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A colour-tunable, weavable fibre-shaped polymer light-emitting electrochemical cell

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The emergence of wearable electronics and optoelectronics requires the development of devices that are not only highly flexible but can also be woven into textiles to offer a truly integrated solution. Here, we report a colour-tunable, weavable fibre-shaped polymer light-emitting electrochemical cell (PLEC). The fibre-shaped PLEC is fabricated using all-solution-based processes that can be scaled up for practical applications. The design has a coaxial structure comprising a modified metal wire cathode and a conducting aligned carbon nanotube sheet anode, with an electroluminescent polymer layer sandwiched between them. The fibre shape offers unique and promising advantages. For example, the luminance is independent of viewing angle, the fibre-shaped PLEC can provide a variety of different and tunable colours, it is lightweight, flexible and wearable, and it can potentially be woven into light-emitting clothes for the creation of smart fabrics.

Light-emitting electrochemical cells[1–6], in particular polymer light-emitting electrochemical cells (PLECs), have been widely studied for various applications, including flexible flat panel displays, signage and lighting[4–6]. Like organic light-emitting diodes (OLEDs) and polymer light-emitting diodes (PLEDs), PLECs have a structure that is usually composed of two metal electrodes connected to an organic semiconductor. However, PLECs differ in that mobile ions are incorporated into the organic semiconductor, thereby offering promising advantages such as low operating voltage, high electron/photon conversion efficiency and high power efficiency compared with OLEDs[7–18]. More importantly, PLECs do not require the use of low-workfunction cathodes composed of calcium or magnesium (which are sensitive in air). In contrast, PLEDs require a low-workfunction cathode and high-workfunction anode to realize efficient charge injection[19–21]. In a typical PLEC, the electroluminescent polymer layer forms an *in situ* light-emitting p-i-n junction for the injection of both electrons and holes from the electrodes[4,5,22]. This means that PLECs can be effectively operated with relatively rougher surfaces than is generally possible with OLEDs and PLEDs, which is advantageous when scaling them up for practical applications with low cost and high efficiency[23–25].

Based on these described advantages, the PLEC is particularly promising for use in portable and wearable electronics, which are being developed for a wide range of applications, from microelectronics to biomedicine, transport and areospace[26–32]. Conventional planar light-emitting devices, including both rigid and flexible films, cannot satisfy the basic requirements for such an application, including softness, light weight and weavability[33,34]. To this end, advances in the textile industry have suggested a useful direction in which to pursue a solution: if a PLEC is made into a continuous fibre using a melting or all- solution-based process, it can be woven into various flexible textiles or integrated into soft substrates for use in portable and wearable electronic devices[35].

For practical applications it is also important to be able to emit various colours from a single device, so modifications such as polymer blending and electrochemical doping can be made to develop the desired light-emitting devices[36]. However, it is difficult to independently tune colours as well as their intensities. To truly realize *in situ* colour tunability, tandem structures with two to three sub-cells connected in series have been widely explored, although these are limited by the available colours and complex fabrication procedures, low efficiency and high cost[37,38].

In this Article, a novel fibre-shaped PLEC is reported using all-solution-based processes. The fibre-shaped PLEC has a coaxial structure that includes a modified metal wire cathode and a conducting aligned carbon nanotube (CNT) sheet anode, with an electroluminescent polymer layer sandwiched between them. The fibre shape has unique and promising advantages, such as the luminance being independent of the observation angle. Furthermore, the use of an aligned CNT sheet as the anode can significantly decrease the light loss compared with the indium tin oxide of a conventional planar OLED[37,38]. A wide variety of colours are achieved by assembling two fibre-shaped PLECs that emit different colours, and the luminance of each can be continuously and independently tuned by varying the external current source. As expected, the fibre-shaped PLEC is lightweight, flexible and soft, and it can be woven into light-emitting textiles for large-scale applications.

