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Enhancing antibacterium and strength of cellulosic paper by coating triclosan-loaded nanofibrillated cellulose (NFC)

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ABSTRACT

The nanofibrillated cellulose (NFC) was used as substrates to carry triclosan (TCS), which was then applied as a coating agent for impacting antibacterial property to paper while also improving its strength. The TCS-loaded NFC material was further characterized. UV–vis spectra results showed that a characteristic absorption band at 282 nm was observed, which was attributed to triclosan, confirming its successful loading onto NFC. The antibacterial activity tests indicated that the coated paper exhibited excellent antibacterial activity against *Escherichia coli*, and the growth inhibition of bacteria (GIB) increased as the loading amount of triclosan coated on paper increased. The GIB can reach 98.7% when the 0.023 g TCS-loaded NFC was coated on paper. Meanwhile, the tensile and tear index of the coated paper increased by 18.0% and 26.4%, respectively compared to the blank paper. Therefore, the triclosan-loaded paper could be potentially used in the medical field.

1. Introduction

Nanofibrillated cellulose (NFC), disintegrated from wood pulp, have great potential for a wide variety of applications due to its renewable nature, biocompatibility, biodegradability and high specific strength (Liu, Lin, Chen, Huang, & Cao, 2014; Dash, Cateto, & Ragauskas, 2014; Liu et al., 2013; Fernandes et al., 2010; Trovatti et al., 2012; Zhu et al., 2014a; Zhu, Fang, Preston, Li, & Hu, 2014b). Among these application, NFC as a carrier for nanoparticles has attracted more attention in recent years because of its nano-dimensional network structure (Arola, Tammelin, Setälä, Tullila, & Linder, 2012; Valo et al., 2013). For example, Li et al. (2013) prepared a uniform, flexible, magnetic nanopaper by the immobilization of Fe_3O_4 nanoparticles in an NFC network in an aqueous medium, and found the resulting transparent magnetic nanopaper possessed excellent transparency and magnetic properties combined with outstanding mechanical performance and flexibility. Moreover, NFC can be used as reinforcing agent for a variety of composite materials because of its high stiffness and network structures (Huq et al., 2012; Martins et al., 2012; Srithep, Turng, Sabo, & Clemons, 2012; Besbes, Vilar, & Boufi, 2011; Lee et al., 2012; Zimmermann, Bordeanu, & Strub, 2010). For example, Alcalá et al. used the NFC as reinforcement in the preparation of biocomposites in the form of paper handsheets. They found that the presence of NFC induced an increase in the density of biocomposites and significant enhancement of the mechanical properties (Alcalá, González, Boufi, Vilaseca, & Mutjé, 2013).

In the previous work of our group, we studied nanocrystalline cellulose (CNC) as carriers for hydrophobic spirooxazine (SO)-based dye and alkenyl succinic anhydride (ASA), it was found that the use of CNC could improve the stability of SO-based dye and ASA, thus resulting in a significant improvement in the SO coloration efficiency and ASA sizing efficiency (Sun, Hou, He, Liu, & Ni, 2014a; Sun, Hou, Liu, He, & Ni, 2014b). This indicated that nanocrystalline cellulose would be a promising carrier for chemicals that have specific end-user application.

Triclosan (5-chloro-2-(2,4-dichlorophenoxy)phenol), a synthetic and broad-spectrum antimicrobial compound, is commonly used in a variety of personal care products (Anger et al., 2013). Triclosan possesses antibacterial properties as well as some antifungal and antiviral properties, so it can be used in papermaking process for enhancing the antimicrobial property of paper. For example, Soares et al. produced reinforced antibacterial paper packaging using the antimicrobial triclosan and organically modified montmorillonite via coating process. They found that the presence of 1% triclosan in the coated papers exhibited inhibitory effects against

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Staphylococcus aureus and *Escherichia coli* (Soares, Moreira, Fialho, & Melo, 2012). Obviously, triclosan is an effective antimicrobial agent for paper. However, triclosan can not be easily retained in paper because of its hydrophobic property under the paper making conditions of slightly acidic or near neutral pH.

In the present study, NFC was used as a carrier for loading triclosan so that triclosan can be dispersed and used in an aqueous system at a mild pH without the aid of an organic solvent, the triclosan-loaded NFC was then coated on paper to induce the antibacterial property to paper while enhancing its mechanical strength. The antibacterial property was tested against *Escherichia coli* (*E. coli*, ATCC 11229). Such paper products may have the potential in the medical field.

