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ORIGINAL RESEARCH

One-step deposition of hydrophobic coatings on paper for printed-electronics applications

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Abstract The ability to pattern highly conductive features on paper substrates is critically important for applications in radio frequency identification (RFID) tags, displays, sensors, printed electronics, and diagnostics. Ink-jet printing particle-free reactive silver inks is an additive, material efficient and versatile strategy for fabrication of highly conductive patterns; however, the intrinsic wetting properties of cellulose based papers are not suitable to serve as substrates for this process. This study reports one-step and practical modification of the surface of paper substrates using industrially available materials. The paper substrates were dip-coated with films of hydrocarbon and fluorocarbon based polymeric resins. Ink-jet printing particle-free reactive silver inks on the modified paper substrates followed by fast thermal annealing resulted in highly conductive patterns. The coatings improved the conductivity of the patterns and reduced the number of printing layers required to obtain conductivity. We finally demonstrated fabrication of a printed RFID tag on the coated paper substrates operating at the frequency range of 865–870 MHz.

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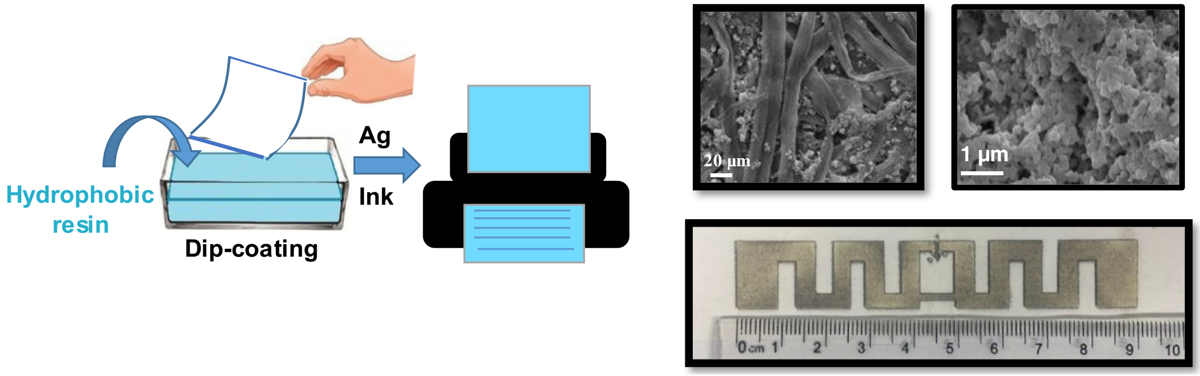
Graphical abstract



Keywords Printed electronics · Paper · Surface modification · Nanoparticles · Silver

Introduction

Emerging applications in wearable sensors (Özdemir and Barshan 2014) and devices (Özdemir 2016), bio-integrated electronics (Kim et al. 2012), disposable electronics (Lee et al. 2017), wireless electromechanical sensing devices (Zhang et al. 2018) and foldable displays (Nakagaito et al. 2010) require fabrication of electronic devices in unconventional forms. The ability to pattern conductive tracks is one of the most important steps in fabrication of these kinds of electronic devices (Ma et al. 2018). The rigid, planar and thermally stable silicon substrates used in conventional electronics allow for a variety of lithographic processes in conjunction with etching and deposition methods to define conductive features (Xia et al. 1999). These processes are not applicable to flexible, non-planar, and low-thermal budget substrates that are necessary for these emerging applications (Chang et al. 2017; Torvinen et al. 2012). A highly effective approach to fabricate electronic devices on such substrates is one-step printing of conductive materials in an additive manner (Onses et al. 2015; Tekin et al. 2008). Recent developments in preparation of inks based on metallic nanoparticles and carbon nanostructures have presented practical routes for fabrication of printed electronic devices (Kamyshny and Magdassi 2014). A particularly interesting approach developed by Walker and Lewis (2012) involved preparation of particle-free aqueous inks that lead to highly conductive patterns upon heating at temperatures as low as 90 °C. The ink is based on a solution of reactive silver precursors and largely overcomes the clogging problems encountered in nanoparticle-based inks. The silver particles form upon printing during evaporation of volatile components. The thermal annealing ensures removal of organic species and results in patterns with conductivities that approach to the bulk conductivity of silver. The broad adaption of this and similar inks in practical applications requires proper engineering of the surface of the low-cost, environmentally benign and flexible substrates.

