Cellulose Nanocrystal:Polymer Hybrid Optical Diffusers for Index-Matching-Free Light Management in Optoelectronic Devices

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A novel optical diffuser based on cellulose nanocrystals (CNCs) embedded in polydimethylsiloxane (PDMS) matrix, or cellulose nanocrystal:polymer (CNP) is reported. By offering simple low-cost fabrication process as well as compatibility with large-scale production, the proposed optical diffuser is a better choice for integration into optoelectronic devices for light management compared to other cellulose-based diffusers due to its physical durability and the lack of requirement for index-matching between the diffuser and the optoelectronic device thanks to the unique surface properties, mechanical flexibility, and optical transparency offered by PDMS as the matrix material. It is demonstrated that CNCs are an excellent filler material to pair up with PDMS in an optical diffuser providing highly efficient broadband light diffusion in visible and near-infrared range of light at very low concentrations (≈1 wt%). At its optimized form, a CNP hybrid optical diffuser is capable of achieving very high haze values (up to 85%) while maintaining a high degree of transparency (≈85%) at the same time. As a proof of concept, light management capabilities of CNP hybrid optical diffusers are leveraged to demonstrate their potential for light extraction improvement in organic light-emitting diodes and light absorption enhancement in thin-film solar cells.

1. Introduction

Optical diffusers provide soft light with uniform spatial and directional intensity distribution. They have been extensively exploited for the purpose of uniform backlighting, brightness enhancement, efficiency improvement, and increased sensitivity in liquid crystal displays (LCDs),[1–3] light-emitting diodes (LEDs),[4–6] solar cells,[7–9] and photodetectors,[10–12] all via excellent light scattering properties of optical diffusers.

As a nontoxic and biodegradable material, cellulose nanocrystals (CNCs) are environment friendly. They are directly extracted from natural resources such as wood and other fiber supplies available in plants. As a high-molecular-weight linear polymer formed of monomers linked together by glycosidic oxygen bridges,[13] they offer desirable bulk and nanoscale properties (e.g., high tensile strength, high surface area for interaction with surrounding species)[14] that make them suited for use as substrates in devices such as sensors,[15] solar cells,[16] LEDs,[17] and transistors.[18] In addition, recently, cellulose nanofiber (CNF)-based films and papers have attracted attention for their light scattering capabilities.[9,19,20] Both CNCs and CNFs are very similar in terms of chemical composition and can be derived from the same source but are morphologically different in that CNFs are long (in order of micrometers) and flexible composed of both crystalline and amorphous parts while CNCs are short rod-like crystals with lengths in the range of a few hundreds of nanometers.[21]

Here, we propose, fabricate, and characterize a new class of optical diffusers based on CNP hybrid films. While some of the recently proposed volumetric diffusers are made of rare earth materials such as cerium[22] which can drive up the cost of fabrication, the proposed diffuser relies on a ubiquitous and environment-friendly material, cellulose, which is cost-effective to produce. Our hybrid diffuser achieves high haze values (up to 85%), which is significantly higher than cellulose nanofiber/wood pulp diffusers,[9,20] while maintaining a high degree of transparency (≈85%). In addition, unlike previously reported cellulose nanofiber/wood pulp optical diffusers which require an additional index-matching layer between the diffuser and the device for efficient light coupling,[9,20] no index-matching is required for the diffuser here due to the hydrophobic surfaces

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offered by polydimethylsiloxane (PDMS) that allows direct attachment onto device substrates.

Through extensive characterization including spectrophotometry, laser beam delivery, and angular power distribution, we prove that CNCs are an excellent filler material candidate in a volumetric optical diffuser thanks to their rod-like shape and wavelength-scale size in the order of a couple of hundreds of nanometers,[23] which offer excellent broadband light softening in both the visible and near-infrared regime. Where most volumetric diffusers require a filler material with concentrations up to 15 wt% for efficient light scattering,[24,25] CNP hybrid diffusers offer highly efficient light diffusion at CNC concentrations as low as 1 wt%. We further leverage the light management capabilities of CNP hybrid optical diffusers to demonstrate their potential in light absorption enhancement in semiconductor thin films and light extraction improvement in organic LEDs.

