EFFECT OF ALKALI-TREATED COCONUT FIBER IN CHITOSAN FILM ON PHYSICAL AND MECHANICAL PROPERTIESFOR MEDICAL APPLICATION

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ABSTRACT

This study aimed to compare the physical and mechanical properties of chitosan films mixed with cellulose fiber derived from coconut husks under various conditions. These conditions included different concentrations of NaOH used for treating the cellulose fiber and varying amounts of fiber incorporated into the chitosan film. The experiments, testing and characterized physical and mechanical properties of the resulting films were conducted in a laboratory setting. The treatment of coconut fiber with NaOH enhanced the tensile strength of the chitosan films, while simultaneously reducing their %elongation. Additionally, films containing 0.25g of cellulose fiber exhibited a lower swelling ratio compared to those with 0.10g of fiber. However, variations in fiber quantity and NaOH concentration did not significantly affect thermal stability. Based on the findings, it can be concluded that the chitosan film formulated with 4% NaOH and 0.25g cellulose fiber exhibited the most favorable physical and mechanical properties for future development.

Keywords: Chitosan films, coconut fibers, tensile strength, swelling ratio

1. INTRODUCTION

Chitosan has seen remarkable advancements as a film material in medical applications over recent decades. Its unique structure and beneficial properties, including biodegradability, antimicrobial action, and biocompatibility, have made it a versatile tool in areas like wound healing, drug delivery, tissue engineering, and infection control. Recent innovations, such as combining chitosan with other materials or modifying it chemically, have further enhanced its performance and extended its use across various medical fields. Originally investigated in the late 1980s for drug delivery systems, chitosan's applications have since expanded to include wound healing, tissue engineering, and regenerative medicine. Its biocompatibility**,** biodegradability**,** and antimicrobial properties[1] make it ideal for use as scaffolding in tissue repair and as a carrier for controlled drug release, effectively supporting tissue regeneration and repair[2, 3]. Recent advancements aim to enhance its antimicrobial effects and refine its physical properties, improving its versatility in various medical fields, such as ophthalmology and dentistry, and reinforcing its role as an essential biomaterial in modern medicine[4]

Natural fibers present numerous advantages over traditional reinforcing materials since their suitable specific strength, low cost, low density, biodegradability, favorable thermal properties, enhanced energy recovery. Fibers like sugar crane, sisal, pineapple, and abaca[5-14] have been widely studied as reinforcements and fillers in composite materials. However, coconut husk has few studied researched in biodegradable composites, despite its abundance as a by-product of the coconut industry (e.g., coconut oil, coconut milk) and its use as a fuel source in the food industry. Alkali treatment is considered an effective technique to improve the mechanical properties of these composites by modifying fiber surfaces, enhancing adhesion between the fiber and the matrix. Ray et al..[15-17] reported the physical and mechanical properties of composites reinforced with alkali-treated jute fibers. Similarly, Samal et al. [18] treated jute fibers with 2% NaOH solution for 1 hour, and Rout et al.[19] observed increasing of tensile strength 26%, flexural strength at 15%, and a improvement in Charpy impact strength 20% after treating coir fibers with a 2% NaOH solution for 1 hour.

This study aimed to develop films with enhanced strength and sustained swelling properties in solutions, utilizing coconut fiber as a matrix filled with natural fibers synthesized in situ. The resulting films were characterized and investigated in this report. Ensuring that the film characteristics within an appropriate range for medica applications[20] was also a key focus of this study.

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2. MATRIALS AND METHODOLOGY

2.1 Materials and Methods

Chitosan powder from SN crab shell, high viscosity (*M*W: 125-500 kDa, LOT NO. SNCS-210120/01) was purchased from BIO21 THAILAND. Coconut husk was purchased from Thailand's tree market. Sodium hydroxide (NaOH) and acetic acid were purchased from RCI LABSCAN THAILAND.

