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ORIGINAL PAPER

Morphology control for tunable optical properties of cellulose nanofibrils films

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Abstract Flexible cellulose nanofibrils film substrates with high smooth surface and high transparency are attractive for next- generation flexible transparent electrical device applications. In recent years, tuning optical properties of the substrates has become more and more important for the fabrication of the transparent electronic devices. In this study, a simple depositing process with micro-scale TEMPO-oxidized wood fibers was utilized to tune top surface morphology of the cellulose nanofibrils films. The influence of the surface morphology on the optical properties was also investigated. As the upper surface roughness increased, the optical haze of the transparent films increased. The obtained films, with total transmittance ranged from 83% to 88%, exhibited relatively low haze of 3.8% to high haze of 62.3%. In addition, the lower surface of cellulose nanofibrils films has a super flat surface, which is required for applications in electronics and optoelectronics.

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Introduction

Substrates are the fundamental building blocks for flexible electronic and optoelectronic devices, such as touch screens, display screens, solar cells, and light-emitting diodes (Bai et al. 2015; Guo et al. 2015; Hoeng et al. 2016; Kim et al. 2015; Madaria et al. 2011; Roth et al. 2015). Typical flexible plastic substrates including polyethylene terephthalate (PET), polyethylene naphthalate (PEN), and polycarbonate (PC), have been utilized for flexible transparent devices widely (Hui et al. 2017; Leppaniemi et al. 2017; Miettunen et al. 2009). However, the high coefficient of thermal expansion and poor thermal stability are clearly major obstacles to the fabrication of electronic devices with a high processing temperature (Mecking 2004). Recently, it was found that a novel transparent film constituted of "green" TEMPO-oxidized cellulose nanofibrils (TOCNs) could replace those flexible plastics due to their excellent mechanical properties, transmittance and thermostability (Nogi et al. 2010; Su et al. 2016; Zhu et al. 2013a). As indispensable building blocks for the electronic devices, transparent conductive films are produced by depositing tin-doped indium oxide (ITO), aluminum doped zinc oxide (AZO) or coating graphene, carbon nanotubes and silver nanowire on the surface of the substrates (Hu et al. 2013; Kang et al. 2015; Koga et al. 2014; Sadasivuni et al. 2014). In general, the thickness of the conductive layer is lower than 1 µm. To guarantee excellent application properties for the electronic devices, transparent substrates must possess a smooth surface. Transparent films constituted of TOCNs with diameter of 5–10 nm exhibit an extremely low surface roughness because of the small fibers diameter, which is suitable for fabricating transparent electronics (Huang et al. 2013; Zhu et al. 2013c). The TOCN films based on nanoscale fiber have high transparency and usually a low transmission haze values due to denser structure (Zhu et al. 2013b). However, in many applications, highly transparent but also a high haze or an extremely low haze films are required. For example,the optical clarity is critical in high-definition displays that require an extremely low haze value (less than 1%) (Fitz-Gerald et al. 2000). A high haze (more than 40%) endows anti-glare function for outdoor displays, which guarantee the displays can be used under strong sunlight (Chung and Lu 2003).

Various inorganic materials including SiO_2, TiO_2 nanoparticles and silver nanowires have been reported to manage the optical haze of the optoelectronic devices because of the different refraction coefficient between the substrates and inorganic materials (Hassinen et al. 2015; Kim et al. 2009). While the light scattering caused by the difference in index of refraction is limited, incorporating these materials requires complex process and additional cost. Recently some novel “green” methods have been proposed to achieve tailored optical properties for the optoelectronic devices. In contrast to the filtration method, cellulose nanopapers prepared by solution-casting possess a lower transmission haze, which is the result of a lower surface roughness (Yang et al. 2017). Opaque mesoporous wood fiber-based paper can be filled with cellulose nanofibrils (CNFs) to create a higher optical haze, lower cost and better shape stability transparent substrate (Fang et al. 2013). Transmittance and haze values for cellulose nanopapers could also be tuned with the cellulose fiber diameter and nanopapers packing density (Zhu et al. 2013b). Recently transparent nanopapers with a tailored optical haze were prepared by a facile filtration method with regulating the weight ratio of TEMPO-oxidized fibers to CNF (Zhu et al. 2013a). In a word, a simple and efficient approach tuning optical haze of transparent substrates has drawn abundant attention.

