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Soft electronics by inkjet printing metal inks on porous substrates

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Soft electronics by inkjet printing metal inks on porous substrates

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Abstract

Soft electronic devices enable new types of products for an ergonomic interaction of humans with a digital environment. The inkjet (droplet on demand) printing of electrically conductive ink in plural on soft substrates such as paper, textile, and polymers is a promising route for the prototyping and small-scale production of soft electronics that is efficient, cost-saving, and provides a rapid turnaround due to its fully digital workflow. The choice of materials and processing parameters is challenging, however, due to the combined complexity of metal-containing inks, their dynamics during droplet ejection, the active role of the porous substrate, and possible post-deposition steps. This review focuses on recent developments in inkjet printing of metal inks onto soft, porous substrates and their applications. The first section discusses the general principles in the inkjet printing of metal inks, including drop formation and jetting, wetting, and post treatment processes. The second section deals with the effect that the porosity of substrates has on the drying, diffusion, and adhesion of inks. Finally, current challenges and achievements of inkjet-printed, metal-containing inks are discussed.

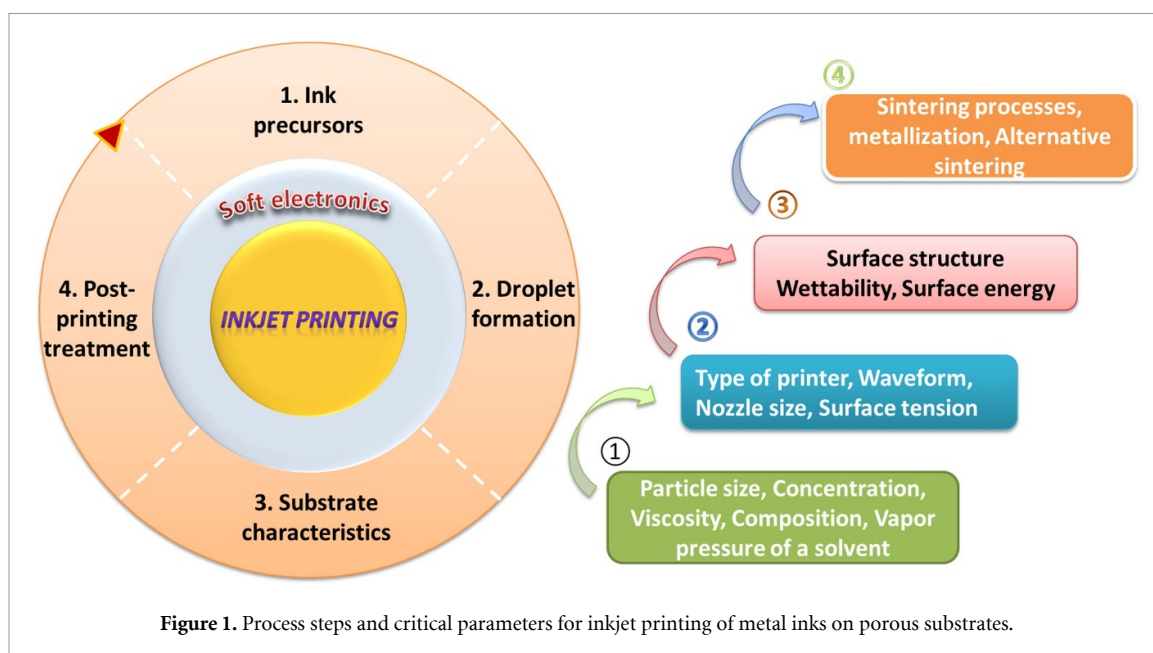
1. Introduction

Flexible and stretchable ‘soft’ electronics enable new types of displays, wearables, and biomedical devices. They are attractive for applications close to the human body, where traditional hard materials would be less ergonomic, and for new devices that directly interface with living tissues and cells. Functional conductive materials and their processing on flexible substrates have been intensively investigated for several decades in order to develop advanced soft electronics [1–4]. Here, we review the inkjet printing of electronic materials on deformable, porous substrates for soft electronic devices.

Porous substrates are common: paper, textiles, and hydrogels have pores at different length scales. Printing on porous surfaces is a proven strategy to improve the adhesion of thin functional films to the substrate, a property that is particularly important if the device will be bent or even folded. The embedding of a functional printed material inside a porous substrate leads to a composite and can increase the resistance against abrasion, cracking, chemical corrosion, and other degradation mechanisms.

Inkjet printing is suitable for porous substrates. It can deliver sufficient liquid ink to create interconnected, electrically conductive features of material on such substrates. As a digital technology, it provides rapid turn-over from pattern design to production. The resolution of standard inkjet printers is on the order of tens of micrometers with typical film thicknesses up to a micrometer, a range that is suitable for many relevant devices [5, 6]. Most importantly, inkjet printing can deposit stacks of layers from a wide variety of materials without masks, including electrically conductive materials. This has been used to print radio-frequency identification tags [7], transistors [8], sensors [9], solar cells [10], wearable electronics [11], and lab-on-a-chip systems [12].

