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Water-Enabled Healing of Conducting Polymer Films

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The conducting polymer polyethylenedioxythiophene doped with polystyrene sulfonate (PEDOT:PSS) has become one of the most successful organic conductive materials due to its high air stability, high electrical conductivity, and biocompatibility. In recent years, a great deal of attention has been paid to its fundamental physicochemical properties, but its healability has not been explored in depth. This communication reports the first observation of mechanical and electrical healability of PEDOT:PSS thin films. Upon reaching a certain thickness (about 1 μm), PEDOT:PSS thin films damaged with a sharp blade can be electrically healed by simply wetting the damaged area with water. The process is rapid, with a response time on the order of 150 ms. Significantly, after being wetted the films are transformed into autonomic self-healing materials without the need of external stimulation. This work reveals a new property of PEDOT:PSS and enables its immediate use in flexible and biocompatible electronics, such as electronic skin and bioimplanted electronics, placing conducting polymers on the front line for healing applications in electronics.

Materials able to heal damage caused by external agents are highly desirable for applications in the electronics, biomedical, automotive, and aerospace fields.[1] The healing can be autonomic or nonautonomic, depending on whether or not an external stimulation is required. In particular, self-healing electronic materials can find applications in wearable, stretchable, and bendable electronics, as well as in artificial electronic skin.[2] The major challenges for self-healing electronic materials is to combine fast and reproducible healing characteristics, high conductivity, facile processing and, in the the case of applications for electronic skin, biocompatibility.[2,3] In this context, Bao and co-workers reported the first example of an intrinsic self-healing conductive composite that heals at room temperature.[4] This composite, consisting of nanostructured nickel microparticles (μ Ni particles) embedded in a hydrogen-bonded polymer matrix, showed a conductivity of 40 S cm^{-1}. The conductivity of this material was restored within 15 s after damage by applying pressure to the separated slices. The same group recently reported a stretchable and healable semiconducting polymer, where cracks could be healed by post-treatment consisting of heating (150 °C for 30 min) and solvent annealing ($CHCl_3$ vapor), which are able to promote polymer chain movement.[5] Sun and co-workers obtained a self-healing conductor by coating conductive Ag nanowires on nonconductive healable polyelectrolyte films.[6] The film conductivity was similar to that of indium thin oxide (ITO), while the response time required for healing after damage was more than 2 min. White Moore and co-workers developed a self-healing conductor using micrometer-sized polymeric urea-formaldehyde capsules containing conductive gallium–indium eutectic (EGaIn).[7] Upon mechanical damage, the microcapsules broke and the encapsulated EGaIn leaked and diffused to the damaged area, resulting in a fast healing process. More recently, Shi et al. reported an electrically self-healable hybrid gel based on a self-assembled supramolecular gel and nanostructured polypyrrole.[8] This material showed a conductivity of 12 S cm^{-1} and a healing time of more than 1 min. Despite progress in the field, however, a material that can synchronously combine electrical healability with a fast healing time and biocompatibility for practical bioapplications is still highly required.

Conducting polymers have been the object of intense research since Shirakawa et al. demonstrated a high electrical conductivity in iodine-doped polyacetylene.[9] In the late 1980s, Bayer AG research laboratories developed a large-scale synthesis of the conducting polymer polyethylenedioxythiophene doped with polystyrene sulfonate (PEDOT:PSS),[10] which has become commercially available since then. Despite that, PEDOT:PSS has encountered enormous success due to its remarkable properties such as ease of processing, high conductivity, and high stability in air;[11] however its healability has not been explored in depth. A healable material in the form of a dough has been obtained from a blend containing 20 wt% PEDOT:PSS and 80 wt% of the surfactant Triton X-100.[12] However, no self-healing was observed after decreasing the surfactant percentage to 70 wt%, thus proving that the healing properties were mostly due to the surfactant rather than to PEDOT:PSS itself. Besides that, the dough had a conductivity of about 60 S cm^{-1} due to the presence nonconductive surfactant, and a fairly high thickness of 50 μm was required for the healing.