In this work, a simple depositing of TEMPO-oxidized wood fibers (TOWFs) was directly utilized to tune the optical haze of the TOCN film. The coating of micro-sized TOWFs with an average diameter in 18.6 µm and an average length in 63 µm significantly increases the light scattering on the upper surface of TOCN films and dramatically improves the optical haze. At the same time, a superior smooth surface of TOCN films was also observed, which was suitable for depositing continuous conductive thin layer and endowing excellent electronic properties for devices. A facile solution-casting method was proposed to fabricate transparent films with a tunable transmission haze and smooth surface. The mechanism for the existence of vary light scattering behaviors was illustrated by charactering the optical properties, surface morphology and roughness of the films.

Experimental and methods

Materials

All commercial chemicals were analytical reagents, and were used without further purification. Sodium hydroxide (NaOH), hydrochloric acid (HCl), Sodium hypochlorite (NaClO), Potassium bromide (KBr) were purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). Tetramethyl-1-piperidinyloxy (TEMPO) was obtained from Sigma-Aldrich. The raw materials of bleached softwood kraft pulp were purchased from Suzano.

Preparation of TOWFs and TOCNs

5 g of bleached softwood kraft pulp was suspended in 500 mL of deionized water containing 0.08 g NaBr. 35 mL NaClO was added to induce the TEMPO mediated oxidation reaction. The pH was maintained at 10.5 using HCl (20% V/V) and NaOH (0.5 mol/L). The obtained TEMPO-oxidized wood fibers (TOWFs) was dialyzed using a regenerated cellulose membrane with 12,000–14,000 (D) after centrifuging at 5000 rpm three times, and stored in cold environment at 5 °C for future using and treatment. Then the

TOWF suspension was treated with a homogenizer (FB-110X, ShangHai LiTu Mechanical Equipment Engineering Co. Lid, china) for 10 cycles under 950 bar pressure to obtain TOCNs.

Fabrication of transparent TOCN films with varying degree of optical haze

Tuning optical haze of the clear TOCN films was conducted with regulating the weight of the coated TEMPO-oxidized fibers. The 0.5 wt% aqueous TOCN suspension was used to fabricate transparent TOCN films by a solution-casting method using a petri dish (60 mm, PS) at room temperature for few days, until a thin wet films formed. Then the TEMPO-oxidized fibers were directly coated on the wet TOCN films using drop-casting method with various grammages ranged from 0 to 20 g/m^2, and drying under room temperature. The obtained dried films were place at 23 °C, 50% RH for 24 h before measurement. The details are showed in Fig. 1.

Analysis

The dimension and morphology of the TOWFs were characterized using Mofri Compact (LDA02, OpTest Equipment Inc., Canada) and optical spectroscope (U-TV1X-2, JAPAN OLYMPUS, Japan). Atomic force microscopy (AFM) was used to characterize the dimensions of TOCNs under tapping mode (Dimension Edge, Bruker, Germany). A droplet of 0.001 wt% suspension was deposited on cleaved mica and air-dried. Transparency and transmission haze of the films were obtained using a UV/VIS/NIR spectrophotometer (Lambda 950, PerkinElmer, USA) according to the ASTM1003-13 standard method (Plastics), and the measured wavelength ranged from 400 to 1100 nm. Field emission scanning electron microscope (FE-SEM) was used to observe the surface morphology and fracture section of the transparent films (JSM-7600F, JEOL Ltd., Japan). AFM (Dimension Edge, Bruker, Germany) was utilized to characterize the surface roughness of the films samples. The thickness of films was analyzed by the field emission scanning electron microscope (FE-SEM) and micrometer caliper.

Results and discussion

Morphologies of TEMPO-oxidized wood fibers and TEMPO-oxidized cellulose nanofibrils

In this study, high transparent films with a tailored optical haze have been prepared by coating TOWFs on the top surface of ultra-smooth TOCN films. TOWFs were prepared by oxidizing wood fiber using TEMPO-mediated oxidation method (Saito et al. 2007). Then the nano-sized TOCNs were extracted from the TOWFs via a homogenization process. The morphology of the TOWFs were observed by optical microscopy. As shown in Fig. 2a, the wood fibers were cleaved into smaller fibers during the TEMPO-mediated oxidation treatment and obvious fibrillated fibers were observed. The inducing of the carboxyl groups provides electrostatic repulsion to stabilize wood fiber aqueous slurry system. The diameter of the TOCNs is around 6–8 nm, which was characterized by the atomic force microscopy (AFM) (Fig. 2b). As shown in Fig. 2c, TOWF aqueous slurry system exhibited excellent dispersity and no obvious precipitates. According to FQA (fiber quality analyzer) data, the size of the TOWFs is around 63.0 μm length and 18.6 μm width, details shown in Fig. 2e, f. When the laser penetrates the slurries of TOWFs, incident light

