatures on PALF at 90°C to 130°C [15]. Again, the initial stage of non-wood fibres occurs at temperatures less than 140°C, with the main stage of lignin removal occurring between 140°C to 170°C [5], which was investigated by Daud et al., at 170°C and found that it resulted in higher pulp yield [12]. Cooking times enhance breakdown of lignin and hemicellulose but excessive cooking degrades the fibre. Cheirmakani et al., treated PALF with 5% NaOH for 2 hours [33], while Munthoub et al., also investigated the effects of different cooking times between 30 to 180 minutes and found that the 90 minutes treatment gave the best fibre quality [11]. In this study, the pulp was obtained through a semi-mechanical alkaline treatment with NaOH solution in a laboratory bioreactor. The leaves were mixed with water at a substrate to liquor ratio of 1:10 with varying NaOH concentrations (C) of (2%, 4% and 6%). Each batch was treated at different cooking temperatures (CT) of (130°C, 140°C and 150°C) and for different cooking times (Ct) of (60, 90 and 120) minutes. After treatment, the fibres were washed thoroughly and subjected to a 1% acetic acid solution for 20 – 30 minutes to neutralize the residual base. The fibres were then rinsed thoroughly under clean running water to remove any residual chemicals and air-dried until the pulp is completely dried.

2.3. Kraft Paper Production

PALF has a high cellulose content of (70-82%) [34], and a low microfibrillar angle, which improves the tensile

strength of matrix reinforced biocomposites. Higher fibre content in composites leads to reduction in tensile strength due to lower polymer content in it. Low polymer composites possess lower surface coverage, leading to inappropriate strength at the bonding interface [33], [35]. Treated fibres provide better tensile strength, due to the removal of hemicellulose on the surface and the improvement of fibre and matrix interface bonding [33]. NaOH treatment removes lignin and hemicellulose, exposing hydroxyl groups for stronger fibre bonding [34], [36]. Gum Arabic acts as a natural binder to enhance fibre bonding, tear resistance and flexibility, forming a more durable paper structure. Siregar et al. studied the physical properties of short pineapple leaf fibre (SPALF) reinforced high impact polystyrene (HIPS) composites and used similar levels of PALF concentration adopted in this study at 10 – 50% to improve the properties of HIPS composites [32]. The combination of PALF, NaOH treatment, and Gum Arabic further enhances paper strength and durability for packaging applications. Beating time refines the fibres by strengthening fibre to fibre bonding which increases surface area and flexibility. Studies have shown that increasing beating time resulted in greater fibre fibrillation and largely improved paper strength and flexibility [37]. In this study, the pulp obtained from the semi-mechanical alkaline treatment with NaOH solution was mixed with Gum Arabic as a binder in the kraft paper production. Gum Arabic with concentrations of 30% and 50% by weight of pulp were dissolved in water for 30 to 45 minutes, using a water bath heated to a temperature of 95°C, which ensured a faster rate of dissolution. Gum Arabic is a binding agent that improves the fibre bonding, paper flexibility and surface smoothness [38], [39]. The dissolved Gum Arabic was then added to the pulp and subjected to different beating times, Bt of (4, 8 and 12 minutes) using a laboratory disintegrator or blender with a rotation speed of 2,840 RPM. The beaten pulp was formed into paper sheets using a hand paper-making mold. The synergy between the processing combinations resulted in a paper that is well bonded, strong and flexible, making it a suitable alternative to conventional packaging materials. The sheets were air-dried, removed from the mold and pressed with a slip rolling machine at a temperature of 80°C and a pressure of 80 psi for 3 seconds. The paper samples were prepared and conditioned at an atmosphere of 23°C and 50% relative humidity before carrying out a tensile strength test in accordance with (ISO 187:1990 and ISO 1924-2) standards.

2.4. Characterization of Pulp and Kraft Paper

Pulp Yield: The pulp yield was calculated as a percentage of the initial mass of raw pineapple leaves, using the formula:

$$\text{Pulp Yield (\%)} = \frac{\text{Weight of Dried Pulp}}{\text{Initial Weight of Raw Leaves}} \times 100 \quad (1)$$

FTIR Analysis: Fourier Transform Infrared Spectroscopy (FTIR) was performed on the untreated and treated fibres to identify the chemical changes, particularly the removal of lignin and the preservation of cellulose for the different concentrations of NaOH [40], [41].

Tensile Strength and flexibility: Tensile strength test were conducted using a universal testing machine in accordance with the (ISO 187:1990 and ISO 1924-2) standards. The maximum strain calculated over the entire area, expressed as a percentage during the tensile strength test is synonymous to the tensile strain at break, which represents the total elongation of the paper just before it breaks. The tensile strain at break was used to infer the flexibility of the kraft paper. Higher tensile strain at break means that the kraft paper can stretch more without tearing which denotes better flexibility.

2.5. Optimization of the Processing Conditions for PALF-based Kraft Paper

Grey Relational Analysis (GRA) was conducted on the response values with the aim of compressing the multi-response variables into a single response. The first stage of GRA involved experimental design using Taguchi analysis technique in Minitab 19 statistical software, followed by signal-to-noise (S/N) ratio calculation of responses using larger-the better for Tensile Strength and Flexibility quality characteristics shown in Eq. (2). The results of the GRA are shown in Tables 3 – 6.