Paper has attracted considerable interest as a substrate for printed electronics applications, thanks to its low cost, mechanical flexibility, recyclability, and scalable manufacturability (Punpattanakul et al. 2018; Siegel et al. 2010; Tobjörk et al. 2012). As a result, different devices including solar cells (Li et al. 2018), touch pads (Mazzeo et al. 2012), batteries (Jabbour et al. 2013), and sensors (Cao et al. 2018) were fabricated on paper substrates. A challenge for printing particle-free aqueous silver and similar inks on paper substrates is its absorbent nature that arises from its structure composed of hydrophilic cellulose fibers. This type of wetting behavior results in spreading of the printed aqueous inks followed by wicking into the substrate (Tobjork and Osterbacka 2011). Besides the enlargement of the printed feature sizes resulting in deterioration of the resolution, the formation of conductive tracks with particle-free inks upon annealing is adversely affected by the intrinsic wetting behavior of the paper substrates (Lessing et al.

2014). The common approaches to reduce the adsorption of liquids on paper involved laminating with polymers, such as polyethylene (Andersson et al. 2002). Commercial photo papers are good examples to such papers and have been extensively used in printed electronics applications (Allen et al. 2008; Wang et al. 2016). These approaches typically require special infrastructure (e.g. lamination tool) and reduce the flexibility of the substrate. Other strategies to increase impermeability include deposition of hydrophobic particles and plasma processing (Balu et al. 2008; Torun and Onses 2017). These strategies; however, typically bring in additional surface topography and result in extremely repellant surfaces that are not suitable for printing continuous linear features. The solution and vapor-phase deposition of silanes is another effective strategy to improve the wetting properties of paper substrates (Glavan et al. 2014; Tang et al. 2016). The work by Lessing et al. (2014) demonstrated that vapor-phase deposition of silanes made the paper substrate highly suitable for inkjet printing of particle-free reactive inks. The resolution and conductivity of the printed features improved with the increasing hydrophobicity of the paper. This study showed the importance of the wetting properties of the paper for fabrication of conductive features with particle-free aqueous silver inks. Printed electronics applications will greatly benefit from development of solution-processing based complementary strategies for fabrication of hydrophobic paper substrates using versatile, roll-to-roll compatible, low-cost processes and industrially available materials.

In this study, we present a simple and inexpensive approach based on dip-coating of industrially available and ecofriendly resin materials for fabrication of hydrophobic paper to serve as substrates in printed electronics applications. Two types of resins, fluorinated and non-fluorinated, are used in this study. The fluorinated resin is based on alkyl chains of six carbon in length, and therefore conforms to recent regulations on the use of fluorinated compounds. The modification of the surface can be accomplished in durations that are shorter than 5 min and the method is suitable for a roll-to-roll type processing. Ink-jet printing particle-free reactive silver inks on the modified paper substrates followed by fast thermal annealing results in highly conductive patterns. The conductivity of printed patterns is studied as a function of the number of printing layers on bare and modified paper substrates. We finally demonstrate fabrication of a radio frequency identification (RFID) tag on the modified paper substrates.

Experimental

Deposition of water-repellant coatings

Office grade paper was purchased from Alkim Inc. and used as received. Two different types of commercially available water repellant resins from Tanatex Chemicals were used in this study. Fluorinated resin, FR, (Baygard® Clean 01) is based on alkyl chains of six-carbon in length. Non-fluorinated resin, NFR, (Baygard® WRS) is a fluorine-free and hydrocarbon based polymeric resin. The deposition of the resins was performed from an acidic aqueous solution. The pH of the purified water was adjusted to 5.5 by adding acetic acid. The resins were then added to the acidic water solution at a concentration of 100 g/L. The resins were deposited on the paper substrates by dip-coating. The paper substrates were dipped into the resins for few seconds and then pulled out of the solution at a speed of 20 mm/s. The substrates were heated at 170 °C for 2 min after the coating process.