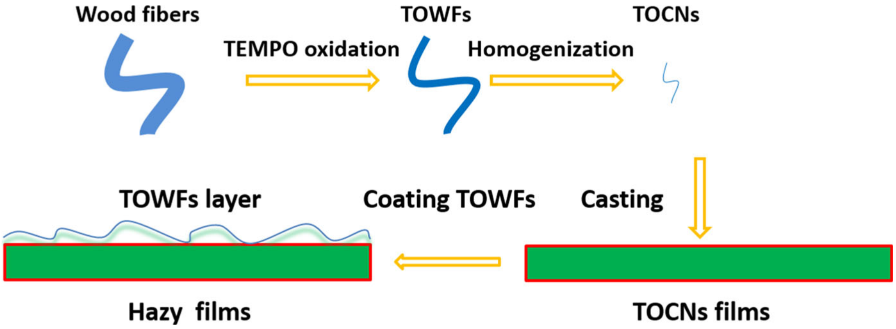

Fig. 1 The whole processes of the fabrication of hazy TOCN-based films

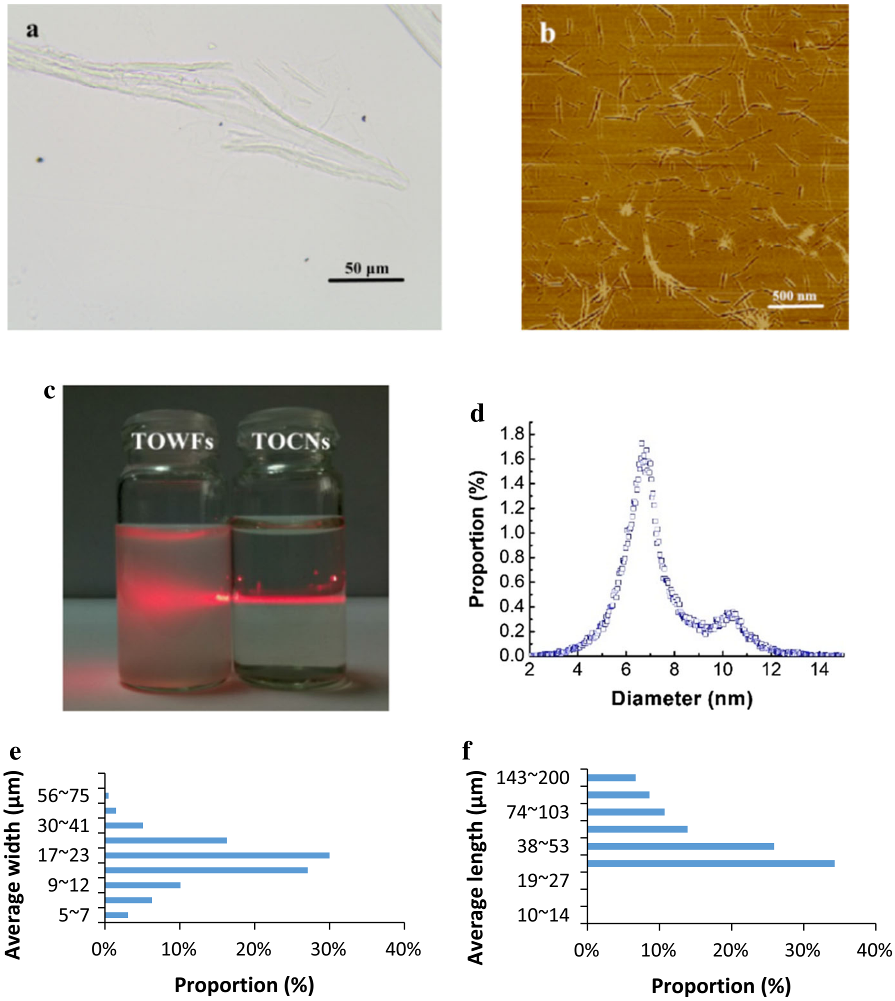

Fig. 2 **a** Optical microscopy image of TOWFs. **b** AFM image of TOCNs. **c** Digital photograph of TOWF and TOCN suspensions with 0.5 wt% concentration and a red laser beam with a wavelength of 540 nm travels through the TOWF and TOCN suspensions. **d** The corresponding diameter of TOCNs measured from AFM topographic data. **e**, **f** Width fraction and length fraction of TOWFs

is strongly scattered because of the micro-sized dimension of the fibers. That is the reason for the limiting light transmittance of TOWF slurry. As the TOCNs extracted from the TOWFs via a homogenization process, the light scattering behavior is limited due to the fact that the dimension of the TOCNs is much smaller than the visible Wavelength (Qing et al. 2015).