Larger-the better Characteristics:

$$S/N = -10 \log \frac{1}{n} \left(\sum_{i=1}^n \frac{1}{x^2} \right) \quad (2)$$

Where, x = Responses of given process-parameter level combination and n = number of experimental samples.

S/N ratio calculation was followed by calculation of Grey Relational Generation (GRG) using Eq. (3), (larger-the-better attributes). GRG was conducted to

normalize the S/N ratio values in the range between 0 and 1 [42], [43].

$$\text{Larger-the-better attributes: GRG} = \frac{X_{ij} - \bar{X}_i}{\bar{X}_i - X_j} \quad (3)$$

Where, x_{ij} is the individual response value and $\bar{x}_i = \max\{x_{ij}, i = 1, 2, \dots, m\}$ and $\bar{x}_j = \min\{x_{ij}, i = 1, 2, \dots, m\}$.

The GRG procedure is followed by the calculation of grey relational coefficient (GRC) using Eq. (4)

$$\text{GRC} = \frac{\Delta_{\min} + \lambda \Delta_{\max}}{\Delta_{ij} + \lambda \Delta_{\max}}$$

$$(i = 1, 2, \dots, m \text{ and } j = 1, 2, \dots, n) \quad (4)$$

Where $\Delta_{ij} = x_{0j} - x_{ij}$ while $\Delta_{\min} = \min(0)$ and $\Delta_{\max} = \max$ (1)

λ is the distinguishing coefficient, $\beta \in [0, 1]$.

Distinguishing coefficient (λ) is used to expand or compress the range of the GRC and 0.5 is the accepted value [44].

The final stage of GRA is the calculation of grey relational grades which was obtained using Eq. (5)

$$\text{Grade} = \frac{\text{Individual GRC}}{\text{Number of responses}} \quad (5)$$

The GRA was used to determine the optimal processing conditions for the PALF-Based Kraft Paper.

3. Results and Discussion

The result of this study shows the synergistic effects of Gum Arabic and beating time on the mechanical properties of kraft paper produced from pineapple leaf fibre. Through systematic experimentation, it was found that the multi-response optimal process parameter combinations; CT at 130°C, C at 4% and 50 % GAC with a Ct of 90 minutes and a Bt of 12 minutes provided the best balance between pulp yield, tensile strength, and flexibility. These conditions produced kraft paper with superior mechanical properties. Gum Arabic, known for its film-forming and adhesive properties, significantly improved fibre bonding and flexibility of the paper [38], [45] but excessive use of it reduces the tensile strength. The higher beating time ensured adequate fibrillation, enhancing the surface area of the fibres, promoting better fibre-to-

fibre bonding and improving paper strength and flexibility [46]. Fig. 1 shows samples of the resulting Gum Arabic integrated PALF-based kraft paper.



Fig. 1 Samples of the PALF-based kraft paper

3.1. Pulp Yield of PALF

Table 1 Pulp Yield of PALF under varying conditions

Run	CT (°C)	Ct (min)	C (%)	PY (%)
1	130	120	6	14.25
2	130	90	6	17.74
3	140	60	6	22.87
4	140	90	6	17.71
5	140	120	2	25.14
6	140	120	4	14.12
7	140	120	6	14.02
8	150	60	6	34.78
9	150	120	6	14.76

Table 1 presents pulp yield of PALF, influenced by the varying concentration of NaOH, cooking time and temperature. During the pulping process, NaOH breaks ether and glucosidic bonds separating chains of lignin carbohydrates to remove lignin, hydrolyzes hemicellulose, and causes cellulose fibres to swell, which improves fibre bonding [36]. Overexposure of fibres to high cooking temperatures and alkali concentrations will however lead to depolymerization of cellulose which further reduce pulp yield and fibre strength [11]. In Table 1, experimental runs 1, 6, 7 and 9 with higher NaOH charge, and longer cooking time produced very low pulp yields suggesting cellulose degradation which was also reported by Munthoub et al. [11]. Experimental run 8 however produced a higher pulp yield, which may suggest a possible incomplete removal of lignin [47].

Empirical Regression Equation for Pulp yield of PALF

$$-PY(\%)^{-0.5} = -0.518 + 0.00315 CT (^{\circ}C) - 0.000906 Ct (mins) - 0.01326 C (\%) \quad (6)$$

The empirical regression equation for the pulp yield of PALF shown in Eq. (6), was obtained using an interactive analysis with Minitab 19 on the experimental data of pulp yield. The analysis indicates that increasing the cooking temperature increases pulp yield while increasing the cooking time and alkali charge (NaOH concentration), decreases the yield of pulp yield. Process parameter combinations of CT (150°C), Ct (60mins) and C (6%) achieved a high pulp yield. While it is economical to maximize pulp yield, higher yields suggest inadequate removal of lignin which makes paper brittle and affects the paper value [47]. Lower yields suggest over exposure of cellulose and harsh alkali treatment which may damage cellulose structure and interfere with the quality of the resulting kraft paper. There should be a balance between the lower and higher yield for quality pulp and better paper properties, wherein yields must be kept as moderate as possible for better fibre quality. Hence the optimal processing conditions for improved pulp quality as shown in the main effects plots in Fig. 6 after Grey Relational Analysis are cooking temperature at (130°C), cooking time of (90mins) and an alkaline charge or NaOH concentration of (4%).

