PAPER

A graphene-based electro-thermochromic textile display

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Electronic textiles (e-textiles) are rapidly emerging as key enablers for wearable electronics. Graphene and 2D materials have played a major role in enabling truly wearable e-textiles. We demonstrate a textile-based display by using the Joule's heating of a screen printed few layers graphene ink to drive the colour switching of thermochromic polyurethane on cotton fabric. The average temperature of the few layers graphene ink on fabric was voltage-controlled reaching about 43 °C in 45 s at a bias of 12 V and a recovery of < 20 s, with negligible degradation after several heating/cooling cycles. This is used to demonstrate several electro-thermochromic textile displays, representing a breakthrough in e-textiles technology.

Introduction

Wearable electronics is an emerging field, which offers huge potential in healthcare and wellbeing, Internet of Things (IoT) and biosensing applications[1–3]. Electronic textiles (E-textiles) are a very promising technology, which promises to revolutionize wearable electronics offering breathability, conformability and comfort to wear[4]. Despite all-textile individual electronic components[5] and integrated circuits have been demonstrated[6], only few reports have attempted to achieve textile-based displays[7]. Thermochromic textile devices have already been demonstrated using metal oxide (such as CoO and Pb_3O_4), leuco dye or polymer (such as polydyne) through various methods of textile integration, such as coating[8], spinning[9], and dyeing.[10]
Thermochromic dyes are composed of thermochromic molecules (TMs). These molecules have the property to change colour when exposed to an increase of the surrounding temperature (above a certain critical temperature, T_c). The change in colour is caused by the variation of the structural conformation of the molecule, induced by the thermal energy[10]. Thermochromic dyes have attracted much attention in medicine[11], wearable devices[12] and security materials because of its application as storage element in memories[13], temperature switches[14], colour changing paints[15] and anti-counterfeiting devices[15,16]. The leuco dye is one of the most common thermochromic dyes consisting of a colour former (i.e. conjugated molecule, which is responsible for the colour combination) and a developer (typically weak acids such as bisphenol A, gallates, hydroxybenzoates and hydroxcoumarin derivatives) in a nonvolatile organic solvent (such as methyl stearate) [17–19]. The weight ratios of the colour former, the colour developer and the solvent is key in engineering the thermochromic dye. If the solvent is too much, the thermochromic dye will be too light or even colourless below T_c. On the contrary, if the solvent is too little, the thermochromic dye will change colour when above Tc. However, the presence of bisphenol A in leuco dyes, generates major environmental concerns and the solvent leakage upon heating[20] (when the lecuo dye reach thermcohromic temperature, the solvent is changed to liquid from solid[18]) make lecuo dyes unsuitable for textile applications[21]. Stimuli-responsive functional materials have been investigated as a suitable alternative. Ref[22] combined red-green-blue (RGB) thermochromic materials (such as DEBN, cyan-6, yellow-1, and blue-220) and luminescent materials (such as Eu(III)(TTA)3phen and Alq_3) to obtain thermoresponsive dual-mode (define as red-green-blue colouration and fluorescence) representations in RGB colours with large contrast (from RGB to white). A polyurethane-based fluorescent dye was synthesized by attaching a fluorescent dye (such as 4-amino-N-cyclohexyl-1,8-naphthalimide) to polyurethane chains by capping reaction (through a reaction of –NH2 and –NCO) resulting in a fluorescence intensity increase with above T_c [23]. The thermochromic polyurethane is mainly prepared by mixing pure polyurethane with thermochromic dyes. A waterborne UV-curable polyurethane acrylate/SiO_2