2.2 Preparation of pure chitosan films

One g of chitosan powder was dissolved in 100 mL of 0.5% (v/v) acetic acid solution and stirred continuously while heating at 60°C for 1 hour. After this period, the solution became fully homogeneous and colorless. A volume of 50 mL was then poured into a 90x15 mm petri dish, and the chitosan films were dried at 60°C for 24 hours. The result was a round, homogeneous, and colorless chitosan film.

2.3 Preparation of alkali-treated coconut fibers

To prepare alkali-treated coconut fibers, 20 g of raw coconut husk were mixed with 100 mL of NaOH at concentrations of 2% and 4%, then heated at 50°C for 2 hours to remove impurities and organic residues. The alkali-treated coconut fibers was separated, thoroughly washed with distilled water, and adjusted to pH 7 using 0.5%(v/v) acetic acid. Next, the alkali-treated coconut fibers were dried at 70°C for 72 hours. To obtain fibers sized 400-600 µm, the dried fibers were ground and sieved. These prepared fibers were then combined with chitosan film for further use.

2.4 Preparation of chitosan films with alkali-treated coconut fiber

Chitosan powder (1g) was dissolved in 100 mL of the 0.5% (v/v) acetic acid solution at 60ºC for 1h. After the solution was completely dissolved, added 0.1 and 0.25g of alkali-treated coconut fibers coconut fibers. Stirred the suspension until well combined. Next, the solution was poured into petri dish (size 90x15 mm) 50mL and dried at 60°C for 24hs.

2.5 Film Characterizations

2.5.1 Analysis of chemical components with Fourier-transform infrared spectroscopy (FT-IR)

FT-IR)Fourier-transform infrared spectroscopy(data for the samples were records by Thermo Scientific™ Nicolet™ iS™5 FT-IR Spectrometer. Use the powder obtained from fiber separation, including untreated powder as well as powder treated with 2% and 4% NaOH.

2.5.2 Surface Morphology

The cellulose morphology was studied by scanning electron microscopy (SEM) techniques. The samples were prepared into 1x1 cm per piece and coated with Au. To observed using an accelerating voltage of 5 kV. SEM revealed porosity and surface roughness of coconut fiber.

2.5.3 Physical properties

2.5.3.1 Thickness

The thickness of chitosan film with alkali-treated coconut fiber was measured by vernier caliper and reading accurately at different area of films and averaging it.

2.5.3.2 Swelling Ratio

The sample were cut in 1x1 cm size and dried at 60ºC for 10 mins. After initial weighing, they were kept in PBS water (pH 7.4) for 3hs. Then take it out and removed the excess water from surface of film with Kimwipes paper. The swelling ratio of the films were calculated using the equation below (1).

$$\frac{Ww - Wd}{Wd} \times 100 \quad \text{.....(1)}$$

where *W*w and *W*d are the sample mass at wet and dried state, respectively. All the swelling experiments were taken in triplicate and average values are presented in the data.

2.5.4 Mechanical properties

Tensile strength and %Elongation at break of the samples were measured by Universal Testing Machine Shimadzu, AGS-X 500N The samples were cut in dimension of 1 X 5 cm with speed of 50 mm/ min and 500 N load cell.

)stress($\boldsymbol{\sigma = \frac{F}{A}}$ ……(2)

)strain) $\boldsymbol{\varepsilon = \frac{\Delta l}{l_0}}$ ……(3)

%Elongation $\boldsymbol{\varepsilon = \frac{\Delta l}{l_0} \times 100}$ ……(4)

Modulus $\boldsymbol{= \frac{\sigma}{\varepsilon}}$ ……(5)

3. RESULTS AND DISCSSIONS

3.1 Fourier-transform infrared spectroscopy (FT-IR)

The treated coconut fiber was analyzed the chemical components and molecular function by using FT-IR (Figure. 1). The result demonstrated a vibration peak of acetyl group (C=O) in hemicellulose at the wavelength 1720 cm^{-1}. Furthermore, the absorbance was decreased to 1580 cm^{-1} as the vibration of the aromatic bond in lignin. Therefore, increasing of sodium hydroxide concentration for coconut pretreatment able to remove more lignin and hemicellulose from coconut fiber.

