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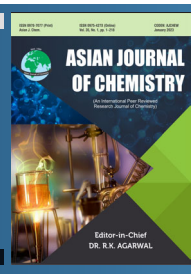


# Preparation and Characterization of Cattail Cellulose Films as Environmentally Friendly for Drug-Controlled Release System

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The objective of this work was to extract cellulose from cattail plants by treatment with 10 % (w/v) sodium hydroxide for 3 h at 80 ºC and bleached with 2% (v/v) sodium hypochlorite for 1 h. The sample was cut with 1% (v/v) sulfuric acid for 1 h to obtain 27.88% cellulose. The extracted cellulose was then prepared as films by casting method in a petri dish. The cellulose films were light brown and transparent with random direction of an orientation. The cellulose film increased transparency and flexibility by adding glycerol. The FTIR results clearly revealed the cellulose functional groups and some peaks being shifted when mixed glycerol. The cellulose films have a maximum decomposition temperature ($T_{d,max}$) of 310-340 ºC and the decomposition temperature decreases slightly when mixed with glycerol. Mixing methylene blue, a sample hydrophilic drug, the cellulose films have increased their texture brittle and found micro-porous on surfaces. In addition, the differences in peaks of the cellulose functional groups were also observed. These might be caused by the interaction formation of methylene blue functional groups and cellulose. The interaction resulted in an increase in the $T_{d,max}$ of the films. The release of the methylene blue pattern concerned the amount of mixed methylene blue. These results can be used as a basis for extracting cellulose from cattail plants and preparing cellulose films for specific applications such as drug-controlled release system.

**Keywords: Cattail, Cellulose extract, Film, Drug-controlled release system.**

## INTRODUCTION

The use of plastics has increased dramatically, which has resulted in a buildup of garbage made of plastic [1,2], resulting in the environment pollutions [3]. Eco-friendly materials have interested in studying and widely developed recyclable or biodegradable products from renewable sources [4]. The macromolecules, such as polysaccharides, are widely found in nature and can be used as materials alone, combining another polymer as well as blends and polymer composites. In recent, many works have been suggested to use lignocellulosic fibers as renewable raw materials as high value-added materials or polymers [5-8]. They have unique structures and properties, which are considered to instead the synthetic polymers [4]. The lignocellulosic materials (cellulose, lignin and starch) are the major constituents found in plants cell walls, especially in cotton, flax, hemp, jute, kenaf, sisal, ramie, curaua, pineapple, bamboo and coir [9]. However, their content and properties vary depending on various parameters such as origin and type of fibers, plant species and plant growing environment [7]. Among them, cellulose is the most abundant lignocellulosic fiber with a content of approximately 45%. It is organized and stabilized laterally by inter- and intra-hydrogen bonds from hydroxyl groups in the structure [10]. Cellulose is a homopolymer of glucose linked together *via* β-1,4-glycosidic bonds [11] mixed with hemicellulose and lignin [12]. The cellulosic fibers after the isolation process and can be used in different fields such as textiles [13] and polymer composites [14], food ingredient [15], pulp and paper [16], pharmacy [17], water pollution treatment [18], wine and beer [19]. These was due to cellulose has many good properties that make it suitable for applications such as biodegradation, transparent, ductile, low moisture content and easily plasticized [20]. Previously reports revealed that there are different methods to isolate cellulose from the natural plants including physical, mechanical and/or chemical treatments [7].

Cattail (*Typha angustifolia*), an aquatic plant which grows in wetlands and is widely spread in many countries including Thailand. It has been used in traditional Chinese medicine [21].

The cattail composed high biomass and has been used to remove heavy metal [22,23] or nitrogen compounds [24,25] in wastewater. Comparison to other natural fibers, the cattail fibers have not been used wildly as materials even its composed of over 70% of lignocellulosic fibers. This aquatic plant would be a source of a cheap and renewable raw material for cellulose production. The novel composite based-cattail fibers were firstly reported in 2011 [26]. So far, cattail fibers have not been reported as a source of cellulose as well as cellulose-based cattail fibers for further applications. Therefore, this work used cattail plants as raw material for extraction of cellulose for preparation films. The films were then characterized for their properties. The cattail based cellulose mixed glycerol as plasticizer films were also prepared as comparison. The extraction process of cellulose from cattail would be proposed and expand the range of cattail cellulose applications.

