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# Green-Chemical Nanofibrillated Cellulose from Pineapple Cores using Microfluidization

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**Abstract**

The production of nanofibrillated cellulose (NFC) from pineapple cores (*Ananas comosus* (L.) Merr.) involves green-chemical cellulose extraction followed by microfluidization. The chemical composition of pineapple cores was initially analyzed to assess their suitability for cellulose production. The analysis revealed that the pineapple cores contained 94.18 ± 0.20 % insoluble dietary fiber (IDF), while soluble dietary fiber (SDF) comprised only 0.31 ± 0.01 %, and consequently suitable for cellulose extraction. The cellulose extraction from pineapple cores involved removing non-cellulosic components: 1) Control (washed with water), under a green-chemical method with 0.1 M citric acid for 20 min at different temperatures, 2) Boiling (100 °C), and 3) Autoclaving (121 °C). Autoclaving with citric acid achieved the highest cellulose content (72.77 %), with minimal hemicellulose (21.44 %), lignin (13.33 %), galacturonic acid (3.42 %), and residual total sugar (1.82 %). The short extraction duration of only 20 min produces high-purity cellulose comparable to that obtained through alkaline and bleaching methods. Scanning electron microscopy (SEM) indicated that this method effectively removed non-cellulosic materials, increasing fiber porosity. The cellulose was then microfluidized at 25,000 psi (172 MPa) through 1 - 5 cycles, reducing the fiber diameters from 80.25 to 62.46 nm. Rheological testing revealed that NFC exhibited a higher storage modulus (G') than loss modulus (G"), suggesting gel-like behavior. NFC suspensions at 0.5 and 1 % concentrations showed viscosities of 3.35 - 3.85 cPs and 3.75 - 4.40 cPs, respectively. Therefore, NFC can function as a stabilizing agent for food without significantly altering the viscosity.

**Keywords:** Pineapple core, Nanofibrillated cellulose, Citric acid, Autoclaving, Microfluidization, Mechanical treatment, Viscosity, Galactulonic acid

## Introduction

Cellulose nanomaterials (CNMs) have recently gained greater attention for use in various products, including foods, cosmetics, packaging materials, and pharmaceuticals. Generally, CNMs are derived from agroindustry waste and extracted into nanoscale materials through chemical and physical methods. CNMs can be classified as nanocrystalline cellulose (NCC) and nanofibrillated cellulose (NFC), respectively, based on their preparation technique [1].

Recently, there have been several references to the use of NFC in food. Consequently, NFC has the potential for application as dietary fiber (a functional food ingredient) and a food stabilizer [2]. In food production, NFC has several functional properties, including water-holding capacity (WHC), gelling ability, and emulsifying properties [3]. These properties make it suitable for use in food products such as salad dressings and mayonnaise [4] or during the production

of ice cream, whipped cream [5], and sausages to improve the gel strength of the texture [6].

NFC can be derived from the by-products of agroindustry processing due to their high cellulose content, cost-effectiveness, and support for sustainability. Examples include lime residues [7,8], banana rachis [9], grapefruit peels [5], and palm-pressed fiber [10].

Pineapple (*Ananas comosus* (L.) Merr.) is one of the world's most economical crops. The canned pineapple production industry generates a large volume of by-products, particularly pineapple cores, with approximately 247,089.9 tons left over from the production process [11,12]. The chemical composition of the pineapple core has been reported to include lignin (12.9 %), cellulose (11.5 %), and hemicellulose (14.1 %) [13]. Suebsuntorn and Jirukkakul [12] investigated the production of cellulose from pineapple cores by chemical treatment, demonstrating that it could be modified to produce carboxymethyl cellulose (CMC), which exhibits high viscosity and up to 98 % solubility, comparable to commercial CMC. The pineapple core is an attractive and high-quality raw material for cellulose production. Utilizing agricultural by-products helps reduce the cost of cellulose production. However, the pectin and sugars present in pineapple cores are important for the quality of cellulose and should be considered [14].