Inkjet inks that contain colloidal particles or molecular precursors of silver or other metals are widely used in soft electronics, mainly because they yield layers that are more conductive than carbon-based materials and conductive polymers. Such ‘metal inks’ usually require sintering, in which the printed features are heated to remove organic components and merge particles. This implies high temperatures (>250 °C) that are incompatible with



many polymer and paper substrates [13–17]. Thus, recent research activities have focused on development of non-toxic and low temperature processable metal inks that can be printed to create deformable conductive structure on soft, porous substrates [18].

Printing with metal inks on porous substrates leads to complex process-structure-property relations that have not been fully understood yet. Microstructure and quality of the product both depend on properties of the ink such as viscosity, surface tension, colloidal stability, and solid content; and on properties of the substrate such as wettability, charge, and pore size distribution as described in figure 1. If, for example, the pores in the surface are larger than the metal particles, and if charge does not prevent it, particles may penetrate the bulk and form a discontinuous, non-conductive structure. Capillarity and viscosity can counteract this mechanism and lead to microscale bridging of pores by the metal particles, but these may have limited mechanical strength.

In this review, we survey the metal inks and porous substrates that have been used and identify mechanisms that set the quality of the product. First, we briefly discuss fundamental aspects of inkjet printing that are relevant for metal-based inks. Then, we summarize the effect of porous substrates on ink uptake, drying, diffusion, and adhesion of metals. We present selected cases of inkjet-printed soft electronics on paper and textiles. Finally, we discuss current challenges and comment on the future of the field.

2. Inkjet printing of metals

Conductive inks for inkjet printing have been formulated from 1D- and 2D-carbon materials [19, 20], conductive polymers [21, 22], metal precursors [23, 24], and metal nanoparticles (NPs) [15, 25].

Carbon materials and conducting polymers lead to typical conductivities of $10\text{--}10^2\text{ S cm}^{-1}$, 2–4 orders of magnitude below that of metal inks [26]. Metal inks based on molecular precursors or NPs lead to highly conductive films at relatively low processing temperatures (below $250\text{ }^{\circ}\text{C}$) due to the melting point depression associated to their small size [27]. Thus, they are popular materials for printed electronics.

Drop on demand (DOD) inkjet printers are commonly used to print metal inks [28]. Their print heads use either thermal or piezoelectric actuation to eject an ink droplet as shown in figure 2(a). In thermal inkjet printers, a small heater behind a ink-filled cavity close to the nozzle is activated by an electrical current pulse for approximately $1\text{ }\mu\text{s}$, resulting in the formation of a vapor bubble that ejects the ink from the nozzle [29]. Piezoelectric DOD inkjet printers release ink using the mechanical deformation of a piezoelectric transducer. The ejected volume depends on the driving pulse voltage and the resulting pressure wave that has to be adjusted for good accuracy and reliability [6]. The ink can only be ejected by a positive pressure wave with enough kinetic energy to overcome the surface energy of droplet formation; the remaining energy sets the velocity of the ejected droplet. Tuning the pulse voltage profile thus provides control over droplet size and velocity [30]. This review will focus on piezoelectric printing, the most common technology used in functional inkjet printing today [30–32].

2.1. Droplet formation from metal inks

Metal particles can clog inkjet print heads. A common rule of thumb is that the particle diameter should be at least 50 times smaller than the micron-sized nozzles of typical piezo heads and that the ink should have a viscosity in the range of $8\text{--}20\text{ mPa s}$.