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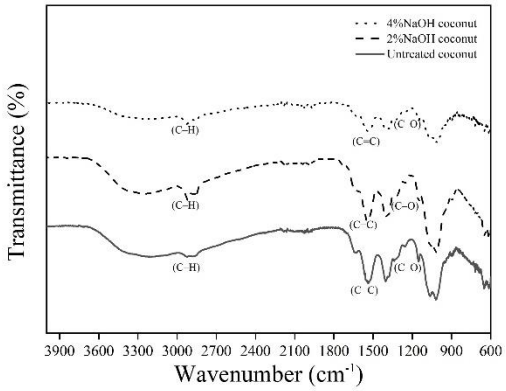


Figure 1. FT-IR spectra of untreated and treated coconut fiber with 2% and 4% NaOH

3.2 Surface Morphology Characterization

The surface morphology of coconut fiber was studied using SEM to measure the porosity of coconut fiber as show in fig 2. The results reveal that the surface structure of untreated showed smooth and non-porous on coconut fiber (figure.2 a). After treated at 2% and 4% NaOH, the cellulose from coconut fiber showed more roughness and some porous on their surface (figure.2 b,c). From these results, it might be These might be hemicellulose and lignin that were removed[19].

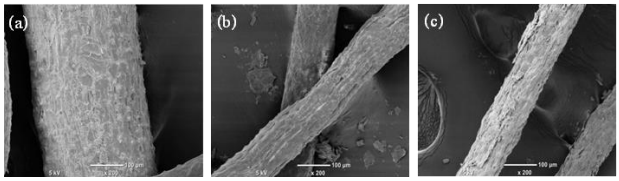


Figure. 2. SEM image of coconut fiber surface: (a)non-treated, (b)treated with 2% NaOH and (c)treated with 4% NaOH.

Due to the hydroxyl group of hemicellulose, it could be oxidized by sodium hydroxide, and the structure was broken, resulting in the fiber being dissociated and more dispersed; this was called “fibrillation”[19]. Nanofibrils separated from the fiber surface. In samples without alkali-treatment, they remained embedded within the chitosan film, while in sodium hydroxide-treated samples, they were more uniformly dispersed.

3.3 Physical properties

The thickness of sample was measured by vernier caliper and reading accurately at different area of films and averaging it. The thickness was showed in Table 1. The degree of swelling ratio of chitosan film with alkali-treated coconut fiber was calculated from that comparing between 0.1g and 0.25g of alkali-treated coconut fiber are summarized in Table 1. The swelling ratio were measure by immersion in water due to partial dissolution of chitosan[24]. The results showed that at 0.25g swelling less than 0.1g because of cavity in fiber was replaced, causing less water to penetrate. These results increase moisture resistance of chitosan films[25].

Table 1. Physical properties of Chitosan films and chitosan films with alkali-treated coconut fiber.

Acetic Acid (%)	Average size of fiber (μm)	Coconut fiber (g)	Thickness (mm)	Swelling Ratio (%)
Untreated	400	0.1	0.015	49.2
2%	400	0.1	0.013	75.5
4%	400	0.1	0.011	74.6
Untreated	400	0.25	0.016	58.1
2%	400	0.25	0.015	65.0
4%	400	0.25	0.016	66.7
Untreated	600	0.1	0.012	64.5
2%	600	0.1	0.015	70.6
4%	600	0.1	0.012	74.7
Untreated	600	0.25	0.017	51.6
2%	600	0.25	0.014	63.3
4%	600	0.25	0.018	67.9