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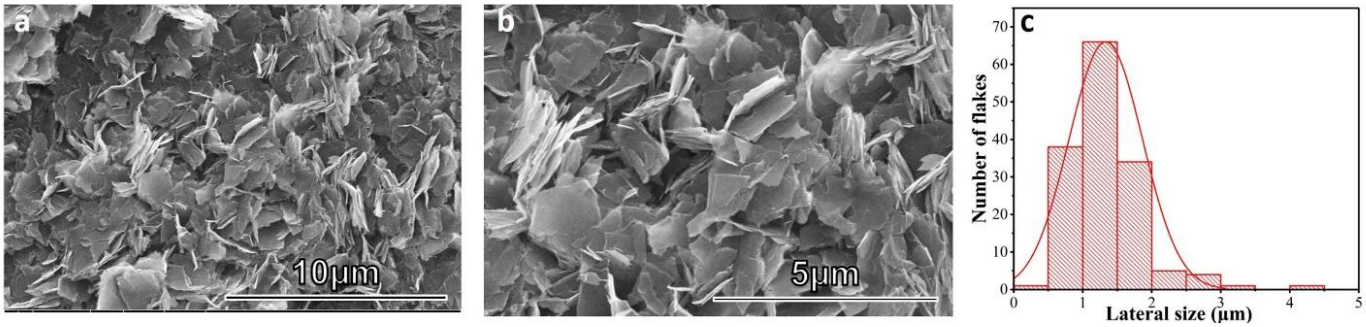

Fig.1 (a) and (b) SEM images of GNP flakes; (c) Histogram of the lateral flake size for the GNP ink after 70 cycles of microfluidic exfoliation.

nanocomposites incorporating nanosilica was prepared using a new form of sol-gel method and mixed with inorganic thermochromic dyes to make a polyurethane-based thermochromic ink[8]. Recently ref[24] demonstrated a thermochromic textile based on UV-induction polyurethane coating mixed with Rhodamine thermochromic dye. The textile showed thermochromic behavior at ~ 50 ℃ under magnetic heating with UV induction.

Electrically-driven thermochromic switching was also proposed by ref [25]. The device was achieved by weaving metallic yarns within the fabrics and then paint thermochromic inks on top them. Ref [26] deposited a thermochromic ink on one side of a textile substrate and use metal wires as heating elements on the other side, achieving a temperature, T ~ 50 ℃ at a voltage bias (V_{bias}) of 7 V. Ref[27] also prepared an electro-thermochromic paper with a commercial leuco dye as the thermochromic ink, which achieved switching at T ~ 45 ℃ at V_{bias} ~ 4 V.

However, this strategy has its own drawbacks since the colour change can only happen in the region close to the conductive yarn when a current passes through it. Recently, the combination of electric heaters from conductive coatings and colour-changing dyes from TMs has been exploited to demonstrate an electro-thermochromic textile. This process allows coating or printing of specific pattern enabling electro-thermochromic image and text. For example, ref.[28] described the production of conductive non-woven mats of poly-3,4-ethylenedioxythiophene (PEDOT) using electrospinning. In this case, when an electric current passes through the PEDOT film producing sufficient heat (T ~ 100 ℃ at V_{bias} ~ 10 V), the coated thermochromic ink undergoes a colour change. Recently, electromagnetic interference shielding fabric, and stable Joule heating response based on CNTs network-embedded poly (vinyl alcohol) (PVA) composite film were also reported.[29-31] More recently, ref. 32 prepared a coaxial multilayer monofilament whose conductive core layer could trigger the colour change of the external thermochromic layer upon resistive heating. However, the conductivity of the monofilament is low (1×10^{-2} S/cm), requiring a high voltage to induce a thermochromic colour change, becoming unsuitable for wearable applications. Graphene and graphene nano-platelets (GNPs) also have been the focus of intense research as suitable materials for electric heating[33], as well as biocompatible[34,35] and wearable electronics [6,36,37].

Following on from previous works on UV-induced thermochromic textile [24] using Rhodamine B ethylenediamine derivative molecules, here we demonstrated a screen-printed electro-thermochromic textile (ET) on cotton fabric, consisting of a UV-induced thermochromic polyurethane layer based on Rhodamine B ethylenediamine derivative molecules and GNP electrodes. The electro-thermochromic switching of the fabric from magenta to white was controlled via Joule's heating from an applied voltage, reaching T ~ 43 ℃ at V_{bias} ~ 12 V within 45 s and a recovery of < 20 s. We then used this technology to demonstrate an electro-thermochromic textile display (ETD).

Results and discussion

The voltage-controlled heating of the screen printable GNP ink is key to enable the electro-thermochromic devices on textile. Here we first fabricate the electrically and thermally conducting fabric by screen printing the GNP ink on cotton fabric and then investigate the surface morphology, the electrical properties of the printed GNP ink, and the performance as a heater GNP-coated fabric.