2. Materials and methods

2.1. Materials

Nanofibrillated cellulose (NFC) (2%, w/w) was from Tianjin Haojia Cellulose Co., Ltd. (China). Triclosan (99%) was purchased from Alfa Aesar reagent Co., Ltd. Phosphate buffered saline and LB Broth with agar were purchased from Sigma-Aldrich reagent Co., Ltd. All other chemicals were of analytical grade and used without further purification.

3. Methods

3.1. Preparation of triclosan-loaded NFC

Triclosan solution was prepared by dissolving 0.001, 0.003, 0.005, 0.007, 0.01, 0.05, and 0.1 g of triclosan in 5 ml of 1% NaOH solution at room temperature, respectively. Then 15 g of 2% (w/w) NFC was added and stirred at a stirring rate of 8000 rpm. After 5 min, 1.8 ml of 3.7% HCl solution was added into the above suspensions and stirred for 10 min at a stirring rate of 15,000 rpm. The amount of triclosan loaded on the NFC was 0.0033, 0.01, 0.017, 0.023, 0.033, 0.17, and 0.33 g/g NFC.

3.2. Coating of paper

The NFC loaded with different amount of triclosan was coated on a filter paper (dia. 18.5 cm, 100 g/m^2) using a laboratory coater machine (K303 Multicoater, RK Print coat Instruments Ltd., U.K.), the coated paper was then kept in a drying oven at 105 °C for 20 min. The other side of the paper was also coated with the triclosan-loaded NFC and kept in a drying over at 105 °C for another 20 min. The total coating amount of triclosan-loaded NFC on paper was 300 ± 20 mg/g paper.

3.3. Characterization of the triclosan-loaded NFC and coated paper

The triclosan solution (0.04 g/l in 1% NaOH solution) and the NFC loaded with different amount of triclosan were analyzed using an Ultraviolet–visible (UV–vis) Spectrometer (Genesys 10-S, Thermo Electron Corporation, USA). UV–vis spectra were recorded between 200 and 500 nm at room temperature. Thermogravimetric analysis (TGA) of the blank paper, triclosan, and coated paper were performed on a DSC-TGA instrument (SDT Q600, TA Instruments, USA). Approximately 5 mg of sample was weighed and heated from room temperature to 600 °C at a heating rate of 10 °C/min under nitrogen flow rate of 100 ml/min.

3.4. SEM analysis

The surface morphology of the blank paper and coated paper was investigated using a scanning electron microscope (SEM) (JSM-6400, JEOL, Japan). The surface of the sample was carbon coated and then gold coated for conductivity, and the accelerating voltage of SEM was 15 kV.

3.5. Antibacterial activity of the paper coated with triclosan-loaded NFC

The shaking flask method was used to detect the antimicrobial activity of the coated paper, and the Gram-negative bacteria *Escherichia coli* (*E. coli*, ATCC 11229) was chosen as the test microorganism. The test method was conducted as follows (Zhang & Xiao, 2013): about 4.5 ml of 0.01 M phosphate buffered saline solution was mixed with 0.5 ml *E. coli* (10^5 CFU/ml) in a sample tube, and 0.1 g blank paper or coated paper was added into the above solution. The sample tube was then immersed in a water bath shaker and shaken at 37 °C for 1 h. About 0.1 ml of each culture was transferred and seeded on an agar plate, the plate was then kept in an incubator at 37 °C for 24 h. The number of the colonies was counted, and the inhibition of the bacteria growth was calculated using the following equation:

$$\text{Growth inhibition of bacteria (GIB) (\%)} = \frac{A_0 - A_1}{A_0} \times 100 \quad (1)$$

where A_0 and A_1 are the number of the colonies detected from the blank paper and coated paper, respectively.

3.6. Strength properties

The tensile properties and stretch of the paper and coated paper were tested using a tensile strength tester (L&W, Sweden). Tensile strength and stretch were measured in accordance with TAPPI Test Method T 494. The tear strength of the paper and coated paper was tested using a tearing tester (L&W, Sweden) according to the ASTM D 689 test method.