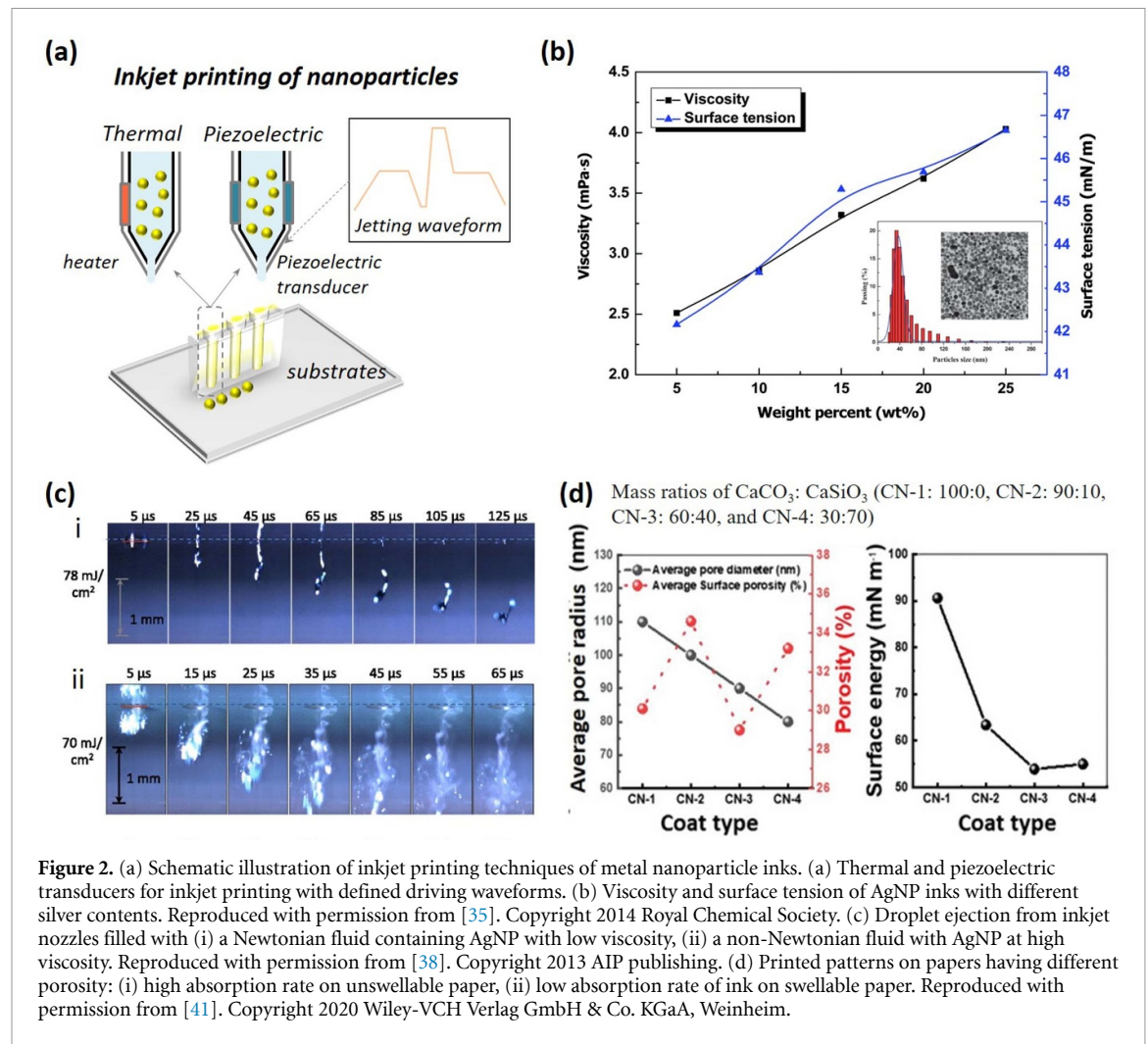


Figure 2. (a) Schematic illustration of inkjet printing techniques of metal nanoparticle inks. (a) Thermal and piezoelectric transducers for inkjet printing with defined driving waveforms. (b) Viscosity and surface tension of AgNP inks with different silver contents. Reproduced with permission from [35]. Copyright 2014 Royal Chemical Society. (c) Droplet ejection from inkjet nozzles filled with (i) a Newtonian fluid containing AgNP with low viscosity, (ii) a non-Newtonian fluid with AgNP at high viscosity. Reproduced with permission from [38]. Copyright 2013 AIP publishing. (d) Printed patterns on papers having different porosity: (i) high absorption rate on unswellable paper, (ii) low absorption rate of ink on swellable paper. Reproduced with permission from [41]. Copyright 2020 Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim.

[33]. More detailed analyses consider the surface tension (γ), viscosity (η), and density (ρ). Printability is then often determined using dimensionless groups: the Reynolds number (Re) that relates inertial forces with viscosity and the volumetric flow rate (Q) (equation (1)), the Weber number (We) that compares droplet velocity (v) and surface energy (equation (2)), and their combinations, the Ohnesorge number Oh (equation (3)) and its inverse (Z). They depend on physical properties of the fluid and the dimensions of the ejecting nozzle that is usually close to the drop diameter, but not on droplet velocity [34]:

$$Re = \frac{v\rho L}{\mu} = \frac{4Q\rho}{\pi\eta D_{noz}} \quad (1)$$

$$We = \frac{v^2\rho L}{\gamma} \quad (2)$$

$$Oh = \frac{1}{Z} = \frac{\sqrt{We}}{Re} = \frac{\mu}{\sqrt{\gamma\rho D_{noz}}}. \quad (3)$$

Newtonian metal inks are well-described by these dimensionless groups. Shen *et al* investigated the

printability of silver nanoparticle (AgNP) inks based on deionized water and ethylene glycol as function of their Ag content [35]. The inks were Newtonian fluids with viscosities that increased from 2.51 mPa s to 4.03 mPa s and surface tensions that increased from 42.16 mN m⁻¹ to 46.65 mN m⁻¹ as the AgNP content increased from 5 wt% to 25 wt% as shown in figure 2(b). The small size of the silver particles (mean diameters ranging from 30 to 50 nm, figure 2(b)), the good dispersibility in polar solvents, and the absence of additives explain the relatively small viscosity increase at higher particle content. Consequently, Z decreased from 9.94 to 7.00 but remained in the range of $10 > Z > 1$ that is often deemed most suitable for DOD printing without satellites (that occur for $Z > 10$) or drop formation problems (for $Z < 1$). Hoeng *et al* reported the printability of silver-crystalline nanocellulose (CNC) nanoparticle (CNC-Ag) inks as a function of Ag concentration and Z value. They observed that adding 0.15 wt% of the surfactant dioctyl sulfosuccinate decreased surface tension and led to an acceptable value of Z ($=13.3$) at a surface tension of 29.1 mN m⁻¹ [36].