Graphene nano platelets ink

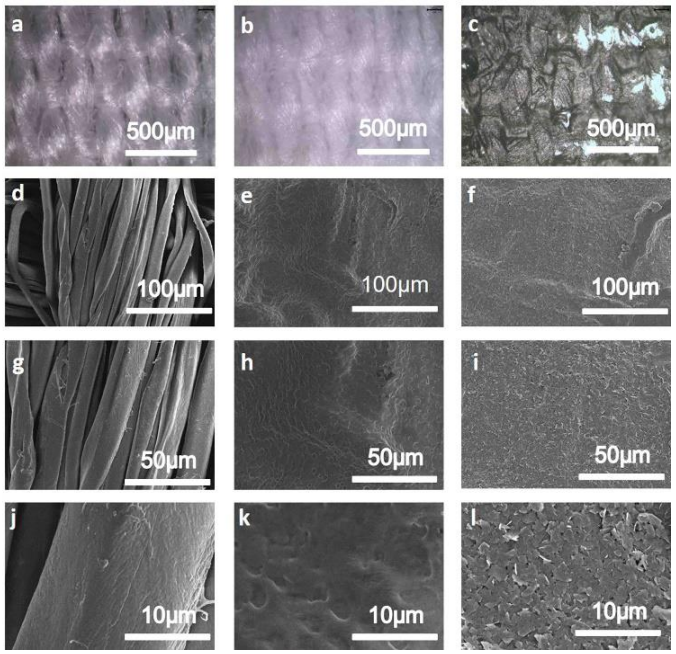

Fig. 2 Optical microscope images of: (a) control cotton fabric, (b) PU-treated cotton fabric, (c) GNP fabric, SEM images of (d, g, j) control cotton fabric, (e, h, k) PU-treated cotton fabric, (f, i, l) GNP fabric

The GNP ink was prepared via microfluidic exfoliation of graphite (see methods). Scanning electron microscopy (SEM) was used to investigate the surface topography and the lateral size of the graphene flakes. Fig.1 (a) and (b) show SEM micrographs with the typical surface topography of GNP ink. Statistics of the lateral size (defined as the longest dimension) of the GNP flakes (Fig.3 c) reveals that mainly this is peak at 1-1.5 µm.

Graphene nano-platelets fabric (GNP fabric)

The white-coloured control cotton fabric is shown in Fig.2 (a). After polyurethane treatment, the cotton fabric is dried in the oven at 60 °C for 10 min to remove any residual water, resulting in a layer film on the surface of the cotton fabric as the Fig.2 (b). The GNP fabric (shown in Fig.2 c) is prepared by screen printing the GNP ink on polyurethane (PU-)treated cotton fabric forming a GNP ink layer (7.6 µm thick) on the surface. Figs 2 (d-l) show the optical SEM images of the control cotton fabric (Figs 2 d, g, j), PU -treated cotton fabric (Figs 2. e, h, k) and GNP fabric (Figs.2 f, i, l) at different magnifications. The SEM images (Figs.2 d, g, j) show a typically fibrous structure with the fibres twisting with each other, producing a quite rough fabric surface. Comparing Fig. 2 (e) with Fig. 2 (d), the surface of the cotton fabric is evenly covered with a film and the texture of the fabric is faintly seen. The surface of the pre-treated cotton fibre is covered with a layer of polyurethane film. The SEM in Fig. 2 (f) shows how the GNP ink uniformly conforms to the pre-treated cotton fabric. Compared with the control cotton fabric, the C content of the GNP fabric was increased (shown as Figure S1).

