



Effect of Ultrasonication Time on Structural, Morphological, and Functional Properties of Sugar Palm (*Arenga pinnata* Merr.) Flour Nanofibers

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ABSTRACT

Sugar palm (*Arenga pinnata*) flour has the potential to be developed into nanofibers for use as natural biopolymer materials. Ultrasonication is a promising method for modifying particle size and structural properties; however, the effects of different ultrasonication times on the characteristics of sugar palm flour nanofibers have not been fully explored. This study analysed the effect of different ultrasonication times on the physicochemical properties of sugar palm flour nanofibers produced with an ultrasonic probe homogeniser. A Completely Randomized Design (CRD) was applied with five ultrasonication times: 0 (control), 30, 60, 90, and 120 minutes, each with three replications. The evaluated parameters included morphology (SEM), particle size, crystallinity (XRD), viscosity, and color characteristics (CIE L*, a*, b*). Increasing ultrasonication time significantly reduced particle size from 177.33 ± 85.55 nm to 19.8 ± 14.89 nm, resulting in a smoother, more homogeneous nanofiber morphology. XRD analysis revealed an amorphous diffraction pattern within the 2θ range of $13-25^\circ$, indicating the absence of an ordered crystalline structure. The viscosity remained largely stable ($9522-9524$ mPa·s) between all treatments. Color analysis showed stable L*, a*, and b* values, suggesting that ultrasonication did not induce pigment degradation or noticeable visual changes. These findings demonstrate that prolonged ultrasonication effectively produces uniform, stable, and nano-sized sugar palm flour fibers.

Contribution to Sustainable Development Goals (SDGs):

SDG 3: Good Health and Well-being

SDG 9: Industry, Innovation and Infrastructure

SDG 12: Responsible Consumption and Production

1. INTRODUCTION

1.1. Research Background

Indonesia is rich in plant-based resources with significant potential for development into functional food ingredients, one of which is sugar palm (*Arenga pinnata* Merr.). The fruit of the sugar palm is widely consumed in traditional food products; however, its utilization remains largely limited to conventional

forms with minimal value-added processing. The high moisture content of sugar palm fruit contributes to its short shelf life and low handling stability, thereby constraining its broader application in food systems. To address these limitations, processing the fruit into flour has been proposed as an effective strategy to enhance storage stability, facilitate handling, and expand its potential as a functional ingredient [1].

Sugar palm fruit flour is known to contain high levels of carbohydrates and dietary fiber, with galactomannan as its major



functional polysaccharide. Galactomannan is a water-soluble hydrocolloid that has been widely reported to exhibit antioxidant activity, anti-ageing potential, and physiological benefits associated with dietary fibre. These properties make it a promising candidate for application in functional foods and biopolymer-based systems. Nevertheless, in its native flour form, galactomannan often demonstrates limited functionality due to poor dispersion, particle aggregation, and restricted surface area, which reduce its effectiveness in aqueous systems and limit its industrial applicability [2].

To overcome these limitations, structural modification approaches are required to enhance the physicochemical and functional properties of galactomannan. One promising approach is the application of nanotechnology, particularly the development of nanofibers. Reducing particle size to the nanoscale can significantly increase surface area, improve dispersion, and enhance interaction with surrounding media, thereby improving the overall functional performance of the material in food and biopolymer applications.

1.2. Literature Review

Nanofiber technology has been widely applied to modify biopolymers, improving their solubility, stability, and functional properties. Among various techniques, ultrasonic probe homogenization has gained considerable attention as an effective physical method for producing nanostructured materials. This technique utilises acoustic cavitation, generating intense shear forces and microstreaming that promote particle size reduction without chemical modification, making it particularly suitable for food-grade materials such as polysaccharides [3, 4].

