FTIR analysis of polyethylene glycol treated bacterial cellulose pellicle

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Abstract. This research identified the effect of additional polyethylene glycol (PEG) in bacterial cellulose made from pineapple peel waste. The produced pellicles were soaked in PEG solution with 0%, 1%, 2.5%, and 5%. Then, they were homogenised using an ultrasonic homogeniser for 15 minutes before dried using the freeze-drying method. The functional groups were observed using FTIR. The functional groups showed the presence of cellulose I and cellulose II at 439.08 cm⁻¹, 2895.15 cm⁻¹, 1963.53 cm⁻¹, 1633.71 cm⁻¹, 1315.45 cm⁻¹, 839.03 cm⁻¹ wavenumbers. Additional PEG caused an increase of the C-H bond at 1963.53 cm⁻¹ wave number.

1. Introduction

Nowadays, people become aware of waste management, be it biodegradable or non-biodegradable waste. Indonesia is a country that produces the largest pineapple globally besides Thailand, the Philippines, Malaysia, Hawaii, Ivory Coast, Kenya, Brazil, Taiwan, Australia, India and South Africa [1]. High pineapple production means high pineapple peel waste. An effort to manage pineapple peel waste is by using it as a culture to develop bacterial cellulose (BC). Pineapple peel has fructose, glucose, and sucrose that can be used as bacterial cellulose cultivate.

Bacterial cellulose is a polymer network measuring 20–50 nm and has a 3-dimensional structure derived from 3-dimensional hydrogel bonds [2]. Bacterial cellulose has the same structure as plants cellulose. Besides, it also has high mechanical strength, crystallinity, and hydrophilicity [3]. It also has purity, high biocompatibility, and degraded properties [4–6]. Hence, cellulose is often applied to packaging, gas barriers, novel drug delivery systems, antibacterial membranes, composite membranes, implants, wound dressings, and magnetic films [7–14]. Bacterial cellulose can also be used as an air filter without generating non-recyclable waste.

Producing bacterial cellulose membrane for filter application requires attention in the drying method. The freeze-drying method [15–19] with a perforated surface of the specimen is suitable for air filter applications. Besides the drying process, the material as a surfactant also requires attention because it can influence the bond characteristic of bacterial cellulose and other metal. Infrared spectroscopy is a method to analyse a bond produced by a material, bacterial cellulose included. Thus, a further review of
the additional surfactant effect on bacterial cellulose bond characteristic was needed using infrared spectroscopy. This research studied the effect of additional surfactant on bacterial cellulose properties using additional Poly Ethylene Glycol (PEG) 4000 with 0%, 1%, 2.5%, and 5% variations. This study aimed to observe those additional PEG as surfactant on bacterial cellulose pellicle bonds using the Fourier Transform InfraRed (FTIR).

2. Materials and methods

2.1. Materials
Pineapple peels were obtained from a plantation in Blitar, East Java, Indonesia. The bacteria to make the cellulose were *Acetobacter xylinum*, Glucose, NaOH, and Poly Ethylene Glycol (PEG) 4000, and they were obtained from CV Makmur Sejati, Malang, East Java, Indonesia.

2.2. Bacterial cellulose synthesis
300 gram of pineapple peel was washed, crushed, and extracted into juice. The extraction used 2 L of water, 10% (w/v) sugar, and acetic acid to achieve a 4.5 pH. Then, 20% of the *A. xylinum* bacteria was added into the culture before fermentation for ten days at 30°C temperature. The produced pellicles were transparent and floated on the solution. After harvesting, the pellicles were rinsed with distilled water to remove bacteria and other impurities. The pellicles were soaked in 1% NaOH for 2 hours, stirred using a magnetic stirrer at 350 rpm speed, and rinsed with distilled water until they reached 7.0 pH.

2.3. Mixing process
The celluloses were cut in 5 cm x 5 cm dimensions, soaked in the PEG solutions (0%, 1%, 2.5%, and 5%), then put into ultrasonic homogeniser for 15 minutes in 300 W power 20 kHz frequency.

2.4. Drying process
The bacterial cellulose pellicles were put into vacuum freeze-drying (Berkley Scientific, China) at 1 Pa and were condensed at -30°C for two days. The dried samples were stored at 58% humidity and 25°C temperature.