The droplet formation of non-Newtonian fluids has been investigated, too. Reis *et al* studied droplet

velocities and sizes formed from alumina suspensions (average diameter $0.3\ \mu\text{m}$; the viscosity at 20 vol% was 7 mPa s, at 30 vol% 22 mPa s, and at 40 vol% 42 mPa s) as a function of pulse width, driving voltage, and solvent (mixtures of paraffin wax and kerosene) that affected the amount of shear thinning [34]. They noted that the viscosity exhibited asymptotic behavior at high shear rates and could be considered constant at the high shear rates typical for ink-jet printing. The velocity of ejected drop was governed by the displaced volume of the print head as predicted with the dimensionless quantity Tsai *et al* studied non-Newtonian AgNP inks containing 30 wt% silver. The high viscosity of the silver suspension at this high particle concentration required larger pulse voltages for droplet formation and led to Re and We in the ranges of 5–8 and 2.0–4.7. Single droplets were ejected in the range of ± 33 and ± 43 V, while ± 46 V caused satellites formation [37]. Mathews *et al* visualized the ejection of silver NP and silver nanopaste inks with viscosities of 10 mPa s ($Z = 2\text{--}4$) and >100 Pa s ($Z > 20$), respectively. Nanopaste ink at very high viscosities (500 Pa s) behaved solid-like and did not show a jetting behavior at all (figure 2(c)) [38].

2.2. Printing metal inks on porous substrates

Classical inkjet substrates are porous. Paper, cardboard (CB), fabrics, and other materials widely used in graphical printing do not have a flat, hermetic surface. Their surfaces range from microporous (2 nm average pore diameter or below) and mesoporous (2–50 nm) to macroporous (50 nm or above); in most cases, all three pore types are present. The porosity affects the penetration of the printed ink and its spreading on the surface. The distribution of dried metal ink on and inside the substrate affects adhesion and electrical properties. An understanding of the porous microstructure and its interaction with the liquid ink therefore is crucial for optimal performance.

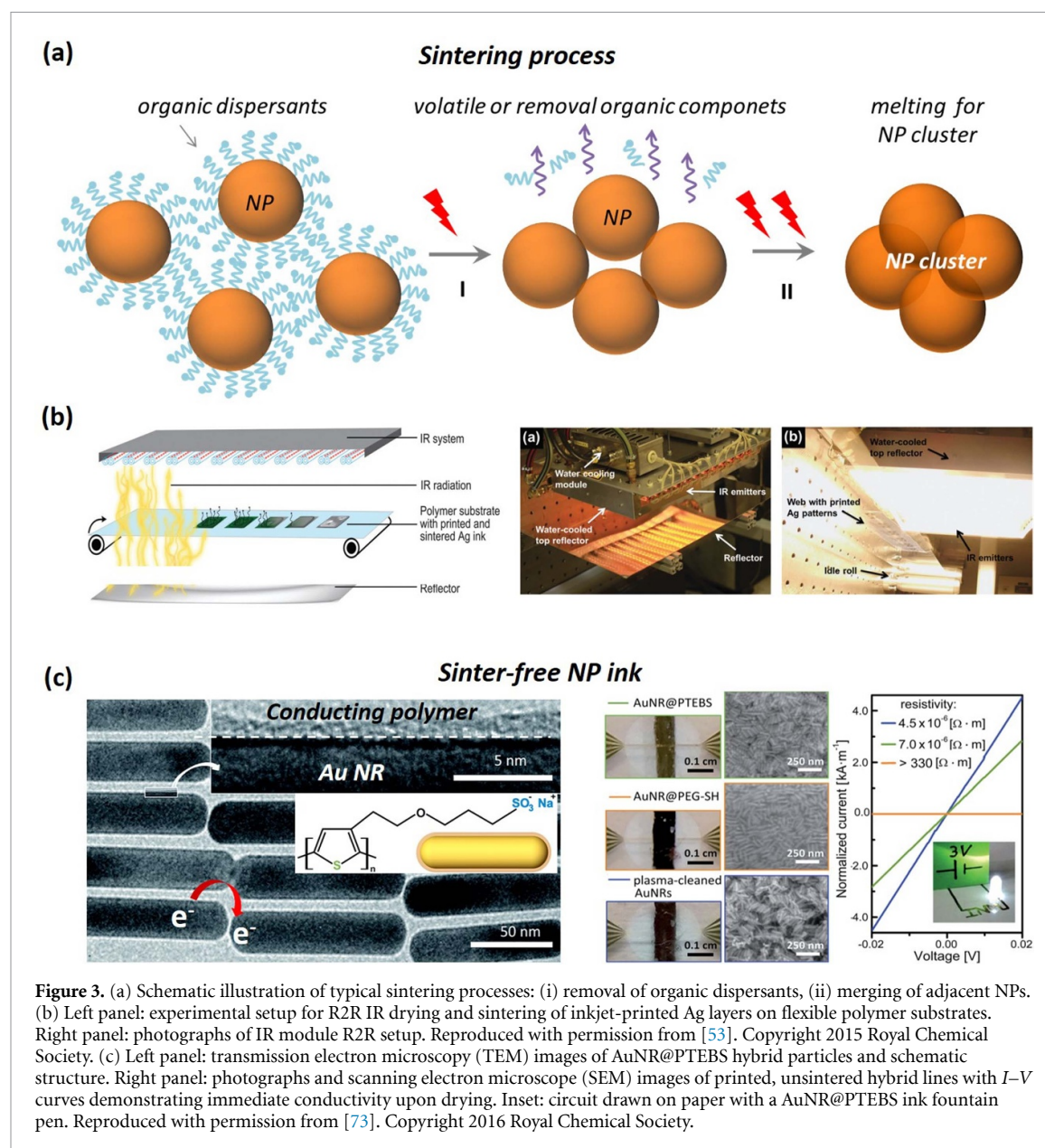
The transfer and setting of ink during gravure printing and other graphical printing techniques with relatively viscous inks have been studied and reviewed in the 1990s [39]. The spreading of low-viscosity inkjet inks has been quantitatively analyzed later. Wijshoff provided a comprehensive picture that combines different length scales and mechanisms from initial spreading to kinetic and capillary spreading and complete wetting [40] that considers the deposition of particles, too. The group of Song *et al* exploited the porosity of the substrate to increase the attainable resolution [41], but the role of porosity is only briefly considered. Other groups increased the printing speed by optimizing the interaction of the ink with the porous substrate [42], the group of Toivakka studied the spreading of the liquid and exploited it for analytical purposes [43]. Only few published studies discuss the spreading and penetration of metal inks on porous substrates

