

## Effect of Homogenization Pressure on Bacterial Cellulose Membrane Characteristic Made from Pineapple Peel Waste

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### ABSTRACT

Many studies were conducted to maintain the environment by reducing the waste, especially pineapple peel waste. This study aims to explore the effect of various pressure of the homogenization process on bacterial cellulose membrane surface morphology and structure produced using extract of pineapple peel waste. The methods include the preparation of pellicle samples from the product of the fermentation process of *Acetobacter xylinum* using a medium from the extract of pineapple peel waste. Bacterial cellulose pellicles were crushed using a blender. Mashed bacterial cellulose pellicle was homogenized in High-Pressure Homogenizer with pressure variation of 150 bar, 300 bar, 450 bar, dan 600 bar and then cast into a mold. The bacterial cellulose solutions were dried in an oven at 60°C for 8 hours. The dried bacterial cellulose membrane was analyzed using XRD for the structure and SEM analysis for the morphology. The results indicate that the crystalline properties of BCM were shifted after being treated by various pressure processing in a High-Pressure Homogenizer. It was found that the High-Pressure Homogenizer with higher pressure reduced the peak intensity, decreased crystalline index from 87% to 70%, and decreased the degree of crystalline from 88% to 77% without changing the cellulose structure. The higher pressure of the homogenization process causes the porosity of the membrane to be decreased.

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**Keywords:** Bacterial cellulose, high-pressure homogenizer, morphology, pineapple waste, XRD

## I. Introduction

Cellulose is an abundant biopolymer resulted in almost plants. It is composed of many compounds such as lignin, pectin, and hemicellulose, so its need many process and energy to purify plant cellulose. In this case, bacterial cellulose (BC) offers a higher purity source of cellulose than plant cellulose [1]. BC is produced from bacteria secretion in a fermentation medium that acts as a nutrient source, and the most famous bacteria used in the fermentation process for producing BC is *Acetobacter xylinum* instead of *Gluconobacter xylinum* [2].

Nowadays, many studies have been conducted to maintain the environment by reducing waste. Agricultural waste has been used as carbon source for many applications. Indonesia is a big product of pineapple till to 200,000 tons per year [4]. Pineapple peels are types of substrates that can be utilized as a carbon source. Thus, it is big opportunities to develop pineapple peel waste as a nitrogen and carbon source [3] to produce BC. Therefore, this



study was conducted to apply pineapple peel waste extract as a medium source in BC production and for the waste's utilization.

BC, as natural fibers, has biodegradable properties. It makes BC as one of the favorite materials for various applications. In its natural state, BC has very good properties with a purity of almost 100%. It is getting advantages for getting pure cellulose compared to plant cellulose for future development. The BC structure is constructed of a network of fibrils with a high surface area, making it porous. Its hydrophilic property causes BC has a high water holding capacity. The high purity of BC implied a high crystallinity property causing high mechanical properties [1]. But, BC still has some limitations in the utilization of this biopolymer. So, a composites system was introduced to overcome the limitations of BC. By composting the BC, it can get advantages functions such as; photocatalyst, optical, anti-bacterial, anti-fungal, bio-regeneration, and conductivity properties [5]. In this case, nanoparticles are a way to make nanocomposites by dispersion methods and BC regeneration. A regeneration method is needed to disintegrate BC fibers into nano-size.

High Pressure Homogenizer (HPH) is a method to fibrillate BC into nanosized. HPH promotes traditional non-thermal processes for emulsion stabilization and improves color uniformity, viscosity, taste, and texture in the food industry [6]. HPH has been applied for the treatment of micro-fibrillated cellulose suspensions in cellulose [7][8], pulp, and bagasse [9]. It was reported that the HPH process was applied in the isolation of cellulose nanofibers to reduce the diameter of the nanofibers from 117 to 67 nm through crushing force, shear stress, and cavitation [10][11]. Furthermore, ease of upgrading and continuous operation are benefit of the HPH process. The problem of homogenizer clogging, which occurs cause of the higher fiber diameter, can be suppressed either by increasing the number of cycles or the pressure in the HPH process.

Mechanisms of cavitation, friction, shear, turbulence, rapid pressure drop, velocity, compression, and heat initiate the HPH process [12]. Its process is free from organic solvent and has highly efficiency. Repeatedly application of HPH with pressure treatment can produce high cellulose nanofibers and high fibrillation rates [13][14]. HPH leads to increased entanglements because of nanoformation in the networks structures [15]. HPH processes on BC reduce dimensions from microscales to nanoscales [16]. Microstructural information is needed in engineering BC film to find out the film structure relationship to mechanical properties and parameters in expansion, water release, and water absorption. This study purposed for analyzing the influence of the pressure in HPH process on the characteristics of the BC membrane (BCM).