Optical properties of transparent hazy films

The fiber dimensions play an important role in tailoring the optical properties of the transparent films. Obviously, regular papers constituted of mirco-sized wood fibers are opaque, because of the serious light scattering of the fibers and mircro-scaled pores (Hsieh et al. 2017). To modify the morphology of wood fibers, TEMPO oxidation system and homogenization were used. The obtained fibers with nano-sized dimension show limited light scattering and high transparency. Photographs of transparent TOCN films coated with different grammages of TOWFs are shown in Fig. 3a, b. All the films exhibited a high transparency and the pattern under the transparent films could be clearly observed by human eyes. These phenomena illustrated that the coated layer of TOWFs doesn't inhibit the transparency of the TOCN films. When the films were lifted up with a certain distance of 6 cm, the pattern behind the neat TOCN films still can be clearly observed. With an increase in TOWF layers grammage, the pattern gradually became obscure due to the intensive light scattering effect mainly caused by surface-relief (Nogi et al. 2009). To further illustrate the optical properties of the transparent films, total

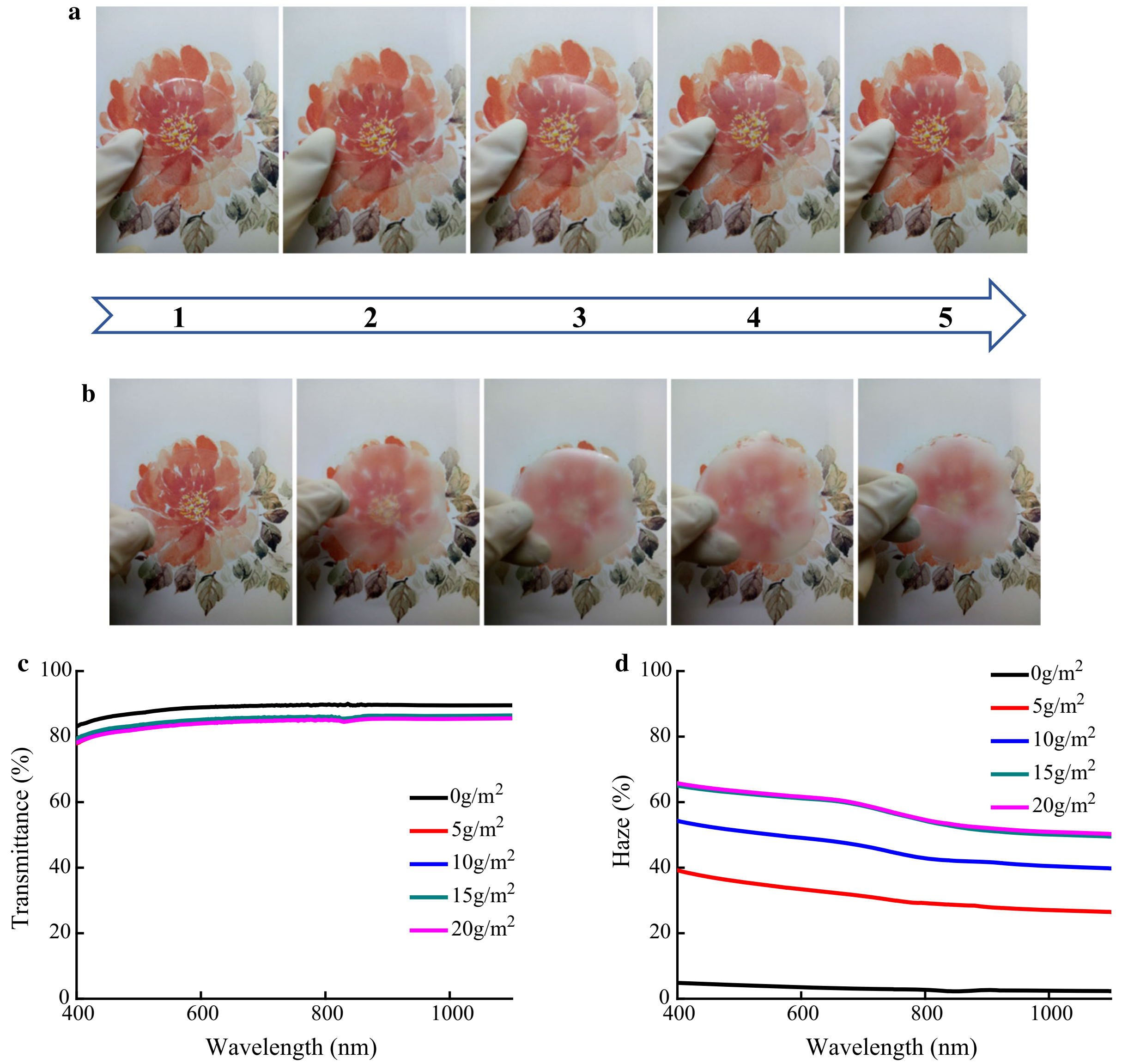

Fig. 3 **a** Visual appearance of TOCN films with different grammages of TOWF layer closely contact underneath color pattern. **b** Digital photographs of TOCN films with different grammages of TOWF layer were lifted up with a certain distance of 6 cm from under color pattern. The numbers of 1, 2, 3, 4, 5 represent the TOWF layer grammages of 0, 5, 10, 15, 20 g/m^2 respectively. **c**, **d** optical properties of TOCN films with different grammages of TOWF layer, including total transmittance and optical haze

transmittance and transmission haze were measured using a UV–Vis spectrometer in wavelength of 400 nm to 1100 nm. The total transmittance and optical haze curves are shown in Fig. 3c, d respectively, and the values are shown in Table 1. The neat TOCN films have the highest transmittance of 88% and the lowest optical haze of 3.8% at the wavelength of 550 nm. The results are similar to those of flexible glass and polyethylene terephthalate (PET) films (Yao et al. 2017). As the TOWFs were deposited, the TOCN films exhibit a tailored optical haze from 3.8% to 62.3% and a high total transmittance of 88% to 83% at the wavelength of 550 nm. These tailored optical properties clarify the potential applications of transparent substrates in optoelectronic devices, including indoor displays and solar cells (more than 40%).