3.2. FTIR Analysis on PALF

A typical pineapple leaf fibre comprises 68.5 % cellulose, 18.8 % hemicellulose, 6.04% lignin, 3.2% fat and wax, 1.1% pectin and an ash content of 0.9% [16]. The observations made from the FTIR spectra of the untreated and alkaline treated PALF suggest that alkaline treatment with NaOH progressively removes hemicellulose and lignin while retaining and exposing cellulose, as presented in Fig. 2. In the untreated PALF, a peak at 1102.87 cm⁻¹ corresponds to the C—C stretching vibrations found in both cellulose and hemicellulose [46]. After 2% NaOH treatment, this peak becomes sharper, indicating partial removal of hemicellulose and increased exposure of cellulose. In the 4% and 6% NaOH treated sample, the peak is even more pronounced at 1057.68 cm⁻¹ and 1027.27 cm⁻¹ respectively, which suggests further removal of hemicellulose and greater retention and exposure of cellulose. For O—H stretching, untreated PALF has a broad peak at (3436.97 cm⁻¹), which sharpens at (3412.82 cm⁻¹) after 2% NaOH treatment, reflecting greater cellulose exposure, [46], [48], [49] and becomes even sharper with 4% NaOH treatment at (3325.86 cm⁻¹). These peaks demonstrate that treating PALF at higher NaOH concentrations more effectively remove hemicellulose and lignin while retaining more cellulose but may also damage the cellulose structure if the concentration of NaOH becomes too high. Fig.2 shows FTIR spectra for untreated and treated PALF with NaOH at varying concentrations.

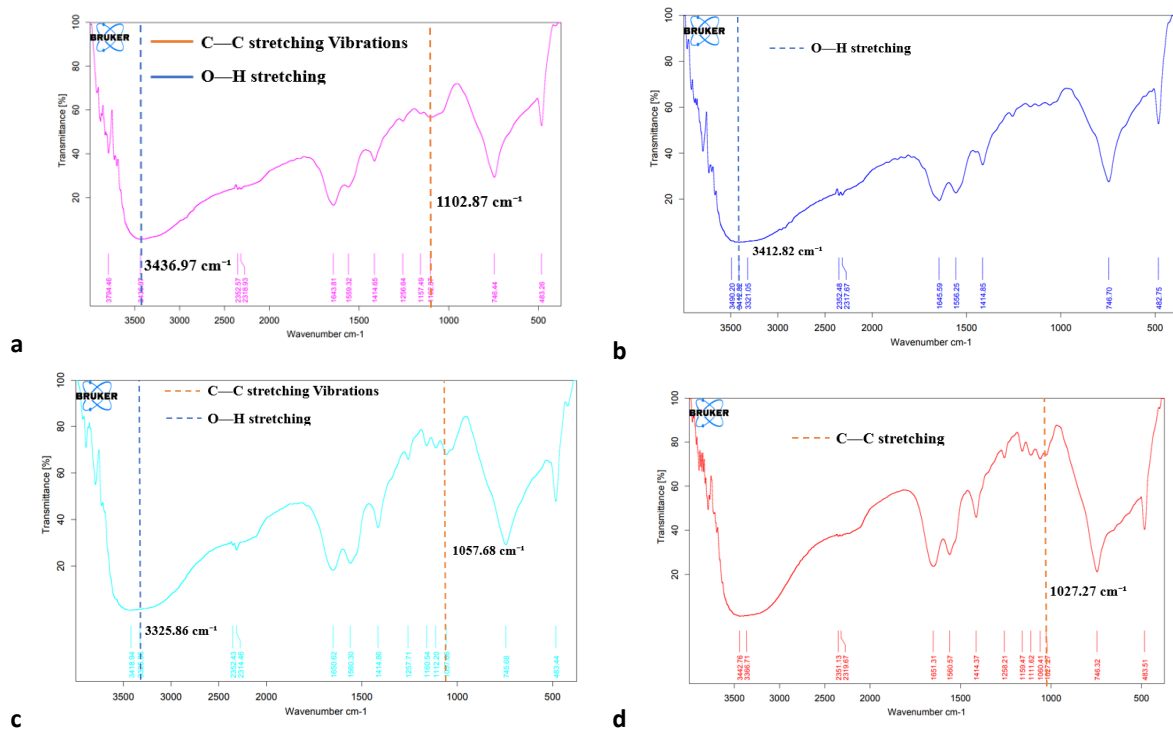


Fig. 2 FTIR Spectra of (a) Untreated PALF and treated PALFs at (b) 2% NaOH, (c) 4% NaOH and (d) 6% NaOH