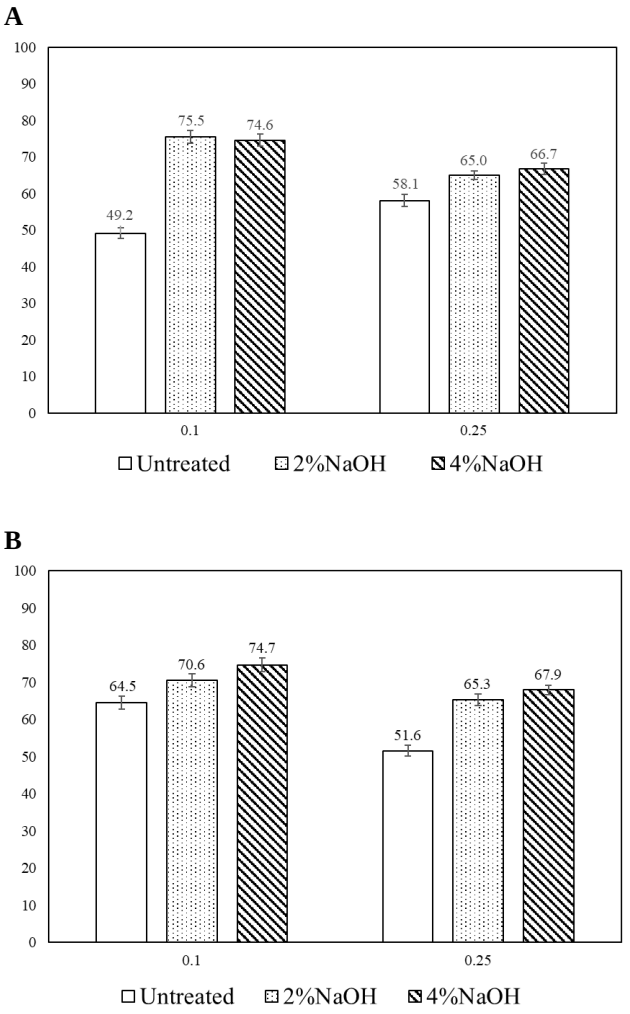


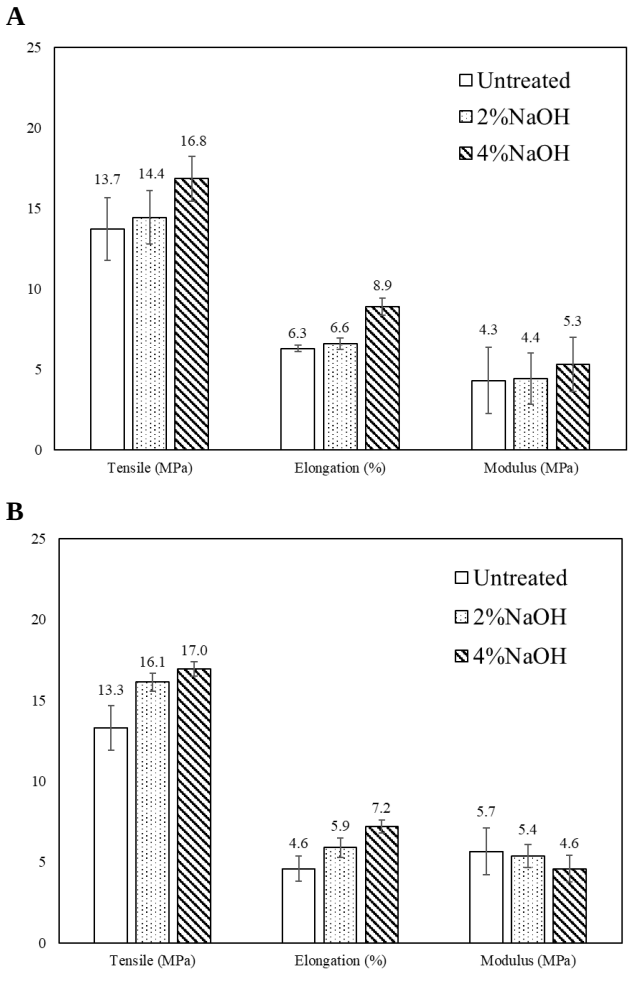
Figure 2. Swelling percentage of material treated with different NaOH concentrations at fiber sizes of **(**A**)** 400 and **(**B**)** 600µm **(**where 0.1 and 0.25 represent the amount of coconut fiber added (g))