Preparation of particle-free conductive inks and ink-jet printing

Particle-free conductive inks were prepared following the method developed by Walker & Lewis (Walker and Lewis 2012). To prepare the precursor solution, 1 g of silver acetate (Sigma-Aldrich) was dissolved in 2.5 mL of ammonia solution (25%, Merck) followed by mixing with a stir-bar for tens of seconds at room temperature. Formic acid (95%, Sigma-Aldrich) with a volume of 0.2 mL was then slowly added to the precursor solution at a rate of $\sim$ 4.4 μL/s. To settle down and remove the silver particles that formed with the addition of formic acid, the solution was centrifugated at 4000 rpm for 30 min. The clear supernatant was then taken and stored in a fridge (+ 4 °C) and used as the ink in the printing experiments.

An office-type ink-jet printer (HP Deskjet Ink Advantage 2060) was used for fabrication of conductive patterns on the resin-coated paper substrates. The cartridge was thoroughly cleaned by repeatedly washing in ethanol under sonication and drying with air. The particle-free ink was then loaded in the cartridge and printing was performed in the high-quality mode (720 dpi) on the paper substrates preheated to a temperature of 170 °C. Upon printing, the paper substrates were annealed at 170 °C for 2 min.

Structural and wetting characterization

The morphologies of the paper substrates and conductive tracks were imaged with a Zeiss EVO LS10 scanning electron microscope (SEM) at 25 kV. Before SEM imaging, the paper substrates were sputter-coated with a film of gold. The chemical composition of the samples was analyzed using energy-dispersive X-ray (EDX) spectroscopy (Bruker) attached to the SEM and X-ray photoelectron spectroscopy (XPS, Thermo Scientific, K-Alpha). The thin film X-ray diffraction (XRD) analysis was performed using a Rigaku SmartLab diffractometer with Cu Kα radiation. The static water contact angle of the paper substrates was measured using a contact angle meter (Attension, Theta Lite). The volume of the water droplet was fixed at 4 μL. At least three measurements were performed over different regions of the substrate.

Electrical resistance of the conductive patterns

The resistance of the conductive patterns was measured using a two-point probe method. We designed our own probe using four brass alloy spring pins (DESC: spring pins, P/N: ED1420-ND, Manufacturer: Mill-Max Mfg. Corp). This custom designed probe included soft-press springs and enabled accurate measurement of the resistance of the fragile materials. Our probe can precisely follow the material surface, thanks to the flexible spring pins (Supplementary Figure S1). To obtain accurate and reliable results, we used the direct method to determine the resistance using three calibrated precise resistance meter devices (TTI 1906 Digital Multimeter). Our custom design probe has four pins and the first resistance meter was connected between the first and second pins, and the second resistance meter was connected between the second and third pins and the third resistance meter was connected between the third and the fourth pins serially (see Supplementary Figure S1). The distance between the first and fourth pins was ~ 1 cm and the distance between the consecutive pins was ~ 0.33 mm. The resistance measurements were performed by placing the probe on the paper substrate with the printed conductive pattern with an area of 1 × 1 cm^2.

Fabrication and characterization of the printed RFID tag

The RFID tag was fabricated using the size and geometry of a commercially available tag (Invengo XCTF-8030A). This ultra-high frequency RFID tag operated at a range of 860-960 MHz. The dimensions of the label were 94 mm × 20 mm × 0.21 mm. Received signal strength indicator (RSSI) measurements were obtained via Impinj Speedway reader using an ultra-high frequency near-field antenna (MTI Wireless edge Ltd. MT-249568/NRH) at a frequency range of 865–870 MHz. The strength of the received signal was determined using the power level in terms of dB losses. The RFID chip was cut from the commercial tag and fastened with two plastic clips to the printed tag.

Results and discussion

Figure 1 presents a schematic description of our approach to fabricate conductive patterns on cellulose based paper substrates. The first step consisted of modifying the surface of the paper with a hydrophobic coating. This step was simply performed by dipping the office-grade paper into a solution of the resin solutions. Two types of industrially available resins were used in this study: one based on hydrocarbon and other based on fluorinated hydrocarbon resin, referred as NFR and FR, respectively. After the coating process, the paper substrates were heated at a temperature of 170 °C by placing the substrate in between two hot-plates. Particle-free reactive silver ink solution was then ink-jet printed on the coated paper substrates that were preheated to a temperature of 170 °C. A brief annealing at 170 °C was performed after the printing process for the formation of conductive patterns. This heating step ensured removal of volatile compounds and resulted in reduction of silver ions to metallic silver particles.