In this communication, we report the first observation of healable PEDOT:PSS thin film (as thin as 1 μm) with an electrical conductivity as high as 500 S cm^{-1}. We observed instantaneous electrical healing of pure PEDOT:PSS film after damage by simply covering the damaged area of the films with a droplet

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of water. The healing process takes place within an extremely short time (about 150 ms) and enables complete restoration of the initial film conductivity, even after multiple damages. Significantly, PEDOT:PSS films wet with water behave as autonomic self-healing conductors: the current flow in the film is totally insensitive to external damage. This is the first observation in conducting polymers of such self-healing properties, which are generally found only in liquid or gel conductors. We show how the healing can be further controlled by humidity. The rapid electrical healing, combined with high conductivity and biocompatibility, places PEDOT:PSS in a highly competitive position among electrically healable materials for applications in large-area self-healable electronics and electronic skin.

PEDOT:PSS films were obtained by drop-casting a Clevios PEDOT:PSS PH 1000 suspension (with or without conductivity enhancers) on glass, then baking them on a hot plate with the temperature gradually increased (refer to the Experimental Section for details). To perform the healing experiments, we biased the film at a constant voltage of 0.2 V using EGaIn contacts, which resulted in a current of 10^{-5} A (or higher for films containing conductivity enhancers). The film was then cut with a razor blade, which created a gap in the film, thus interrupting the current flow across it (**Figure 1**). Surprisingly, covering the damaged area with a drop of DI water leads to the recovery of the current, up to 100% of its initial value, within a remarkably short time of ≈150 ms (Figure 1, inset). This effect is observed after repeated damage and repair in different regions of the same film (Figure 1), without significant variation of the recovered current and the response time. These results demonstrate the high reproducibility and reliability of the process. We would like to emphasize here that such a rapid healing time is among the fastest reported so far for materials that are both electrically and mechanically healable,[2,4–6,12] and is the fastest for a conducting polymer thin film.[5] Remarkably, the current remained stable even after baking (140 °C, 1 h) the healed film, thus proving that the presence of water is not required to ensure conduction in the film. The water-induced healing behavior was also observed for PEDOT:PSS films processed with additives, such as conductivity enhancers (glycerol) and plasticizers (Capstone FS-30) on several kinds of substrates, such as flexible polyethylene terephthalate (PET) and polyimide, confirming that the substrate does not play an important role in the healing process.

To gain insight into the healing process, we carried out a scanning electron microscopy (SEM) investigation of the damaged area before and after its exposure to water (**Figure 2**). A gap in the film, with a width of about 40 µm is clearly visible after film cutting (Figure 2a). Strikingly, the gap is mechanically healed after adding water (Figure 2b) and the healing remains after baking above 100 °C. A schematic representation of the water-enabled healing process is illustrated in Figure 2c.

We investigated the healing effect for PEDOT:PSS films of variable thickness and we found that a thickness greater than 1 µm is needed to trigger a fast electrical healing (≈150 ms) of the 40 µm gap (Figure S1, Supporting Information). For film thicknesses ranging from 1 to 10 µm, the healing time does not significantly depend on the thickness (see Figure S1, Supporting Information).

We demonstrated the healing properties of PEDOT:PSS films by damaging and repairing a film connected in a simple circuit to a commercial light-emitting diode (LED) bulb (Figure 2d; and Video S1, Supporting Information). The LED switched off after the film was cut and switched on immediately after it was healed with water. The process could be observed for repeated cuts (Video S1, Supporting Information).