3.3 Tensile Strength and Flexibility Analysis of PALF-Based Kraft Paper

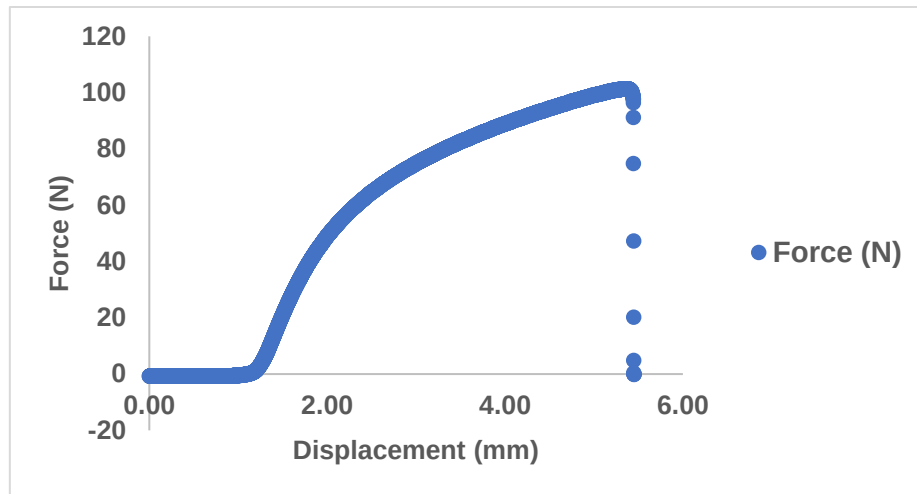


Fig. 3 Tensile Strength Test Curve of a PALF-based kraft paper

Fig. 3 shows a force-displacement graph of PALF-based Kraft Paper, where the force increases with displacement until the ultimate tensile strength is

reached, followed by a sharp drop, indicating a break.

Table 2 Tensile Strength and Flexibility of PALF-based Kraft Paper

Run	CT (°C)	Ct (mins)	C (%)	Bt (mins)	GAC (%)	TS (N/mm ²)	F (%)
1	130	120	6	12	0	20.0530	3.11930
2	130	120	6	12	50	25.0359	3.25595
3	130	90	6	12	0	16.5735	3.42010
4	130	90	6	12	50	15.9310	4.89178
5	140	60	6	8	0	17.2959	2.14678
6	140	60	6	8	50	15.9072	2.45594
7	140	60	6	12	0	15.1071	1.87637
8	140	60	6	8	30	11.2274	1.97014
9	140	60	6	12	50	17.4356	2.92680
10	140	90	6	12	0	11.7345	2.31760
11	140	90	6	12	30	13.5863	2.00012
12	140	90	6	12	50	14.7633	0.89097
13	140	120	2	12	0	22.5653	2.66805
14	140	120	2	12	30	14.8998	2.70929
15	140	120	2	12	50	12.9691	1.40012
16	140	120	4	12	0	25.7879	2.77552
17	140	120	4	12	30	26.6333	3.01930
18	140	120	4	12	50	23.1770	3.01470
19	140	120	6	12	0	13.9258	2.67427
20	140	120	6	12	30	18.4647	1.68677
21	140	120	6	12	50	17.0524	2.69803
22	150	60	6	12	0	15.0239	2.10805
23	150	60	6	12	30	12.9574	2.08345
24	150	60	6	4	50	9.91490	1.83972
25	150	120	6	12	0	12.2256	3.93555
26	150	120	6	12	30	8.32560	3.41802

27 150 120 6 12 50 9.87054 4.67388

Table 2 presents the tensile strength and flexibility of PALF-based kraft paper produced under varying processing conditions. The results show that PALF-based kraft paper far outperformed papers produced from non-wood fibres studied by Adutwum et al. 2024 [50]. The tensile strength of the papers produced from Corn Husk, Pineapple Crown and Plantain Stalk, are (25.675, 21.05 and 23.625) kN/m² respectively [50]. The high strength of the PALF-based kraft paper in this study was influenced by improved processing conditions and the integration of Gum Arabic which enhanced fibre bonding. Again, in a study by Cheirmakani et al., the tensile strength of PALF composites reported ranged from 13.64MPa to 25.10MPa [33], which corresponds with the tensile strength results of the PALF-based kraft paper, presented in Table 2.

Empirical Regression Equation for Tensile Strength

$$-TS \text{ (N/mm}^2\text{)}^{-0.5} = 0.264 - 0.00354 \text{ CT (}^\circ\text{C)} - 0.000077 \text{ Ct (mins)} -$$

$$0.00789 \text{ C (\%)} + 0.00272 \text{ Bt (mins)} - 0.000146 \text{ GAC (\%)} \quad (7)$$

The empirical regression equation for the tensile strength of PALF-based kraft shown in Eq. (7) was obtained using an interactive analysis with Minitab 19 on the experimental data presented in Table 2. The analysis indicates that NaOH concentration has the highest influence on the tensile strength of PALF-based kraft paper, followed by cooking temperature, beating time, cooking time and Gum Arabic concentration respectively. Decreasing the cooking temperature and Gum Arabic concentration with a moderate NaOH concentration while increasing cooking and beating times significantly maximized the tensile strength of the kraft paper. This observation is consistent with earlier findings that treated PALF under similar conditions and improved the tensile strength of PALF by 18–53% [8]. An improvement in strength with increasing beating time confirms that longer beating time increases fibre fibrillation, promoting better bonding and strength [17], [24].