Previous studies have demonstrated that ultrasonication can effectively reduce particle size and enhance the dispersion properties of various biopolymers. However, the effectiveness of this method is highly dependent on processing parameters, particularly ultrasonication time. Increased ultrasonication time generally leads to greater particle size reduction due to prolonged exposure to cavitation forces. On the other hand, excessive ultrasonication may induce structural degradation of polymer chains, negatively affecting functional properties as viscosity, gel-forming ability, and stability [5, 6].

Although numerous studies have explored the application of ultrasonication in polysaccharide modification, most have focused on commonly studied materials such as starch, cellulose, and other plant-derived gums. Research specifically addressing the optimization of ultrasonication conditions for galactomannan derived from sugar palm fruit flour remains limited. In particular, there is a lack of systematic investigation into the effect of ultrasonication time on the physical and structural characteristics of nanofibers produced from this local resource. This gap underscores the need for further research to establish optimal processing conditions that maximise functionality while minimising structural damage.

1.3. Research Objective

Based on the identified research gap, this study aims to investigate the effect of ultrasonication time on the production of nanofibers from sugar palm fruit flour using ultrasonic probe homogenization. Specifically, this research evaluates ultrasonication durations of 30, 60, 90, and 120 minutes and examines their influence on the physical and structural characteristics of the resulting nanofibers. The findings of this

study are expected to contribute to the optimization of processing conditions for sugar palm-based biopolymers and to support the development of value-added functional ingredients derived from local Indonesian resources.

2. MATERIALS AND METHODS

2.1. Materials and tools

Sugar palm fruit (*Arenga pinnata* Merr.) obtained from West Sumatra, Indonesia, was used as the primary raw material. The main instruments used included a Particle Size Analyzer (Horiba SZ100Z2, Japan), Scanning Electron Microscope (Carl Zeiss EVO 10 with EDX, Germany), Ultrasonic Probe Homogenizer (Sonics VCX 750, USA), Fourier Transform Infrared spectrometer (Shimadzu FTIR Tracer-100, Japan), viscometer (NDJ-8S, China), X-ray diffractometer (PANalytical PW3040/60 X'Pert PRO, Netherlands), and colorimeter (HunterLab ColorFlex EZ, USA). Standard laboratory equipment, such as grinders, sieves, glassware, and a food dehydrator, was also used.

2.2. Research Design

This experiment followed a Completely Randomised Design (CRD) with five ultrasonication treatments, each performed in triplicate. Ultrasonication was performed using an Ultrasonic Probe Homogenizer at durations of 0 (control), 30, 60, 90, and 120 minutes.

2.3. Research Procedure

Sugar palm fruit was washed, soaked in water for 48 h with water replacement every 24 h, rinsed, sliced to approximately 0.5 cm thickness, and dried at 80 °C for 18 hours using a food dehydrator until the moisture level was under 10%. Subsequently, the sample was ground and sieved using a 60-mesh screen to produce sugar palm fruit flour [7].

Nanofibers were prepared according to the [6] method with modifications. Sugar palm fruit flour (20 g) was dispersed in 400 mL of solvent and ultrasonicated for 30, 60, 90, and 120 minutes using an ultrasonic probe homogenizer. The resulting suspensions were subsequently used for physicochemical characterization.

FTIR analysis was performed by mixing dried samples (2 mg) with potassium bromide (KBr) and pressing them into pellets, followed by spectral acquisition in the range of 4000–400 cm^{-1} to identify galactomannan functional groups (Ihsan & Anggraini, 2023). Particle size distribution was determined using dynamic light scattering after dispersing samples in 3 mL of distilled water (pH 7) [8]. Structural properties were analysed by X-ray diffraction using a diffractometer equipped with a Cu anode tube, and the diffraction patterns were processed using EVA software [9]. Viscosity was measured using a viscometer with rotor number 4, in which 200 g of nanofiber suspension was placed in a 250 mL beaker before measurement [10]. Color characteristics were evaluated using a colorimeter based on the CIE Lab* system to obtain L^* , a^* , and b^* values, which were used to assess color stability after ultrasonication [11].