Table 1 Properties of clear TOCNs films and transparent hazy films with vary grammages of TOWF layer

Entry	Grammage[a] (g/m^2)	Thickness (μm)	Upper surface roughness (nm)	Bottom surface roughness (nm)	Total transmittance (%)	Optical haze (%)
1	0	29.6 ± 5.1	6.25	6.25	89	3.8
2	5	32.6 ± 2.3	451	6.25	84	35
3	10	39.6 ± 1.8	827	6.25	84	50
4	15	41.0 ± 2.5	918	6.25	85	62
5	20	42.5 ± 1.9	926	6.25	83	62

[a] Grammage of TOWF coating layer

As shown in Fig. 4, the mechanism of light management for TOCN films was illustrated. When incident light through the neat cellulose nanofibrils films (clear TOCN films) with a super smooth surface and dense structure, transmitted light is mainly concentrated in the incident direction. The limited light diffusing endows a highly transparent and extreme low haze for clear TOCN films. Then micro-sized TOWFs were coated on TOCN films surface to manage the light performance for TOCN films substrates. Evidence from Fig. 2c indicates that: the micro-sized fiber scatters light. After deposition process, a rough surface was achieved. With the increase of grammages of the TOWF depositing layer, surface roughness increases. When incident light through the extremely rough surface, an extensive light scattering was observed. The rougher the surface, the more incident light was scattered. In comparison with the rough upper surface, the bottom surface of the TOCN film were ultra-smooth property, beneficial for conductive layer deposition and endows an excellent performance for transparent devices. The clear and hazy transparent films were covered on the screens of display devices to evaluate the anti-glaring performance under strong sunlight respectively (Fig. 4). As shown in Fig. 4, under strong sunlight, the clear TOCN film presents glare, which seriously influence visual perception. In comparison of clear TOCN film, hazy TOCN film exhibits excellent anti-glaring characteristics. In a word, a highly transparent and hazy film was prepared by a facile solution-casting method, which is good match for “green” optical electronic devices fabrication.