for applications that require electrical conductivity. Öhlund *et al* inkjet-printed AgNP dispersed in triethylene glycol monoethyl ether on 11 different paper types and found that the surface energy had only a minor effect on the electrical conductivity [44]. Surface porosity was dominating the electrical conductivity of the printed patterns. The relatively faster absorption rate of porous substrates compared to non-porous substrates reduced spreading movement of particles toward edges. The conductivity of electrodes printed on porous paper was three times above that printed on a non-porous polyimide (PI) film even after sintering at $110\ ^\circ\text{C}$. Kattumenu *et al* observed a similar role of the absorption rate using an ink based on silver flakes: rapid penetration into the substrates at higher surface porosity helped to create narrow conductive lines [45]. Our group recently reported on the inkjet printing of hybrid gold nanoparticle (AuNP) inks on coated CBs [46]. Porosity, absorption rate, and surface tension were adapted using top coats with different ratios between (more polar) CaCO_3 and (less polar) CaSiO_3 particles. We found smaller pore sizes and lower absorption rates for coats with larger fractions of CaSiO_3 as shown in figure 2(d). The slower penetration led to lower electrical resistivity, probably because AuNPs had enough time to pack densely. We also found that the increased wettability of coats with a larger fraction of CaCO_3 facilitated spreading of the water-based ink and improved the connectivity between the conductive particles.

The penetration of metal inks into substrates affects the mechanical adhesion and stability of inkjet-printed conductive layers, too [44, 47, 48]. Tobjörk *et al* reported that thermally annealed AuNP inks were less prone to crack formation upon bending on porous paper substrates than on non-porous polymer and glass. The microstructure that formed during penetration increased the metal-substrate adhesion [49].

3. Post-processing of printed metal NP inks on porous substrates

Silver is a popular material for nanoparticles in conductive inks because of its high conductivity ($1.59 \times 10^{-8}\ \Omega\ \text{m}$ for bulk silver) [50]. Gold has a similar conductivity ($2.44 \times 10^{-8}\ \Omega\ \text{m}$) and is even more stable, but relatively more expensive [51]. Aluminum and copper are considered as cheaper conductive ink materials (aluminum has $2.82 \times 10^{-8}\ \Omega\ \text{m}$, copper $1.72 \times 10^{-8}\ \Omega\ \text{m}$), but they are prone to oxidation [15]. All metals have Hamaker constants on the order of $10^{-19}\ \text{J}$ that imply strong van der Waals attraction between the nanoparticles in the ink. It is therefore necessary to cap the metal particles with stabilizing molecules or ligands, typically organic molecules or polymers, to obtain inks with sufficient colloidal stability. The shell prevents



agglomeration of the particles that would lead to clogging of inkjet nozzles, sedimentation, and inconsistent printing quality.

3.1. Thermal sintering

In the dried ink, organic shells form interfaces between the metal cores that impede electrical transport. Sintering processes are often employed to remove the organic barriers and transform the printed metal NPs to electrically conductive metal films (see figure 3(a)) [8, 37, 52–54]. Typical sintering processes can be divided into two steps. First, the organic component is removed from the surface of the NP. Second, particles coalesce to reduce surface energy, and surface diffusion leads to the formation of bridges and necks with increasingly large necks. Thermal sintering is the most common method because it is simple, fast, and cost effective; most commercial NP-based metal inks require thermal sintering to

achieve conductivity. Typical processing temperatures are above 200 °C for 30 min, which exceeds the thermal budget for common soft and porous substrates such as paper, plastic, and fabric [55–57]. Non-thermal sintering processes are therefore particularly relevant for those materials and covered in detail below.