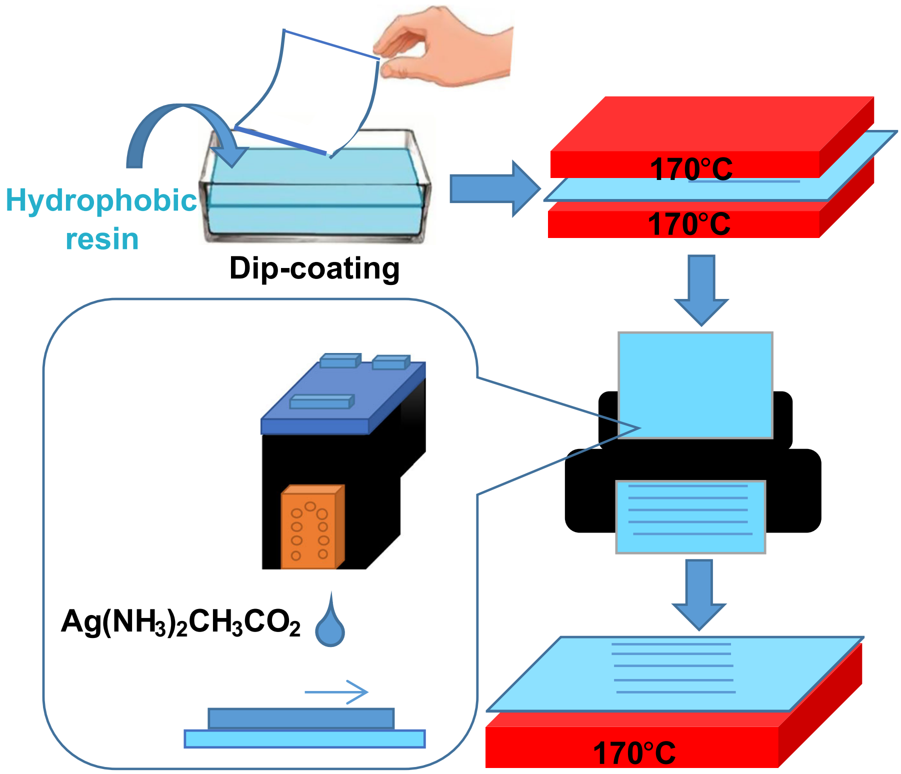


Fig. 1 Schematic description of the process to fabricate conductive patterns on the paper substrate. A hydrophobic polymeric resin was dip-coated on the paper substrate followed by heating in between two hot-plates. Particle-free reactive silver inks were then inkjet printed on the coated paper substrate. A brief annealing following the printing step resulted in the formation of conductive silver patterns on the paper substrate

Deposition of water-repellant coatings

We first studied the effect of the resin coatings on the wetting and morphology of the paper substrates (Fig. 2). The wetting behavior of the surfaces were characterized via static water contact angle measurements. Initially, the static water contact angle of the bare paper substrate was 105° ± 2°. Both resins increased the hydrophobicity of the paper and the static water contact angles of the NFR and FR coated paper substrates were 136° ± 2° and 140° ± 2°, respectively. The contact angle of water was higher on the FR coated substrate in comparison to the NFR coated one. This contrast in wettability likely arises from the lower surface energy of fluorinated compounds than hydrocarbons (Kota et al. 2014). SEM images (Fig. 2b) of the substrates showed the presence of the polymeric resins between the cellulose fibers (see Supplementary Figure S2 for a large area image). The hydrophobicity of the FR and NFR coated substrates remained mostly the same with increasing the concentration of the resins and repeating the dip-coating process to deposit multiple layers.

We studied the chemical composition of the paper substrates using XPS and EDX analysis. XPS is a surface sensitive technique, which provides information from the top 5 nm. EDX complements XPS by collecting data from a depth of couple of micrometers and probes both the thin film and substrate. Figure 3a presents the XPS spectra of the coated paper substrates. Both substrates exhibited characteristic peaks associated with C, O and Ca elements. In the case of the FR coated paper substrate, there was a strong F peak at around 688 eV that relates to the fluorocarbon content of the resin. XPS analysis further showed the presence of Cl containing species in the FR coated paper. O and C peaks of NFR were found at the same peak positions in comparison to the NFR; however, the intensities of these peaks were stronger. On the other hand, a new peak at around 104.6 eV appeared for the