The most common cellulose extraction step uses chemical agents such as an alkaline treatment (NaOH or KOH) and bleaching (sodium chlorite, sodium hypochlorite, hydrogen peroxide, and ozone) to separate cellulose fiber from non-cellulosic components (hemicellulose, lignin, pectin, waxes, proteins, and soluble sugars). Interestingly, environmentally friendly extraction is attractive due to consumer awareness of health and safety. Chemical-free cellulose extraction has been proposed to remove non-cellulosic components. From this perspective, hydrothermal treatment, such as steam explosion and autoclaving, employs water as liquid or vapor at high temperatures (100 - 300 °C). Autoclaving is accelerated to break down the hemicellulose and lignin structure, further enhancing high-purity cellulose [8]. Mild acid extraction using citric acid and other weak organic acids has been proposed as a method for reducing toxicity [15], commonly referred to as green-chemical cellulose extraction.

NFC production involves physical processes such as microfluidization, high-pressure homogenization (HPH), grinding, and cryocrushing. Microfluidization is an alternative mechanical process that uses shear force to defibrillate the cellulose fiber into nanometer scale as NFC. High pressure (200 MPa) and shear force are used to break down the hydrogen bonds of fiber structure, releasing free hydroxyl groups to enhance the water binding capacity [16]. This characteristic promotes intermolecular interaction between NFC to bind cellulose fiber with water and other components in a food matrix, resulting in a web-like structure [17].

In this study, cellulose was extracted from pineapple cores using a green-chemical method with citric acid. The reaction was conducted under varying thermal conditions, including boiling and autoclaving, to assess their impact on cellulose purity, with a one-step process for recovery, thereby minimizing the energy, cost, and waste generated during processing. This cellulose extraction method is environmentally friendly, safe for consumers, and suitable for food applications. Additionally, the study investigated the conversion of cellulose into NFC through mechanical processing using a microfluidizer. Morphological and rheological properties were analyzed to evaluate the flow behavior of pineapple core cellulose fibers for potential use in food products.

## Materials and methods

### Materials

Pineapple cores (*Ananas comosus* (L.) Merr.) were obtained from Banka District, Ratchaburi Province in Thailand. Fresh pineapple cores are by-products of pineapple processing. The pineapple cores were washed and separated as pulp and juice using a fruit pulp separator. The pineapple core pulp was stored in a commercial refrigerator (Sanden Intercool, SPB-0300, Japan) at approximately 5 °C. Citric acid was used as a food-grade reagent (Union Chemical, Thailand). Sodium hydroxide (technical grade), sodium chlorite (technical grade), and glacial acetic acid (technical grade) were purchased from J.T. Baker Chemical Company (Pennsylvania, USA).

## Methods

### *Chemical composition of pineapple core*

The chemical composition of pineapple core pulp was determined using methods proposed by the Association of Official Analytical Chemists (AOAC). The moisture, crude protein, fat, ash, total dietary fibers (TDF), IDF, and SDF content of pineapple core pulp were measured on a dry weight basis following the methods of AOAC 925.10, 991.20, 2003.05, 940.26, 985.29, 991.42 and 993.19, respectively [18]. Sampling was carried out in triplicate.

### *Preparation of pineapple core cellulose*

Pineapple core pulp was extracted using a fruit pulp separator (Philips, SK-4000, China). Three processes were compared in this study to remove pectin, sugars, and non-cellulosic components from the pineapple cores to increase the purity of the cellulose. The processes were as follows: Water washing (control) until the soluble solid content reached 0 °Brix. Extraction with 0.1 M citric acid solution at a citric acid to pineapple core pulp ratio of 3:1, followed by boiling at 100 °C (low temperature) or by heating in an autoclave at 121 °C (high temperature) (modified from Jongaroontaprangesee *et al*. [8]) for 20 min. The treated pineapple cores were then washed with tap water through a nylon cloth bag until the soluble solid content reached 0 °Brix, verified using a refractometer (Atago, MASTER-53alpha, Tokyo, Japan). The samples were dried at 70 °C (Memmert, UM500, Germany), ground to a fine powder (passing through a 250-micron sieve), and stored in airtight containers for chemical composition analysis.

### *Determination and characterization of pineapple core cellulose*

*Galacturonic acid content determination*

The galacturonic acid (GalA) content was quantified using high-performance liquid chromatography (HPLC). In this method, 0.2 g of pineapple core cellulose, derived from 3 different processes, was dissolved in 4 mL of distilled water and incubated at 30 °C with shaking at 150 rpm for 1 h. The solution was then centrifuged at 10,000 rpm for 120 min at 4 °C. The supernatant was filtered through a 0.4 µm syringe filter prior to HPLC analysis (Shimadzu Prominence LC-20A, Kyoto, Japan). Compound separation was achieved using a Shodex SUGAR SH1011 column (6 µm, 8.0×300 $mm^2$) maintained at 40 °C. The mobile phase consisted of 10 mM $H_2SO_4$ in deionized water, with a flow rate of 1.0 mL/min. A 10 µL sample was injected, and analytes were detected with a UV detector set at 210 nm. GalA content was determined by measuring peak height, with quantification based on a standard curve generated from GalA concentrations ranging from 1 - 100 mg/L [19].