3.4 Mechanical properties

The mechanical properties measured are tensile strength, %elongation and modulus of the samples. The results were in Table 2. The effect of fiber content on tensile strength were increased from 13 to 30 and from 11 to 28 with increase the content of fiber from no fiber to 0.25g for 400 and 600 µm, respectively. In addition, the results of %elongation and modulus showed consistency value with tensile strength, which decreased values when increased content of fiber. This indicated that the concentration of fiber significantly affected to the strength compared with the pure chitosan film. To evaluate the effect of NaOH concentration on the mechanical properties of chitosan film, specifically its tensile strength, it was found that the tensile resistance of the fiber treated with 4% NaOH was greater than that of the untreated fiber and the fiber treated with 2% NaOH. The increase in tensile resistance is attributed to the surface treatment of the fibers with NaOH, which affects fibrillation and leads to the removal of nanoparticles from the surface. This process enhances the cohesiveness between the cellulose fibers and the chitosan matrix.

Table 2. Mechanical properties of Chitosan films and chitosan films with alkali-treated coconut fiber.

Acetic Acid (%)	Average size of fiber (µm)	Coconut fiber (g)	Tensile (MPa)	Elongation (%)	Modulus (MPa)
Untreated	400	0.1	13.7	6.3	4.3
2%NaOH	400	0.1	14.4	6.6	4.4
4%NaOH	400	0.1	16.8	8.9	5.3
Untreated	400	0.25	13.3	4.6	5.7
2%NaOH	400	0.25	16.1	5.9	5.4
4%NaOH	400	0.25	17.0	7.2	4.6
Untreated	600	0.1	13.3	7.5	4.8
2%NaOH	600	0.1	15.1	8.2	4.8
4%NaOH	600	0.1	17.2	8.9	4.6
Untreated	600	0.25	13.4	5.1	5.3
2%NaOH	600	0.25	16.2	5.5	5.2
4%NaOH	600	0.25	18.5	5.9	5.8



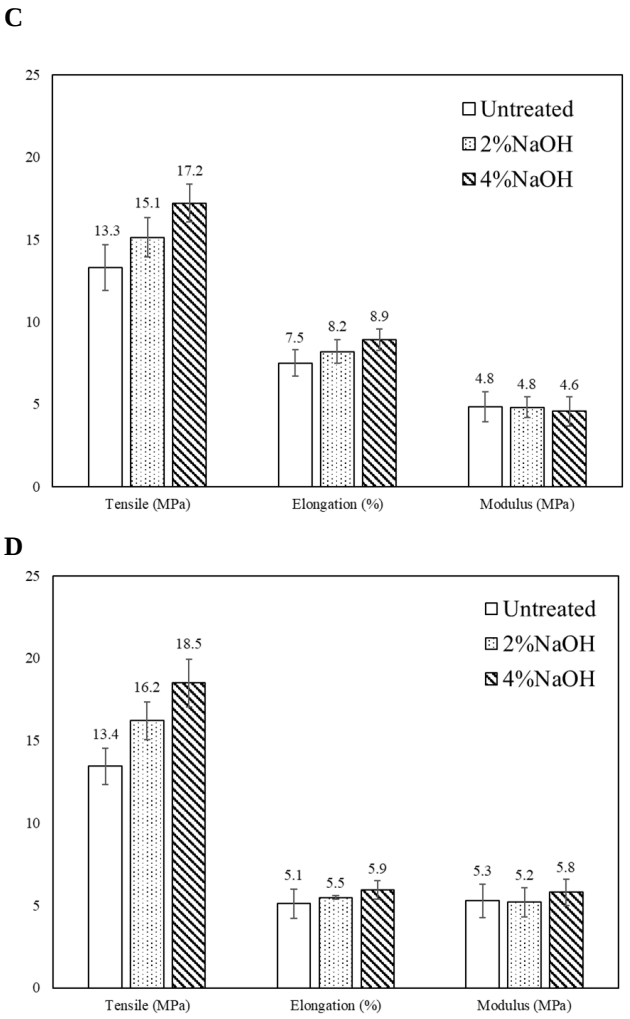


Figure 3. Tensile strength, elongation at break, and modulus of composites containing coconut fiber of (A) 0.1 g – 400 µm, (B) 0.25 g – 400 µm, (C) 0.1 g – 600 µm, and (D) 0.25 g – 600 µm, treated with different NaOH concentrations (Untreated, 2%, and 4%)