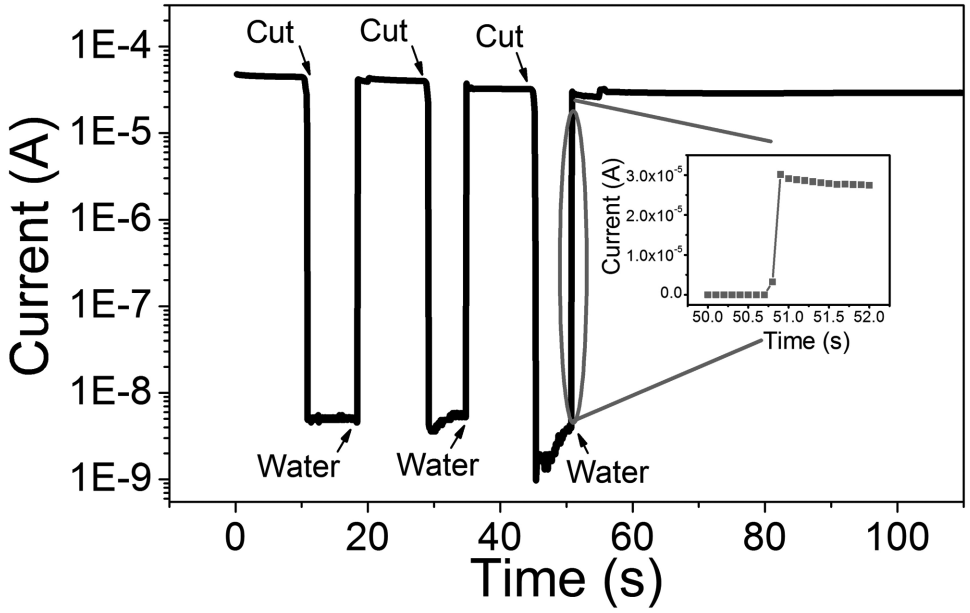

Figure 1. Current versus time measurements (applied voltage 0.2 V) for PEDOT:PSS film (thickness: 10 µm; width: 4 mm; length: 20 mm) showing the effect of damage (cutting with a razor blade) and healing (covering the damaged area with a drop of water). The damage/healing process was repeated three times on different regions of the film. The inset shows the magnified image of the current response time to highlight the rapidity of the healing process.

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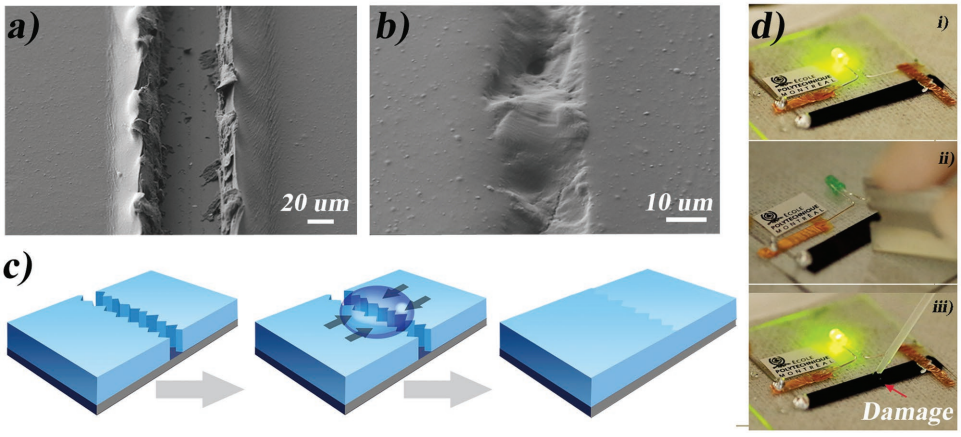

Figure 2. SEM images of the damaged area of a PEDOT:PSS film (obtained adding 5 v/v.% glycerol to Clevios PH1000) before (a) and after (b) healing with a 10 µL drop of deionized water; c) schematic representation of water-induced mechanical and electrical healing of a PEDOT:PSS film (thickness: 10 µm; width: 4 mm; length: 20 mm); d) demonstration of the damage and healing effect on a PEDOT:PSS film connected in a circuit with a LED bulb at a constant voltage of 3 V: i) as-prepared intact film; ii) film damaged with a cut; iii) film healed by dropping DI water on the cut, which enables an immediate repair of the circuit (within 150 ms).

The properties of water healable PEDOT:PSS films can be exploited for water-activated components in integrated logic circuits (Figure S2, Supporting Information), such as inverters and water-erasable read-only memory;[13] water-controlled switches for inflatable life jackets, water-controlled LEDs, and wireless transmitters for maritime search and rescue (Figure S3, Supporting Information), and disposable water detectors for food packaging. Here, we demonstrate a commercial potential ultrasensitive water leak sensor by combining our healable PEDOT:PSS films with a radio frequency wireless device based on low power long range (LoRa) technology (with communication range of over 15 km).[14] Wireless water sensors based on our water-healable PEDOT:PSS films allow a custom-design dimension and an arbitrary shape. They can be used on rigid (Video S2, Supporting Information), as well as on flexible and soft substrates such as human skin (Videos S3 and S4, Supporting Information), which makes them attractive for wearable and biocompatible applications.