2. Results and Discussion

2.1. Optical Diffusion, Transmission, and Haze Analysis

Figure 1a shows a photograph of CNP hybrid optical diffusers with different CNC concentrations (from left to right: 0.5, 1, 2, and 4 wt%). As is obvious from the appearance of the samples in the picture, a change in transparency and light diffusion behavior of the samples is observed with an increase in CNC concentration. The higher concentration of CNC provides lower transparency while offering better light diffusion performance. This is in agreement with other types of volumetric diffusers as it is known that the density of the filler material can directly affect the transparency and the haze (the percentage of total transmitted light that is diffusely scattered) of the diffuser.[24–26] Additionally, it was observed that the flexibility and mechanical properties of PDMS enable the CNP diffusers to withstand harsh physical stresses such as stretch, twisting, and bending (Figure 1b) without any change in their properties, a feature that is not offered by cellulose nanofiber/wood pulp optical diffusers.[9,20] By employing transmission electron microscopy (TEM) of the CNCs (Figure S1, Supporting Information), both individual and aggregated clusters of CNCs were found to be present in the raw material, a feature which we believe could be carried over once CNCs are embedded in PDMS. In addition, helium ion microscopy (HIM) was used to characterize the morphology of CNC embedded films. The HIM images of PDMS samples with embedded CNCs are provided in Figure S2 (Supporting Information). From the obtained images, no air defects resulting from delamination of PDMS from CNCs were observed, emphasizing the role of CNCs as the sole responsible for light scattering effect inside the PDMS matrix. However, due to the similarity of the composition of CNCs and PDMS (one a naturally occurring polymer and the other a synthetic polymer), it was not possible to observe the distribution of CNCs inside PDMS due to the lack of any contrast between the two materials during imaging.

The observed aggregations in TEM images (with sizes in the range of a few micrometers and highly packed densities in contrast to individual CNCs) have the potential to significantly enhance the scattering efficiency of CNCs when embedded in a PDMS matrix. We believe that a combination Rayleigh and Mie scattering can be responsible for light scattering in CNP hybrid diffusers, the former resulting from individual CNCs and the latter stemming from possible clusters of aggregated CNCs in the matrix material. On the other hand, it is intuitive to conclude that Mie scattering, which is known to be far less dependent on the wavelength of light than Rayleigh scattering,[27] is the dominant scattering mechanism here based on the significant insensitivity of the performance of CNP hybrid diffusers to the incident light wavelength as discussed in the following.

In order to optimize the amount of filler material for the hybrid diffuser, we measured the transmittance and haze of various CNP films with different CNC concentrations as a function of wavelength for the visible and near-infrared regions as is depicted in **Figure 2**. Transmittance and haze of a commercially available diffuser (labeled as

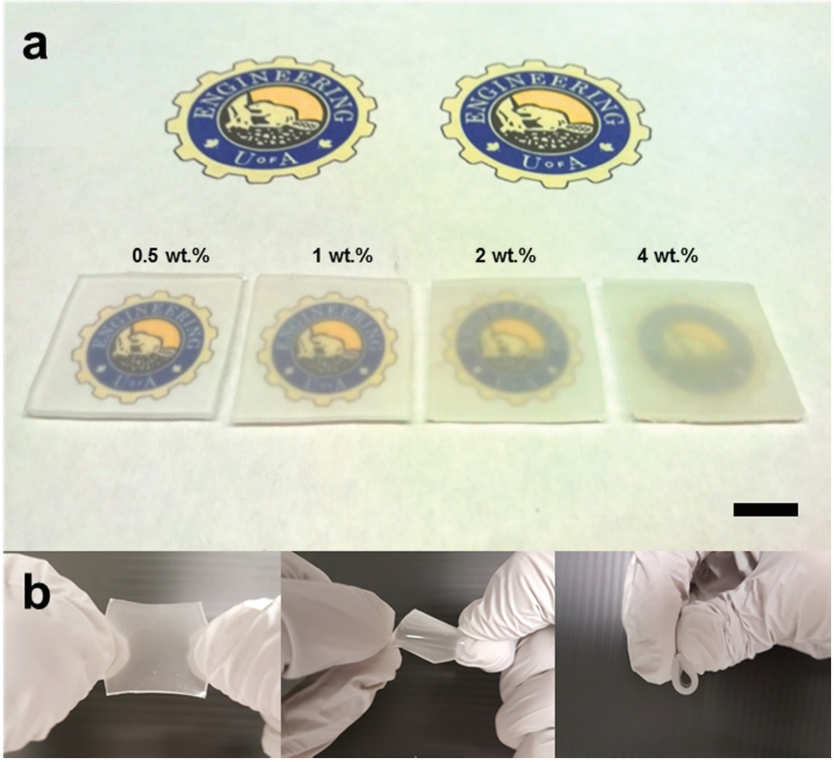

Figure 1. a) Photograph of CNP hybrid optical diffusers with different CNC concentrations (from left to right: 0.5, 1, 2, and 4 wt%). The change in transparency and light diffusion behavior of the samples with an increase in CNC concentration is obvious from the picture. (The scale bar is 1 cm.) b) Physical durability of the diffusers allows application of stretch, twisting, and bending without any effect on their optical properties.