Fabrication of fibre-shaped PLECs

Figure 1a,b shows the fabrication process for the fibre-shaped PLEC. A stainless steel wire was first dip-coated with a thin layer of ZnO nanoparticles, which functions as an electron transfer layer and protects the subsequently coated electroluminescent polymer from fluorescence quenching by the metal matrix. More importantly, the ZnO nanoparticle layer can significantly decrease the leakage current, enhancing the current efficiency and possibly resulting in a more balanced injection of electrons and holes[6,39]. The electroluminescent polymer layer, consisting of a blend of a blue light-emitting polymer (PF-B), ethoxylated trimethylopropane triacrylate (ETT-15) and lithium trifluoromethane sulphonate (LiTf), was deposited on the modified steel wire, also using a dip-coating method. PF-B was selected because of its built-in oligo (ethylene

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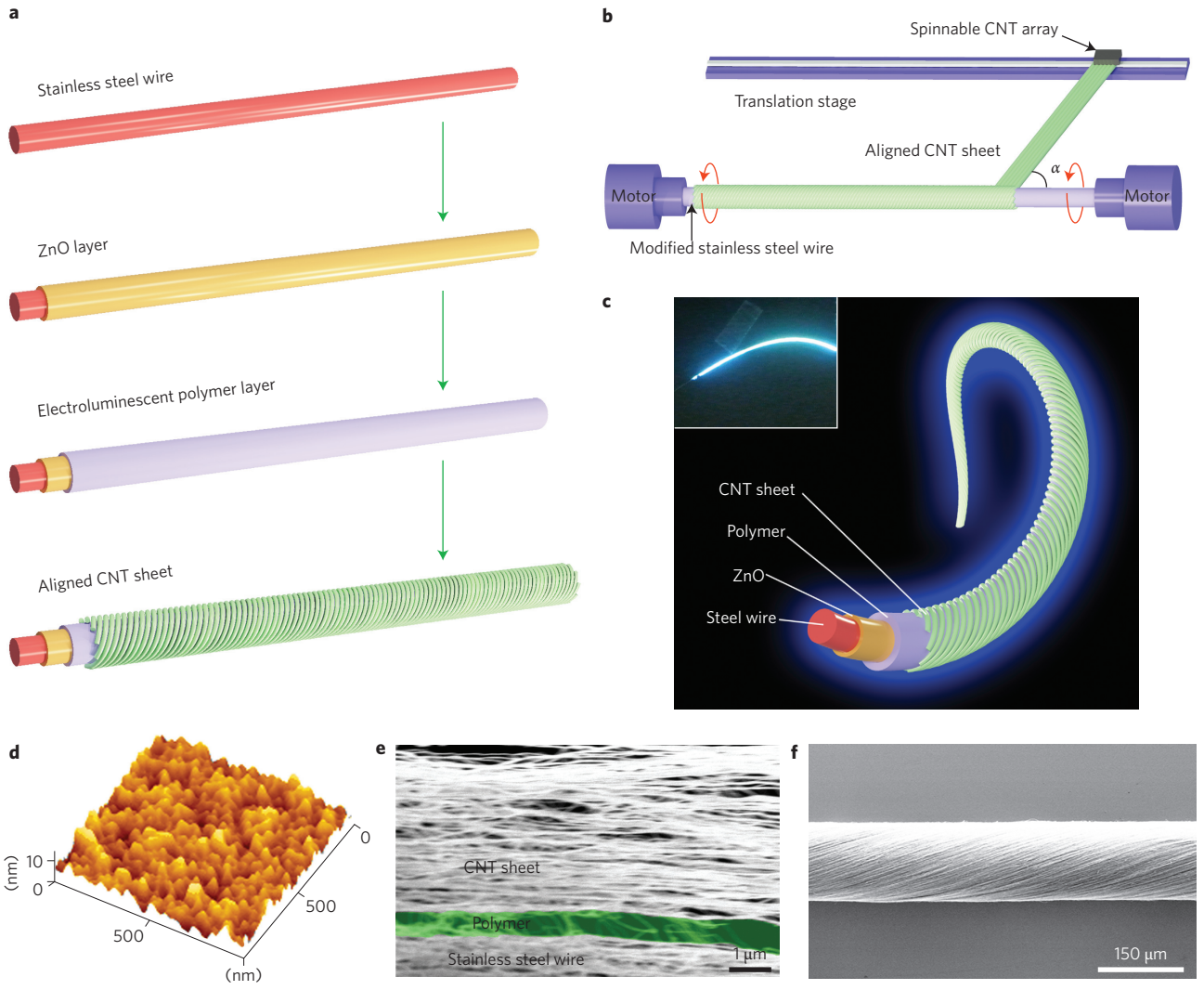