Inspired by the above observations, we investigated the healing properties of wet PEDOT:PSS films, i.e., after soaking them into DI water for 5 s (the water remaining on the film surface after immersion was removed using a lint-free paper and a nitrogen gun). **Figure 3** shows the current versus time characteristics of the wet film upon application of a voltage of 0.2 V. Surprisingly, we observed that the current was totally unresponsive to repeated cuts in different regions of the film and maintained the initial value of ≈10 mA. In other words, wet PEDOT:PSS films show autonomic self-healing. The autonomic self-healing of wet PEDOT:PSS films can be again demonstrated by using them as part of a circuit connected to a LED. This time (as shown in Video S5, Supporting Information) the LED stays in the on state despite the several cuts. This is the first observation on conducting polymer thin films (>1 µm) with such a self-healing property.

The ultrafast electrical self-healing of wet PEDOT:PSS films, together with their other remarkable properties such as mechanical flexibility, ease of processing, and biocompatibility, makes them attractive for in vivo self-healing applications, such as electronic skin,[3] autonomic self-healable interconnects for neurodetectors,[16] and epidermal electronics.[17] The PEDOT:PSS film thickness only shows a decrease after the first time water immersion (1 h) due to the polymer (PSS^-) dissolution in water.[15] No loss in thickness or conductivity observable during the subsequent water immersions (Figure S4, Supporting Information).

As a further potential application, we exploited wet PEDOT:PSS as channel material for a self-healable organic electrochemical transistor (OECT). These devices typically work in aqueous solution, and thus water is always in contact with the PEDOT:PSS channel. OECTs based on PEDOT:PSS has been widely used for in vivo bioelectronics studies.[16,18] The transfer curves of OECT based on our water-healable PEDOT:PSS film are shown in Figure S5 (Supporting Information). Strikingly,

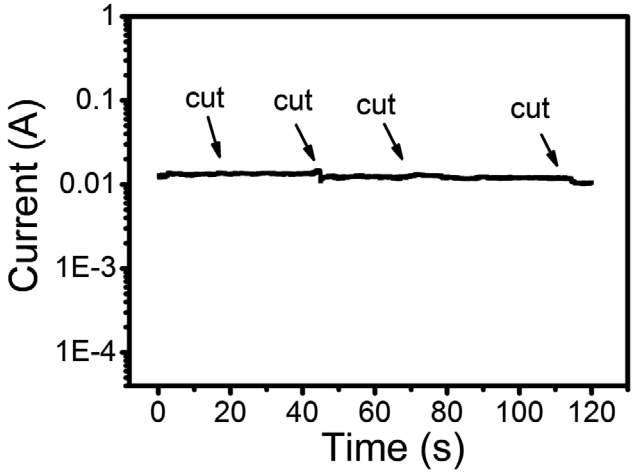

Figure 3. Current versus time profile of wetted PEDOT:PSS film containing the conductivity enhancer glycerol (thickness: 10 µm; width: 4 mm; length: 20 mm) after several cuts in different areas of the film. The applied voltage was 0.2 V. The higher current with respect to Figure 1 is due to the presence of a conductivity enhancer.

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we observed the OECT shows identical transfer curves before and after in situ damage, thus behaving as a self-healable device. We would like to stress that this is the first report of self-healing PEDOT:PSS OECTs. This remarkable observation is of great importance since it improves the device lifetime, especially in conditions where frequent replacements need to be avoided (i.e., in vivo applications).

The healing effect can even be achieved by exposure to water vapor, as shown by damage/healing experiments carried out in a humidity chamber (**Figure 4**). After damage at an initial relative humidity (RH) of 50%, the current dropped to the noise level and did not show any significant recovery even after about 30 min at RH between 50% and 70%. A considerable current recovery started when the RH was increased to 80%. The current gradually increased by about two orders of magnitude in about 5 min and then reached a plateau. A further increase of the RH to 90% brought the current back to its initial value. Overall, the healing process in water vapor is slower than in liquid. However, at high RH (80–90%) the current recovered to its initial value within a few minutes. The humidity-induced healing can be exploited when the direct exposure of the damaged area to liquid water is not desirable, for instance in a self-healable large-scale integrated circuit,[7] to heal simultaneous multiple cuts without the need to precisely identify their locations in the circuit.