Statistical analysis was conducted using IBM SPSS Statistics. The data were evaluated using one-way analysis of variance (ANOVA), followed by Duncan's multiple-range test at the 5% significance level. Results are presented as mean $\pm$ standard deviation ($n = 3$).

3. RESULT AND DISCUSSION

3.1. FTIR Analysis of Galactomannan Functional Groups in Sugar Palm Fruit Flour

FTIR spectroscopy was performed to determine the characteristic functional groups of galactomannan in sugar palm fruit flour before ultrasonication. The spectra were collected over the range of 4000–400 cm^{-1} , and the major absorption bands are shown in Fig. 1 and summarized in Table 1.

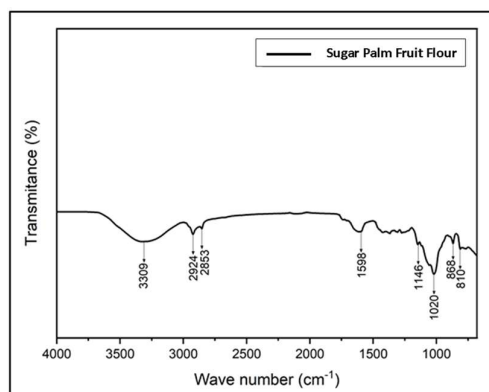


Fig 1. FTIR spectra of Sugar Palm Fruit Flour

The spectrum showed a broad absorption band in the range of 3500–3100 cm^{-1} , attributed to O–H stretching vibrations, which indicates extensive hydrogen bonding characteristic of polysaccharides. Absorption bands at 3000–2800 cm^{-1} were attributed to aliphatic C–H stretching of monosaccharide units, while the band near 1598 cm^{-1} was associated with C=O bending, suggesting the presence of minor protein components [12].

In the fingerprint region (1200–800 cm^{-1}), the band at approximately 1020 cm^{-1} corresponded to C–O–C stretching vibrations. Meanwhile, absorption bands observed at approximately 868 cm^{-1} and 810 cm^{-1} were attributed to glycosidic bond vibrations of α -D-galactopyranose and β -D-mannopyranose units, respectively, thereby confirming the presence of galactomannan [13, 14].

Table 1. FTIR absorption bands of sugar palm fruit flour

Wavenumber of sugar palm flour (cm^{-1})	Wavenumber range (cm^{-1})	Functional group	References
3309	3500-3100	O-H	[13]
2924	3000-2800	C-H	[13]
2853			
1598	~1598	C=O	[12]
1020	~1020	CH ₂ -O-CH ₂	[15]
868	~868	C-O-C	[13, 14]
810	~810	C-O-C	[13, 14]

Overall, the FTIR results confirm that sugar palm fruit flour contains galactomannan functional groups comparable to those reported for commercial galactomannan sources, supporting its suitability as a raw material for nanofiber production.

3.2. Morphological Analysis with SEM

Scanning electron microscopy (SEM) was used to examine the surface morphology and microstructural changes in sugar palm flour nanofibers as a function of ultrasonication duration. SEM images captured at 8,000 $\times$ magnification are presented in Fig. 2.

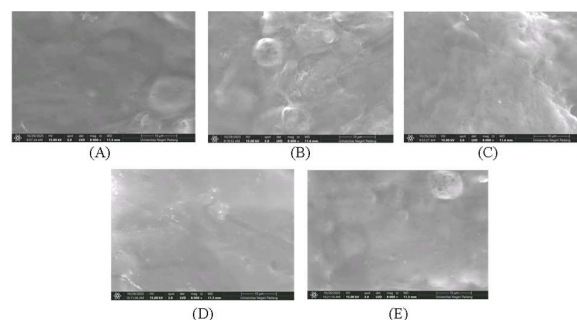


Fig 2. SEM micrographs of sugar palm fruit nanofibers produced at different ultrasonication times: (A) 0 min (control), (B) 30 min, (C) 60 min, (D) 90 min, and (E) 120 min.