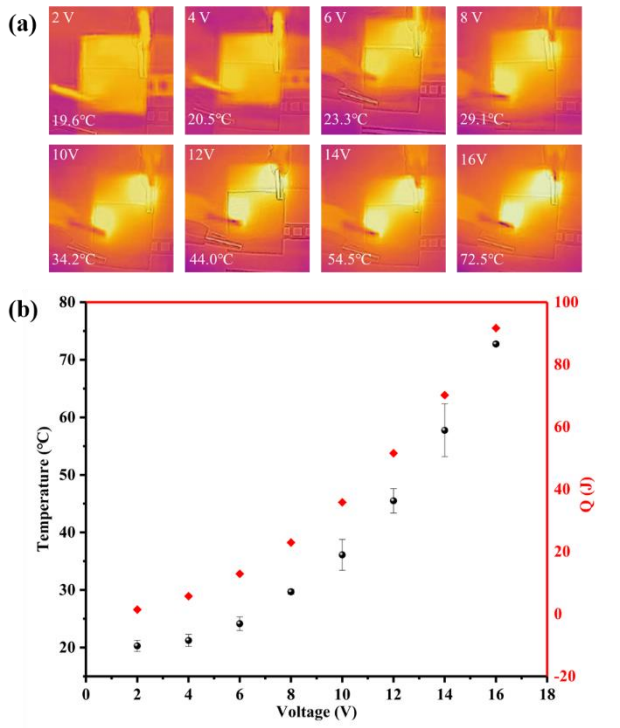

Fig. 3 (a) Infrared thermal images of the conductive fabric at various V_{GNP}; (b) plot of the temperature versus V_{GNP}.

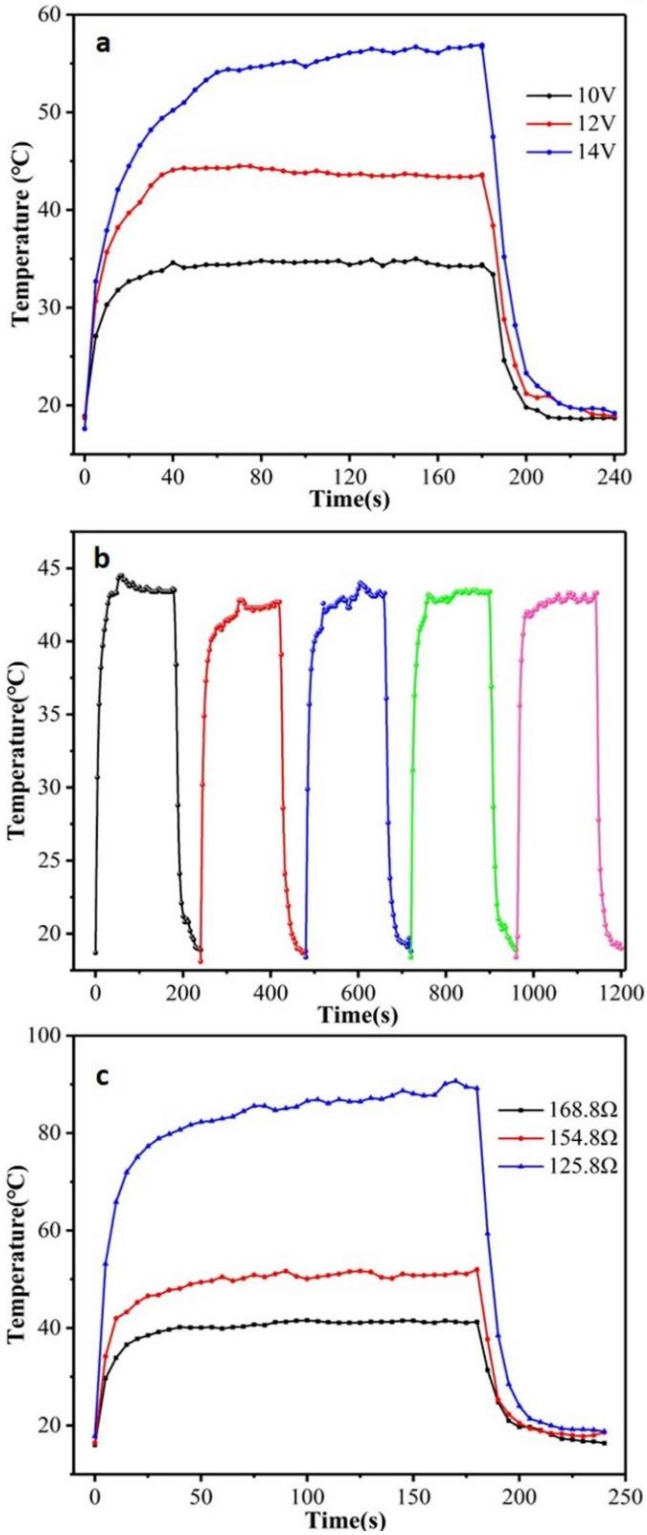

Fig. 4 (a) Temperature profiles of the GNP fabric at different $V_{GNP;}$ (b) Temperature response of the GNP fabric over 5 cycles at V_{GNP} = 12 V, each cycles is 4 min long; (c) Temperature profiles of the GNP fabric operated at different biases;