The details of the mechanism of water healing on PEDOT:PSS films are currently under investigation. We tentatively attribute the healing effect to the swelling of PSS^- chains upon water exposure, leading to an increase of viscoelasticity and softness of the film. The conductive $PEDOT^+$ chains, after drying, are hydrophobic and insoluble in water, while the PSS^- chains are hydrophilic and, as such, can be swelled or dissolved by water.[11] The PEDOT/PSS weight ratio in the commercial suspension Clevios PH 1000 is 1/2.5,[19] therefore an excess of PSS^- is present. We previously reported that a thickness decrease of 30–40% occurs for 100 nm thick PEDOT:PSS films after water immersion and subsequent drying, due to partial dissolution of excess PSS^-.[15] In this work, for films with thicknesses of about 10 µm, we observed only 10–20% (1–2 µm) thickness decrease after water immersion (Figure S6, Supporting Information), indicating that, in percentage, more PSS^- remains in the thick film as compared to the thinner ones. In accordance with previous works,[20] PEDOT:PSS films gain 25% of mass in a moisturized atmosphere due to the water-uptake by PSS^-. Therefore, when water is added to the damaged area of a PEDOT:PSS film, the PSS^- chains quickly uptake water, increase volume (swelling), and propagate toward the damaged site. The swelling of PSS^- simultaneously enables the $PEDOT^+$ chains (where $PEDOT^+$ indicates the oxidized form) to shift, since $PEDOT^+$ and PSS^- are entangled in the film,[21] thus allowing formation of $PEDOT^+$-$PEDOT^+$ conducting paths across the damage, finally restoring the film electrical conductivity (see Figure S7, Supporting Information). This assumption is supported by time-of-flight secondary-ion mass spectrometry (TOF-SIMS) analyses, where there are no noticeable significant differences between the film composition in the healed gap and in the original film (Figure S8, Supporting Information). The fast response is expected to benefit from the rapid PSS^- water uptake, which can also increase ionic conduction. In the wet films, the water uptake of the PSS^- chains causes a significant increase in film softness and a decrease in tensile strength (Figure S9, Supporting Information). Thus, once the film is cut and the material is pushed aside by the razor blade, it instantly flows back to the damaged area, due to increased viscoelasticity, thus explaining the current's insensitivity to damage. The slower current recovery upon exposure to water vapor is due to the lower water absorption rate. The fast healing may also benefit from a water-induced reversible hydrogen bond breaking and restoring. It is a consensus that PEDOT:PSS film is composed of grains with core–shell structure ($PEDOT^+$ core and PSS^- shell).[21,22] The $PEDOT^+$ and PSS^- chains interact via electrostatic (Coulomb) forces, while the individual grains are held together by hydrogen bonds formed between the sulfonate groups ($R\text{-}SO_3^-$) of PSS^- (Figure S7, Supporting Information). These hydrogen bonds, as reported by Dauskardt and co-workers may break after exposure to water.[21] This will cause decohesion among the grains with consequent increase of their mobility in water, which, in turn, accelerates the separation and propagation of PSS^- (and thus $PEDOT^+$) grains to the damaged area. The broken hydrogen bonds, after water evaporates, will reform, and hence the cohesion between grains will be restored. The nature of healing in this way would be a dynamic hydrogen bond controlled by water.

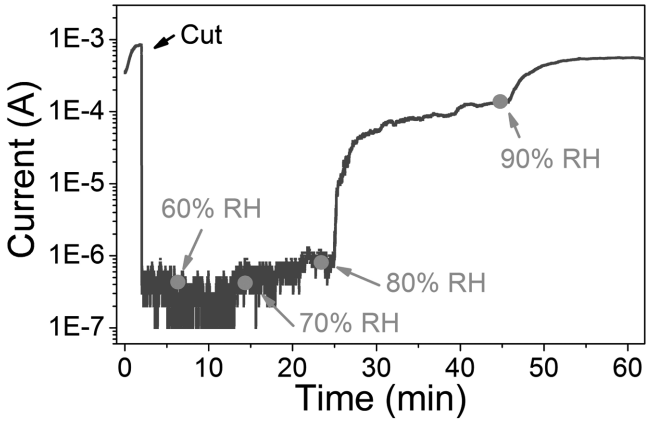

Figure 4. Current versus time plot of PEDOT:PSS film containing the conductivity enhancer glycerol (thickness: 10 µm; width: 4 mm; length: 20 mm) measured in a humidity chamber with RH varying from 60% to 90%, at a constant voltage of 0.2 V. A complete current recovery was observed within 5 min after increasing the humidity to 80% RH and 90% RH.