Figure 1 | Schematic illustration of the preparation and structural characterization of the PLEC. a, Schematic of fabrication of a fibre-shaped PLEC. **b**, Schematic of wrapping an aligned CNT sheet around a modified stainless steel wire. **c**, Schematic of the structure of a flexible fibre-shaped PLEC. Inset: photograph of a fibre-shaped PLEC biased at 10 V. **d**, AFM image of the polymer layer coated on the ZnO nanoparticle layer. **e**, SEM side-view image of a fibre-shaped PLEC. **f**, Aligned CNT sheet wrapped around the modified stainless steel wire with an angle of 15°.

oxide) side groups, which are beneficial for ionic conductivity and high electroluminescent performance (Supplementary Fig. 1). ETT-15 serves as the ionically conductive component, and LiTf provides the ionic dopant for the doped polymer in the formation of the p-i-n junction in the PLEC. An aligned CNT sheet was uniformly wrapped around the modified steel wire to produce the designed fibre-shaped PLEC (see inset of Fig. 1c for a photograph of a fibre-shaped PLEC with arcuate shape). The entire fabrication was carried out in air and is suitable for large-scale production.

The aligned CNT sheet plays a critical role in the successful fabrication of the fibre-shaped PLEC. The CNT has a multi-walled structure with a diameter of ~11 nm (Supplementary Fig. 2). The sheet was dry-drawn from a spinnable CNT array that was synthesized by chemical vapour deposition (Supplementary Figs 3 and 4)[40,41]. The wrapping of the aligned CNT sheet onto the modified steel wire is shown schematically in Fig. 1b. The two ends of the modified steel wire were fixed by two motors and a spinnable CNT array was fixed onto a precisely motorized translation stage. A continuous, aligned CNT sheet was drawn out of the spinnable CNT array and attached onto the modified steel wire at an angle α. The thickness of the aligned CNT sheet on the modified steel wire was accurately controlled by varying the helical angle and width of the CNT sheet.

The flexibility of the aligned CNT sheet is important in order to achieve close and stable wrapping on the fibre-based substrate, and had been investigated by winding it on a flexible polymer fibre (Supplementary Fig. 5). The resistance of the resulting fibre varied by less than 6% after bending for 1,000 cycles (Supplementary Fig. 6). The CNTs were highly aligned, thereby providing the sheet with high electrical conductivities on the order of 10^2 to 10^3 S cm^{-1}. The aligned CNT sheet (thickness of 18 nm) was optically transparent with a transmittance of over 87% at wavelengths above 550 nm (Supplementary Fig. 7) and a symbol located under the CNT sheet could be clearly observed (Supplementary Fig. 8). As a result of the high contact area between the aligned CNT and electroluminescent polymer layer, the aligned CNT sheet is closely attached to the modified steel wire predominantly by van der Waals forces.