Electrical and thermal characterization of the GNP fabric

The electrical and heating properties of the GNP fabric are characterized on a 2×2 cm sample revealing a sheet resistance,

R_s ~ 169 Ω and the conductivity σ ~ 778.6 S/m, which is consistent with the conductivity values reported by previous works[35] The heating properties of the GNP fabric are investigated by monitoring the evolution of the temperature by an infrared camera (see methods) as a function of the applied voltage (V_{GNP}) across the GNP fabric (between 2 V and 16 V), Fig.3. The thermal images show a clear heating of the GNP fabric as a function of V_{GNP}, increasing from T ~ 19.6 ºC to ~ 72.5 ºC.

The heating of our GNP fabric can be described by Joule's law which defines the heat (Q) dissipated from a resistive material when voltage (or current) is applied as follows[36,37]:

$$Q = V^2/R\,t$$

where V is applied voltage, R = 335 Ω is the resistance of the conductive fabric (see methods) and t is the operating time. The measured temperature change and calculated Q as a function of voltage is shown in Fig. 3 (b). Temperature profiles of the conductive fabric under different biases are measured as shown in S2. At V_{GNP} = 6 V, only ~ 19% increase of temperature was detected on the GNP printed pattern. A change of regime, takes place at higher voltage and the temperature began to rise significantly going from T ~ 23.3 ℃ at V_{GNP} = 6 V to T ~ 72.5 ℃ at V_{GNP} = 16 V. This large temperature modulation of the GNP fabric was ascribed to the remarkable electrical conductivity of the GNP ink and the heat dissipation of cotton fibres.

Fig. 4 (a) shows the temperature profiles of the GNP fabric, as a function of the t for V_{GNP} = 10 V, 12 V and 14 V. All curves show a similar trend with a time response (τ, defined as the time required to reach the steady-state temperature which was above 90% of highest temperature from room temperature) of τ = 45 s, τ = 45 s and τ = 60 s for V_{GNP} = 10 V, 12 V and 14V respectively. In this case, when V_{GNP} = 10 V, only about T ~ 15 ℃ increase was observed reaching about 35℃ after τ = 40 s. At V_{GNP} = 14 V, the GNP fabric reached ~ 60 ℃ after τ = 60 s. The steady-state temperatures were reached for all samples in less than 60 s, demonstrating a faster response[41,42] than graphene-based electro-thermal films which reaches T ~ 42 ℃ when 60 V is applied for 2 min[43]. In all our GNP fabrics, the temperature remained stable upon constant voltage applied and then returned to room temperature (T ~ 20℃) after about 60 s when power was off. This cooling rate can be explained by the nature of the cotton fabric that allows the heat to be very effectively dissipated by the material (the thermal conductivity of cotton fabric is 0.026-0.065 W/mK[44]).

To investigate the stability of the GNP fabric, repeated heating and cooling cycles are carried out (as show in Fig. 4 b and Figure S3). The temperature change of the GNP fabric was recorded over a period of 4 min (heating 3 min at V_{GNP} = 12 V and cooling 1 min at V_{GNP} = 0 V) for 5 cycles. As showed in Fig. 4 (b), the heating/ cooling curve remained almost unchanged after several cycles, reaching constantly T ~ 42 ℃ at 12 V for 40s, after several heating and cooling cycles, indicating a good stability and repeatability. During each heating process, the temperature increased from room temperature (T ~ 20℃) to a steady value of T ~ 43 ℃ in the initial 1 min, revealing a high heating rate of the GNP fabric. In addition, in this case the temperature of the GNP fabric gradually dropped to ambient temperature after 60 s since the voltage was set to V_{GNP} = 0V, thanks to the thermal conductivity of the cotton fabric. The time-dependent temperature profiles of the GNP fabrics with different R_s values is shown in Fig. 4 (c). The temperature change of the GNP fabric was recorded with a period of 4 min (heating 3 min at V_{GNP} = 12 V and cooling 1 min at V_{GNP} = 0 V). The value of Q results affected by the resistance of the GNP fabric. At same voltage, the temperature of the GNP fabric is inversely proportional to R_s, thus confirming the heating by Joule effect of the GNP fabric. For R_s ~ 126 Ω, the temperature of the GNP fabric reaches T ~ 90 ℃, while it only reaches about 40 ℃ for R_s ~ 169 Ω. The conductivity of our GNP fabric is higher that that reported for graphene anodes and cathodes, (e.g. in microbial fuel cells)[45].