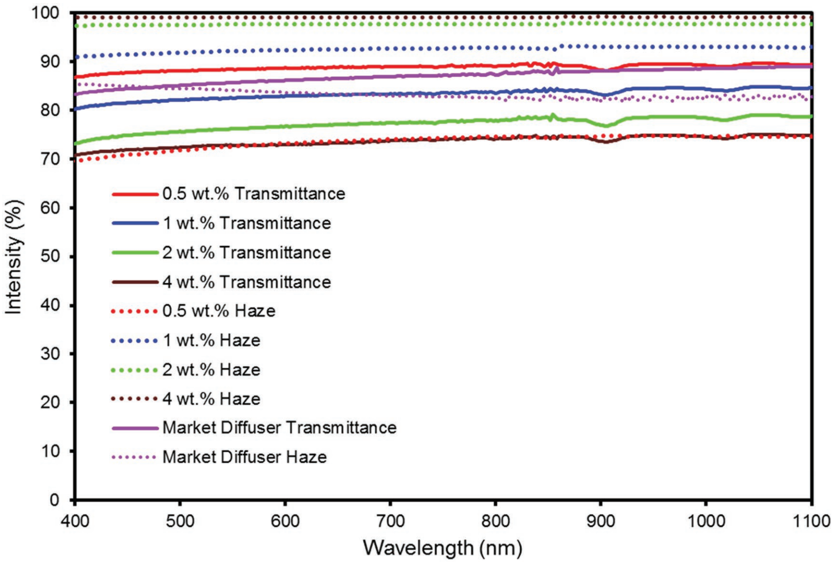

Figure 2. Transmittance and haze of CNP hybrid optical diffusers with different concentrations (wt%) of CNC as a function of wavelength. High transmittance and haze values are achievable across a wide range of wavelengths with the proposed diffuser structure. Transmittance and haze of a commercially available diffuser are also included for comparison.

market diffuser) from a solar simulator system were also measured for comparison. In the CNP device, both high transmittance and high haze values are achievable across a wide range of wavelengths (400–1100 nm) with the proposed diffuser structure, which can be attributed to the difference in refractive index of cellulose (≈1.55–1.6)[28] and PDMS (≈1.4).[29] No significant absorption was observed from the samples. Indeed, it is known that PDMS and CNCs have negligible absorption in the visible and near-infrared range of light. Compared to other cases, a 1 wt% concentration for CNC in PDMS provides a high level of haze (more than 90%) while maintaining a high level of transparency at the same time. Furthermore, it provides performance comparable to that of a commercial diffuser. In all the other cases, either the transparency or the haze level is traded. For instance, in the case of a 4 wt% CNC concentration, despite the fact that almost all light that is passing through the sample is being diffused (≈100% haze), the transmittance is significantly lower than the acceptable level for an optical diffuser. In addition, from the results obtained, it can be concluded that the performance of CNP optical diffusers is almost independent of the wavelength of incident light which is a direct consequence of the randomness of their light scattering structure. Moreover, the calculated scattering mean free path for various concentrations of CNCs (Figure S3, Supporting Information) revealed that the average distance between scattering events is reduced with increasing the concentration of CNCs but this reduction becomes less significant at higher concentrations.

Optical diffusion of a 635 nm laser beam by CNP hybrid optical diffusers with different concentrations of CNC and their comparison with a market diffuser are demonstrated in **Figure 3**. The addition of CNC filler to PDMS as bulk material produces the observed light scattering, since no noticeable change on the laser beam was observed when passing through a PDMS film with no CNC addition (i.e., 0 wt%). Better light softening performance and wider scattering angles were observed with the increase in CNP ratio, in agreement with the observations from wavelength-dependent haze measurements in Figure 2. The sample with 1 wt% CNC offers almost the same performance as the market diffuser while providing wider scattering angles. This is attributed to the superior light scattering capabilities of CNCs as the filler material.