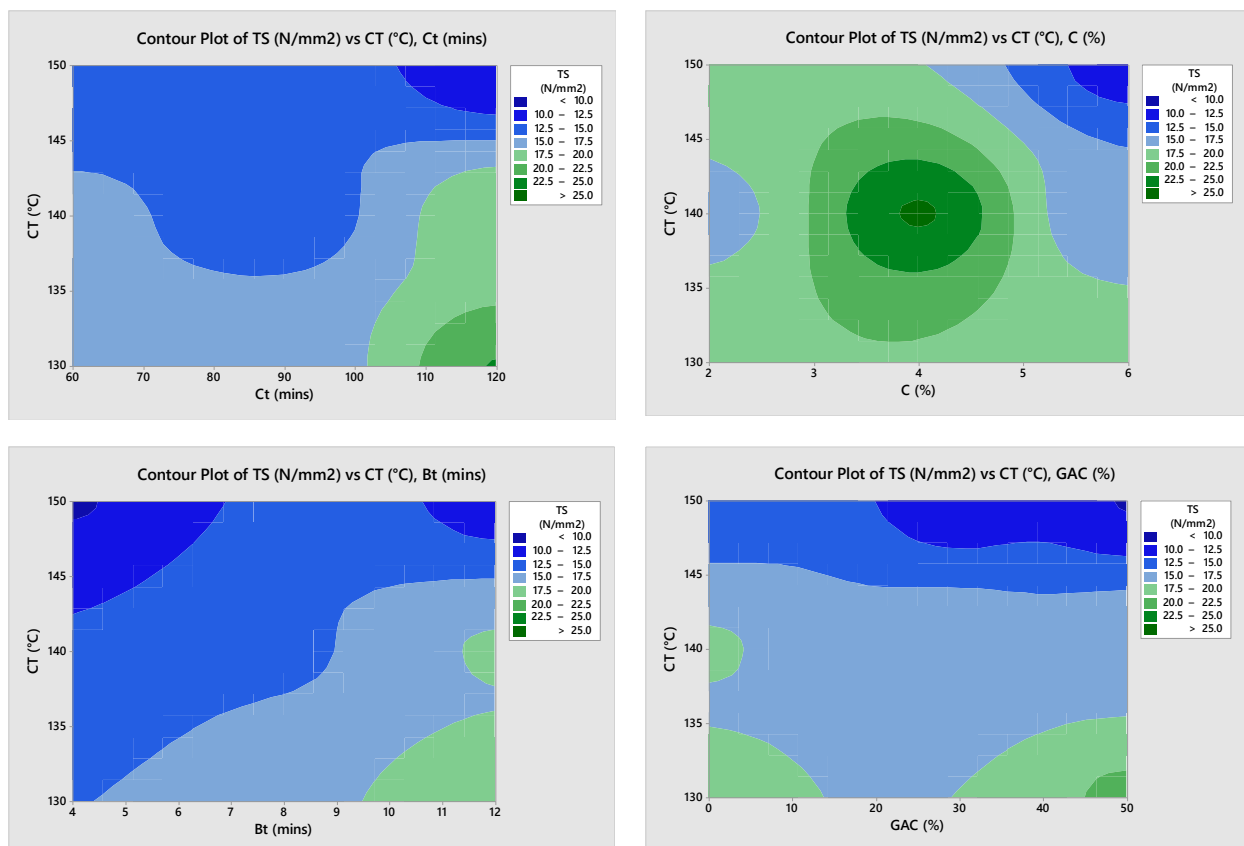


Fig. 4 Contour Plots showing tensile strength of PALF paper at different factor levels

The contour plots for tensile strength of PALF-based paper at varying process parameters as shown in Fig. 4 indicates that, higher beating times and higher Gum Arabic Concentrations across lower temperatures, achieved higher tensile strength values between (20 to 25) N/mm². This suggests that higher beating time combined with increased Gum Arabic Concentrations enhances fibre bonding, resulting in stronger, more cohesive pulp.

3.3.2. Flexibility Analysis

Empirical Regression Equation for Flexibility

$$F(\%)^{0.5} = 1.15 - 0.00294 \text{ CT } (^{\circ}\text{C}) + 0.00471 \text{ Ct (mins)} + 0.0617 \text{ C } (\%) + 0.0077 \text{ Bt (mins)} - 0.00009 \text{ GAC } (\%) \quad (8)$$

Equation (8) presents empirical regression equation from an interactive analysis for flexibility. The results reveal that cooking temperature has a higher influence on the flexibility of PALF-based kraft paper, followed by beating time, NaOH concentration, and cooking time with Gum Arabic concentration having the lowest influence. When the cooking temperature decreased, moderate NaOH concentration was maintained and the Gum Arabic concentration, cooking and beating times were increased, a significant improvement in the flexibility of a PALF-based kraft paper was recorded. Thus, Gum Arabic improves paper flexibility but will reduce the tensile strength at high concentrations. Moderate concentrations of Gum Arabic are therefore desired to strike a balance between the tensile strength and flexibility of PALF-based kraft paper.