*Total sugar determination*

Total sugar content (by weight) was determined using the phenol sulfuric acid method. The sugar content was analyzed by preparing glucose standard solutions at 0, 0.04, and 0.08 g/100 mL, along with a 5 % phenol solution. A pipette containing 10 µL of the sample was placed into a test tube, with 1 mL of distilled water and 1 mL of 5 % phenol added, followed by 5 mL of concentrated sulfuric acid. The mixture was vortexed by allowing it to stand for 10 min, vortexed again, and cooled in a 30 °C water bath for 10 min. The absorbance was measured at 490 nm. A blank was prepared by adding 2 mL of distilled water to 10 µL of the sample. A 5 % (w/v) pineapple core cellulose sample was prepared by weighing 3 g into a 125 mL Erlenmeyer flask and adding 60 mL of distilled water. The mixture was shaken at 30 °C and 150 rpm for 1 h using an incubator shaker (Memmert, WTB35, Germany). An empty centrifuge bottle was weighed and recorded before transferring the shaken sample, ensuring most of the residue was transferred. The sample was centrifuged at 4 °C and 10,000 rpm for 20 min. The sample was filtered using Whatman No.1 filter paper. For total sugar analysis, 1 mL of supernatant was diluted as needed, transferred to a test tube, and mixed with 1 mL of 5 % phenol solution following the same procedure. The blank absorbance from the sample absorbance is subtracted, compared with a standard curve, and the sugar concentration is calculated [20].

*Cellulose, hemicellulose, and lignin content determination*

The chemical composition of pineapple core cellulose was analyzed, focusing on the content of extractives, lignin, holocellulose, and alpha-cellulose, following the analytical standards of the Technical Association of the Pulp and Paper Industry (TAPPI).

The hemicellulose content was calculated as the difference between holocellulose and alpha-cellulose. The extractive content was determined by extracting with an ethanol-benzene mixture (according to TAPPI T264 om-88 and TAPPI T264 cm-97 standards). Lignin content was analyzed by digesting with 72 and 3 % sulfuric acid (by weight), following TAPPI T222 om-98. Holocellulose was analyzed by reacting the extractive-free pineapple core cellulose samples with sodium chlorite and acetic acid, yielding white holocellulose through Browning's acid chlorite method. The alpha-cellulose content was determined by reacting the holocellulose sample with 17.5 % sodium hydroxide solution (by weight to volume), following TAPPI T 203 om-88 [21]. The moisture content of pineapple core cellulose was measured on a dry weight basis following the methods of AOAC 925.10 [18]. Sampling was carried out in triplicate.

***Scanning electron microscopy***

Each pineapple core cellulose sample was dried at 70 °C (Memmert, UM500, Germany) and finely ground. The pineapple core cellulose samples were then fixed on a stub and coated with gold. The microstructure was examined with a scanning electron microscope (JEOL, JSM-IT300, Tokyo, Japan) at 15 kV, with a magnification of 1,000×.

***Preparation of NFC from pineapple core***

NFC was prepared using the mechanical microfluidization technique adapted from Jongaroontaprangesee *et al*. [8]. Briefly, a cellulose suspension derived from pineapple cores was prepared in distilled water with a solid content of 1 % (w/w, wet basis). Prior to microfluidization treatment, the suspension was mixed using a high-speed homogenizer (T25 Digital Ultra-turrax model, IKA, Germany) at 10,000 rpm speed for 15 min. The suspension was then processed through a microfluidizer (LV1 Low volume model, G10Z chamber type, Microfluidics, USA) at 25,000 psi (172 MPa), varying the number of passes from 1 to 5. The processed suspension was collected for further analysis.

***Field emission scanning electron microscopy (FE-SEM) of NFC***

The morphology of each NFC sample was analyzed using a field emission scanning electron microscope (FE-SEM) (JEOL, JSM-7610F, Tokyo, Japan). A droplet of diluted nanocellulose suspension was placed on a glass slide and air-dried. FE-SEM images were captured at a magnification of 50,000×.