Fabrication of the fibre-shaped PLEC was tracked by scanning electron microscopy (SEM). The stainless steel wire had a uniform diameter along the axial direction, with a smooth outer surface (Supplementary Figs 9 and 10), and the ZnO nanoparticles were uniformly coated on the stainless steel wire with an average thickness of 45 nm (Supplementary Fig. 11). Supplementary Fig. 12 presents an SEM image after the electroluminescent

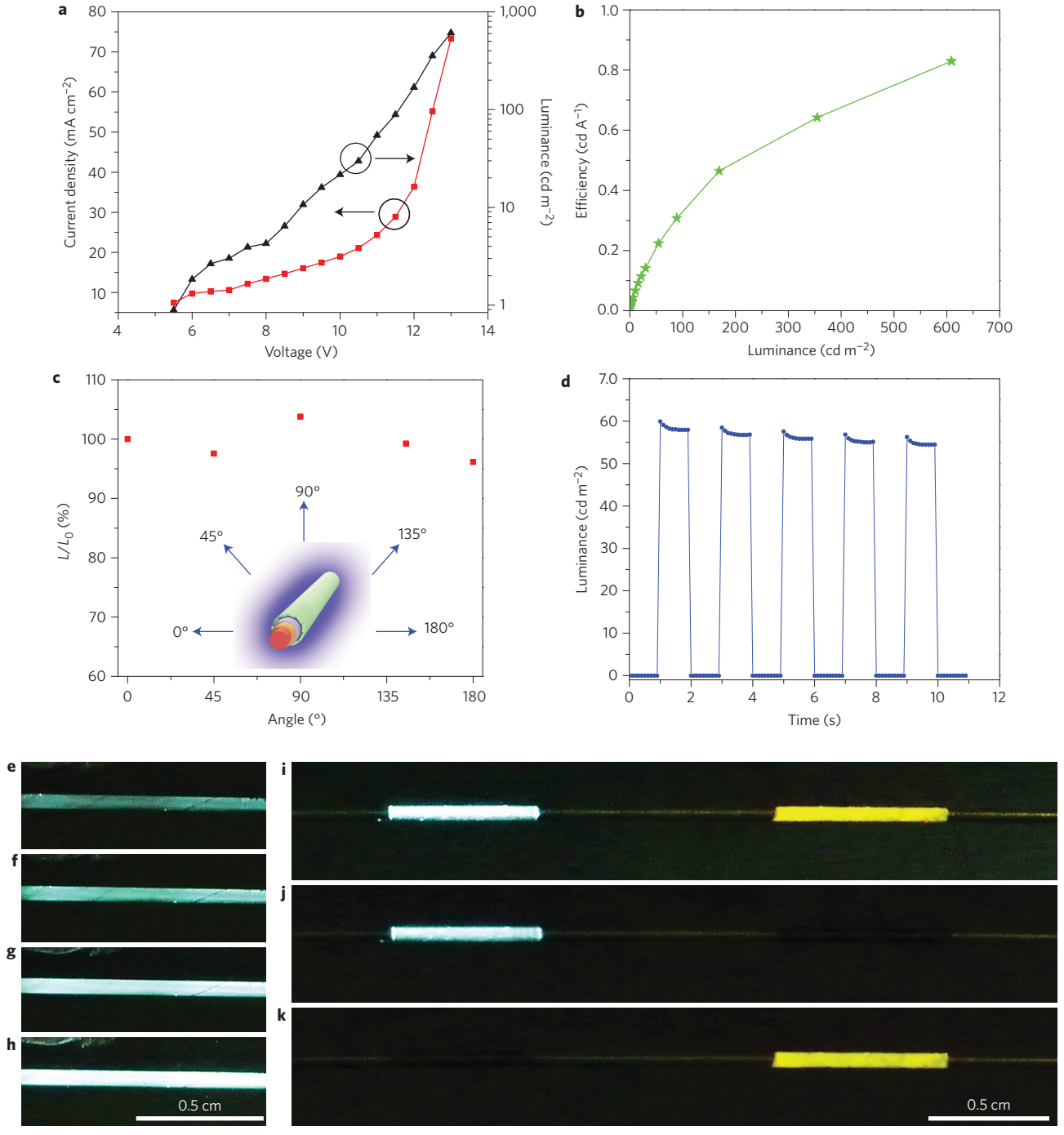

Figure 2 | Characterization of a fibre-shaped PLEC. a, Current density–luminance–driving voltage characteristics of a fibre-shaped PLEC. **b**, Current efficiency–luminance characteristics of a fibre-shaped PLEC. **c**, Dependence of luminance on angle of a fibre-shaped PLEC. L_0 and L correspond to luminance measured at 0° and the other angle, respectively. **d**, Transient light emission response under voltage pulses between 0 and 11 V with a 50% duty cycle for an initially charged fibre-shaped PLEC (50% duty cycle at 0.5 Hz). **e**–**h**, Photographs of a blue-light fibre-shaped PLEC biased at increasing voltages of 6 V (**e**), 7 V (**f**), 8 V (**g**) and 9 V (**h**). **i**–**k**, Fibre-shaped PLEC displaying blue and/or yellow light at its ends when biased at 9 V.

polymer layer has been applied. Importantly, the outer surface appears uniform and smooth, without obvious aggregates or curved structure, favouring a close and stable wrapping of the aligned CNT sheet. The surface smoothness of the electroluminescent polymer layer was also characterized by atomic force microscopy (AFM), and the roughness varied by less than 5 nm (Fig. 1d). The side view presented in Fig. 1e also shows the formed uniform electroluminescent polymer layer (thickness of ~500 nm), while Fig. 1f and Supplementary Fig. 13 present typical SEM images after the CNT sheet has been wrapped around the modified steel wire (low and high magnifications, respectively). The CNTs remain highly aligned, maintaining their high electrical conductivity during wrapping. Supplementary Figs 14 and 15 show photographs of the resulting fibre-shaped PLEC, which can be bent easily into various forms. Metal wires with different diameters may be used to fabricate these fibre-shaped PLECs (although wires with a diameter of 510 μm have been studied here if not specified). Note that some attempts have