2.5. FTIR analysis
The bacterial cellulose functional group was analysed using the Fourier Transform Infrared spectrometer/FTIR (Shimadzu IR Prestige-21). The spectrums were recorded in 4 cm⁻¹ resolution within the 400–4000 cm⁻¹ range.

3. Results and discussion

3.1. Synthesis results
Researchers succeeded in synthesising bacterial cellulose using pineapple peel waste. Fibre cellulose, as the production of *Acetobacter xylinum* bacteria, had a different structure than plant fibre. Bacterial cellulose has a higher purity of up to 100% than cellulose in plants containing hemicellulose and lignin [20]. However, bacterial cellulose also contained impurities such as *Acetobacter xylinum* bacteria, nucleic acid, and protein from pineapple skin extract that needed to be removed using NaOH. Figure 1a shows the wet cellulose after cleaned with NaOH and cut into 5 cm x 5 cm. Figure 1b displays the cellulose after soaked in PEG 4000.

Figure 2 exhibits the plasticisation mechanism of bacterial cellulose with PEG 4000. During the drying process, the bacterial cellulose chain bond tissue increased the hydrogen bonds, limiting the relative movement between the fibres. This condition caused bacterial cellulose to have robust and brittle properties [21]. PEG 4000 not only acted as surface coverage but also penetrated the bacterial cellulose tissue. As a result, the hydrogen interactions between bacterial cellulose were reduced, and the relative movement between the fibres became freer [21].
Figure 1. Bacterial cellulose synthesis results (a) freeze dry without PEG (b) with additional 1% PEG (c) with additional 2.5% PEG (d) with additional 5% PEG

Figure 2. Bacterial cellulose tissue illustrations (a) without PEG (b) with PEG [21]

3.2. FTIR analysis

Table 1 and Figure 3 show the FTIR results of pure bacterial cellulose specimen and additional PEG 4000. Based on the images, before and after additional PEG 4000 bacterial celluloses were dominated
with O-H bond at around 3439.08 cm\(^{-1}\). It indicated a cellulose type I. There was the C-H bond at 2895.15 cm\(^{-1}\) wavenumbers, which indicated cellulose type II. There was an intensity increase of the C-H bending bond. The O-H characteristics of water absorption are also shown in the appearance of the C-O bond at 1633.71 cm\(^{-1}\). The wavenumber of 839.03 cm\(^{-1}\) saw a transmittance peak following the percentage increase of PEG 4000.

In the O-H bond at 3459 cm\(^{-1}\) wavenumbers, the more curved graph indicated the change in intensity value. The graph identified the crystallinity of cellulose I and cellulose II on the O-H bond [22]. Following the additional PEG 4000, the hydroxyl group bond decreases. The O-H graph change between 3750–3200 cm\(^{-1}\) and intensity increase in C-H bending bond at 1936.53 cm\(^{-1}\) showed the additional PEG 4000 identity. Adding PEG 4000 did not produce a new bond but intensity improvement in some bonds. This occurrence was because the functional groups of cellulose ((C\(_6\)H\(_{10}\)O\(_5\))\(_n\)) and PEG 4000 (H(OCH\(_2\)CH\(_2\))\(_n\)OH) consist of mostly the same bonds and do not have much difference in wavelengths [23].

**Table 1. Bacterial cellulose functional group analysis**

| Wavenumber (cm\(^{-1}\)) | Bond          | Component                        |
|--------------------------|---------------|----------------------------------|
| 3439.08                  | O-H bond      | Cellulose I and Cellulose I      |
| 2895.15                  | Alkanes C-H bond | Cellulose I                      |
| 1963                     | C-H           | Polyethylene Glycol              |
| 1633.71                  | C-O bond      | Cellulose I and Cellulose II     |
| 839.03                   | C-H           | Cellulose I                      |

![Figure 3. FTIR analysis result of bacterial Cellulose with additional PEG](image)

**Figure 3.** FTIR analysis result of bacterial Cellulose with additional PEG

### 4. Conclusion

Researchers succeeded in adding PEG 4000 into bacterial cellulose bond using immersion with ultrasonic homogeniser method. Additional PEG 4000 did not change the initial bond of bacterial cellulose, only influenced the intensity values of the bonds. Particularly in C-H bending bond and signed by deeper intensity valley graph at 1963.52 cm\(^{-1}\) wave number. In the future, this material from pineapple peel waste will be developed as filtering material for air and water treatment.
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