***Rheological properties of NFC***

The rheological properties of 1 % (w/w) NFC suspensions, diluted with deionized water, were analyzed using a Physica MCR 300 rheometer (Messtechnik GmbH, Stuttgart, Germany) with parallel plates (40 mm diameter, 1 mm gap). A thin layer of paraffin oil was applied to prevent evaporation. Measurements were conducted at a constant temperature of 25 °C. Frequency sweep tests were performed over an angular frequency range of 0.1 to 100 rad/s. The storage modulus (G') and loss modulus (G") were recorded as functions of frequency to assess the linear viscoelastic range (LVR) and predict potential behavior in food products [8]. All measurements were within the LVR region, with data averaged from 3 replicates per sample. Steady shear viscosity was measured and plotted against NFC suspension concentrations (0.5 and 1 % w/w), with flow curves obtained at shear rates from 0.1 to 100/s. The relationship between microfluidization cycles (1 - 5) and apparent viscosity was also examined. Three replicates were used for each sample.

***Statistical analysis***

A completely randomized design (CRD) was used to compare the physicochemical properties of cellulose extracted from pineapple cores using physical and chemical methods combined with high and low-temperature treatments. The dependent variables included the chemical composition of pineapple core fibers, galacturonic acid content, and total sugar content. The experiment was conducted in triplicate. Data variance was subjected to Analysis of Variance (ANOVA), and mean differences were compared using Duncan's New Multiple Range Test at a 95 % confidence level, employing the Statistical Package for the Social Sciences (SPSS) version 28 (IBM, USA).

## Results and discussion

### Chemical composition of pineapple core

The chemical composition of a pineapple core is shown in **Table 1**. The pineapple core mainly comprises 94.48 ± 0.12 % of TDF (on a dry basis). This type of dietary fiber is resistant to hydrolysis reactions by enzymes in the digestive system. It is a healthy dietary fiber that can be divided into 2 groups: Soluble and insoluble, based on its solubility in water [22]. A pineapple core contains approximately 94.18 ± 0.20 % of IDF, while SDF makes up only 0.31 ± 0.01 %. The majority of IDF is cellulose, composed of glucose units connected by β-1,4 glycosidic bonds, while soluble fibers, such as pectin, are attached to cellulose. Its function is to bind the cell walls of fruits and vegetables may result in high water permeability [8]. As can be observed from **Table 1**, the core of the pineapple core contains the most IDF. Therefore, the pineapple core has suitable potential as a raw material for extracting cellulose fibers.

**Table 1** Chemical composition of the pineapple core.

| Chemical composition | Amount (g/100 g, dry basis) |
|---|---|
| Total dietary fiber (TDF) | 94.48 ± 0.12 |
| Insoluble dietary fiber (IDF) | 94.18 ± 0.20 |
| Soluble dietary fiber (SDF) | 0.31 ± 0.01 |
| Moisture | 5.25 ± 0.41 |
| Protein | 1.83 ± 0.05 |
| Ash | 1.33 ± 0.10 |
| Fat | 0.60 ± 0.11 |

### Chemical composition of pineapple core cellulose

The separation of cellulose fibers from the pineapple core requires the removal of pectin, sugar, and other compounds to obtain pure and high-quality cellulose, as shown in **Table 2**. Galacturonic acid (GalA) is the main component of pectin. A pineapple core washed with water had a GalA content of 35.18 %. Pectin extraction with citric acid at low temperature (boiling, 100 °C) and high temperature (autoclaving, 121 °C) for 20 min resulted in 9.74 and 3.42 % GalA remaining, respectively. The total amount of sugar remaining in the pineapple core after the water-washing process was 0.87 %, followed by extraction of pectin with citric acid at high temperature (1.82 %) and at low temperature (2.19 %). If a small amount of sugar remains, it will help reduce the browning reaction in subsequent production processes. Washing with water was found to be the most effective at dissolving and removing most of the sugar from the pineapple core.