## EXPERIMENTAL

**Extraction of cattail cellulose:** The cattail plants were collected from the shallow fresh pond in Mahasarakham University, Maha Sarakham province, Thailand. The samples were washed with tap water, then cut into small pieces. Following a 24 h period of oven drying, the specimens were subsequently subjected to crush. The cellulose was extracted according to previously reported [27] with minor modifications. The sample powder ( 10 g) was pretreated with 100 mL of 15% NaOH (w/v) with stirring and warming at 80 ºC for 3 h. The slurry was then washed with distilled water until pH was neutral. After drying at room temperature for 24 h, the sample was then bleached by 5% NaOCl (w/v) at room temperature for 24 h. The bleached samples were washed to neutral pH. Finally, the bleached samples were hydrolyzed by 5% $H_2SO_4$ at 60 ºC for 8 h to obtain the cellulose. The cellulose solution was then stirred, washed with distilled water until it reached neutral pH and filtered. The extracted cellulose was kept in the refrigerator at 4 ºC before use.

**Preparation of cellulose films:** After placing the cattail cellulose (0.1 g) in the beaker, 100 g of deionized distilled water was added. The mixture was then stirred at 600 rpm for 30 min to obtain homogeneous texture. The prepared mixture was then cast onto a 4.5 cm diameter petri dish and left at room temperature allowing solvent evaporation. After drying, the cast films were peeled off and then kept in a desiccator. In case of glycerol mixed cellulose, 1.4 and 2.8% (v/v) of glycerol was added into the extracted cellulose, then stirred together. The process to form films was followed by the native cellulose films. The cellulose mixed with different concentrations of methylene blue (0.5, 1 and 2% w/w) was also prepared before pouring onto the petri dish as the same process discussed above. The later films were proposed for releasing profile study.

**Transparency observation:** The light transparency of the constructed films was determined using a UV-Vis spectrophotometer (Lambda 25, Perkin-Elmer, USA) as reported method [28]. Briefly, the films were cut into rectangular pieces and placed directly in the spectrophotometer cell. Then, the percentage transmittance of light at 660 nm through each film was measured in triplicate to calculate the average film transparency.

**Morphological observation:** All the dehydrated films were cut into small pieces with square shape. They were then observed their morphology under a scanning electron microscope (SEM) (JEOL, JSM-6460LV, Japan). Before observing, the pieces of film were fixed on the stub and then sputtered coated with gold to enhance conductivity before scanning.

**Secondary structure investigation:** All films were analyzed for their secondary structure by attenuated reflection Fourier transform infrared (ATR-FTIR) spectroscopy (Perkin Elmer-Spectrum Gx, USA) in the spectral region of 4000-400 $cm^{-1}$.

**Thermal analysis:** A thermogravimetric analyzer (TGA) (SDTQ600, TA-Instrument Co. Ltd., USA) was used for testing the thermal stability of the prepared films. In this experiment, thin films with a mass of 3-5 mg were subjected to a heating process under a nitrogen atmosphere ranging from 50-800 ºC, with a heating rate of 20 ºC/min.

**Releasing profile of methylene blue:** For releasing study, the cellulose-methylene blue blended films were immersed in distilled water with shaking at room temperature. After the interval times of 1, 2, 3, 4, 8, 16, 24 and 48 h, 2.5 mL of water was collected. The same new volume of water was added instead of the collecting volume and then the absorbance was measured by UV-Vis-spectrophotometer at 640 nm. The concentration of methylene blue released from the blend films was calculated by comparison to a standard curve.

## RESULTS AND DISCUSSION

In this work, the extraction yields of cellulose from cattail raw materials was found to be 27.88 ± 0.61 %. The extracted cellulose yield from this work obtained in higher content than the extracted cellulose from apple and kale pomaces as previously reported [29]. However, it was about 2-fold lower than the yield of the microcrystalline cellulose (55 %) extracted from sugarcane bagasse [30,31]. The variable cellulose content might be from plant varieties [32] and the chemicals and extraction methods used [30]. Additionally, the obtained cellulose yield was also influenced by several other factors including acid concentration, time, temperature and the ratio of the acid to cellulose [30].