4. Results and discussion

4.1. Preparation of triclosan-loaded NFC

A simple method was used to load triclosan using NFC as a carrier. In the method, the triclosan was firstly soluble in the alkaline aqueous solution and dispersed in NFC suspension, a certain amount of hydrochloric acid solution was then added to precipitate triclosan particles on the surface of NFC. The formation of triclosan particles loaded on the NFC was confirmed by UV–vis spectral analysis. From the UV–vis spectra of the triclosan solution and triclosan-loaded NFC (Fig. 1), a characteristic absorption was observed at 282 nm for triclosan-loaded NFC, and its intensity increased as the amount of triclosan loaded increased. Lavecchia and Zuorro (2009) reported that the absorption spectrum of triclosan in aqueous ethanol had a sharp peak at 282 nm. This indicated that triclosan has been loaded on the NFC successfully. However, at an alkaline condition, the characteristic absorption of triclosan occurs at 292 nm, instead of 282 nm, due to the reason that the phenolic hydroxyl group of triclosan was present as phenolate ion (Vukovic, Jurišic Grubešic, Kremer, & Spaic, 2013). This explains the red shift of the characteristic absorption of triclosan from 282 to 292 nm.

4.2. Thermal property of the coated paper

Thermal stability of the triclosan-loaded NFC is very important to the coated paper during drying process. The thermogravimetric

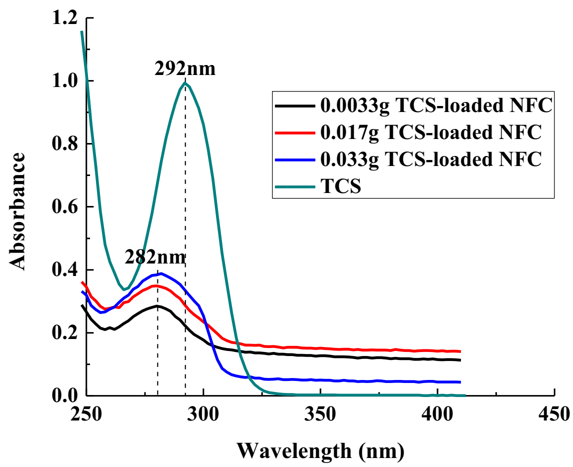

Fig. 1. UV–vis absorption spectra of TCS and TCS-loaded NFC.

(TG) and derivative thermogravimetric (DTG) curves of the blank paper, triclosan, and coated paper were shown in Fig. 2. It can be seen from Fig. 2a that the TG curve of triclosan exhibited only one step, and the degradation during this step was about 95.3 wt% due to the degradation of triclosan. However, the TG curves of the blank paper and coated paper exhibited three main steps. The blank paper degradation during the first, second, and third steps was 3.5, 79.6, and 2.5 wt%, respectively. The coated paper degradation during the first, second, and third steps was 5.6, 61.5, and 8.8 wt%, respectively. The mass loss of the blank paper and coated paper during the first step can be attributed to evaporation of the free water came from paper. The second mass loss of the blank paper and coated paper was ascribed to the thermal decomposition of cellulose and triclosan.

In addition, the degradation temperature of the blank paper and triclosan were found to be 352 and 244 °C, respectively (Fig. 2b). The lower degradation temperature of triclosan than blank paper indicated that triclosan possessed worse thermostability than did the cellulose of paper. However, the degradation temperature of the coated paper was 337 °C, which was between that of triclosan and blank paper. This may be the reason that the triclosan with poor thermostability had a lower decomposition temperature, resulting in a lower peak decomposition temperature for paper coated with triclosan-loaded NFC. Many studies for thermal stability of composites indicated the similar results. For example, Salam et al. studied the thermal stability of modified starch nanoparticles, chitosan, and their composites. They also found that the maximum degradation temperature of the modified starch nanoparticles/chitosan composites was between that of them (Salam, Lucia, & Jameel, 2013).