Figure 4a–d illustrates the angular intensity distribution of a 635 nm laser beam after passing through a CNP hybrid optical diffuser with different concentrations of CNC. As the concentration of filler material increases, the light scattering angles are wider and the intensity distribution becomes more uniform, consistent with the visual observations of light diffusion from Figure 3. In the case of the sample with 4 wt% CNC concentration, the intensity distribution is very close to an ideal diffuse surface with Lambertian distribution (dashed line). This emphasizes the light diffusion ability of the hybrid diffuser being able to offer a Lambertian-like distribution with only 4 wt% concentration of the filler material.

Since it is expected from an optical diffuser to maintain its performance at different incident light angles, the dependence of the performance of CNP hybrid optical diffusers on the incident light angle for different concentrations of CNC was investigated as shown in Figure 4e. It is clear that at oblique angles, CNP hybrid optical diffusers offer even higher performance than a normal light incidence. The incident light has to travel a longer path inside the light scattering medium at oblique angles and, in this case, the chance of light being scattered increases due to the high density of scattering sites created by CNCs embedded into PDMS matrix. The

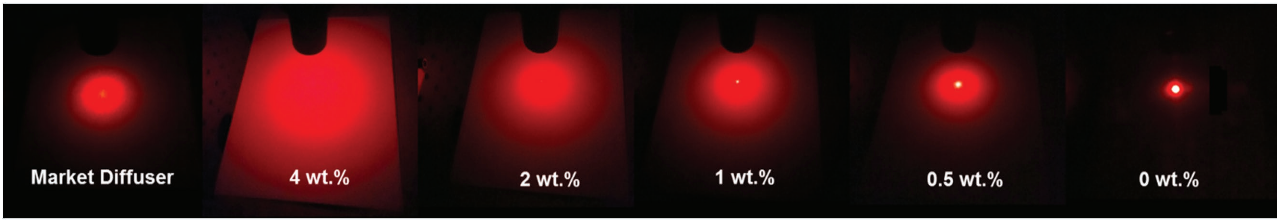

Figure 3. Optical diffusion of a 635 nm laser beam by CNP hybrid optical diffusers with different concentrations (wt%) of CNC and comparison with a market diffuser.