Fig. 1 shows film transparency from digital images. The native cellulose film (a) has smooth and homogeneous throughout the film texture as like the cellulose-mixed glycerol films (b,c). All films have white pale, thin and can peel off from the petri dish. With glycerol, films increased their flexibility which could be bent more than the native cellulose film. Generally, glycerol is a common plasticizer that is used to increase flexibility. This was due to glycerol helping to increase polarity and decrease crystallinity of the polymers. The prepared films have light transmittance about 25%. This indicates that the glycerol does not interfere with film transparency.

General characteristic of cellulose films loaded methylene blue is shown in Fig. 2. The results indicated that film could be formed in all methylene blue concentrations. However, the film texture showed higher brittle than the native cellulose film (Fig. 1a). The brittle texture slightly increased when the concentration of methylene blue was increased. Addition of methylene blue might decrease the gap between molecules of

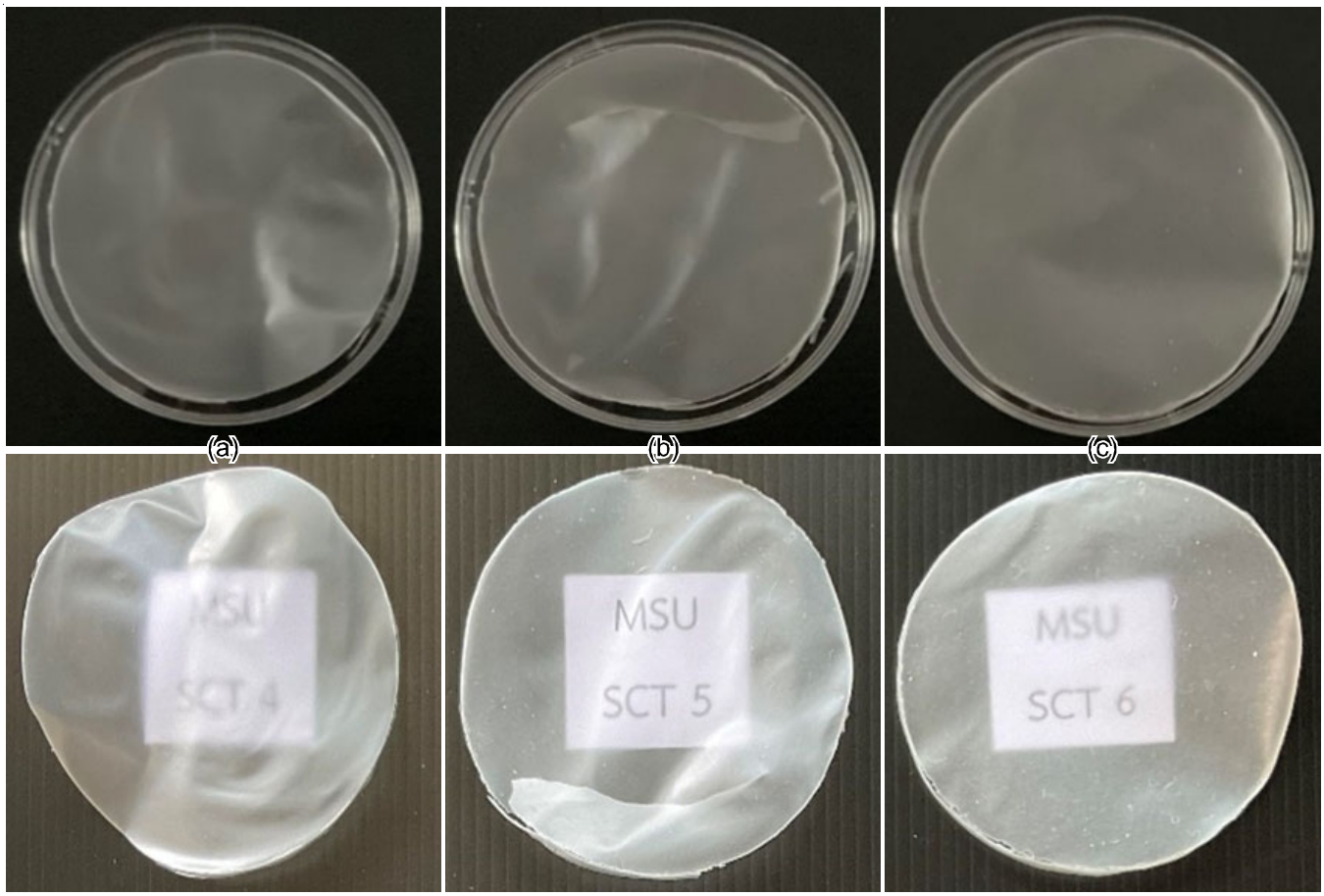


Fig. 1. Digital images of films transparency; raw cellulose (a), cellulose-mixed 1.4% glycerol (b) and cellulose-mixed 2.8% glycerol (c)


Fig. 2. Digital images of cellulose films loaded methylene blue with different concentrations: 0.5 (a), 1 (b) and 2% (w/w) (c)

glucose in cellulose chains, resulting in form crystalline parts, which results in the brittleness of the film.