3.2. Alternative sintering processes

Optical sintering with laser [18], infrared (IR) [58], plasma [59], or flash light [60] at room temperature have emerged as attractive alternatives to overall thermal treatment due to its high speed and compatibility with soft and porous substrates. Balliu *et al* studied the effect of laser sintering on inkjet-printed AgNP layers (850 ± 150 nm thick) on seven different types of papers with a galvanometric scanning mirror system at 250 kW cm^{-2} [61]. They found that layers on CB and light-weight coated paper (LWC)

exhibited lower conductivities than layers on instant-drying photo papers due to residual solvent that led to damage of the Ag films by the intense laser light. The highest conductivity of $1.63 \times 10^7 \text{ S m}^{-1}$ (nearly 26% of bulk silver) was observed for a ‘photographic’ paper with a coating system based on aluminum hydroxide oxide, which has relatively high optical absorption compared to CB and LWC. Gaspar *et al* used IR light to sinter inkjet-printed AgNP layers (thicknesses 250–400 nm) on different fiber-based substrates [62]. They reached electrical conductivities of up to 40% of bulk silver with IR light at a short exposure time of below 2 min. Low-pressure Ar plasma sintering at 190–300 W and 0.4 mbar was applied by Sanchez-Romaguera *et al* to sinter inkjet-printed AgNP layers (thicknesses $\geq 18 \text{ }\mu\text{m}$) on two different paper substrates [63]. Highly porous tattoo paper led to unreliable and comparatively high resistances ($11.6 \text{ }\Omega$ along a $8 \times 0.4 \text{ mm}^2$ printed line); PEL Nano-P60 paper (Printed Electronics Ltd, UK) with an inorganic microporous coating had $3.3 \text{ }\Omega$ along the same geometry after 30 min in a plasma. They reported that optimal pulses had energies of 900–1100 J. Lower pulse energies did not significantly improve sheet resistance, whereas higher energies increased the risk of partial ablation of the silver from the substrate.

Metal NPs can be ‘chemically’ sintered by controlled exposure to liquid or gaseous agents. Solvents or surfactants can be used to dissolve the stabilizing molecules and remove them from the printed patterns [64, 65]. Chemical removal of the stabilizers can induce coalescence of the metal cores and increase electrical conductivity. Magdassi *et al* reported coalescence and sintering of AgNPs at room temperature upon exposure to oppositely charged polyelectrolytes. The resulting conductivity was sufficient to inkjet-print an electroluminescent device on paper [66]. Dai *et al* reported chemical sintering at room temperature via ligand exchange of Cu–Ag core-shell nanoparticles (Cu@AgNPs) [67]. The oleylamine stabilizer was replaced by 1-amino-2-propanol by galvanic displacement. A NaBH_4 solution induced particle coalescence within a few minutes to reach a final conductivity of $36.3 \text{ }\mu\Omega \text{ cm}$.

3.3. Sinter-free metal inks

An alternative to metal sintering is to print metal precursors that can be reduced on the substrate to form metals [68–73]. For example, modified Tollens reagents based on silver nitrate, sodium hydroxide and ammonia have been used to prepare silver complexes. The ammonia evaporates upon drying, and metallic silver is formed in-place by the reduction of Ag^+ [74]. Walker *et al* developed a related Ag precursor ink of silver acetate in aqueous ammonium hydroxide and formic acid that has a low viscosity and yields silver films with a conductivity of $6.25 \times 10^5 \text{ S cm}^{-1}$ at $90 \text{ }^\circ\text{C}$ for 15 min [70]. Chen

et al reported silver citrate as precursor in an inkjet-printable ink that was prepared with ethylenediamine, water, ethanol, and ethylene glycol to tailor the viscosity and surface tension [24]. They used the ink to print Ag precursors with a commercial DOD printer of Epson KGT-3290A on a flexible PI substrate as catalyzing seed to deposit copper in a site-selective electroless plating bath. The resulting copper patterns had a conductivity of $2.80 \text{ }\mu\Omega \text{ cm}$ and were immersed in a 1,2,3-benzotriazole solution (0.02 mol l^{-1}) for 2 min to prevent oxidation in air. Black *et al* used a reactive organometallic inks originally developed for atomic layer deposition to print silver [75]. The authors formulated the commercial silver hexafluoroacetylacetonate cyclooctadiene with propan-2-ol in anhydrous toluene to obtain an inkjet ink. Propan-2-ol acted as a catalyst for the removal of hexafluoroacetylacetonate ligands from the silver precursor. The optimal sinter-free deposition of conductive silver films was achieved at a printing temperature of $110 \text{ }^\circ\text{C}$, the electrical resistivity was as low as $4.1 \times 10^{-8} \text{ }\Omega \text{ m}$, which is 39.2% that of the resistivity of bulk silver ($1.59 \times 10^{-8} \text{ }\Omega \text{ m}$).