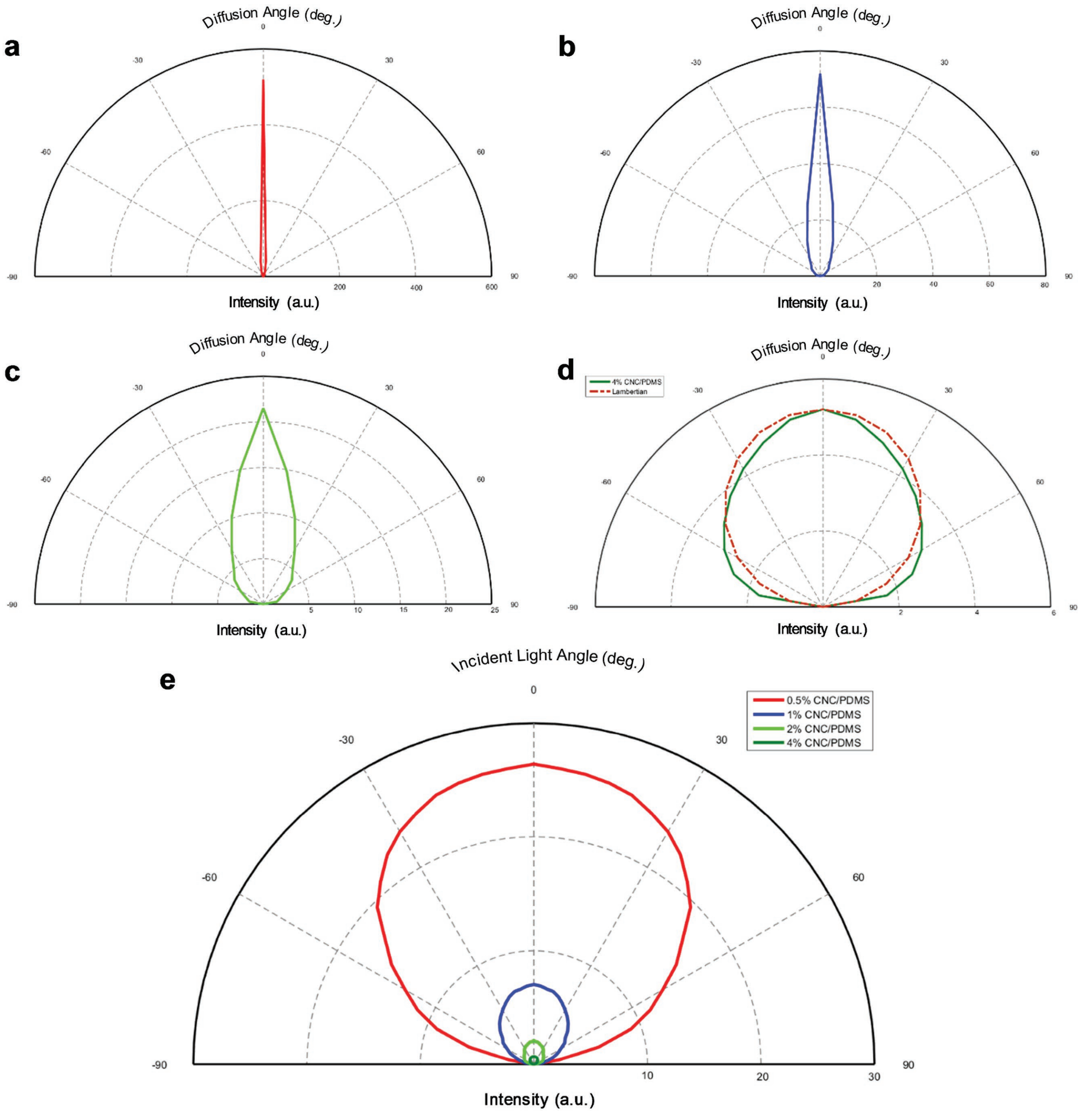

Figure 4. Angular intensity distribution of a 635 nm laser beam after passing through a CNP hybrid optical diffuser with different concentrations of CNC: a) 0.5, b) 1, c) 2, and d) 4 wt%. As the concentration of CNC increases, the distribution becomes more uniform. The sample with 4 wt% concentration is very close to an ideal diffuse surface with Lambertian distribution (dashed line). e) Dependence of specular transmission of CNP hybrid optical diffusers on the incident light angle for different concentrations of CNC. At oblique angles, more light diffusion can be observed from the diffusers.

maintenance of light diffusion efficiency at different incident angles suggests that the CNP hybrid diffusers can be useful in ambient light harvesting applications since in the case of ambient light, the incident direction can be randomly oriented most of the times.

In a volumetric diffuser, because the light scattering material is distributed inside the bulk material, the thickness of the diffuser can play an important role in light management performance. Therefore, the thickness optimization of CNP hybrid films for the desired transparency and haze is an important step in the design of an optimally performing optical diffuser. Based on the results in Figure 2, it was concluded that a 1 wt% concentration of CNC offers an optimal combination of transparency and haze. Therefore, this configuration was used for the thickness optimization. As shown in Figure S4 (Supporting Information), a thin diffuser layer with thicknesses less than or close to the scattering mean free path (≈0.5 mm for 1 wt% CNC as depicted in Figure S3, Supporting Information) is not efficient in light scattering due to small probability of scattering events to happen. On the other hand, although thicker diffuser film provides a higher degree of haze due to the longer light traveling path created inside the scattering medium, the transmittance of the diffuser is reduced. Thus, as a figure of merit, an optimized optical diffuser should provide the highest transmittance × haze value possible. In that regard, our 1 mm thick diffuser provides a significantly higher average figure-of-merit value (72.25%) than that of cellulose nanofiber/wood pulp optical diffusers[9,20] (59.85 and 63.75%, respectively). This shows the superiority of CNP hybrid optical diffuser in light management compared to other cellulose based diffusers.

2.2. Index-Matching-Free Light Management in Optoelectronic Devices

Due to the excellent light scattering capabilities offered by CNP hybrid optical diffusers, one potential application of the proposed optical diffuser is the enhancement of light extraction properties of LEDs. LEDs are known to suffer from light trapping inside the substrate due to total internal reflection contributed by the substrate/air refractive index contrast.[30,31] The proposed optical diffuser can be exploited to enhance light extraction efficiency in an organic LED by being directly attached to the glass substrate of the device. This can provide local scattering sites and refractive index change to suppress part of the total internal reflection at the substrate/air interface as shown in **Figure 5**a. Unlike reported cellulose-based optical diffusers,[9,20] no index-matching layer between the diffuser and the device is required in this study due to the strong adhesion of PDMS film onto device substrates that leaves no air gap in between.