**Morphological studies:** The SEM image depicted in Fig. 3 displays the film morphology of the prepared cellulose films. The native cellulose film (a) found some short fibers in rod shape embedded in surface texture (Fig. 3aI). With cross-section (Fig. 3aII), a homogeneous surface was observed and small gaps between its fibers also appeared. The short fibers with slight flat shape were found in the film surfaces in the cellulose-mixed glycerol (Fig. 3bI), whereas the texture cemented between cellulose fibers by glycerol and seemed to be smoother than the native film (Fig. 3bII). The surface of cellulose film loaded methylene blue is shown in Fig. 3(c-d). Both films are covered by the cellulose short fibers in flat shape affecting the rough surfaces. At low methylene blue content, the film surface was rougher than the film contained high methylene blue (Fig. 3cI), while containing methylene blue, films have small pores appearing in the texture (Fig. 3c,dII). Moreover, the short flat shape fibers were found in the film surface containing high amount of methylene blue (Fig. 3dI). As shown in Fig. 3dII, high content

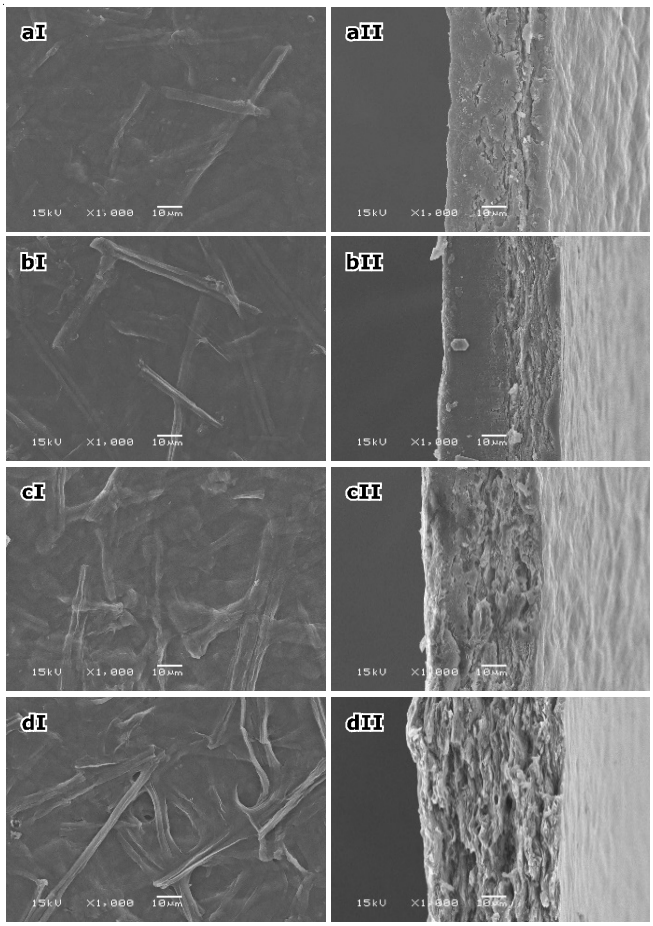


Fig. 3. SEM micrographs of different films; native cellulose (a), cellulose-mixed 1.4% glycerol (b), cellulose-mixed 0.5% methylene blue (c) and cellulose-mixed 2% methylene blue (d) at 1,000X magnifications (I present as film surface and II as cross-section)

of methylene blue affected the porous formation in the film texture. This indicated that methylene blue might be interacted with moisture in the film preparation process, which often evaporates in the drying step to form these pores.