Electro-thermochromic cotton fabric performance

We developed a colour-changing smart fabric and a textile display by combining the colour changing property of the

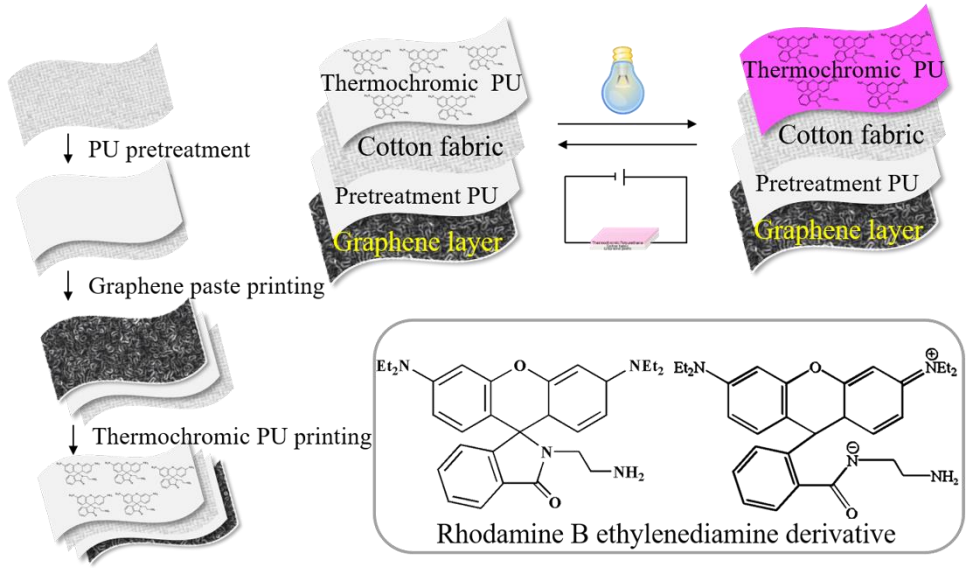

Fig. 5 The schematic diagram of the procedure used to fabricate the ET. In the frame the molecular structure of the two Rhodamine B derivatives.

thermochromic polyurethane ink and the GNP fabric heater as shown in Fig.5. The Rhodamine B ethylenediamine derivative thermochromic polyurethane ink was screen-printed on one side of the PU -treated cotton fabric, while the GNP ink was screen-printed on the other side (Fig. 5). The choice of depositing the two inks on opposite sides aims to maximize the colour changing property of the thermochromic dye. The layer of polyurethane reduced the surface roughness[38] of the fabric and prevented the absorption of the inks into the cotton fabric. The colour-changing property of the ET was investigated by monitoring colour parameter (i.e. K/S, where a^* corresponds to the red and green colour combination and b^* corresponds to yellow and blue colour combination) as a function of V_{GNP}. The darker the red, the larger the a*. Fig. 6 (a) shows how the colour of the thermochromic coating gradually disappears as the applied voltage increases.

This is the result of the UV induction which modifies the conjugate structure of Rhodamine B ethylenediamine derivative in the chemical composition of the polyurethane ink, hence resulting in a change of the polyurethane optical properties[46]. Our transparent thermochromic polyurethane starts reflecting in the region around magenta (λ_{max} = 550nm) following the UV induction. It then returns colourless upon heating. The optical images of our ET at different voltages are shown in Fig. 6(a). Firstly, the thermochromic coating on the ET was activated by a treatment irradiation at λ = 254 nm for 3 min. This allows the lactam ring structure of Rhodamine B ethylenediamine derivative moiety in the thermochromic polyurethane to gradually change to an open-loop state with a quinolone structure[24]. Then we apply an increasing voltage,from V_{GNP} = 2 V to V_{GNP} = 16 V. We can see the ET returning white as the applied voltage increased above V_{GNP} = 10V. This is because the Q generated by the GNP ink is sufficient to degrade the quinolone structure in the thermochromic ink, back-converting it to the colourless lactam ring when V_{GNP} > 10 – 12 V. A V_{GNP} = 12 V corresponds to a higher temperature than T_c, which consistently gives a transparent thermochromic coating. In the heating process the GNP coating is the heat source, which transfers heat to the thermochromic polyurethane by radiation and conduction, as shown in the schematic of ET in Fig. 5.