Figure 5b,c shows the performance of the LEDs as a function of applied bias voltage without and with diffusers. The luminance is substantially improved when the optical diffuser is attached, although devices show the same current density–voltage (*J*–*V*) behavior. This confirms that the integration of optical diffuser into the LED can improve the light extraction capability. A deeper look into the brightness of the devices with optical diffusers having various concentrations of CNC reveals that a 1 wt% CNP hybrid optical diffuser provides the highest light extraction performance. This can be attributed to its optimized transparency and haze. It was found that a 0.5 wt% concentration makes almost no difference in light extraction from a LED possibly because of its low level of haze and a 4 wt% concentration can result in lower luminance than the control device without a diffuser probably due to its lackluster degree of transparency.

The external quantum efficiency (EQE) and power efficiency of the device as a function of luminance without diffuser and with diffusers having different concentrations of CNC are illustrated in Figure 5d and Figure S5 (Supporting Information), respectively. The results are consistent with the observations in Figure 5c: 1 and 2 wt% CNP hybrid diffusers offer almost the same light extraction enhancement while a 0.5 wt% device results in fairly the same performance as the control device with no diffuser and the 4 wt% device provides a lower performance than the control device. Thanks to its superior light extraction capabilities, the proposed CNP hybrid optical diffuser is able to enhance the EQE of the device by about 15% on average which showcases its great potential for light extraction enhancement and superiority to cellulose nanofiber/wood pulp optical diffusers. These hybrid films are applicable to any previously fabricated LED device since the light extraction structure is not part of the device structure and can easily be laminated on any device and could be replaced in case of degradation.

The application of CNP hybrid optical diffusers can be extended to the enhancement of light absorption inside the active layer of thin-film solar cells and photodetectors. To demonstrate the capability of light absorption enhancement, CNP hybrid optical diffusers were integrated with a 200 µm thick silicon slab as illustrated in Figure S6a (Supporting Information). Figure S6b (Supporting Information) depicts the amount of absorption in the silicon slab versus incident light wavelength without diffuser and with diffusers having different concentrations of CNC. The absorption in the silicon slab can be significantly enhanced with the addition of a CNP hybrid optical diffuser. The optical diffuser extends the light path length inside the slab, and this increases the chance for light absorption. In addition, a portion of the observed absorption enhancement could be the result of lower light reflection from the surface of silicon since the optical diffuser can reduce the refractive index contrast between air (1) and the silicon slab (≈3) by its intermediate refractive index (≈1.5). However, the high concentrations of CNC are not as effective in enhancing the absorption due to the reduction in transmittance of the diffuser at high concentrations as shown in Figure 2. For example, a lower transmittance is observed from the 2 wt% CNC sample although it offers higher haze compared to its 1 wt% counterpart which can be considered the reason behind observing similar light absorption enhancement in silicon slab for both concentrations (Figure S6, Supporting Information). From these results, we conclude that 1 wt% for CNC is an optimized concentration for light management applications.

3. Conclusion

In summary, we reported on a volumetric optical diffuser based on CNP hybrid materials. By offering a simple and low-cost fabrication process as well as compatibility with large-scale production using CNC as filler, the proposed optical diffuser is well chosen to integrate into optoelectronic devices for light management especially due to its insensitivity to physical damage at its surface and the lack of requirement for an index-matching layer between the diffuser and the optoelectronic device thanks to the unique surface properties, mechanical flexibility, and optical transparency offered by PDMS as the bulk material. This CNP diffuser provides excellent capabilities for broadband light softening in both visible and near-infrared regime and highly efficient light diffusion at concentrations as low as 1 wt%. It was shown that, in its optimized form, a CNP hybrid optical diffuser is capable of achieving super-high haze values (up to 85%) while maintaining a high degree of transparency (≈85%). We leveraged the light management capabilities of CNP hybrid optical diffusers to demonstrate their potential for light absorption