A new approach that entirely avoids post-processing of the printed structures is the use of hybrid NPs. Small molecules and polymers containing π -conjugated bonds can be used as stabilizing ligands of metal NPs that enable carrier transport between particles in the dried film. In 2012, Kanehara *et al* used large aromatic phthalocyanine (H_2pc) derivatives as ligands of AuNPs with an average diameter of 14.3 nm. The AuNPs were synthesized using citrate as reducing agent and stabilizer, and a H_2pc derivate was added to the dispersion to replace the citrate on the NP’s surface. The conductivity of drop-casted samples was determined to be 1560 S cm^{-1} and 6604 S cm^{-1} depending on the H_2pc employed [76]. The same group later demonstrated the use of these inks to fabricate transistor devices printed at room temperature [77]. Improved colloidal stability can be obtained by using polymers as ligand. Some of us reported a ligand exchange reaction that results in gold nanorods (AuNRs) stabilized by conjugated polymers such as (poly[2-(3-thienyl)-ethyloxy-4-butylsulfonate] (PTEBS) and poly(3,4-ethylenedioxythiophene):polystyrene sulfonate (PEDOT:PSS) as shown in figure 3(d) [78]. The polymers lent colloidal stability to the ink and formed conductive particle-particle junctions upon drying that led to a bulk conductivity 10^3 times above that of bulk PEDOT:PSS.

4. Porous substrates for inkjet-printed soft electronics

Porosity is a common property of many substrates traditionally used in writing and printing; papyrus, vellum, and conventional paper all take up considerable amounts of ink into their structure.

Table 1. Combinations of metal inks and porous substrates reported for soft electronics.

Particle type	Dispersion medium	Substrate polarity	Structure of substrate	Sintering process	Application	References
AgNPs	(1) Mixture of ethanol and ethanediol (2) Nonpolar organic solvent mixture	Hydrophilic	Nanoporous surface coating on cellulose paper	Thermal sintering at 150 °C for 30 min	Thin-film transistors	[79]
Ag based catalyst ink	Aqueous solution	Hydrophilic	With/without surface treatment of micro-porous paper	—	Electronic circuit	[80]
AgNP	Aqueous solution	Hydrophilic	Mitsubishi resin coated paper (micro-porous)	Room temperature sintering	Thin film electrodes for light-emitting diodes	[14]
AgNP	Polar based alcohol mixture	Hydrophilic	Thermal treated micro-porous paper	Laser irradiation	Humidity sensor	[23]
AgNW	Aqueous solution	Hydrophilic	Cellulose nanofibril (CNF)-mediated nanoporous paper	UV irradiation	Supercapacitor (SC)	[81]
Single crystal of SnS ₂	Polar based alcohol mixture	Hydrophilic	Plasma treated commercial fibrous photo paper	—	Gas sensor	[7]
AgNP	Deionized (DI)-water	Hydrophilic	Nanocellulose coated nanoporous CB	Thermal sintering at 180 °C for 30 min	Electrode	[82]
AgNP	Polar based alcohol mixture	Hydrophilic	Commercial fibrous photo paper	Laser irradiation	Flexible electronic	[61]
Reactive Ag	Aqueous solution	Hydrophilic	Interconnected porous fabric substrates (knit, woven, and nonwoven fabrics)	Heat-cured at 140 °C for 5 min	Smart textile	[83]
Reactive Ag	Aqueous solution	Hydrophilic	Two different interconnected porous polyethylene terephthalate(PET) knit fabrics	Heat-cured at 150 °C	Electronic textile	[84]
Reactive Ag	Aqueous solution	Hydrophilic	Microporous synthetic textiles	Heat-cured at 90 °C	Textile heating actuator	[52]
Ag and CuONP	Polar based alcohol mixture	Hydrophilic	40 different micro-porous papers	Photonic sintering	Flexible paper electronic	[85]
AgNP	Tetradecane	Hydrophobic	Porous PI films	Thermal sintering at 220 °C for 60 min	Flexible electrode	[86]

Modern polymer coatings and foils, in contrast, are sufficiently flat and compact to prevent ink take-up almost completely. Porosity dramatically changes the printing and drying processes, the adhesion of the printed ink, and—in case of conductive inks—the conductivity of the resulting structures.

Porosity alone does not imply that ink is taken up. Surface chemistry and microstructure strongly affect wetting and absorption. Matching substrate and ink properties therefore is an important research topic; some examples of successful combinations used for

conductive printing are summarized in table 1. The following chapter reviews important aspects of the substrate-ink interplay and summarizes the current understanding of the relevant interactions.

4.1. Paper

Paper is the most common flexible substrate used for printing [87]. It is lightweight, low in cost, highly accessible, and can be recycled using a fully developed infrastructure that is already available in many countries. A well-established technology exists to modify

the surface of paper in order to tune its porosity and surface chemistry. It is not surprising, therefore, that paper has been used in many studies to prepare flexible electronics [7, 65, 79–81, 88, 89].