SEM observations revealed clear morphological changes in sugar palm flour nanofibers with increasing ultrasonication time. The control sample showed large, irregular aggregates with rough, inhomogeneous surfaces, indicating strong particle agglomeration driven by intermolecular hydrogen bonding between galactomannan chains [16]. After 30 minutes of ultrasonication, partial aggregate disintegration produced smaller particles with slightly smoother surfaces, although residual large particles remained, suggesting limited disruption of intermolecular interactions [17].

At 60 minutes, nanofibers exhibited a more distinct fibrous morphology with reduced particle diameter, improved surface smoothness, and enhanced homogeneity, indicating more effective dispersion. This effect became more pronounced at 90 minutes, where finer diameters and more regular morphology were observed due to intensified cavitation effects [13]. The most uniform morphology occurred at 120 minutes, characterized by well-defined nanofibers with smooth surfaces, reflecting extensive disruption of hydrogen bonding and improved polysaccharide chain rearrangement [5, 16].

Overall, SEM results confirm that increasing ultrasonication time reduces particle aggregation and enhances morphological uniformity, demonstrating the effectiveness of ultrasonic probe homogenization for producing homogeneous sugar palm flour nanofibers. SEM micrographs were qualitatively interpreted to compare morphological differences among treatments, including aggregation, fibrillation, and surface uniformity, while quantitative particle size measurements were obtained using PSA.

3.3. Particle Size Analysis

Particle size is a critical parameter in nanofiber characterization, as it directly influences specific surface area and functional performance. The particle size distribution of sugar palm flour nanofibers was analysed using a particle size analyser (PSA) to evaluate the effectiveness of ultrasonication in reducing particle size. Results of particle size analysis are presented in Table 2.

Particle size results showed a significant reduction with increasing ultrasonication time (Table 2). The control sample (A) exhibited an average particle size of 177.2 ± 85.55 nm, indicating

strong particle aggregation and limited dispersion, which can be attributed to intermolecular hydrogen bonding among galactomannan chains that promotes aggregation and restricts particle separation [5]. Ultrasonication for 30 minutes resulted in a marked decrease in particle size to 66.76 ± 57.50 nm; however, no significant difference was observed between samples treated for 30 and 60 minutes ($p > 0.05$), suggesting that initial cavitation effects mainly disrupted loosely bound aggregates.

A further significant reduction occurred at longer ultrasonication times, with particle size decreasing to 45.40 ± 19.77 nm at 90 minutes and reaching 19.80 ± 14.89 nm at 120 minutes. Compared to the control, ultrasonication for 120 minutes achieved an approximately 89% reduction in particle size, confirming the effectiveness of prolonged cavitation in fragmenting galactomannan-based structures [18]. These findings are consistent with SEM observations, which demonstrated increased homogeneity and finer fiber morphology at extended ultrasonication times.

Compared with previous studies, sugar palm flour nanofibers exhibited more efficient size reduction than celery extract under similar ultrasonication conditions [6]. This difference is likely related to the molecular structure and flexibility of galactomannan, which facilitates more effective chain disruption under ultrasonic cavitation [4]. Overall, these results indicate that ultrasonication times exceeding 60 minutes are required to achieve a statistically significant reduction in nanoscale particles in sugar palm flour nanofibers.

3.4. Viscosity Test

Viscosity is an important parameter for evaluating the rheological behavior of polysaccharide-based nanofiber suspensions. The viscosity values of sugar palm flour nanofibers at different ultrasonication times are presented in **Table 2**.

Viscosity values ranged from 9522.33 to 9524.33 mPa·s and remained relatively stable across treatments. No significant differences were observed among samples treated for 0–60 minutes, while a slight increase was detected at 120 minutes of ultrasonication ($p < 0.05$).