**FTIR studies:** The ATR-FTIR spectrum of the native cellulose film is presented in Fig. 4a. The typical bands at 3333 (O-H *str.*), 2897 (C-H *str.*), 1428 (C-H *vib.*, 1028 (C-O-C pyranose ring skeleton) and 870 $cm^{-1}$ (β-(1-4)-glycosidic bond of cellulose) were detected [2,33-35]. The bands at 1640 and small peak at 1160 $cm^{-1}$ were assigned to the C=O ester and arabinoxylan groups of hemicellulose, respectively [36]. Additionally, the C=O stretching of the hemicellulose peak should have appeared at about 1727 $cm^{-1}$ [37]. In addition, the small peak at 1314 $cm^{-1}$ for aromatic residues in lignin was also observed. This indicates that the lignin was not fully removed from the fiber by treatment with alkali and bleaching.

The spectrum of cellulose-mixed glycerol as shown in Fig. 4b. The major characteristic bands of O-H *str.* (3333 $cm^{-1}$) and C-O-C (1028 $cm^{-1}$) *str.* were clearly observed. This means that the hydroxyl group of glycerol had H-bond interactions with plenty of hydroxyl groups in cellulose [38]. However, the bands at 1160 $cm^{-1}$ are also associated with glycerol in the films [39-41]. The ATR-FTIR spectrum of cellulose loaded 2% methylene blue is shown in Fig. 4c. Overall, the most absor-

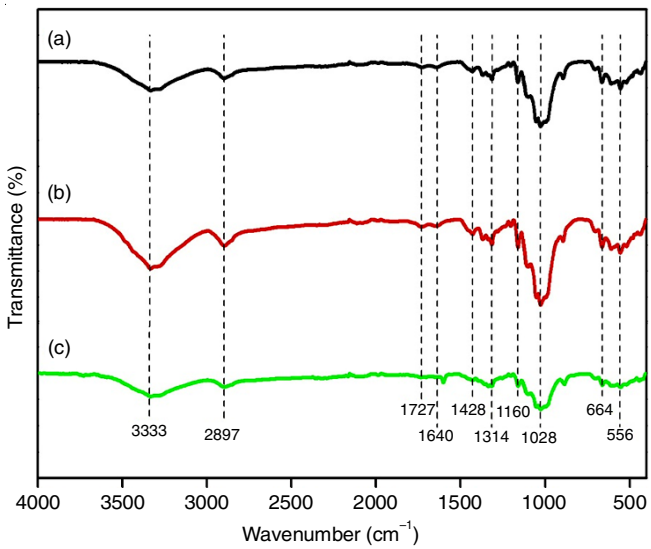


Fig. 4. ATR-FTIR spectra of different films; native cellulose (a), cellulose-mixed 1.4% glycerol (b) and cellulose-mixed 2% methylene blue (c)

ption bands have the same wavenumber as those that appeared in the native cellulose film (Fig. 4a). This indicate that methylene blue did not exhibit any destructive or transformative effects on the structure of the cellulose chain. However, loading of methylene blue causes the transition change of some cellulose characteristic peaks, which might be thought that some interactions between hydroxyl groups in the cellulose chain and polar groups in the methylene blue were formed.

**Thermal studies:** Thermal property of the native cellulose and cellulose-mixed glycerol films are shown in Fig. 5. The results show the decomposition at least 3 points. The first is the temperature less than 100 ºC responds to water evaporation [42]. The second point was in the range of 250-270 ºC, which involved the breakdown of glycerol molecule. However, this decomposition point has also been suggested for hemicellulose and lignin breakdown. The third point appeared at 310-330 ºC, which was attributed due to the cellulose decomposition peaks. The maximum temperature of decomposition rate ($T_{d,max}$) of the native cellulose and cellulose-mixed glycerol films were 324 and 312 ºC, respectively. The addition of glycerol as a plasticizer contributed to the enhanced flexibility of the film by intramolecular bonding with glucose inside the cellulose

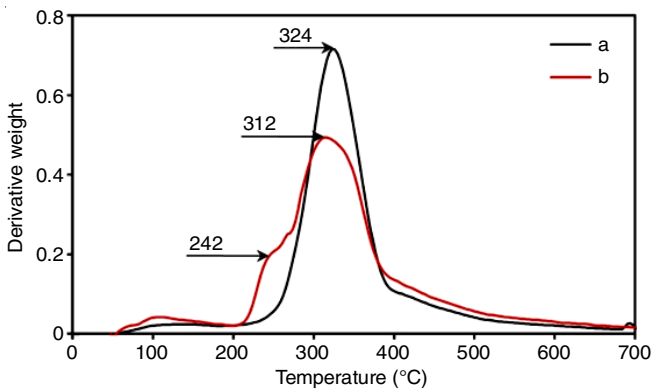


Fig. 5. DTG thermograms of the native cellulose (a) and cellulose-mixed glycerol (b) films

chain. Consequently, this led to a reduction in both the structural strength and thermal stability of the film.