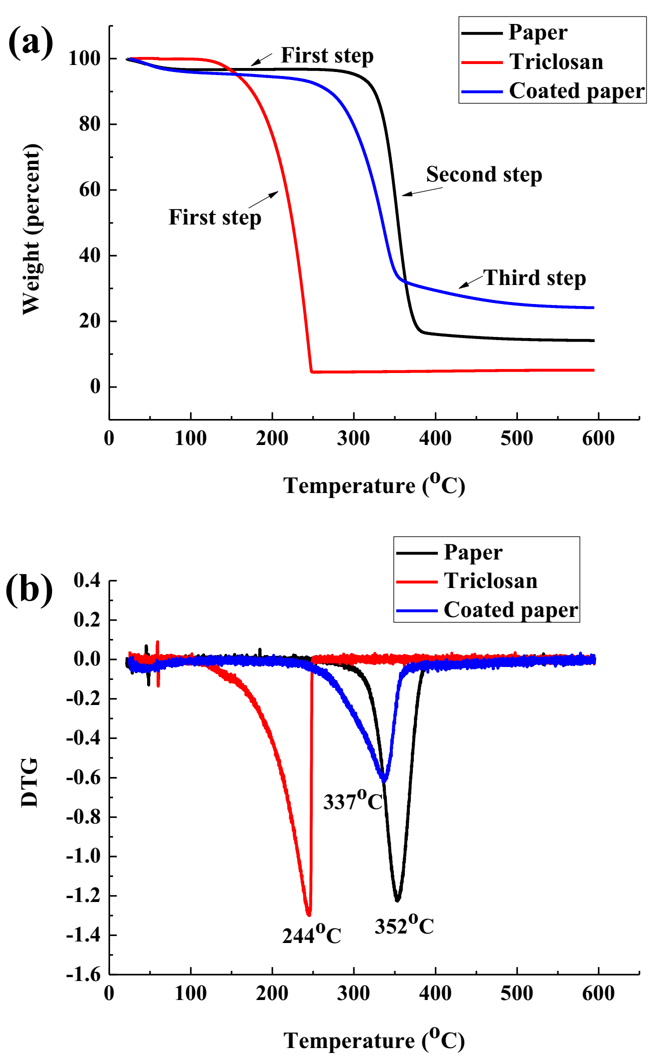

Fig. 2. (a) TG and (b) DTG curves of the blank paper, triclosan, and paper coated with 0.33 g TCS-loaded NFC.

4.3. SEM studies

SEM was used to confirm the coating of triclosan-loaded NFC on paper, and the SEM images of blank paper and coated paper were shown in Fig. 3. It was found that lots of pores between fibers existed on the surface of the blank paper (Fig. 3a-1), and there were not any particles loaded on fibers (Fig. 3a-2). However, some pores between fibers were covered after the triclosan-loaded NFC was coated on the blank paper (Fig. 3b-1). Meanwhile, a lot of particles can also be observed on the fiber of the coated paper (Fig. 3b-2). This indicated that the triclosan-loaded NFC was coated on the surface of the paper and the triclosan particles were kept on the paper.

4.4. Antibacterial activity studies

The shaking flask method was used to test the antibacterial property of the paper coated with triclosan-loaded NFC, and the *Escherichia coli* (*E. coli*), a common bacteria harm to human health, was chosen as the test object (Zhang & Xiao, 2013). The test results were shown in Fig. 4 and Table 1. It was found that a lot of colonies have grown on the plate for the blank paper (Fig. 4a). This indicated that the cellulose of blank paper has no antibacterial property. However, the number of colonies on the plate decreased greatly for the paper coated with 0.023 g triclosan-loaded NFC (Fig. 4b), and there were no colonies on the plate for the paper coated with 0.33 g triclosan-loaded NFC (Fig. 4c). According to the calculation of GIB, it was found that the GIB of the coated paper increased as the loading amount of triclosan coated on paper increased (Table 1), and the GIB reached 100% when the 0.33 g TCS-loaded NFC was coated on paper. This indicated that the coated paper had excellent antibacterial activity against *E. coli* when sufficient level of triclosan was coated on the paper. It was mainly due to the strong antibacterial property of triclosan. Soares et al. (2012) evaluated the antibacterial property of the paper coated with triclosan and modified montmorillonite composites and found the presence of 1% triclosan in the coated papers exhibited inhibitory effects against *E. coli* and *S. aureus*. The different between his results and the results in this study was the experimental method and conditions used to test the antibacterial activities. The antibacterial