According to the tentative healing mechanism described above, solvents with a low chemical affinity with PSS^- should not be able to heal the film. To verify this assumption, we used as the healing agent a fluorinated solvent (Orthogonal 700), which has a similar viscosity to water but is totally inert toward PSS^-. In line with our expectations, the fluorinated solvent neither electrically restores the current nor mechanically heals the film (see Figure S10, Supporting Information). A similar result was obtained with glycerol and with the ionic liquid 1-Ethyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imide (EMIM-TFSI), indicating these materials are not able to efficiently swell PSS^- or break the hydrogen bonding between PSS^- chains. These experiments further point to a chemical nature of the healing process (PSS^- swelling) and exclude the possibility

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that the current recovery results from mechanical movements of the film or by the transport of conducting debris, created during the cut, to the damaged area.

We would like to stress here that although the water healable films require a thickness greater than 1 µm, they maintain a high conductivity (≈500 S cm^{-1} for 1 µm film). This high conductivity, together with the rapid healing time, makes PEDOT:PSS films much superior to other electrically healable materials reported so far. In addition, the fact that the films maintain their healability after additives are introduced is also of great importance since it allows property modification (such as hydrophobicity, stability, and elasticity) of the PEDOT:PSS films by using cosolvents, thus extending their applications.[15,23]

To fully take advantage of the healability of these films, it is important to obtain them in a free-standing form, to enable transfer onto various substrates. We obtained free-standing films with a high yield by using water-assisted delamination from glass substrates (**Figure 5**a).[24] We immersed the thick films on glass in DI water and, after 1 h of gentle stirring, we were able to detach them from the substrate with tweezers without any rupture (Figure 5b,c). These free-standing films maintain their high conductivity after drying. The detached wet free-standing films can be easily shaped with scissors and plastered on objects with irregular shapes, such as fingers and even fingertips (Figure 5d,e), hence they have potential applications as conformable and healable electrodes or electrical patches. For instance, they show high conformability to fingers and are even able to reflect fingerprint details on their surface (Figure 5e). The wet films can be further used for erasable imprinting (Figure S11, Supporting Information) or shape-memory materials[25] because the shaped films can recover their smoothness after absorbing water (Video S6, Supporting Information). They can also be developed as a healable conductive patch to repair a damaged Au connection on an elastomer substrate by simply pasting it on top of the damaged region (Figure S12, Supporting Information). The free-standing patch showed perfect conformability to the elastomer surface and led to a current recovery of 100% of its initial value, which remained stable even after crumpling the polydimethylsiloxane (PDMS). In comparison, when a copper conductive tape was used for the same repair, a much lower current was achieved after repair, due to the poor conformability of the tape to the PDMS surface.

In summary, we have demonstrated healability of PEDOT:PSS films. After increasing the thickness to a threshold of about 1 µm, the films are electrically healed within ≈150 ms upon the addition of a water droplet. The healed damage can be more than 40 times larger than the film thickness and the healing ability remains after multiple damages. Significantly, a wet film became an autonomic self-healing conductor. Moreover, instead of a water droplet, we showed the current healing can be further controlled by humidity, suggesting potential applications in self-healable large-scale electronics. We also introduced an easy and reliable way to obtain free-standing films by using a water-assisted wedging method, and demonstrated their excellent conformability on various surfaces and efficient use as electronic welding patches.

The electrically healable PEDOT:PSS films we presented possess the following outstanding advantages: i) ease of to process from commercially available suspensions; ii) electrical conductivity (500 S cm^{-1}) among the highest reported for electrically healable materials; iii) healing achievable at room temperature and ambient air simply by using water; iv) long-term stability in air and water; v) the films are free-standing, without the need for substrate support, and therefore can be directly laminated on either regular or irregular objects; iv) biocompatibility that can be exploited for biological applications, such as in vivo studies or electronic skin.

As the most successful conducting polymer, PEDOT:PSS is now expanding its importance to the field of self-healable electronics. We believe it will greatly further research in electronic skin, self-healing circuits, water-enabled sensors, and wearable electronics.