**Table 2** Chemical composition of pineapple core cellulose prepared by washing with water, boiling, and autoclaving with 0.1 M citric acid for 20 min.

| Sample | GalA | Total sugar | Chemical composition (% dry basis) | | | |
|---|---|---|---|---|---|---|
| | | | Moisture | Cellulose | Hemicellulose | Lignin$^{ns}$ |
| Pineapple core pulp (washing with water) | 35.18 ± 0.01$^{a}$ | 0.87 ± 0.05$^{a}$ | 5.88 ± 0.12$^{a}$ | 45.64 ± 0.75$^{c}$ | 41.11 ± 1.48$^{a}$ | 14.20 ± 0.21 |
| Boiling with citric acid | 9.74 ± 0.25$^{b}$ | 2.19 ± 0.08$^{c}$ | 4.84 ± 0.08$^{b}$ | 59.61 ± 1.65$^{b}$ | 33.18 ± 0.94$^{b}$ | 13.82 ± 0.13 |
| Autoclaving with citric acid | 3.42 ± 0.10$^{c}$ | 1.82 ± 0.05$^{b}$ | 4.78 ± 0.18$^{b}$ | 72.77 ± 1.06$^{a}$ | 21.44 ± 2.80$^{c}$ | 13.33 ± 0.16 |

Note: a - c mean ± standard deviation in the same column with different letters indicates a significant difference ($p < 0.05$). ns means there is no significant difference ($p \geq 0.05$).

**Table 2** shows that fibers from the autoclaving process had lower hemicellulose and lignin content, at 21.44 % and 13.33 %, respectively. The galacturonic acid content was reduced to 3.42 %, and the remaining

total sugar content was 1.82 %. The extraction of pectin with citric acid at high temperatures resulted in the highest cellulose content of 72.77 %, significantly higher than other processes. These results are consistent with similar investigations on the cellulose content of water hyacinth (72.63 %) and the removal of lignin and hemicellulose by the alkalization process [23]. A similar finding has been reported, demonstrating that autoclaving lime residue after juice extraction via the chemical-free procedures, i.e., autoclaving with water at 120 °C for 2 h, significantly reduces the GalA content and hemicellulose. In contrast, the cellulose content in lime residue was 47.04 % on a dry basis. When autoclaving at 130 °C for 2 h was combined with chemical pretreatment through alkaline hydrolysis and bleaching, the cellulose content of lime residue increased to 76.51 % on a dry basis [8].

Hydrothermal processes, such as steam explosion and autoclaving, are attractive green extraction techniques with the potential to reduce chemical usage [8]. It has also been reported that hydrothermal processes disrupt plant cell structure. Water heated above boiling point (100 °C) and pressurized beyond its saturation (14.6 Psi) creates a hydrothermal condition that effectively removes non-cellulosic components, particularly pectin [24]. In addition to temperature, prolonged extraction time further enhances yield by increasing mass transfer between the solute and solution. However, high temperature, extended heating, and acidic conditions in conventional processes may cleave the glycosidic linkage between GalA residue in pectin, enhancing its solubility [24]. The additional extraction activity of citric acid on chelator-solubilized pectin fractions results in higher yields compared to mineral acids under identical extraction conditions, leading to the partial depolymerization of hemicelluloses and pectins. However, this process does not reduce the lignin content in the fibers. Lignin removal generally occurs through the reaction of sodium chlorite during the bleaching process, resulting in the oxidative fragmentation of lignin, with some portions leaching out as lignin chloride [8].

Extraction using citric acid and other weak organic acids may provide significant advantages for application as food ingredients since strong mineral acids are corrosive, pose health risks, and can increase waste treatment costs. The high efficiency of citric acid in pectin extraction was also compared to the pectin yields determined for hydrochloric, sulfuric, nitric, citric, and acetic acids [15]. Previous studies have investigated chemical-free methods for NFC preparation. Microwave (MW) heating has been proposed as an alternative method for extracting NFC from the lime residue after juice extraction, reaching a maximum sample temperature of 106.9 °C, which may be insufficient for effective hemicellulose hydrolysis [7]. Additionally, MW treatment did not further remove pectin bound to plant cell walls, since this pectin appears to be strongly embedded within cellulose microfibrils. The lignin content in NFC produced via MW heating (2 - 3 cycles) was markedly lower than in this study (3 - 5 % on a dry basis), while the cellulose content remained low at 46 - 49 % (dry basis) [7]. This indicates that autoclaving is optimal for extracting pectin with citric acid since it accelerates the hydrolysis reaction, leading to greater pectin extraction and high cellulose content from the pineapple core.