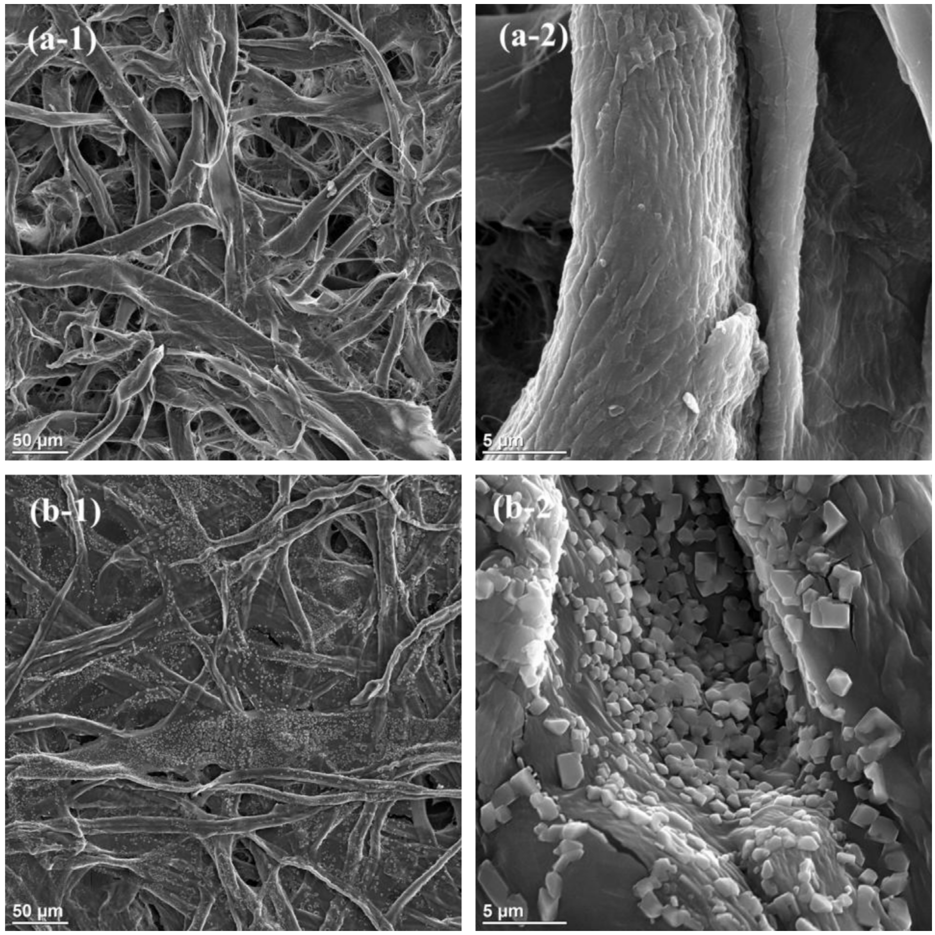

Fig. 3. SEM images of (a-1 and a-2) blank paper and (b-1 and b-2) paper coated with 0.023 g TCS-loaded NFC. (a-1 and b-1) were photographed at low magnifications; (a-2 and b-2) were photographed at high magnifications.

Table 1
Antibacterial property of paper coated with TCS-loaded NFC.

Loading amount of triclosan (g/g NFC)	0	0.0033	0.01	0.017	0.023	0.033	0.17	0.33
GIB (%)	–	12.5	79.2	87.5	98.7	99.8	99.9	100

activity tests in this study clearly demonstrated that the triclosan was successfully loaded on NFC and coated on the paper.

4.5. Strength property studies

The strength property of the blank paper and coated paper were studied by testing the tensile and tear strength, and the results were shown in Table 2. It was found that the tensile index of blank paper was only 24.93 N m/g. However, the tensile index can reach 29.41 N m/g after the triclosan-loaded NFC was coated on paper. Furthermore, the tear index also increased from 7 to 8.85 mN m^2/g. The increase of the tensile and tear strength of the coated paper was attributed to the NFC coated on the surface of the paper. The NFC had smaller size than did the fibers of paper and could form a network structure with a lot of hydrogen bonds between fibers, thus enhancing the strength property for paper. Littunen, Hippi, Saarinen, and Seppälä (2013) prepared the composites of poly(methyl methacrylate) and nanofibrillated cellulose (NFC) by solution blending, injection and compression molding, and found the formation of a percolating nanofibril network between 1 and 5 wt% of NFC by dynamic rotational rheometry in molten state. Our previous studies also pointed out that NFC had great reinforcement effect for a variety of biomaterials because of its nano- to submicron-wide size and the formation of strong interactions between adjacent whiskers by hydrogen bonding (Liu et al., 2013, 2014). In addition, the blank and coated paper had similar stretch, suggesting that the coating of triclosan-loaded NFC had little effect on the stretch of paper.