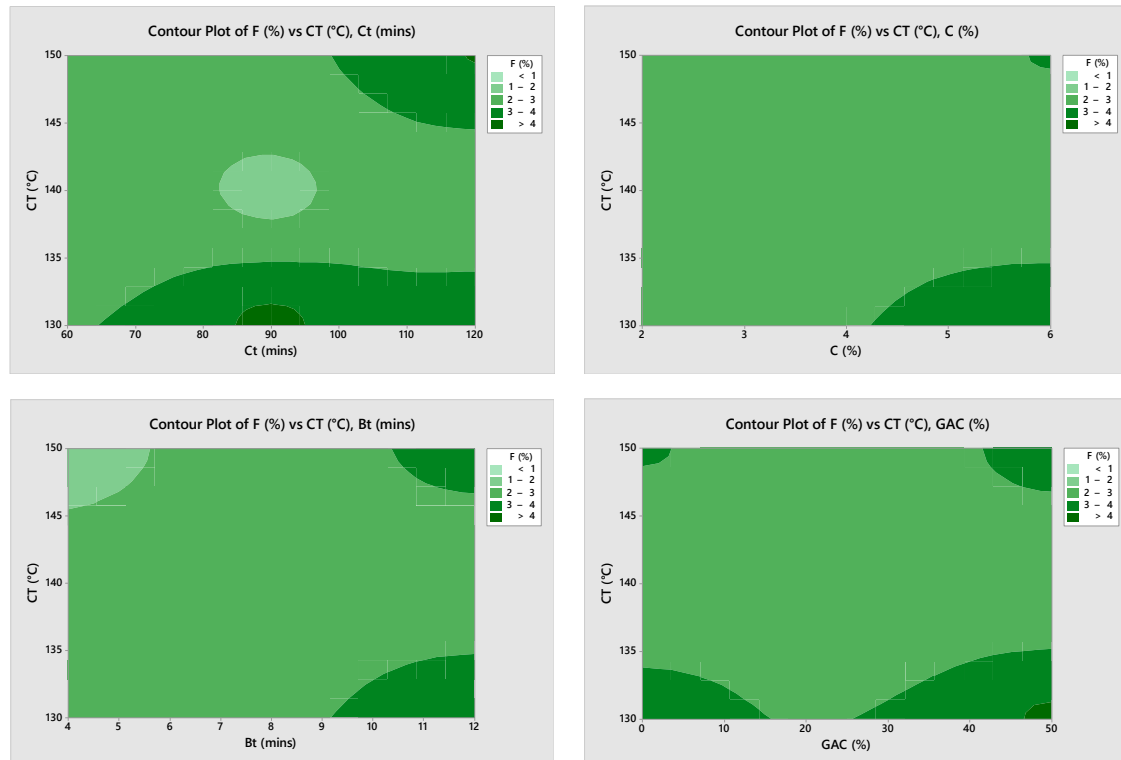


Fig. 5 Contour Plots showing flexibility of PALF- paper at different parameter levels

The contour plot for flexibility of PALF-based kraft paper presented in Fig. 5 shows a uniform distribution with flexibility consistently remaining above 4% across the tested temperature range at higher beating time (Bt) and higher Gum Arabic Concentrations (GAC). This further confirms that variations in beating time and Gum Arabic Concentrations at higher levels have significant influence on flexibility.

3.4. Grey Relational Analysis of Experimental Results

The results of experimental response values along with their corresponding S/N ratio values are shown in Table 3

Table 3 Experimental Responses and S/N values

Run	Experimental Matrix				Experimental Responses			Signal to noise ratios (dB)	
	CT (°C)	Ct (mins)	C (%)	Bt (mins)	GAC (%)	TS (N/mm ²)	F (%)	S/N for TS (dB)	S/N for F (dB)
1	130	120	6	12	0	20.053	3.1193	26.04359	9.88114
2	130	120	6	12	50	25.0359	3.25595	27.97126	10.25355
3	130	90	6	12	0	16.5735	3.4201	24.38828	10.68078
4	130	90	6	12	50	15.931	4.89178	24.04486	13.78934
5	140	60	6	8	0	17.2959	2.14678	24.75886	6.63575
6	140	60	6	8	50	15.9072	2.45594	24.03187	7.80436
7	140	60	6	12	0	15.1071	1.87637	23.58362	5.46637
8	140	60	6	8	30	11.2274	1.97014	21.00558	5.88994
9	140	60	6	12	50	17.4356	2.9268	24.82874	9.32786
10	140	90	6	12	0	11.7345	2.3176	21.38929	7.30077
11	140	90	6	12	30	13.5863	2.00012	22.66202	6.02112
12	140	90	6	12	50	14.7633	0.89097	23.38367	-1.00274
13	140	120	2	12	0	22.5653	2.66805	27.06882	8.52388
14	140	120	2	12	30	14.8998	2.70929	23.46361	8.65711
15	140	120	2	12	50	12.9691	1.40012	22.25820	2.92331
16	140	120	4	12	0	25.7879	2.77552	28.22832	8.86689
17	140	120	4	12	30	26.6333	3.0193	28.50850	9.59813
18	140	120	4	12	50	23.177	3.0147	27.30114	9.58488
19	140	120	6	12	0	13.9258	2.67427	22.87640	8.54411
20	140	120	6	12	30	18.4647	1.68677	25.32685	4.54112
21	140	120	6	12	50	17.0524	2.69803	24.63571	8.62094
22	150	60	6	12	0	15.0239	2.10805	23.53565	6.47762
23	150	60	6	12	30	12.9574	2.08345	22.25036	6.37566
24	150	60	6	4	50	9.9149	1.83972	19.92577	5.29503
25	150	120	6	12	0	12.2256	3.93555	21.74540	11.90011
26	150	120	6	12	30	8.3256	3.41802	18.40831	10.67549
27	150	120	6	12	50	9.87054	4.67388	19.88682	13.39355

Also, the results of GRG, GRC and Grades are shown in Table 4.