## II. Material and Methods

### *Materials*

The BCM synthesis applied bacteria species of *Acetobacter xylinum* (Microbiology Laboratory, UM, Malang, Indonesia). The main medium for the fermentation process used an extract of pineapple peels. Chemical reagents such as glucose ( $C_6H_{12}O_6$ ), distilled water ( $H_2O$ ), sodium hydroxide (NaOH), ammonium sulfate  $(NH_4)_2SO_4$ , and acetic acid ( $CH_3COOH$ ) were used in technical grade [6].

### *Synthesis of BC*

BC production was conducted according to previously published methods [17]. 5 kg pineapple peel waste was blended and then filtered to get the extract. 10 L of water was

added into a container and boiled on hot plate equipment. Ammonium sulfate 0.5% (w/v) and sugar 10% (w/v) were added into suspension and adjusted the pH using acetic acid until about pH 4.5. The boiled medium was then cooled until room temperature. *A.xylinum* 10% (v/v) was added to the culture medium, and after 10 days pellicle was then floated on the medium and then harvested. Pellicle boiled in a sodium hydroxide (NaOH)1% at 90C for an hour, then rinsed with water until neutral.

#### *Synthesis of BCM*

The cleaned pellicle was crushed using a high-speed blender. Water as much as 750 ml was added into suspension of 250 ml, then suspensions were inputted into HPH, then the homogenization process was conducted at a pressure of 0 bar, 150 bar, 300 bar, 450 bar, and 600 bar at 5 cycles. The BC solution from HPH was cast into a mold and dried in an oven at 60°C for 8 hr. BCM was saved in a dry box.

#### *Morphological observation*

Observation of the surface morphology of BCM was conducted under SEM, FEI, Inspect-S50. Before observation, specimens were coated using a gold coater (SC7-620 Emitech).

#### *Structure Analysis*

The structure of BCM includes the degree of crystallinity, and crystalline index was conducted using XRD (PanAnalytical, X-Pert Pro). Cu-K $\alpha$  radiation is used at  $\lambda=1.54 \text{ \AA}$ , at 30 mA and 40 kV. Scanning was conducted in the  $2\theta$  range from 10° to 40°. The degree of crystallinity (%Cr) and crystallinity index (CrI) were calculated using Segal's equation.

### **III. Results and Discussions**

#### *Morphology analysis*

The morphology of BCM before and after HPH treatment with various pressures (0 bar, 150 bar, 300 bar, 450 bar, and 600 bar) are shown in Figure 1. The membrane shows a change in pore size to nanometers, and the cellulose fibers are clearly split and peel off into nano-sized particles with increasing pressure in the HPH process.

Figure 1a, BCM with HPH pressure of 0 bar shows the highest amount of porosity and the fibers are still not completely split. Figure 1b of BCM with HPH pressure of 150 bar shows that the fibers begin to split and peel to form particles to fill the pores between the random fiber structures of BC. Figure 1c BCM treated with HPH pressure of 300 bar showed reduced porosity compared to Figures 1a and b. However, the BC fibers were still not fragmented and completely exfoliated. Figures 1d and 1e, BCM with HPH pressures of 450 and 600 bar show the least amount of porosity. Even with increasing pressure treatment in HPH processes, the BC fibers completely disintegrate into uniform particles.

#### *BCM structure analysis*

The form of diffractogram of HPH treatment with cycle and pressure variations (150 bar, 300 bar, 450 bar, and 600 bar) is presented in Figure 2. Diffractograms of BCM before and after HPH treatment using various pressures results in crystallinity degree, crystallinity index,  $2\theta$  angle, peak height, and crystal size are presented in Table 1.