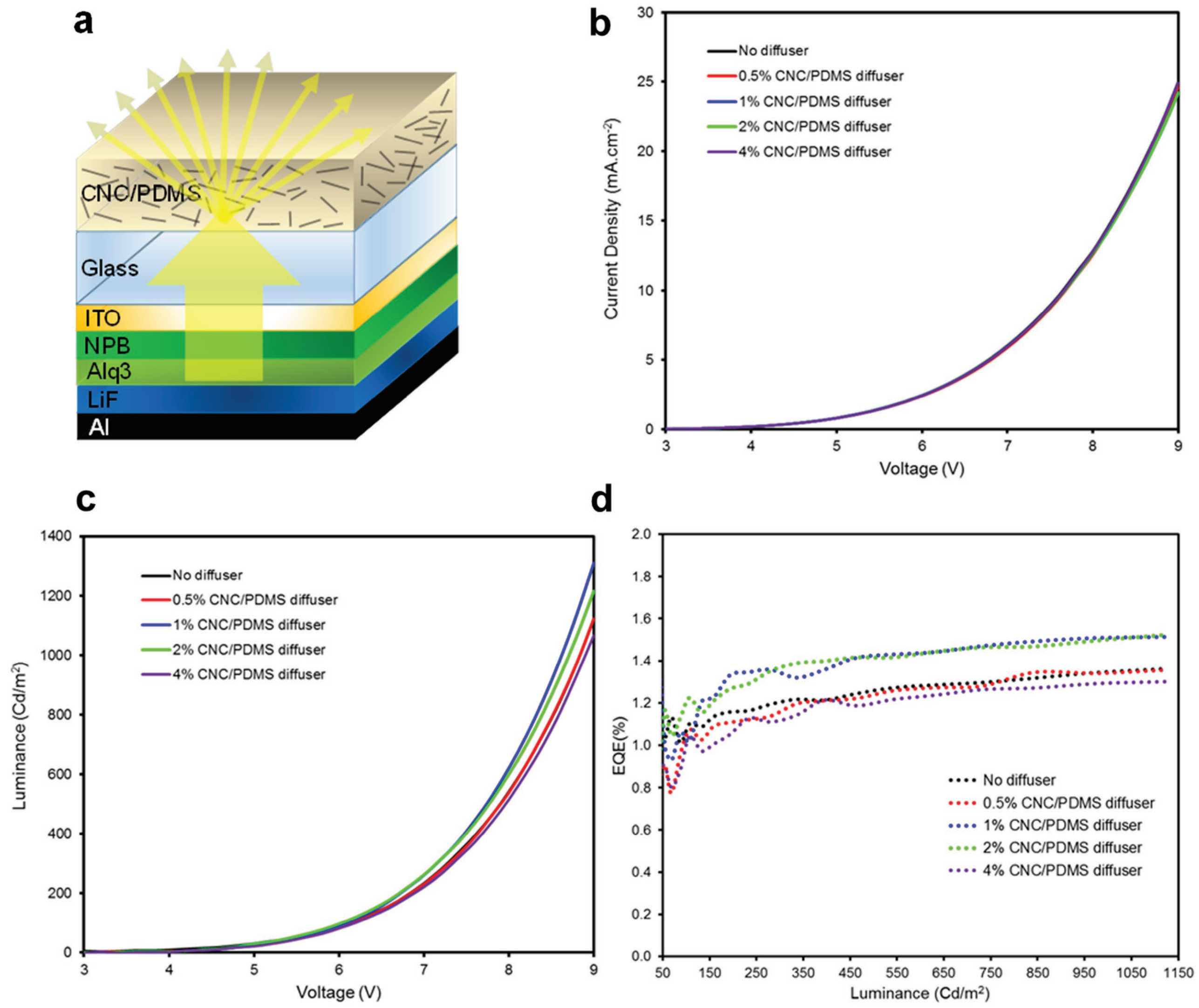

Figure 5. Light extraction enhancement in LEDs using CNP hybrid optical diffusers. a) Schematic of the device structure with the integration of hybrid optical diffuser. b) *J–V* curves of the device without and with diffusers. c) Device brightness as a function of applied bias voltage without and with diffusers. d) External quantum efficiency of the device without and with diffusers having different concentrations of CNC. The diffuser with 1% wt. CNC has the highest enhancement in the efficiency of the device.

enhancement in thin-film silicon solar cells and light extraction improvement in OLEDs.