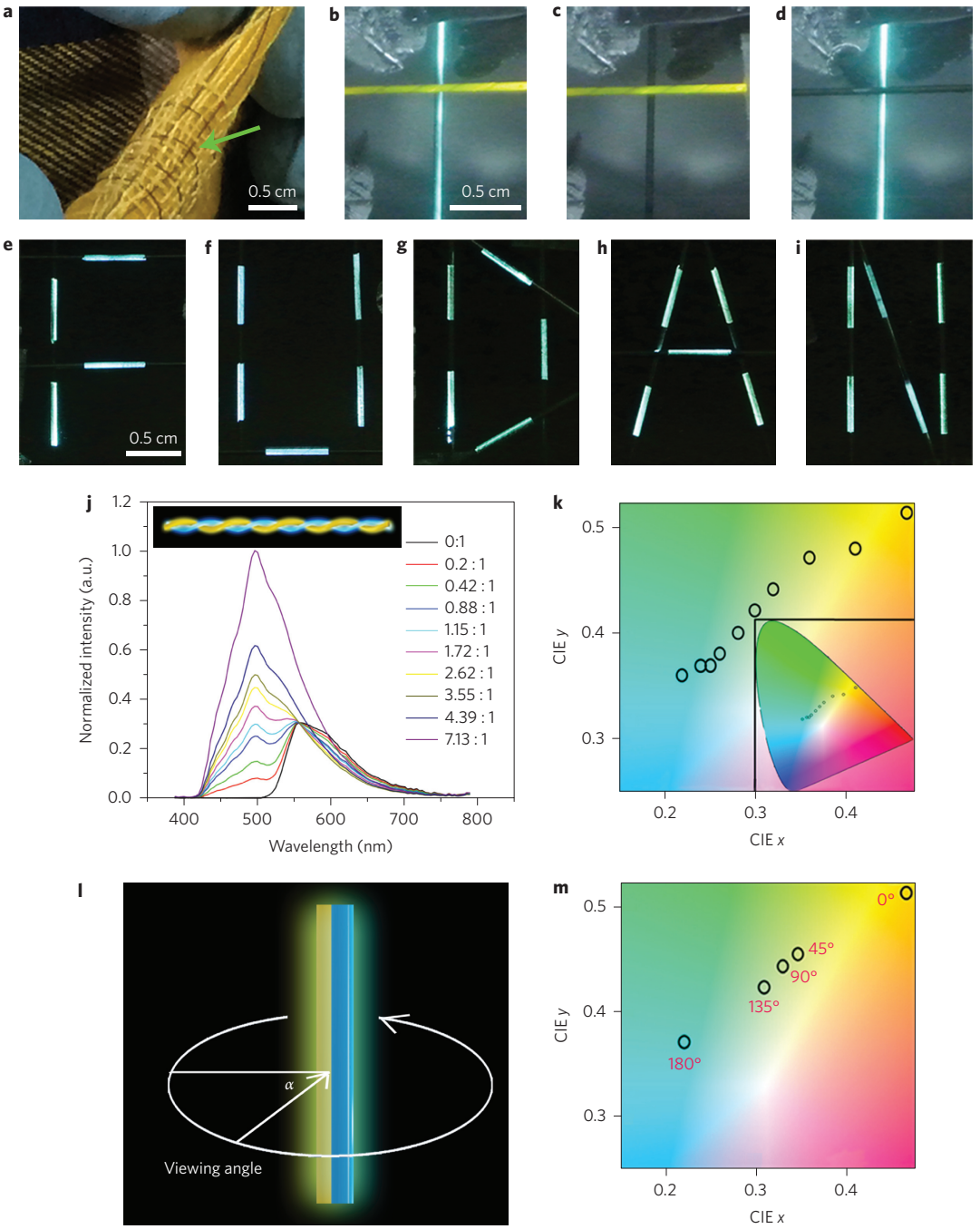

Figure 3 | Integrated PLEC fibres and textiles. a, Textile under bending and twisting. **b–d**, Two fibre-shaped PLECs with different colours being selectively illuminated (biased at 10 V). **e–i**, Fibre-shaped PLECs being woven into a 'FUDAN' pattern (biased at 9 V). **j**, Electroluminescent spectra with the brightness ratios of blue to yellow shown on the right (inset schematic: two co-assembled fibre-shaped PLECs). **k**, *x,y* chromaticity coordinates controlled by adjusting the brightness of two fibre-shaped PLECs of different colours. **l**, Schematic of testing method. **m**, Dependence of *x,y* chromaticity coordinate on viewing angle.

been made to fabricate electroluminescent fibres based on OLEDs and inorganic phosphors (www.laserfocusworld.com/articles/2013/07/thread-becomes-functional-oled-emitter.html and www.lytec-asia.com/products.html)[42], but complicated fabrication processes are typically required.

Properties of fibre-shaped PLECs

The PLEC fibre was first driven to evaluate the time required to establish the p-i-n junction and device lifetime at 30 mA cm^{-2}. As shown in Supplementary Fig. 16, the brightness of the device rises gradually to a peak value of 125 cd m^{-2} due to the gradual formation of a p-i-n junction in the electroluminescent polymer layer in the first 21 min (the time to establish such a p-i-n junction is determined mainly by the speed of ionic migration in the emissive layer). The emission intensity then gradually reduces to 63 cd m^{-2} in the following 4 h. We note that such a rather short operational stability is not uncommon in PLEC devices. Significant efforts have recently been reported in the literature to address this issue, with some success[23,43–46]. Many of those techniques could be introduced into or adapted for the fibre-shaped PLEC.

The fibre-shaped PLEC was further characterized after passing a current of 15 mA cm^{-2} for 10 min. The current density–luminance–driving voltage characteristic curves and current efficiency–luminance characteristic curves are presented in