**Scanning electron microscopy of pineapple core cellulose**

SEM images (**Figure 1**) reveal morphological differences in the pineapple core cellulose obtained via water washing, boiling, and autoclaving with citric acid for 20 min. The porous structure can be observed in **Figure 1(c)**. This may result from high temperature and pressure during autoclaving, which rapidly disrupts plant cell walls. The primary cell wall components are cellulose, hemicellulose, and pectin. Increasing the interaction with citric acid, potentially cleaving the glycosidic linkage between GalA residue, leads to the solubilization and depolymerization of hemicellulose and pectin.

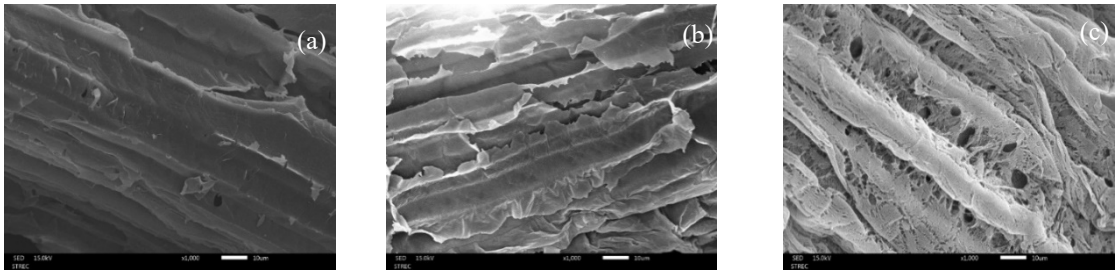


**Figure 1** SEM images of pineapple core pulp (washing with water) (a), pineapple core cellulose from boiling (b), and autoclaving (c) with 0.1 M citric acid for 20 min.

**Properties of NFC from pineapple core**

The morphology of NFC was assessed using a field emission scanning electron microscope (FE-SEM), shown in **Figure 2**. Initial pineapple core cellulose (not passing through a microfluidizer) is sized in the micrometer range on a paper-like surface. Generally, particle sizes should be less than 250 micrometers to pass through the microfluidizer without clogging. The material processed through the microfluidizer for 1 - 5 cycles leads to better dispersion into individual fibers, and the average diameter of the fibers reaches the nanometer scale [25], thus referred to as NFC. The average fiber length decreases to a range of 18.03 - 18.52 micrometers, and the average diameter of NFC from the pineapple core is reduced to the nanometer scale, from 80.25 to 62.46 nanometers. This is due to the increased shear forces passing through the microfluidizer for 1 - 5 cycles, thereby breaking the glycosidic bonds of cellulose. It can be observed that smaller fiber sizes result in better dispersion of cellulose fibers. Previous studies have examined the effect of passing microcrystalline cellulose (MCC) through a microfluidizer at 137.9 MPa for 1 - 20 cycles on nanofiber morphology. The results indicated that 10 - 15 passes increased the aspect ratio of cellulose fiber bundles, while additional cycles (20 passes) caused fiber aggregation due to increased surface area and hydroxyl (OH) group concentration [1]. This research demonstrates that a smaller NFC with a higher surface area can be produced by increasing the number of passes through the homogenizer. However, higher cycle numbers in the microfluidizer resulting in NFC dispersing into finer single fibers, leading to physical entanglement and weaker gel structures, remains a limitation of the microfluidization process.