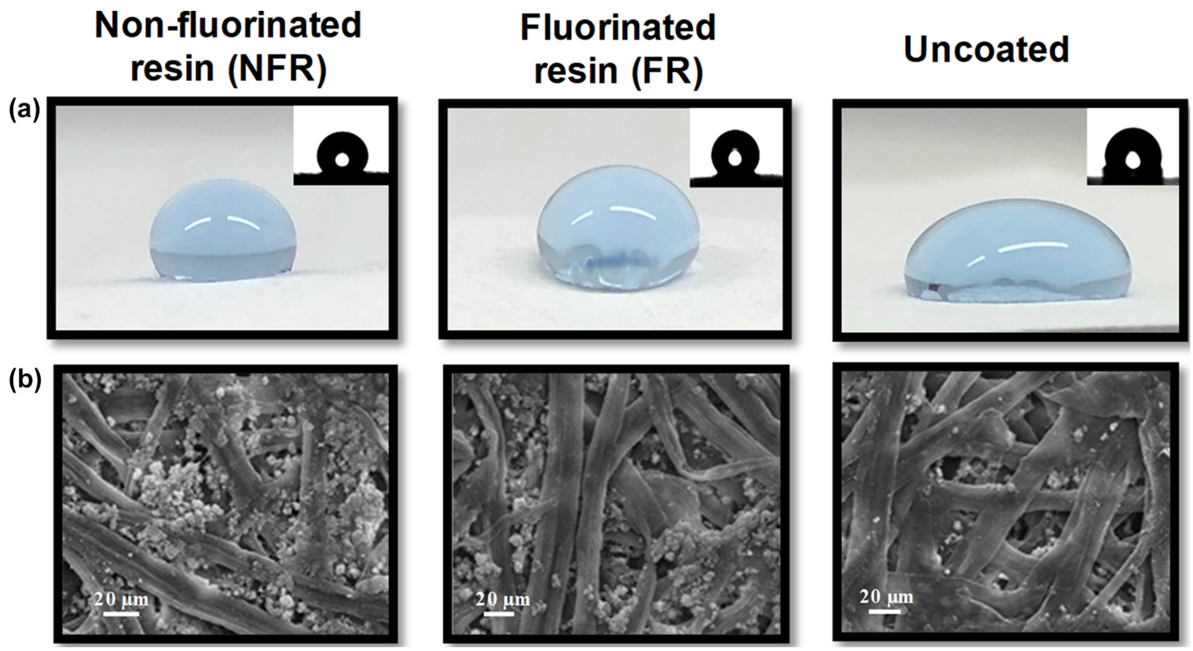


Fig. 2 Wettability and structural characterization. **a** Pictures of water droplets on the bare and coated paper substrates. The insets present images of the water droplets during the measurement of the contact angle. **b** SEM images of the substrates

NFR coated sample, which can be assigned to Si2p, suggesting the presence of Si in the coating layer. EDX analysis (Fig. 3b–d) showed the presence of C, O and Ca elements in the all substrates. The Ca peaks were more discernible in the EDX than XPS for the coated substrates, which suggest that the surface of the paper substrates is depleted with this element. These results imply that the uncoated paper substrate contains calcium carbonate species and the coating of substrates with the resins results in the coverage of the substrate with the resins. The relatively hydrophobic nature of the uncoated paper substrate likely relates the calcium carbonate additives present in the paper (Niu et al. 2014). EDX analysis further supports the presence of F and Cl in the FR coated paper and Si in the NFR coated paper substrates.

Printing of conductive patterns

The resistances of the patterns fabricated by ink-jet printing particle-free silver inks were investigated as a function of the number of printing layers for the coated and uncoated paper substrates. The preheating of the substrates at 170 °C was important to obtain low resistances over the printed pads. Table 1 lists the resistances of the printed patterns fabricated by passing 5–8 consecutive layers over the same region on the paper substrates. The resistance measurements represent the average values obtained from three different measurements over different regions of the pads. The resistance of the printed pads decreased with the increasing number of layers for the all substrates. The coating of the paper substrates with the resins significantly improved the resistance of the printed patterns. The hydrocarbon-based coating led to slightly lower resistances than the fluorinated one. The resistance of the coated paper substrates was roughly 40-fold lower than the bare paper substrate for the range of layers investigated. We could not obtain reliable resistance measurements on the bare paper substrate up to 6 printed layers, where the resistances of the printed patterns on the coated substrates was as high as 2.64 Ω. The lowest resistance was obtained by printing 8 consecutive layers on the NFR coated paper substrates. The significant improvement of the resistances together with the reduction in the number of printed layers required to obtain reliable conductivity showed the promise of the presented coatings.