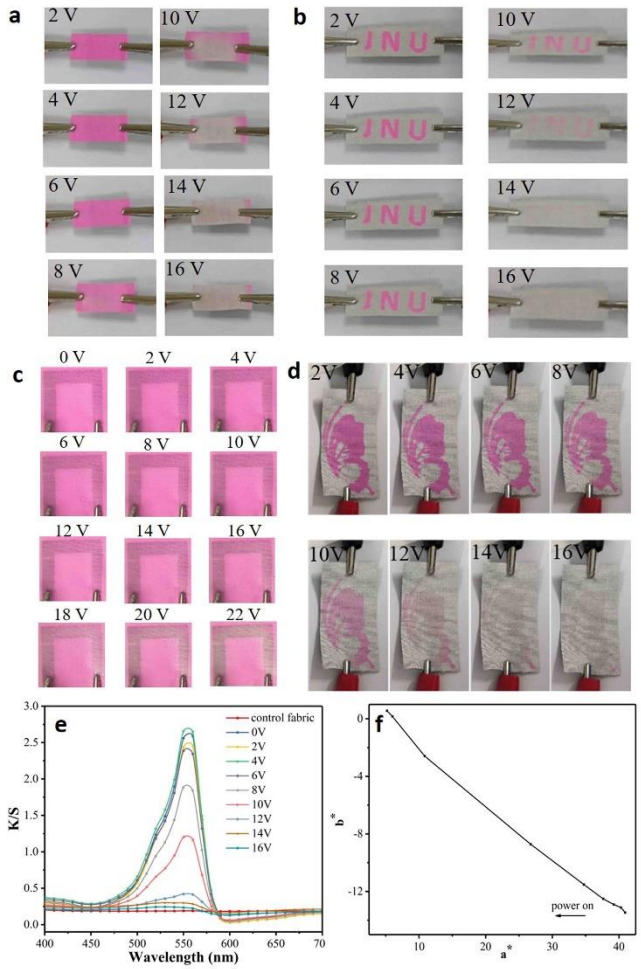

Fig.6 (a) Photographs of ET (1 cm × 2 cm) showing smart colour- changing effect as a function of VGNP (b) Photographs of ET (1 cm × 3 cm) showing smart colour- changing effect as a function of VGNP; (c) Photographs of ET with a horseshoe pattern, showing smart colour- changing effect as a function of VGNP; (d) Photographs of ET with butterfly pattern, showing smart colour- changing effect as a function of VGNP; The (e) The K/S curves of the ET as a function of VGNP; (f) The a* and b* of the ET as a function of VGNP.

An electro-thermochromic textile display

A series of ETDs are prepared by screen printing the GNP ink, following the steps in Fig.5 (Figs 6 b, c, d). The thermochromic polyurethane ink was screen printed on the other side of the PU pre-treated cotton fabric with a series of predetermined patterns, “JNU” (Figs. 6 b), a horseshoe (Figs. 6 c) and a butterfly (Figs. 6 d). The thermochromic pattern is then activated by UV induction at λ = 254 nm giving rise to the patterns in magenta. A voltage V_{GNP} > 10 – 12 V, turns the patterns colourless as shown in Figs 6 b, c, d. Also in this case, we attribute the colour change of the printed pattern to the Joule heat generated by the GNP ink printed at the back of the fabric. Fig 6 (e, f) and Table S1 show the colour parameters (colour difference, ΔE, the K/S value, a^*, b^*) of the ET as a function of λ for a V_{GNP} range between 2 V to 16V. The K/S value shows peaks at 560 nm which are in agreement with the standard peaks for Rhodamine B ethylenediamine derivative. In particular the reference peak at λ = 560 nm decreased as the voltage increased and the a^* was also decreased, which confirms the colour change of the ET and the patterned ETD from magenta to white.