Due to the fact that cellulosic papers are user friendly, biodegradable, cost-effective, among other associated advantages,

Table 2
The comparison of tensile strength, tear strength, and stretch of blank and coated samples. (The amount of triclosan loaded on NFC was 0.023 g/g NFC.).

	Tensile index (N m/g)	Tear index (mN m^2/g)	Stretch/elongation (%)
Blank paper (control)	24.93 ± 0.60	7.00 ± 0.081	2.68 ± 0.35
Triclosan-loaded NFC coated paper	29.41 ± 1.97	8.85 ± 0.26	2.77 ± 0.41

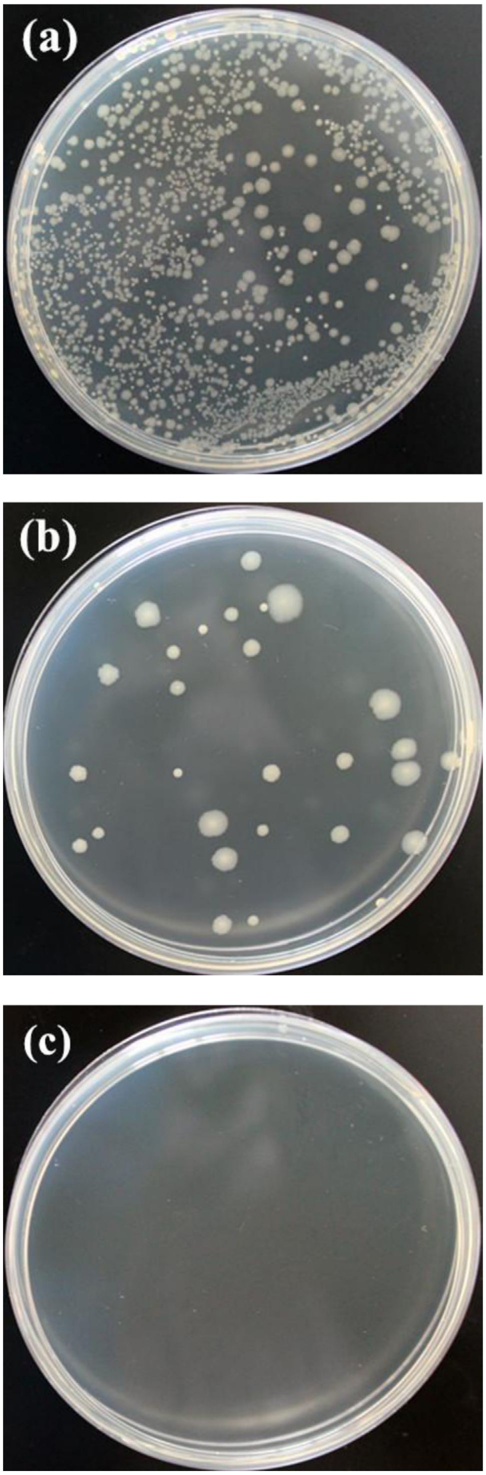

Fig. 4. Photographs of bacterial colonies formed by *E. coli*: (a) blank paper, (b) paper coated with 0.023 g TCS-loaded NFC, (c) paper coated with 0.33 g TCS-loaded NFC.

they are more and more used in our daily lives, for example, tissue products in personal hygiene. One example of the present concept would be the paper/non-woven mat used in the hospital.

5. Conclusions

The trichlosan (TCS) was loaded on NFC using a simple method, and then the TCS-loaded NFC was applied as surface coating onto paper surface for impacting antibacterial properties to paper and enhancing its strength property. The resultant paper-based novel composite was very effective in inducing the antibacterial activity while enhancing its strength properties. The results showed that the coated paper exhibited excellent antibacterial property against *E. coli*, and the growth inhibition reached 98.7% when the 0.023 g TCS-loaded NFC was coated on paper. Meanwhile, the tensile and tear index of the coated paper increased by 18.0% and 26.4%, respectively compared to the blank paper. These results suggest that the triclosan-loaded paper could be a potential bio-material based product that may have applications in the medical field.

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