Table 4 Results of GRG, GRC and Grade

Xo (Ref. Point)	Signal to Noise Ratios (dB)		Grey relational Generation (GRG)		Grey relational Coefficient (GRC)		Grey Relational Grade
	S/N for TS (dB)	S/N for F (dB)	GRG for TS	GRG for F	GRC for TS	GRC for F	
1	26.04359	9.88114	0.7560	0.7358	0.672	0.654	0.663
2	27.97126	10.25355	0.9468	0.7610	0.904	0.677	0.790
3	24.38828	10.68078	0.5921	0.7898	0.551	0.704	0.627
4	24.04486	13.78934	0.5581	1.0000	0.531	1.000	0.765
5	24.75886	6.63575	0.6288	0.5164	0.574	0.508	0.541
6	24.03187	7.80436	0.5568	0.5954	0.530	0.553	0.541
7	23.58362	5.46637	0.5124	0.4373	0.506	0.471	0.488
8	21.00558	5.88994	0.2572	0.4660	0.402	0.484	0.443
9	24.82874	9.32786	0.6357	0.6984	0.578	0.624	0.601

10	21.38929	7.30077	0.2951	0.5613	0.415	0.533	0.474
11	22.66202	6.02112	0.4212	0.4748	0.463	0.488	0.476
12	23.38367	-1.00274	0.4926	0.0000	0.496	0.333	0.415
13	27.06882	8.52388	0.8575	0.6440	0.778	0.584	0.681
14	23.46361	8.65711	0.5005	0.6530	0.500	0.590	0.545
15	22.25820	2.92331	0.3812	0.2654	0.447	0.405	0.426
16	28.22832	8.86689	0.9723	0.6672	0.947	0.600	0.774
17	28.50850	9.59813	1.0000	0.7167	1.000	0.638	0.819
18	27.30114	9.58488	0.8805	0.7158	0.807	0.638	0.722
19	22.87640	8.54411	0.4424	0.6454	0.473	0.585	0.529
20	25.32685	4.54112	0.6850	0.3748	0.613	0.444	0.529
21	24.63571	8.62094	0.6166	0.6506	0.566	0.589	0.577
22	23.53565	6.47762	0.5076	0.5057	0.504	0.503	0.503
23	22.25036	6.37566	0.3804	0.4988	0.447	0.499	0.473
24	19.92577	5.29503	0.1502	0.4258	0.370	0.465	0.418
25	21.74540	11.90011	0.3304	0.8723	0.427	0.797	0.612
26	18.40831	10.67549	0.0000	0.7895	0.333	0.704	0.519
27	19.88682	13.39355	0.1464	0.9732	0.369	0.949	0.659

The summary of GRA-Grade results along with their corresponding process-parameter levels are shown in Table 5.

Table 5 Summary of GRA-Grade values and Process-parameter Levels

Run	Experimental Design				GRA-Grade value	
	CT (°C)	Ct (mins)	C (%)	Bt (mins)	GAC (%)	
1	130	120	6	12	0	0.663
2	130	120	6	12	50	0.790
3	130	90	6	12	0	0.627
4	130	90	6	12	50	0.765
5	140	60	6	8	0	0.541
6	140	60	6	8	50	0.541
7	140	60	6	12	0	0.488
8	140	60	6	8	30	0.443
9	140	60	6	12	50	0.601
10	140	90	6	12	0	0.474
11	140	90	6	12	30	0.476
12	140	90	6	12	50	0.415
13	140	120	2	12	0	0.681
14	140	120	2	12	30	0.545
15	140	120	2	12	50	0.426
16	140	120	4	12	0	0.774
17	140	120	4	12	30	0.819
18	140	120	4	12	50	0.722
19	140	120	6	12	0	0.529
20	140	120	6	12	30	0.529
21	140	120	6	12	50	0.577
22	150	60	6	12	0	0.503
23	150	60	6	12	30	0.473

24	150	60	6	4	50	0.418
25	150	120	6	12	0	0.612
26	150	120	6	12	30	0.519
27	150	120	6	12	50	0.659

3.4.1 Factor Levels of Main Effects

The factor level effects for responses shown in Table 6 were obtained using the Grade values from GRA as presented in Table 5.

Table 6 Resulting Experimental Process-parameter and Level Effects

Process-Parameter	Leve	CT (°C)	Ct (mins)	C (%)	Bt (mins)	GAC (%)
Level 1		0.7115	0.5012	0.5508	0.4179	0.5893
Level 2		0.5637	0.5514	0.7718	0.5085	0.5433
Level 3		0.3423	0.4343	0.5639	0.5943	0.5916