Fig. 2a,b. Light emission occurred at 5.6 V (at a light intensity of 1 cd m^{-2}) and reached a peak value of 609 cd m^{-2} at 13 V. The current efficiency increased with increasing brightness and reached 0.83 cd A^{-1} at the end, with an external quantum efficiency of 0.35%. According to the CIE 1931 standard colour-matching functions, the emitted blue light can be demonstrated by *x*,*y* chromaticity coordinates (0.22, 0.36) (Supplementary Fig. 17). Due to the one-dimensional structure, which offers luminance in all directions, the brightness is almost independent of observation angle (Fig. 2c). The turn-on response of light emission from the p-i-n junction in the active layer was also investigated by means of a pulsed voltage operation (Fig. 2d). The pre-charged PLEC displays a rapid turn-on response that is similar to that of a conventional OLED. Figure 2e–h and Supplementary Fig. 18 also show that the PLEC fibre exhibits increasing blue light emission with increasing voltage. The uniformity of the brightness in the fibre-shaped PLEC was quantitatively compared at different locations biased at 9 V, and was found to vary by less than 7.8%.

The emitting mechanism of the fibre-shaped PLEC is described in existing literature.[4,5,22]. Ions in the electroluminescent polymer layer are redistributed upon application of a voltage, establishing electric double layers at the cathodic and anodic interfaces that allow electron and hole injection, respectively. Electrons are injected into the electroluminescent polymer layer through the ZnO nanoparticles from the stainless steel wire, while holes are injected into the electroluminescent polymer layer from the aligned CNT sheet. The injected electrons and holes are electrostatically attracted, compensating an electrochemical doping process, with the electroluminescent conjugated polymer becoming n-doped at the cathode and p-doped at the anode. Eventually, a light-emitting p-i-n junction is formed in the electroluminescent polymer layer that facilitates electron and hole injection from the steel wire and aligned CNT sheet, respectively.

This fibre shape has many unique and promising advantages. As already mentioned, its brightness is almost independent of viewing angle (Fig. 2c), which is important for the luminance when in use. Although, here, blue light has been investigated to demonstrate the fibre-shaped PLEC, other colours can also be realized by varying the polymer emitter. For instance, a yellow fibre-shaped PLEC has been produced with a similar structure, but with a yellow emissive layer instead. According to the CIE 1931 standard colour-matching functions, the emitted yellow light can be demonstrated by *x*,*y* chromaticity coordinates (0.46, 0.52) (Supplementary Fig. 19). Accordingly, different colours can be integrated into a single fibre device with a similar fabrication process; that is, a steel wire coated with ZnO nanoparticles is successively dip-coated with electroluminescent polymer layers that emit light of different colours, and an aligned CNT sheet is then wrapped onto the modified steel wire. A simple model with two colours at the end is shown in Fig. 2i. The two colours can be selectively lit, depending on the application requirement (Fig. 2j, k). Similarly, a series of fibre-shaped PLECs can also be made alternately on a metal wire (Supplementary Fig. 20).

The fibre-shaped PLEC is flexible, and its brightness was maintained at above 90% of its maximum after bending with a radius of curvature of 6 mm for 100 cycles (Supplementary Fig. 21). The surface structure of the electroluminescent polymer and the aligned CNT layer were further tracked by SEM images before and after bending (Supplementary Fig. 22). No obvious damage was observed in either layer after deformation. This means that the fibre-shaped PLEC can be easily woven into flexible electronic clothes to form patterns (Fig. 3a and Supplementary Fig. 23). Figure 3b–d shows that two crossed fibre-shaped PLECs can be selectively lit to produce various configurations. The fibre-shaped PLECs can also be easily woven into patterns, for example to form the word 'FUDAN', as in Fig. 3e–i, and each letter of the word may be selectively lit (Supplementary Fig. 24 and Supplementary Movie 1).

As previously mentioned, it remains challenging to emit different colours in practical applications. Here, fibre-shaped PLECs were woven into clothes and show tunable colours, as expected. The luminance of each fibre-shaped PLEC can be continuously and independently tuned by varying the external current source. The presented technology also provides a useful platform with which to adjust colours by co-assembling two PLEC fibres that emit different colours (Fig. 3j, inset), for example, yellow and blue. While keeping the brightness of the yellow PLEC fibre unchanged, the voltage applied to the blue PLEC was gradually raised to increase the blue-to-yellow brightness ratio from 0 to 7.13, thereby adjusting the resultant colour (Fig. 3j). The brightness of the blue PLEC was enhanced to achieve *x*,*y* chromaticity coordinates ranging from (0.46, 0.52) to (0.22, 0.36) (Fig. 3k). Based on a similar strategy, the area ratio of blue to yellow lights can be varied by changing the viewing angle to tune the brightness ratio. In this way, the *x*,*y* chromaticity coordinates were changed from (0.46, 0.52) to (0.22, 0.36) by increasing the angle from 0° to 180° (Fig. 3l,m).