We further characterized the conductive patterns fabricated by ink-jet printing particle-free reactive silver inks on the coated paper substrates. SEM images presented in Fig. 4a clearly show the presence of high

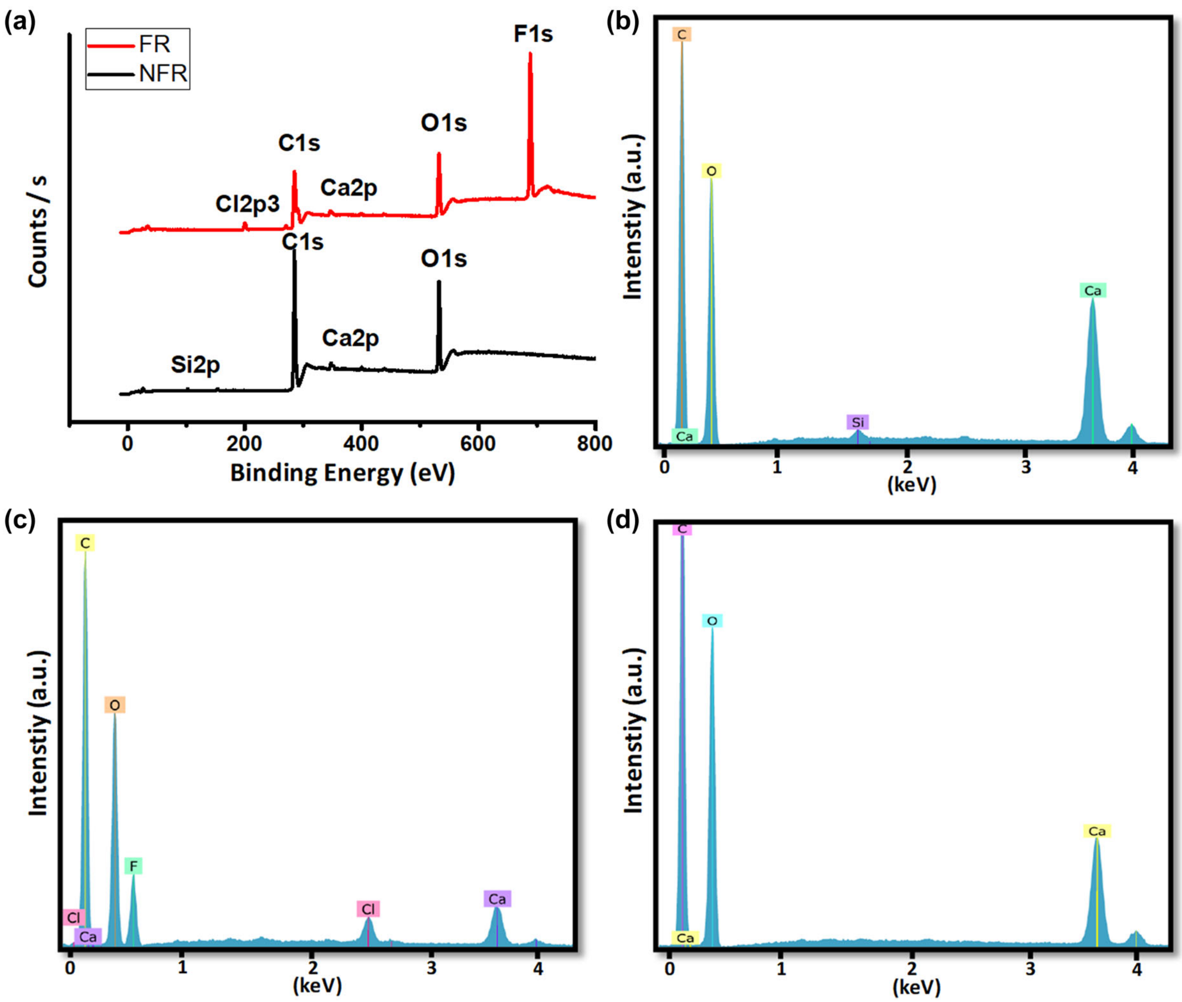


Fig. 3 Chemical composition of the coated paper substrates **a** XPS spectra of NFR coated and FR coated paper substrates. EDX spectrum for **b** NFR coated paper, **c** FR coated paper, **d** uncoated paper substrates