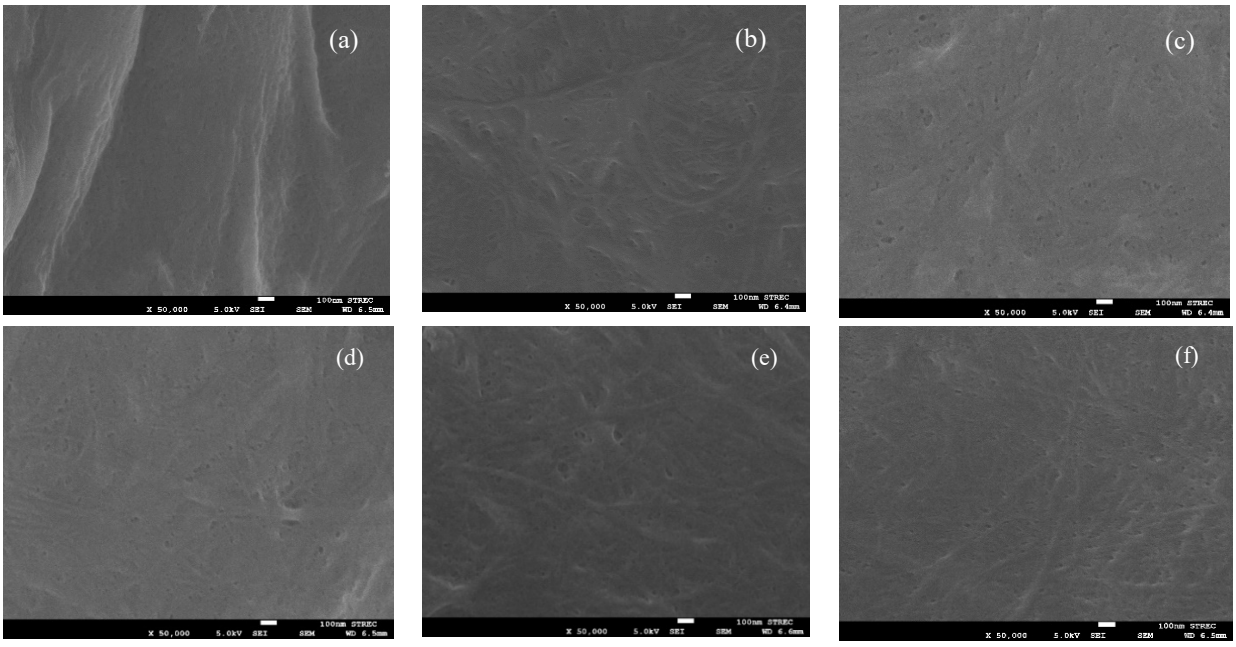


**Figure 2** FE-SEM images of NFC from pineapple cores at a concentration of 1 % (by wet weight) before (a) and after passing through a microfluidizer for 1 - 5 cycles (b) - (f).

The physical appearance of a suspension is shown in **Figure 3**. This indicates that NFC from pineapple cores can be homogeneously dispersed in water without sedimentation or phase separation. Microfluidization also promotes the dispersion of fiber components. The NFC gel structure depends on crosslinks and the mechanical entanglement of fibrils and polymer chains. NFC, with its high aspect ratio, large surface area, and abundance of hydroxyl (OH) groups, exhibits effective WHC and gelation properties.

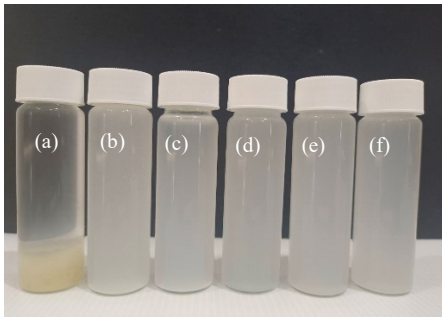


**Figure 3** Appearance of NFC suspension from pineapple cores at a concentration of 1 % (by wet weight) before (a) and after passing through a microfluidizer for 1 - 5 cycles (b) - (f).

**Rheological properties**

The rheological properties of NFC from the pineapple core revealed flow behavior to identify hydrogel structures or polymer-polymer chain interactions. The strength of NFC solution at a concentration of 1 % (by weight) was compared in terms of storage modulus (G') and loss modulus (G"). The frequency sweep test at 0.1 - 100 rad/s showed changes in the G' and G" values of 1 % NFC from the pineapple core, as illustrated in **Figure 4**. The 3 cycles processed through the microfluidizer exhibited higher G' and G" values (2.26 - 4.11 Pa) compared to other levels. The NFC exhibited solid-like or gel-like behavior, with higher G' values indicating stronger gels. Conversely, if G" exceeds G', the sample behaves like a liquid [26]. Cellulose subjected to multiple cycles in a microfluidizer exhibits increased fiber separation into individual fibers and enhanced hydrogen bonding between NFC under applied stress or angular frequency changes. However, excessive microfluidizer cycles further degrade the fibers, leading to fiber disentanglement and liquid-like behavior, weakening the gel structure and reducing G' values.