Conclusion

In summary, a fibre-shaped PLEC has been developed by designing a coaxial structure and incorporating a flexible and conducting CNT sheet anode. We have demonstrated that the entire manufacture of fibre-shaped PLECs can be carried out in air using a simple dip-coating technique that is compatible with high-speed and low-cost roll-to-roll fabrication. The fibre-shaped PLEC provides the same brightness in all directions, and it is lightweight, flexible and wearable. It can be woven into various flexible electronic clothes using well-developed textile technology, so accurately designed and colourful textiles can be produced from these PLEC fibres with different colours. The driving voltages are a little higher than those of conventional OLEDs, but may be reduced after optimization such as increasing the electrical conductivity of the aligned CNT sheet.

Methods

Fabrication of the fibre-shaped PLEC. The stainless steel wire was sequentially washed in acetone, isopropanol and deionized water. The ZnO precursor layer was coated onto the precleaned stainless steel wire after immersion in a ZnO precursor solution[47]. The ZnO precursor was prepared by dissolving 1.46 g Zn $(CH_3COO)_2{\cdot}2H_2O$ and 0.2 ml $NH_2CH_2CH_2OH$ in 25 ml $CH_3OCH_2CH_2OH$ under vigorous stirring for 30 min at 60 °C to achieve a hydrolysis reaction in air. The ZnO nanocrystal layer was produced by thermal annealing at 300 °C for 30 min in air, and this procedure was repeated three times to obtain a continuous nanocrystal film with a thickness of ~45 nm. The polymer layer was then dip-coated onto the modified stainless steel wire, followed by vacuum drying for 1 h. The blue-light-emissive conjugated polymers were dissolved in anhydrous, inhibitor-free tetrahydrofuran, followed by the addition of ETT-15 and LiTf. The weight ratios for the polymer/ETT-15/LiTf were 20/10/1, with the final concentration of conjugated polymer being 40 mg ml^{-1} (ref. 23). The yellow-light-emissive conjugated polymers were dissolved in anhydrous, inhibitor-free tetrahydrofuran, followed by the addition of ETT-15, Poly(ethylene oxide) (PEO) and LiTf. The weight ratios for the polymer/ETT-15/PEO/LiTf were 20/2/2/1, with the final concentration of the conjugated polymer being 3.5 mg ml^{-1} (ref. 6). The aligned CNT sheet was finally wrapped around the polymer-coated stainless steel wire to produce the fibre-shaped PLEC[26] (for the synthesis of the CNT sheet see Supplementary Section 'Synthesis of spinnable carbon nanotube (CNT) arrays'). The procedure for measuring the brightness is shown schematically in Supplementary Fig. 25 and is described further in the Supplementary Section 'Calculation of luminance of the fibre-shaped polymer light-emitting electrochemical cell (PLEC)'. For convenience of characterization, the ends of two electrodes were connected to indium by an ultrasonic soldering mate (USM-V, Kuroda Techno).

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Author contributions

Z.Z., Q.P. and H.P. discussed and designed the experiment. K.G. carried out the performance tests. Y.L., X.L., G.G., H.L., Y.L., F.Z., Q.Z., B.W. and H.P. participated in materials synthesis, device fabrication and data processing. Z.Z. and H.P. wrote the paper. H.P. supervised the project.

Additional information

Supplementary information is available in the online version of the paper. Reprints and permissions information is available online at www.nature.com/reprints. Correspondence and requests for materials should be addressed to H.P.

Competing financial interests

The authors declare no competing financial interests.