The thermal stability of the cellulose loaded methylene blue in different concentrations is shown in Fig. 6. All films showed at least 4 decomposition points. The first point was less than 100 ºC, the second was a broken point of methylene blue at 250 ºC. The third point at 306-320 ºC was a decomposition of glucose unit in the cellulose chain. The last point was found at 350 ºC, which is the decomposed of hydrogen bonds formed between glucose and methylene blue. The result also indicated that the methylene blue concentration affected the thermal stability of the films. The thermal stability of films was gradually decreased when the methylene blue concentration was increased. This might be thought that the arrangement of glucose molecules in film texture would interfere with high concentrations of methylene blue.

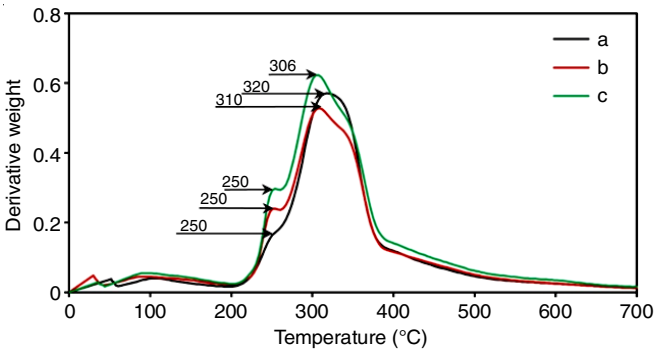


Fig. 6. DTG thermograms of the cellulose films loaded with different concentrations of methylene blue; 0.5% (a), 1% (b) and 2% (c)

**Releasing profiles:** The present study investigated the characteristics of films containing cellulose and methylene blue, focusing on their release patterns when immersed in a buffer solution for a duration of 48 h. As shown in Fig. 7, the 2% methylene blue loaded in the film showed the highest content of methylene blue released (85%) from the film after testing for 48 h while 0.5% methylene blue was in the lowest releasing of about 30% at an initial content. The results indicate that the releasing profile of methylene blue depends on the loading concentration. This is caused by the arrangement of methylene blue in the film texture, which would have interfered bonding formation of substances. At high content methylene blue, it might decrease the texture stability of the film as related to the obtained result from SEM images.

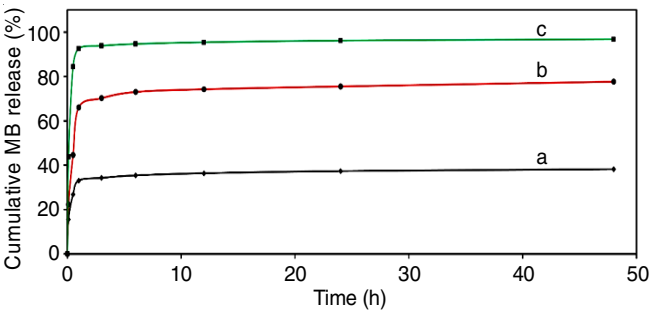


Fig. 7. Releasing profile of methylene blue from the cellulose films loaded with different concentrations: 0.5% (a), 1% (b) and 2% (c)

## Conclusion

This study employed the extraction of cellulose from cattail as a material for film production for releasing profile of methylene blue. The effect of glycerol and methylene blue on the film was also characterized. The native cellulose shows smooth surface, milky and increased flexibility by mixing glycerol. The glycerol helped to cement the gap between cellulose fibers, but it decreased thermal property and transparency of the films. The SEM images of cellulose loaded methylene blue revealed different morphology from the native and cellulose-mixed glycerol. The film texture was separated when the methylene blue was loaded. These changes of chemical structure and the thermal properties of the methylene blue-loaded film compared to others. Interestingly, the release profile of methylene blue from the cellulose films showed controlled release patterns depending on the loading parameters. This finding has an advantage to design the drug-loading material for a drug-controlled release system.

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## CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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