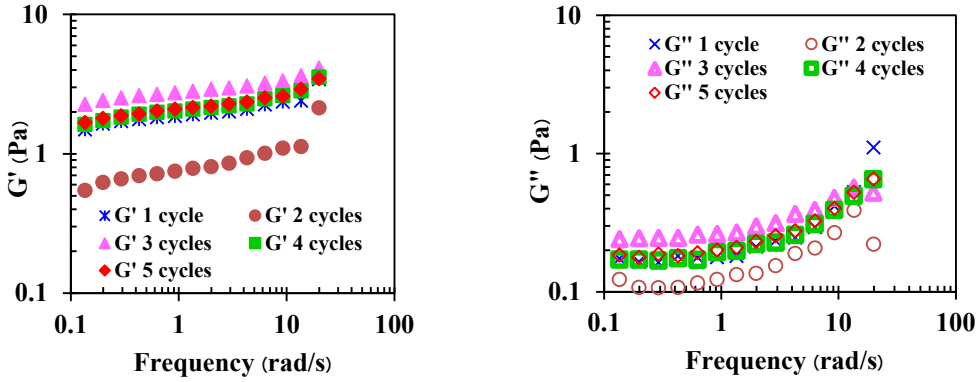


**Figure 4** G' (filled symbols) (a) and G" (open symbols) (b) of NFC suspension from pineapple cores (1 % solid content, by wet weight) after passing through a microfluidizer for 1 - 5 cycles.

The apparent viscosity can be observed with steady shear testing, as illustrated in **Figure 5**. Increasing the number of passes through the microfluidizer resulted in similar apparent viscosities for NFC from pineapple cores at concentrations of 0.5 and 1 % (by weight), ranging from 3.35 - 3.85 cPs and 3.75 - 4.40 cPs, respectively. Increasing the concentration to 1 % (by weight) led to a slight increase in apparent viscosity, which may be attributed to the fiber particle size being in the micrometer or nanometer range. The nanometer-scale size does not significantly affect the increase in apparent viscosity of the NFC from pineapple cores.

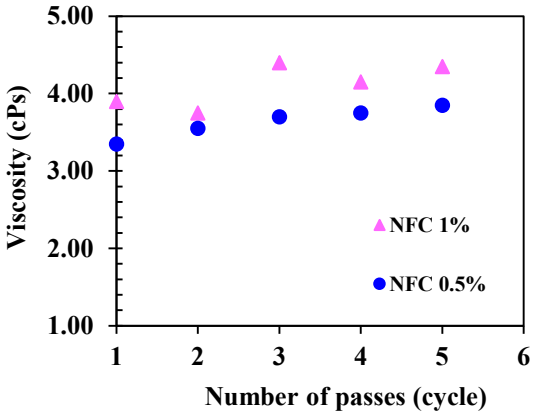


**Figure 5** Relationship between microfluidization cycles (1 - 5) and the viscosity of NFC suspension concentrations (0.5 and 1 % solid content, by wet weight).

Generally, viscosity depends on the shape, size, and charge of molecules, with larger fibers exhibiting higher viscosity compared to smaller fibers of the same type. Larger particles can increase the viscosity of suspensions, while smaller fibers reduce it. Specifically, cellulose fibers smaller than 110 microns do not increase viscosity [14]. Thus, NFC from pineapple cores (with an average fiber diameter of 62.46 - 80.25 nm and average length of 18.03 - 18.64 µm) does not significantly enhance viscosity.

However, increasing the concentration of NFC from 0.5 to 1 % (by weight) shows an increasing viscosity trend, indicating that higher fiber concentrations result in stronger fiber entanglement and higher viscosity. Additionally, NFC from pineapple cores processed through multiple cycles of a microfluidizer displays a stable suspension, suggesting its potential as a stabilizer to maintain food stability.

## Conclusions

The production of NFC from pineapple cores via autoclaving at 121 °C with 0.1 M citric acid for 20 min yields higher cellulose purity than boiling at 100 °C, establishing a green-chemical process suitable for food applications. This rapid 20-minute extraction increases the cellulose content from 45.64 to 72.77 %, producing high-purity NFC comparable in quality to traditional alkaline and bleaching methods. This single-step process reduces chemical complexity, effectively removing hemicellulose, pectin, and sugars, although it does not affect lignin solubility. SEM analysis reveals significant fiber structural breakdown at high temperatures, resulting in a more porous surface. Microfluidization further reduces fiber diameters after 1 - 5 cycles, while rheological analysis shows NFC with G' values exceeding G", indicating gel-like behavior and suspension stability. The results of this study indicate that alternative drying methods should be explored to maintain NFC stability, given the potential hydrogen bond disruption during drying. Future work should investigate NFC interactions with food components, considering factors like pH, composition, and processing temperatures to optimize stability in food systems.

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