Table 1 The resistances (Ω) of the printed silver patterns on different paper substrates for varying number of printing layers

Number of layers	NFR	FR	Uncoated
5	2.64 ± 1.10	15.93 ± 4.63	–
6	1.42 ± 0.65	4.66 ± 3.24	134.5 ± 40.95
7	0.96 ± 0.63	1.33 ± 0.51	37.43 ± 3.68
8	0.29 ± 0.03	0.87 ± 0.03	15.33 ± 4.83

The values represent the average ± standard deviation in the resistance measurements taken from three different regions

density and fine silver particles within the patterns defined by the ink-jet printing followed by the brief thermal annealing. The fibrous structure of the paper was not discernible, since the silver particles merged and formed a continuous layer on the substrate. The silver particles did not exist in the background regions of the coated paper substrate, thanks to the additive nature of ink-jet printing that enables direct delivery of

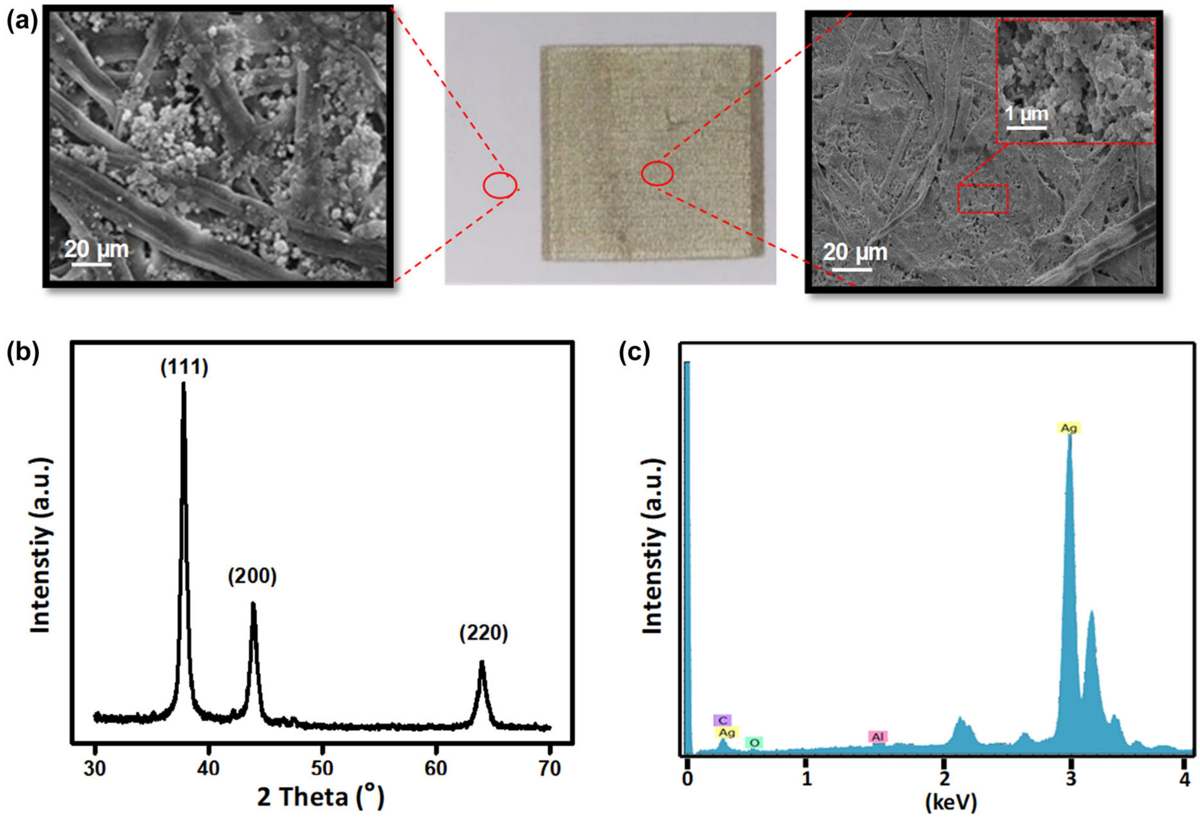


Fig. 4 Structural characterization of conductive patterns fabricated by inkjet printing particle-free reactive silver inks on the NFR coated paper substrates. **a** SEM images of the paper substrates from regions without (left) and with (right) the printed patterns. **b** XRD pattern, **c** EDX spectrum of the conductive patterns printed on the NFR coated paper