The high viscosity of the control sample reflects the characteristic behavior of galactomannan hydrocolloids, which form hydrogen-bonded three-dimensional networks with strong water-binding capacity [19]. Despite the substantial reduction in particle size, viscosity remained largely unchanged, suggesting that ultrasonication primarily induced deagglomeration and fibrillation of sugar palm flour particles rather than extensive depolymerization of galactomannan chains. Similar ultrasonication-induced fibrillation and particle-size reduction have been reported in plant-derived fibrous materials [4, 5]. During acoustic cavitation, the collapse of microbubbles generates localized shear forces and turbulence that preferentially disrupt weak intermolecular associations, such as hydrogen-bonded aggregates and loosely packed fibrillar bundles [20].

In contrast, the covalent backbone of galactomannan likely remained relatively preserved under the applied processing conditions [21]. Since the viscosity of galactomannan suspensions is primarily governed by molecular chain interactions, entanglement, and network formation, retention of the main polysaccharide chains enables the suspension to maintain its rheological behaviour even after a marked reduction in physical particle dimensions [22].

The slight increase in viscosity after 120 min may be associated with the increased specific surface area of finer particles, which can enhance hydration, water retention, and intermolecular interactions, thereby partially offsetting minor structural disruption during prolonged sonication [22, 23]. These findings demonstrate that ultrasonic probe homogenization effectively reduces particle size while preserving the functional viscosity of sugar palm flour nanofibers.

Within the tested range, 120 min appears to be the optimum treatment, as it produced the smallest particle size while maintaining viscosity stability. The slight increase in viscosity at this stage may reflect enhanced hydration and intermolecular interactions. Although no clear evidence of severe galactomannan degradation was observed up to this treatment time, further prolonged ultrasonication beyond 120 min may eventually induce chain scission and reduce functional properties, as prolonged sonication has been reported to cause polysaccharide degradation through cavitation-induced mechanical stress [20].

Table 2. Particle Size and Viscosity of Sugar Palm Flour Nanofibers at Different Ultrasonication Times

Treatments	Particle Size (nm)	Viscosity (mPa.S)
	Mean $\pm$ SD	Mean $\pm$ SD
A (Control)	177.33 ± 85.55^a	9522.67 ± 0.58^a
B (30 min)	66.76 ± 57.50^b	9522.33 ± 0.58^a
C (60 min)	65.03 ± 54.92^b	9522.67 ± 0.58^a
D (90 min)	45.40 ± 19.77^c	9523.33 ± 0.58^{ab}
E (120 min)	19.80 ± 14.89^d	9524.33 ± 0.58^b

3.5. XRD Analysis

XRD analysis was performed to characterise the structural properties and crystallinity of sugar palm flour nanofibers; the diffraction patterns are shown in **Fig. 3**.

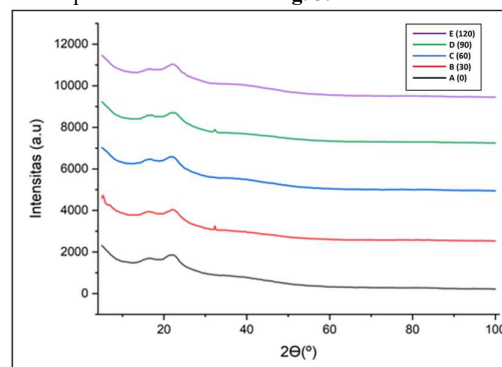


Fig 3. X-ray diffraction (XRD) patterns of sugar palm flour nanofibers

All samples exhibited broad diffraction peaks in the 2θ range of approximately 13° – 25° , without sharp, well-defined peaks typically associated with highly crystalline materials. This diffraction behaviour indicates a predominantly amorphous structure with a very low degree of crystallinity, a characteristic of galactomannan-based polysaccharides [16].