3.4.2 Main Effect Plots for GRA

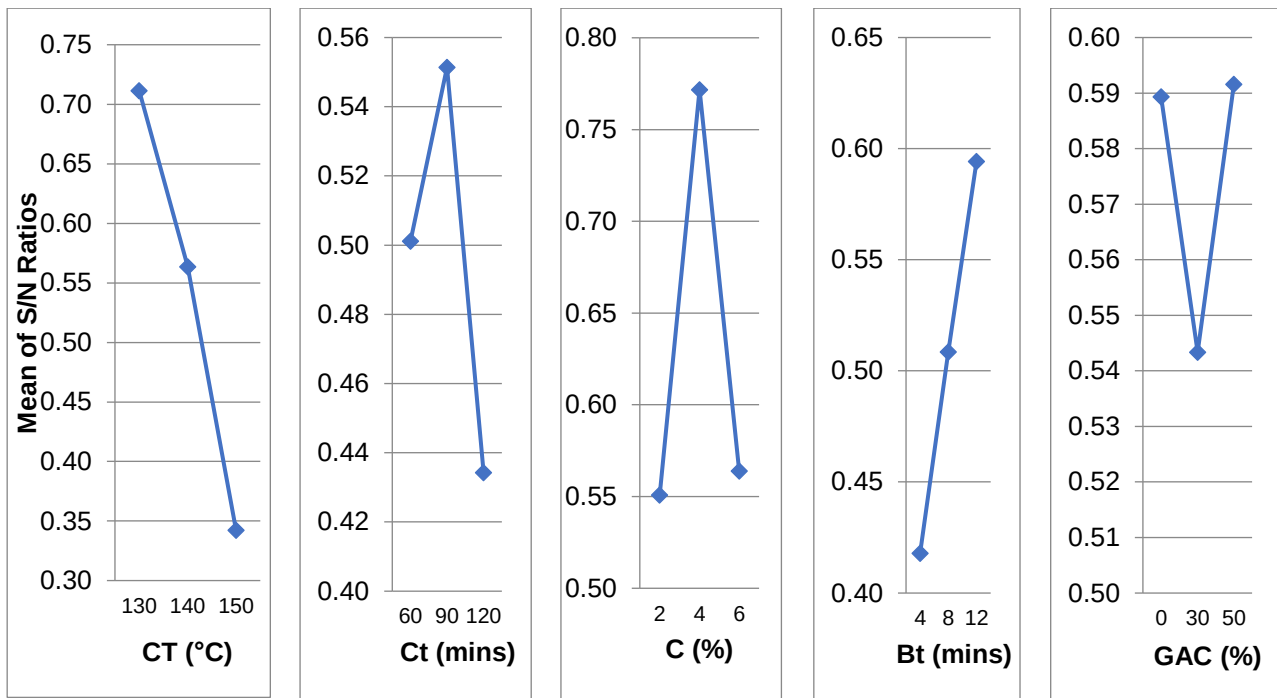


Fig. 6 Main Effect Plot for GRA

The main effect plots for GRA shown Fig. 6 specified the optimal process-parameter levels of the experimental process. These plots were obtained using the factor levels of Main effects shown in Table 6.

$$-TS (N/mm^2)^{-0.5} = 0.264 - 0.00354 CT (°C) - 0.000077 Ct (mins) - 0.00789 C (%) + 0.00272 Bt (mins) - 0.000146 GAC (%) \quad (7)$$

When CT = (130°C), Ct = (90mins), C = (4%), Bt = (12mins) and GAC (50%)

$$-TS (N/mm^2)^{-0.5} = 0.264 - 0.00354(130) - 0.000077(90) - 0.00789(4) + 0.00272(12) - 0.000146 (50)$$

$$TS = 22.82 N/mm^2$$

$$F (%)^{0.5} = 1.15 - 0.00294 CT (°C) + 0.00471 Ct (mins) + 0.0617 C (%) + 0.0077 Bt (mins) - 0.00009 GAC (%) \quad (8)$$

When $CT = (130^{\circ}\text{C})$, $Ct = (90\text{mins})$, $C = (4\%)$, $Bt = (12\text{mins})$ and $GAC = (50\%)$

$$F(\%)^{0.5} = 1.15 - 0.00294(130) + 0.00471(90) + 0.0617(4) + 0.0077(12) - 0.00009(50)$$

$$F = 2.33\%$$

As shown in Fig. 6, it can be observed that the multi-response optimal process parameter combination for the experimental process is Cooking Temperature (130°C), Cooking Time (90mins), C (4%), Beating Time (12mins) and Gum Arabic Concentration at (50%). These optimal conditions substituted into the optimization equations shown in Eq. (7) and Eq. (8), gave optimal response values of 22.82 N/mm^2 and 2.33% for tensile strength and flexibility respectively. This result matches with the tensile strength values of PALF composites reported in literature by Cheirmakani et al., which ranged from 13.64MPa to 25.10MPa [33].

4. Conclusion

The study demonstrates that both the concentration of Gum Arabic and beating time at higher levels significantly impact the mechanical properties of PALF-based kraft paper. Through systematic experimentation and multi-response optimization analysis, it was found that the optimal processing conditions for a PALF-based kraft paper are maintaining a cooking temperature of 130°C with moderate alkali charge or NaOH concentration of 4% and 50% Gum Arabic concentration as well as cooking and beating times of 90 and 12 minutes respectively. This combination strikes a good balance between the response variables; tensile strength and flexibility and results in a kraft paper with superior tensile strength, enhanced fibre bonding, and favorable flexibility for packaging applications.

The multi-response optimal process-parameter combinations achieved optimal response values of 22.82 N/mm^2 and 2.33% for tensile strength and flexibility respectively, exhibiting a well-formed fibrous network. This suggests that the mechanical integrity of PALF-based kraft paper has been greatly improved exhibiting tensile strength values with a mean $\pm$ standard deviation of $16.24\text{N/mm}^2 \pm 5.46\text{N/mm}^2$. Gum Arabic integrated PALF-based kraft has therefore proven to be a viable alternative to conventional wood-based kraft papers for packaging applications.

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