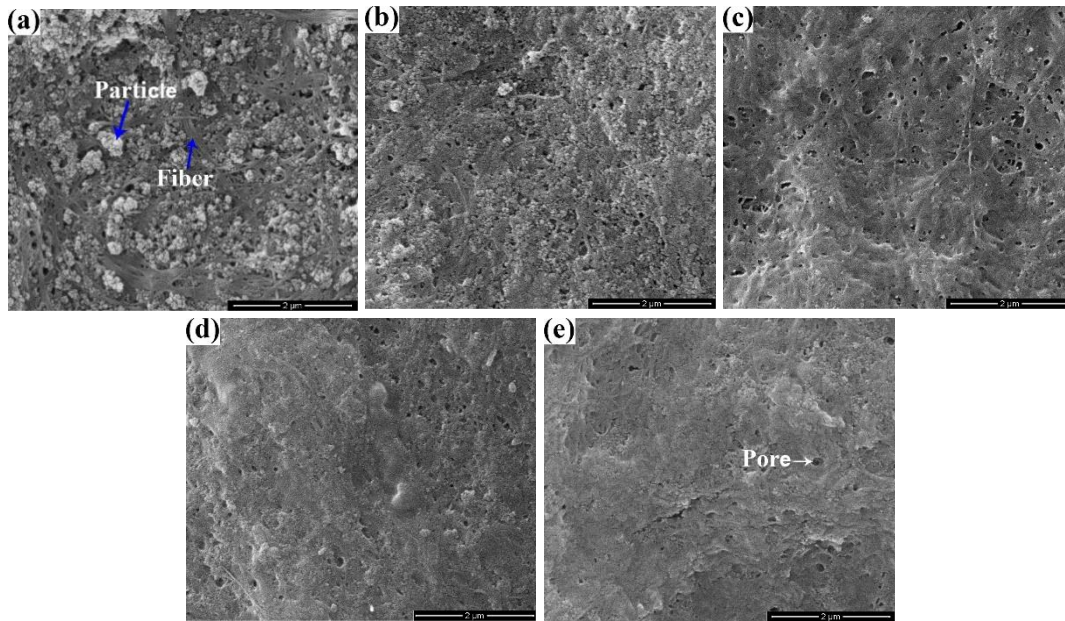


Fig. 1. Surface morphology of BC membrane: (a) HPH 0 bar, (b) HPH 150 bar, (c) HPH 300 bar, (d) HPH 450 bar, dan (e) HPH 600 bar

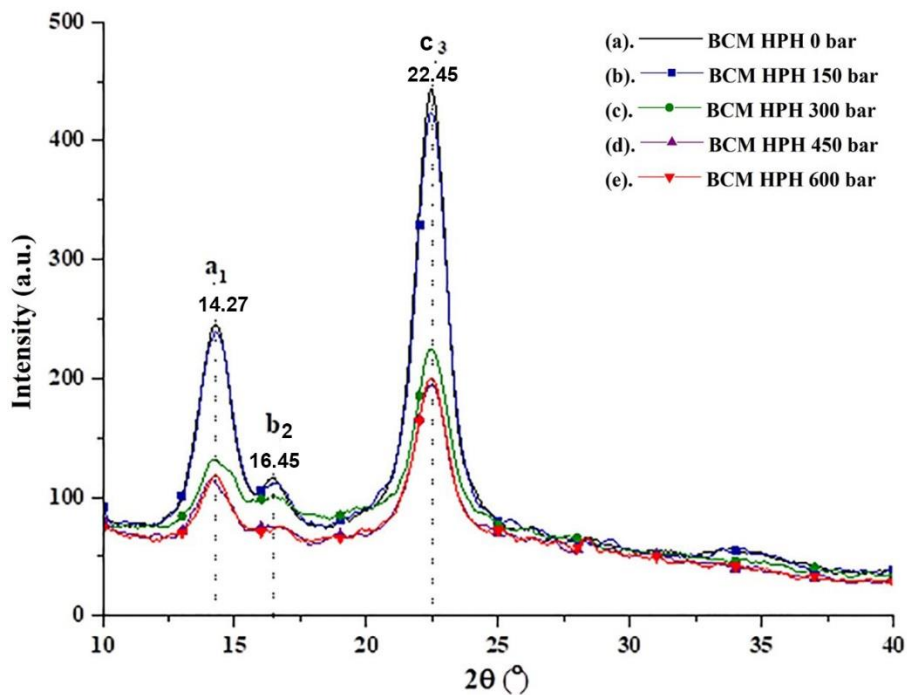


Fig. 2. Diffractogram of BCM produced by various HPH pressure

Table 1 shows the  $2\theta$  angle (a1) is between  $14.27^{\circ}$ – $14.33^{\circ}$ .  $2\theta$  angle (b2) is between  $16.45^{\circ}$ – $16.83^{\circ}$ ,  $2\theta$  angle (c3) is between  $22.45^{\circ}$ – $22.45^{\circ}$ . The lowest peak intensity value (a1) is 116.62, and the highest value is 244.82. The lowest peak intensity value (b2) is 76.76, and the highest value is 115.42. The lowest peak intensity value (c3) is 196.81, and the highest value is 439.41. The lowest crystallinity index value is 62%, and the highest value is 87%.

The value of the lowest degree of crystallinity is 72% and the highest value is 88%. The lowest crystal size value is 4.76 nm, and the highest value is 5 nm.

**Table 1.** Parameter of BCM resulted from XRD analysis

Parameter		High Pressure Homogenizer (HPH) Pressure (Bar)				
		0	150	300	450	600
2 $\theta$ degree	a1	14.27	14.23	14.09	14.33	14.27
	b2	16.45	16.47	16.35	16.83	16.49
	c3	22.45	22.43	22.45	22.45	22.45
Peak Intensity	a1	244.82	213.97	116.62	118.87	120.72
	b2	115.42	107.83	76.76	76.83	90.46
	c3	439.41	391.47	196.81	201.71	213.16
Crystalline Index (CI%)		87	84	62	70	70
Degree of Crystalline (% Cr)		88	86	72	77	77

#### IV. Conclusions

The present study reports the influences of HPH pressure on BCM properties. After different pressure of the HPH process, BCM was prepared. After HPH treatment with various pressure, the HPH with higher pressure cause a reduction in the peak intensity, then the crystalline index decreases from 87% to 70%, and also degree of crystalline decreases from 88% to 77% without raising a new peak in diffractogram. In the future, the engineered BCM could be developed into many applications in fields of engineering such as membrane filter, membrane separator in battery, sensor, active paper, etc.

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