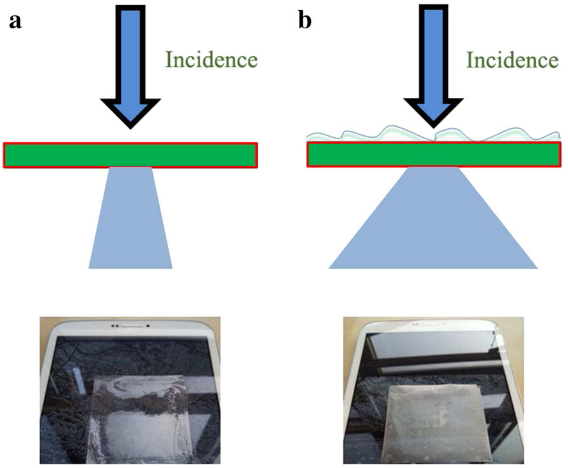

Fig. 4 Diagrammatic sketches of light scattering and anti-glare function, **a** clear TOCN films, **b** hazy TOCN films with TOWF coating layer

Surface morphology of transparent hazy cellulose nanofibrils films

As previously reported, the surface morphology of TOCN films has notable influence on films optical haze properties (Yang et al. 2017). In this study, Field-Emission Scanning electron microscopy (FE-SEM) and Atomic force microscopy (AFM) were used to characterize the top-view morphology of TOCN films with and without TOWFs-coating, and to explore why the transparent films with and without TOWFs-coating exhibit excellent transmittance and tunable optical haze. As shown in Fig. 5a, the neat TOCN film has a compact structure and no obvious pores were observed. Dense TOCN network effectively limits the light scattering induced by the mismatch refraction index between air (1.0) and cellulose (1.5) (Zhu et al. 2013b), which endowing a high transparency for TOCN films. After micro-sized TOWFs depositing,

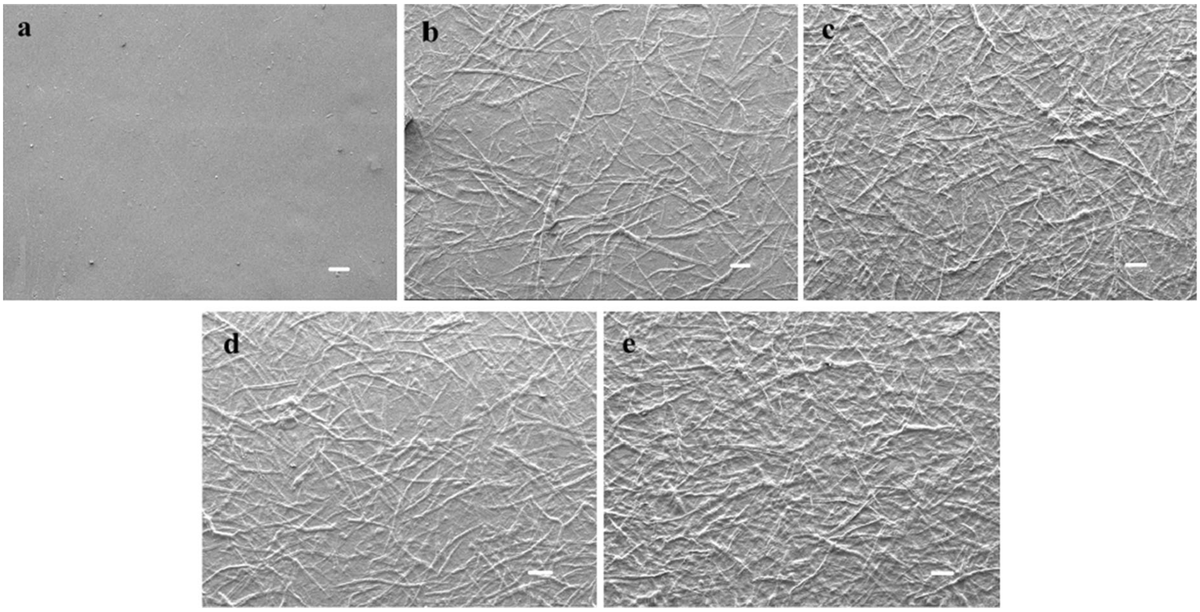

Fig. 5 Top-view FE-SEM images of the transparent films produced by coating vary grammages TEMPO-oxidized fiber layers on the TOCN films. **a** 0 g/m^2, **b** 5 g/m^2, **c** 10 g/m^2, **d** 15 g/m^2, **e** 20 g/m^2, the scale bar is 100 μm

the changes in the upper surface morphology of the TOCN films were observed (Fig. 5b–e). As the grammage of the TOWF layers increased, more micro-sized fibers were observed on the surface of the TOCN films, which increased the surface roughness and promoted light scattering.