3.5 Thermal Stability

Figure. 4 illustrates the impact of fiber content in chitosan films on the residue weight loss curve (STA). The initial weight loss observed in all films is likely due to moisture loss within the film. Subsequently, at temperatures ranging from 220 to 350 °C, weight reduction is attributed to the degradation of cellulose and chitosan. For cellulose, thermal decomposition occurs within the range of 315 to 400 °C, resulting in the breaking of glycosidic bonds and subsequent rearrangement, as well as the removal of water[26, 27]. The chitosan component experiences thermal degradation in the range of approximately 250 to 450 °C[28], during which depolymerization of the chitosan chain occurs, leading to the rupture of glycosidic bonds between glucosamine and N-acetylglucosamine ring[29, 30] Finally, at temperatures between 350 and 550 °C, thermal destruction of the remaining material in the film takes place. Therefore, it can be asserted that the improved thermal stability of the chitosan film containing coconut fiber indicates the beneficial presence of coconut fiber within the film.

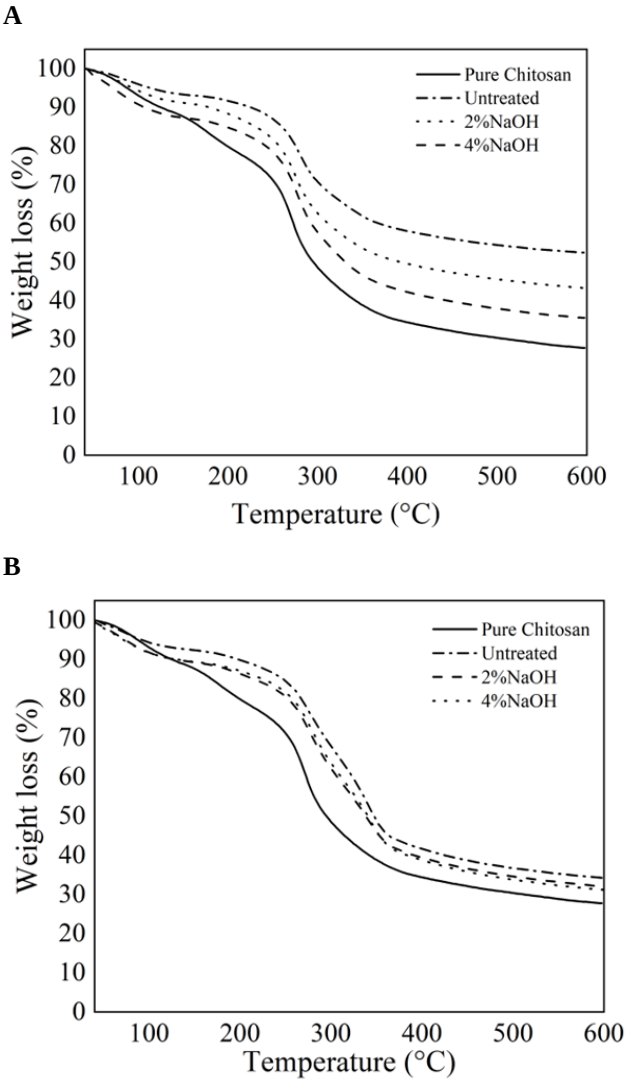


Figure. 4. STA curves of chitosan film with alkali-treated coconut fiber 600µm (A) 0.1g (B) 0.25g

4. CONCLUSION

The chitosan film with alkali-treated coconut fiber at a 4% NaOH concentration and containing 0.25 g of fiber, demonstrates high tensile strength and low swelling. These properties make it a promising candidate for future development. Additionally, it was found that treating the fiber with varying concentrations of sodium hydroxide affects swelling but does not impact thermal stability or tensile strength.

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