Conclusion

We have obtained electrochromic cotton fabrics and electrochromic textile-based displays using the thermal dissipation of a screen printed graphene-based ink to drive the colour switching of a thermochromic polyurethane ink coated on fabric. The surface of graphene-coated fabric was uniformly covered with graphene sheets, which provided the conductivity of the fabric. The graphene-based fabric generated heat by Joule effect upon voltage bias reached T ~ 43 ℃ at 12 V. When further coated by thermochromic polyurethane and activated by UV induction, the composite fabric switched from magenta

to white upon applied voltage of 10 V, demonstrating suitable electro-thermochromic switching. The structure of the thermochromic polyurethane was changed to an open-loop state with a quinolone structure and returned to white when the applied voltage increased with the close-loop state of thermochromic polyurethane. This work paves the way for large area, fast and stable thermochromic textile displays in the future.

Methods

Materials

The UV-induced thermochromic polyurethane ink was homemade[24]. (more details in S2). Timrex KS25 graphite, sodium desoxycholate (SDC) and carboxymethythylcellulose sodium (CMC) were supplied by Aldrich chemical The water-soluble thermal was purchased from Jining Baichuan Chemical Co., Ltd. The printing thickener was supplied by Transfar Co., Ltd.

Formulation of the graphene-based ink

The GNP ink was prepared by dispersing graphite flakes (Timrex KS25) in deionized water (DIW) with Sodium deoxycholate (SDC, 10 mg/mL) followed by a microfluidic exfoliation (M110-P Microfuidizer; Microfluidics) [6] for 70 cycles obtaining a graphene-based dispersion. Sodium carboxymethy cellulose (CMC; 7.5 g/L) was blended with the graphene-based dispersion at 60 ℃ to obtain a GNP ink suitable for screen printing.

Fabrication of the electro-thermochromic cotton fabric and display

The electro-thermochromic cotton fabric ET was prepared by screen printing on a polyurethane coated cotton fabric the graphene heater electrode on the back side of the fabric and the thermochromic UV-induced polyurethane on the front side of the fabric by the following steps:

Cotton fabric pre-treatment

The cotton fabric was pre-treated with water-soluble thermal curing polyurethane (65%), DIW (33%) and thickener (2%). Then the cotton fabric was oven dried at 60 ℃ for 10 min and 100 ℃ for 5 min to prepare the pre-treated cotton fabric, denoted as PU pre-treated cotton fabric.

Printing of GNP ink

The GNP ink is deposited via screen printing on the polyurethane-treated cotton fabric followed by a drying step at 60 ℃ for 10 min. The conductive fabric was denoted as GNP fabric.

Printing of thermochromic polyurethane

The screen printing with UV induction thermochromic polyurethane on the back of conductive fabric. Then the electthermochromic fabric was dried at 60 ℃ for 10 min and baked at 130 ℃ for 5min.

Electron microscopy characterization

The original graphene ink was dried to prepare the sample for scanning electron microscopy. The surface morphology of conductive fabric was also investigated by SEM. A statistical analysis of over 150 flakes of the original GNP ink were used estimate the statistics of the lateral size (defined as the longest side of the flake).

Sheet resistance measurements

The sheet resistance of conductive fabric was measured by four-point probe system (KEITHLEY 2400 Source Meter Unit, US). The final resistance value of the fabric was measured with 10 different positions and averaged.

Thickness measurements

The thickness of the GNP flakes was measured with a fabric thickness test equipment (YG141, Changzhou Second Textile Machinery Factory, China).

Thermal and electro-thermochromic measurements

The average temperature of the fabric surface was monitored with an infrared camera (FLIR Ex). The infrared images of the conductive fabric were acquired at various V_{GNP} applied cross the GNP, with an exposure time of 2 min. The electthermochromic behaviour of the GNP fabric was investigated at various V_{GNP} applied cross the ET and ETDs.

Colour parameters

The colour parameters (K/S value, L^*, a^*, b^*, C°, h°) of the ETs and ETDs were acquired at various V_{GNP} using Xrite-8400 spectrophotometer under a D65 illumination using a 10° standard observer.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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