Further surface roughness analysis of the hazy transparent films was measured using atomic force microscopy (AFM). Avoiding the error caused by small dust particles, a scan area of 5 × 5 μm^2 was used to test neat TOCN films. The scan area of 100 × 100 μm^2 was used to measure other film samples. In comparison of filtration method (Fukuzumi et al. 2008), the casting films has a smoother surface, which effectively limited the light scattering. Hence in this study, a optically clear TOCN film with smoother surface (root-mean-square roughness 6 nm) was prepared using a solution casting method (Aulin et al. 2010). Then a certain amount of TOWFs were coated on the upper surface to tune surface morphology, and the bottom surface without effect still exhibits a smoother surface which is suitable for conductive layers depositing. The root-mean-square roughness (RMS) of the upper surface of high haze transparent films ranged from 451 to 926 nm was observed by the AFM analysis (Table 1). The rough surface caused substantial light scattering, which led to a high transmission haze. The strength of the light scattering depends on the roughness values of the transparent film surfaces. As expected, the optical haze of the transparent films dramatically increased as the surface roughness increased. The 3D morphology of the transparent films is shown in Fig. 6, The diverse morphologies and roughness values confirmed the results drawn from FE-SEM images. When TOWFs were coated on the upper surface of TOCN films, a large spatial variation was observed by the 3D morphology. These rough surfaces with the light scattering behavior, induced the transparent films to appear hazy. Unlike upper surface of TOCN films, super-smooth bottom surface of TOCN films is suitable for fabricating electronic devices.

Cross-sectional structures of transparent hazy films

The loose internal structure will induce serious light scattering, and reduce the film's transparency (Yan et al. 2015). In this study, the cellulose nanofibrils with a dimension around 6–8 nm tightly intertwined with each other to form a uniform films noted cellulose nanofibrils film. Hence a dense and smooth cross section were observed from the cross-sectional structures of transparent hazy films (Fig. 7). Then a TOWF layer was coated on the upper surface of the cellulose

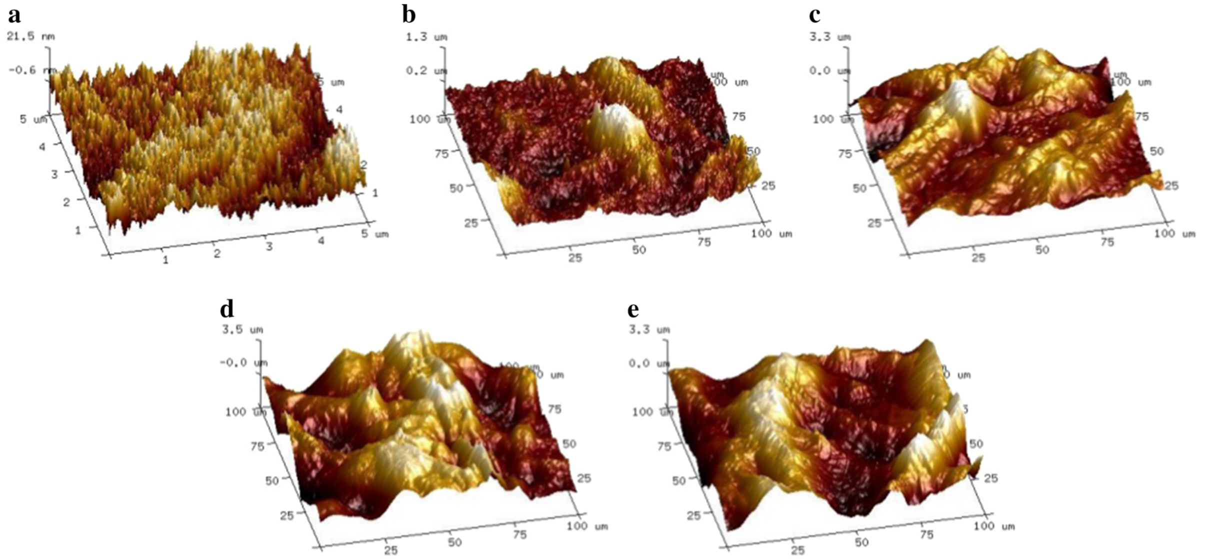

Fig. 6 3D AFM images of the top surface of the transparent films with various amount of micro-sized fiber coating. **a** 0 g/m^2, **b** 5 g/m^2, **c** 10 g/m^2, **d** 15 g/m^2, **e** 20 g/m^2