materials. The crystal structure and the chemical composition of the conductive patterns were further characterized by XRD and EDX analysis. The XRD pattern of the conductive patterns presented in Fig. 4b clearly shows the characteristic peaks that correspond to the (111), (200) and (220) planes of the face-centered-cubic crystal structure (Sakir et al. 2017; Walker and Lewis 2012). The average size of the silver crystallites was 16 nm as calculated from the Scherrer equation (see Supplementary Material for details). EDX spectrum (Fig. 4c) further confirmed the presence of silver within the printed regions.

Fabrication and characterization of the printed RFID tag

We finally demonstrate fabrication of a paper-based RFID tag by inkjet printing particle-free silver inks on the coated paper substrates. For this purpose, we used NFR coated paper substrates and printed 8 consecutive layers of particle-free silver inks. The size of the antenna and frequency of operation impose limitations on maximum attainable gain and bandwidth (Harrington 1960; Wheeler 1975). Compromises have to be made to obtain optimum tag performance and satisfy design requirements. The design (Fig. 5a) of our printed RFID tag followed a commercially available tag to facilitate easy comparison of the performance and use of the off-the-shelf chips. The performances of the printed and commercial RFID tags are compared in

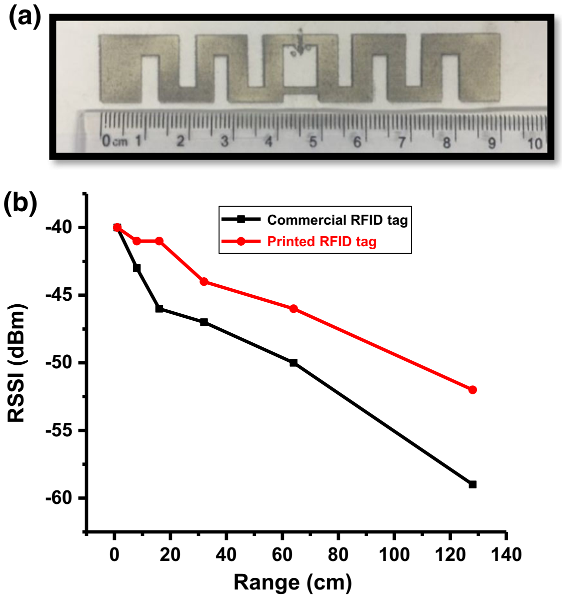


Fig. 5 Printed RFID tags. **a** Photograph of the RFID tag fabricated by ink-jet printing particle-free silver inks on the NFR coated paper substrate. **b** The minimum power level to read the tag as a function of the distance for the commercial and printed RFID tags

Fig. 5b based on the power losses as the RSSI for varying distances (1, 8, 16, 32, 64, 128 cm) between the reader and tag. The performance of the printed tag was better than the commercial tag as indicated by the reduced loss of the power for the entire range of the distances. These results suggest that our printing process is suitable for hardware prototype applications of RF antennas. RF applications are very sensitive for frequency transitions, therefore they are implemented on a printed circuit board, which is time-consuming and costly to fabricate using conventional methods. The ink-jet printing of particle-free inks offers high levels of flexibility in fabrication of conductive patterns overcoming the drawbacks of the conventional methods.

Conclusions

In conclusion, this study presented versatile, roll-to-roll compatible and solution-processing based modification of paper substrates for fabrication of printed electronic devices. The dip-coating of paper substrates with hydrocarbon and fluorocarbon-based resin materials greatly reduced the resistance of the conductive patterns defined by ink-jet printing of particle-free reactive silver inks followed by a brief thermal annealing. In comparison with the bare paper substrates, the conductive patterns could be obtained at much lower number of printed layers, a highly critical parameter for the cost and throughput of the process. The presented approach shows great promise for a variety of applications including sensing, data transmission, wearable electronics and control systems.

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