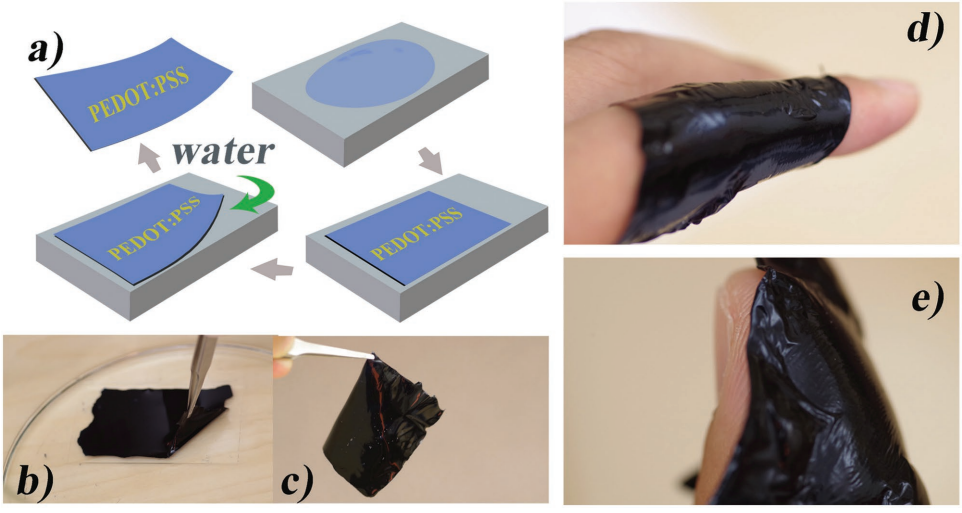

Figure 5. a) Illustration of the water-assisted wedging method used to obtain free-standing PEDOT:PSS films, b) the film is detached from the glass substrate with tweezers, and c) does not deteriorate during the process. d) Excellent film conformability of PEDOT:PSS free-standing films (10 µm thickness) on finger and e) on fingertip. The fingerprint details are clearly visible in (e).

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Experimental Section

The PEDOT:PSS aqueous suspension (Clevios PH1000) was purchased from Heraeus Electronic Materials GmbH (Leverkusen, Germany). Glycerol (99.5+% purity) was purchased from Caledon Laboratories Ltd. (Georgetown, ON). Capstone FS-30 was purchased from Alfa Chemicals. The liquid metal gallium–indium eutectic (EGaIn 495425) was purchased from Sigma-Aldrich. The fluorinated stripper Orthogonal 700 solution (part of a photoresist kit) was supplied by Orthogonal Inc. Glass slides were purchased from Corning. PET sheets were purchased from Policrom Inc. (Bensalem, PA, USA). PDMS (Sylgard 184 silicone elastomer kit) was purchased from Dow Corning. The wireless transmitter and receiver were further designed based on the Demo ZM470SX (supplied by Guangzhou Zhiyuan Electronics co., ltd.). ZM470SX is a LoRa based low power wide area network specification intended for wireless (up to 15 km) battery operated things in a regional, national, or global network.

The films were processed by directly drop-casting the PEDOT:PSS suspension onto glass substrates. The thickness was controlled by spreading different volumes of the suspension on glass. After drop-casting, the films were baked on a hot plate with the following sequence: 80 °C for 1 h, 110 °C for 1 h, and finally 140 °C for 4 h. The gradual temperature increase permitted us to obtain continuous films without bubbles.

Film electrical characterization was performed using a National Instruments NI PXIe-1062Q source-measure unit controlled by Labview software. Electrical contacts were made via two tungsten probes immersed into EGaIn contacts at two sides of the film. The film thickness was measured with a profilometer (Dektak 150) using a 12.5 µm stylus tip with a 10 mg stylus force. The sheet resistance was measured by a Jandel four-point probe with an Agilent B2902A voltage–current source measure unit.

The cuts were performed manually with a standard razor blade. Deionized water (18.2 MΩ cm at 25 °C, Millipore), Orthogonal 700 stripper, the ionic liquid EMIM-TFSI and glycerol were used as healing agents. Electrical measurements at relative humidity between 50% and 90% were performed in a Cole-Parmer humidity-controlled chamber (03323-14).

SEM measurements were performed with a JEOL JSM-7600TFE Field Emission Scanning Electron Microscope with a voltage of 5.0 KV (LEI) under a vacuum of 10^{-4} Pa. The strain–stress characterization was performed with an Instron E-3000 tensile tester. The TOF-SIMS was performed with an IONTOF TOF-SIMS IV. The optical images were obtained with a Carl Zeiss AX10 (10x magnification) microscope. For the repair test on Au contacts, 4 nm Ti and 40 nm Au were deposited by E-beam evaporation on 300-µm-thick PDMS substrates at 1 Å s^{-1}.

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

conducting polymers, organic electronics, PEDOT:PSS, self-healing, wearable electronics

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