4. Experimental Section

Synthesis of Cellulose Nanocrystals: Acid hydrolysis technique was used to synthesize CNCs.[32] In brief, the process was performed in two Pfaudler 50 gallon acid-resistant glass-lined reactors with a steam-heated jacket. Sulfuric acid with the concentration of 64% was used with an initial reaction temperature of 45 °C. This was followed by a centrifuge step using a GEA Westfalia SC-35 separator. The next step in the process was a microfiltration step performed by a GEA filtration-ultrafiltration plant. The wet CNC powders obtained from this step were then dried in an SPX-Anhydro Model 400 spray dryer plant under an inlet temperature of 220 °C and an outlet temperature of 85 °C.

Fabrication of CNP Hybrid Optical Diffusers: CNP hybrid optical diffusers with different concentrations of CNC were fabricated as follows. First, PDMS precursor was made by mixing a silicone elastomer with curing agent from a Sylgard 184 kit (Dow Corning) in 10:1 ratio. Different amounts of CNC powder (0.5–4 wt%) were then added to the PDMS precursor and vigorously mixed together using a combination of manual stirring and a vortex mixer to ensure uniform distribution of CNCs inside the mixture. The CNP mixture was then degassed in a vacuum desiccator for 60 min before being poured into a polystyrene petri dish. The resulting film was then left on a preleveled platform at room temperature overnight to form a uniformly thick film and then was cured on a hot plate at 80 °C for 2 h. The thickness of the film was controlled by carefully adjusting the amount of volume for the mixture that was being poured into the petri dish. After the curing step, the film could easily be peeled off due to the hydrophobicity of polystyrene.

Characterizations: The transmission electron microscopy images were obtained by a Hitachi S-9500 TEM. Helium ion microscopy was performed using a Zeiss Orion HIM. Transmittance and haze results were obtained using the 150 mm integrating sphere module of a Perkin-Elmer Lambda 1050 UV–vis–NIR Spectrophotometer. A custom-made fixture was used to hold the sample at the sample beam entrance port of the sphere. Angle-dependent specular transmissions were measured using the same setup with the sample being located on a rotating stage. For transmittance measurement, all the ports of the integrating sphere except the reference and sample beam entrance ports were kept closed. In the case of haze, the back port of the integrating sphere was opened to prevent the specular transmission from the sample to be captured by the detector. The scattering mean free path was calculated from the measured specular transmittance of the samples using the following equation

$$I = I_o e^{\frac{-d}{l_{\mathrm{MFP}}}} \qquad (1)$$

where I is the specularly transmitted beam intensity, I_o depicts the source beam intensity, d represents the film thickness, and l_{MFP} is the scattering mean free path.

Visual demonstrations of optical diffusion by samples were performed using a 635 nm laser diode (Newport) as the light source with the sample covering the output window of the laser diode. The light passing through the diffuser samples was projected on a white screen located in front of the laser diode. The angular intensity distribution of the laser beam was acquired by a Thorlabs power meter situated in the same horizontal plane as the center of the laser beam. The angular position of the power meter was then changed using a custom-built fixture to measure the light intensity at different angular positions with respect to the center of the laser beam.

The effect of CNP hybrid optical diffusers on light absorption enhancement in silicon was investigated using a 200 µm thick silicon slab with optical diffuser samples being directly attached to the silicon surface. The absorption measurement was accomplished by putting the sample at the center of the integrating sphere of a Perkin-Elmer Lambda 1050 UV–vis–NIR Spectrophotometer using a center mount sample holder.

In order to test the application of CNP hybrid optical diffusers on light extraction improvement in organic LEDs, diffuser samples were directly attached to the back side of the glass substrate of the device fabricated according to a published procedure.[34] The OLED structure was formed of ITO (indium-doped tin oxide)/NPB (*N*,*N*′-di(naphth-2-yl)-*N*,*N*′-diphenyl-benzidine) (70 nm)/$\mathrm{Alq_3}$ (tris-(8-hydroxyquinoline)) (60 nm)/LiF (lithium fluoride) (1 nm)/Al (aluminum) (100 nm). Quantum efficiency, luminance, the power efficiency, and current–voltage characteristics of the OLED device were measured using a HP 4110B source-meter, a calibrated luminance meter (Konica Minolta LS-110), and an Ocean Optics USB2000 spectrophotometer with an integrating sphere in the air and at room temperature.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

cellulose nanocrystals, light scattering, optical diffusers, optoelectronics, polymers

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