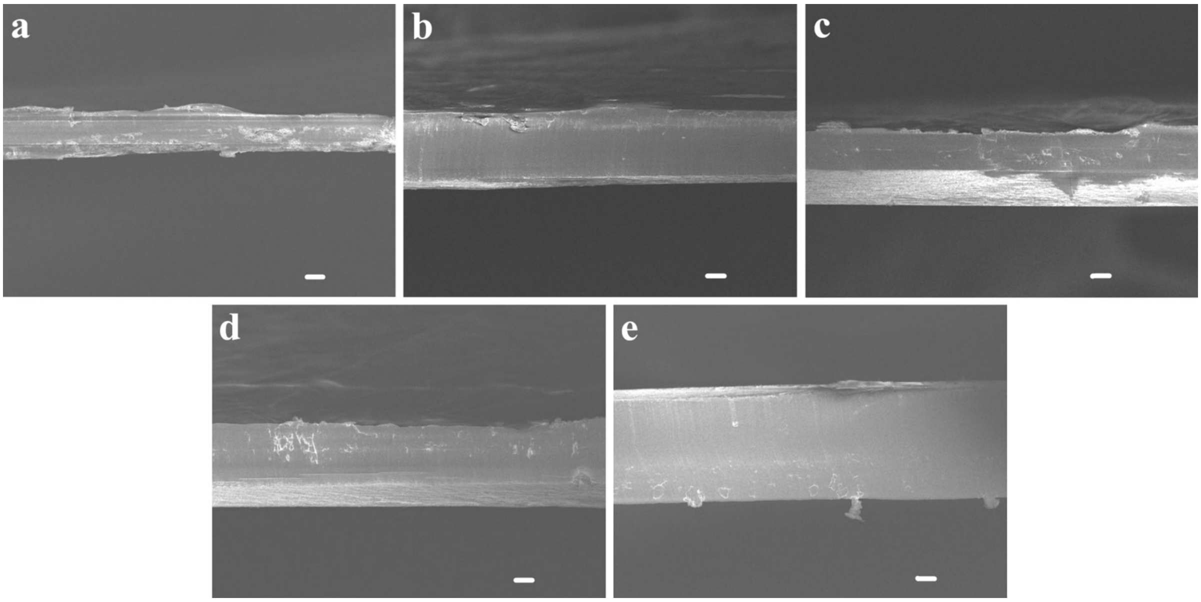

Fig. 7 Cross-sectional structure of the transparent films with various amount of micro-sized fiber coating. **a** 0 g/m^2, **b** 5 g/m^2, **c** 10 g/m^2, **d** 15 g/m^2, **e** 20 g/m^2, the scale bar is 10 μm

nanofibrils film, which has limited influence on the total transmittance and only induces the change of optical haze. In the cross-sectional SEM images, a bi-layer structure cannot be distinctly observed. The reason maybe is that the micro-sized fibers were embedded in the TOCN films. With the increase in the grammage of the TOWF coating layer, the film thickness will increase. The thickness of the transparent films revealed by FE-SEM, ranged from 29.6 to 42.5 μm. The increase of the film thickness will add the pass length of the light, which will increase the light scattering. However, in comparison to the surface morphology, the changing of the thickness has a limited influence on the optical haze.

Conclusions

In summary, highly transparent substrates with tuned optical haze ranging from 3.8% to 62.3% have been fabricated with coating of micro-sized TOWFs on top surfaces of TOCN films. These transparent films by coating method are beneficial for the fabrication of next generation green optoelectronic devices owing to a super-smooth surface with a roughness of 6 nm. According to SEM and AFM figures, we know that the coating layer with a rough surface results in a high haze for films without seriously decreasing total transmission. Such modified TOCN films show an anti-glare function, which is useful for outdoor displays. Substantial light scattering also possesses a potential application in solar cells, and OLED light systems. The TOCN films offer improved durability and flexibility for electronic devices. These transparent, hazy, flexible cellulose-based films are possible next-generation green, optical devices.

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