The absence of distinct crystalline peaks at around 15° , 22° , and 35° , commonly attributed to cellulose crystalline domains, confirms that the nanofibers differ structurally from highly ordered polysaccharides such as cellulose and starch. This behavior is related to the molecular architecture of galactomannan, consisting of a β -(1 $\rightarrow$ 4)-D-mannopyranose

backbone with irregular α -(1 $\rightarrow$ 6)-D-galactopyranose side chains, which interfere with chain packing and prevent the formation of long-range crystalline order [13, 21]

Increasing ultrasonication time led to slight peak broadening and reduced peak intensity, indicating decreased residual short-range order and a more amorphous structure, rather than a complete transition to full amorphisation. Since all samples already showed broad halos typical of predominantly amorphous galactomannan, these changes likely reflect disruption of limited ordered domains by cavitation forces during ultrasonication. Similar behavior has been associated with increased structural disorder in nanoscale materials [24]. Overall, this result indicates that ultrasonication preserved the material's predominantly amorphous nature while slightly reducing residual structural order.

3.6. Color Analysis

Color characteristics of sugar palm flour nanofibers were evaluated using the CIE Lab* system, and the results are presented in **Table 3**. The L* values ranged from 42.91 to 44.31, indicating consistently dark coloration typical of sugar palm flour. No statistically significant differences were observed among treatments, suggesting that ultrasonication did not markedly affect lightness.

The control sample exhibited an L* value of 44.03, comparable to ultrasonicated samples. A slight decrease in L* at 30 minutes of ultrasonication may be attributed to improved particle dispersion, which enhances light absorption. At longer ultrasonication times, L* values increased and stabilized, indicating the formation of a more uniform dispersion structure [25].

The a* values were negative for all samples (−1.18 to −0.62), indicating a weak greenish tone, while b* values were positive (6.81–8.19), confirming the dominance of yellow coloration. The stability of a* and b* values across treatments suggests that ultrasonication did not significantly alter pigment composition or promote browning reactions [26]. Hue angle values ranged from 94.37° to 99.90°, indicating stable yellow coloration with a slight greenish tendency and confirming that all samples remained within the same color region [27, 28]. Minimal variation in hue angle further indicates that ultrasonication up to 120 minutes did not induce pigment degradation or noticeable color shifts.

Table 3. CIE Lab* color parameters of sugar palm flour nanofibers at different ultrasonication times

Treatments	L* $\pm$ SD	a* $\pm$ SD	b* $\pm$ SD	Hue° $\pm$ SD	Color
A (Control)	44.03 $\pm$ 0.17	-0.84 $\pm$ 0.08	8.19 $\pm$ 0.29	95.85 $\pm$ 0.71	Yellow
B (30 min)	42.91 $\pm$ 0.56	-1.18 $\pm$ 0.11	6.81 $\pm$ 0.31	99.90 $\pm$ 1.34	Yellow
C (60 min)	44.06 $\pm$ 0.34	-0.66 $\pm$ 0.01	7.95 $\pm$ 0.23	94.75 $\pm$ 0.07	Yellow
D (90 min)	43.78 $\pm$ 0.20	-0.66 $\pm$ 0.11	7.97 $\pm$ 0.18	94.77 $\pm$ 0.85	Yellow
E (120 min)	44.31 $\pm$ 0.35	-0.62 $\pm$ 0.18	8.18 $\pm$ 0.32	94.37 $\pm$ 1.41	Yellow

Overall, these results demonstrate that ultrasonic probe homogenization produces sugar palm flour nanofibers while maintaining color stability and visual quality during processing.

4. CONCLUSION

Ultrasonication time significantly influenced the structural and functional characteristics of sugar palm flour nanofibers. Prolonged ultrasonication effectively reduced particle size to the nanoscale, improved morphological uniformity, and promoted predominantly amorphous structures without compromising rheological stability or color quality. Ultrasonication for 120 minutes produced the smallest and most homogeneous nanofibers while preserving the molecular integrity of galactomannan. Overall, ultrasonic probe homogenization is a promising physical approach for producing stable, food-grade sugar palm flour nanofibers with potential applications in functional foods and biopolymer-based systems

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