Consider a micron-sized ink droplet that impacts the surface of paper. It wets and spreads on the surface depending on surface tension [90]. Depending on porosity, liquid rapidly enters the bulk, possibly leaving particles behind [91, 92]. Uncoated paper consists of randomly oriented cellulose fibers that are linked by hydrogen bonds, with micro-scale voids that are filled by the liquid [93–95]. Coated papers are covered by ceramic pigments, small molecules, polymers, and composite materials that change optical properties and printability [42, 79, 80, 96–101]. The complex absorption processes into this heterogeneous material have been investigated as a function of paper type and coating both for graphical and conductive inks [102, 103]. Hsieh *et al* studied the effect of porosity on spreading and absorption. The authors compared a commercial AgNP ink that contained 20 wt% of 30–50 nm diameter AgNP in ethanol/ethylene glycol with a particle-free silver-based, metal–organic decomposition (MOD) ink on micro-porous pulp paper, cellulose nanopaper, and PI foils using a piezoelectric inkjet printing system [104]. Spreading of AgNP and MOD inks on micro-porous pulp paper caused wavy pattern boundaries and high resistances because the particles did not form continuous structures (figure 4(a(i))). Mechanically fibrillated nanopapers led to straight boundaries with sharp edges and a resistance below $0.4 \Omega \text{ cm}^{-1}$, sufficient to contact LED lights (figure 4(a(ii))). Nge *et al* studied the effect of cellulose nanofibril (CNF) papers with nanoporous structure and low surface roughness on the printing of a commercial 20–50 nm diameter AgNP ink [105]. Patterns printed on CNF-coated surfaces had lower electrical resistances ($1.57 \pm 0.09 \Omega \text{ cm}^{-1}$) than on PI ($2.07 \pm 0.17 \Omega \text{ cm}^{-1}$) and polyethylene naphthalate foils ($2.10 \pm 0.16 \Omega \text{ cm}^{-1}$) after curing at 150°C for 1 h. Wang *et al* studied a stannous chloride (SnCl_2) paper coat to reduce the high ink absorption rate of an Ag-based ‘catalyst’ ink (0.2 mol l^{-1} silver nitrate dissolved in a mixture of water, ethyl alcohol, ethylene glycol, n-propanol, and glycerol) during inkjet printing [80]. Optical microscopy indicated that the ink penetrated into the coated paper by about $100 \mu\text{m}$, much less than in uncoated paper, because the hydrolysis of SnCl_2 stopped the liquid flow (figure 4(a(iii))). Lessing *et al* reported inkjet printing of high resolution ($\geq 20 \mu\text{m}$) conductive patterns for medical devices on chemically modified paper [106]. Omniphobic (both hydrophobic and oleophobic) paper was prepared using a fast vapor-phase treatment with highly fluorinated organosilanes that reduced the surface free energy of the paper. Two different types of commercial inks (reactive Ag DGP 40-IT-15C

from Advanced Nano Products and Carbon Ink 3801 from Methode Electronics Ink) were printed on different omniphobic papers. The confinement of the material due to the reduced contact angle of the ink on the paper during drying increased the final conductivity and the resolution of the printed patterns. The authors reported an improved mechanical durability because the silver strongly adhered to the cellulose fibers of the silane modified paper. Zhang *et al* used a mixture of surfactant and polymer solution to coat paper and obtained conductive and mechanically durable electrodes using an inkjet printable silver precursor ink (60 mg ml^{-1} of silver salt in glycerol/water) as shown in figure 4(b) [107]. A combination of polyvinylpyrrolidone (PVP) and poly(4-vinyl pyridine) on cellulose papers led to $\sim 2.5 \times 10^{-3} \Omega \text{ sq}^{-1}$ and bending stability up 30% bend rate (calculated as L/L_0 with the true spacing L between terminals and the original spacing L_0).

The potential of conductive inkjet printing on paper has been realized for a range of soft electronic devices including micro-electromechanical system (MEMS), gas sensors, conductive leads, supercapacitors (SCs), and biosensors [7, 61, 81, 106, 108, 109]. Choi *et al* printed flexible SCs on conventional A4 paper using a Ag NW (DT-AGNW-N30-1DI, Ditto Technology Co.) suspension (0.25 wt%) in water and a commercial desktop inkjet printer [81]. The inkjet-printed SCs retained their function over 2000 cycles, provided good mechanical flexibility, and could be adapted for integration into systems (figure 4(c(i))). Tavakoli *et al* fabricated paper-based tattoo-like electronics using a combination of a AgNP ink and a thin layer of eutectic gallium indium (EGaIn) [14]. They showed that the electrical conductivity was increased by six orders of magnitude compared to pure AgNP ink and the printed layers maintained low surface resistance under bending or when stretched to 80% strain. Soft on-skin electronics as shown in figure 4(c(ii)) were fabricated based on the ink system.

4.2. Textiles

Electronics as part of wearable textiles—so called ‘e-textiles’ or ‘smart textiles’—are a flexible, lightweight, and conformable platform that enable new applications in healthcare, sport, fashion, and safety [110–113]. Early examples were based on conducting polymer fibers, metal wires, or carbon nanotubes directly embedded in the fabric [114–116]. It is difficult to digitally pattern using such materials, while nano-sized metal NPs or metal component-inks can be printed directly onto suitable textiles [83, 84, 117, 118]. Nechyporchuk *et al* used wood-derived cellulose nanofibrils (CNFs) as a base coating for textile substrates to improve the inkjet printing of a commercial AgNP ink (15 wt% of Ag in water (NBSIJ-MU01 from Mitsubishi Paper GmbH))

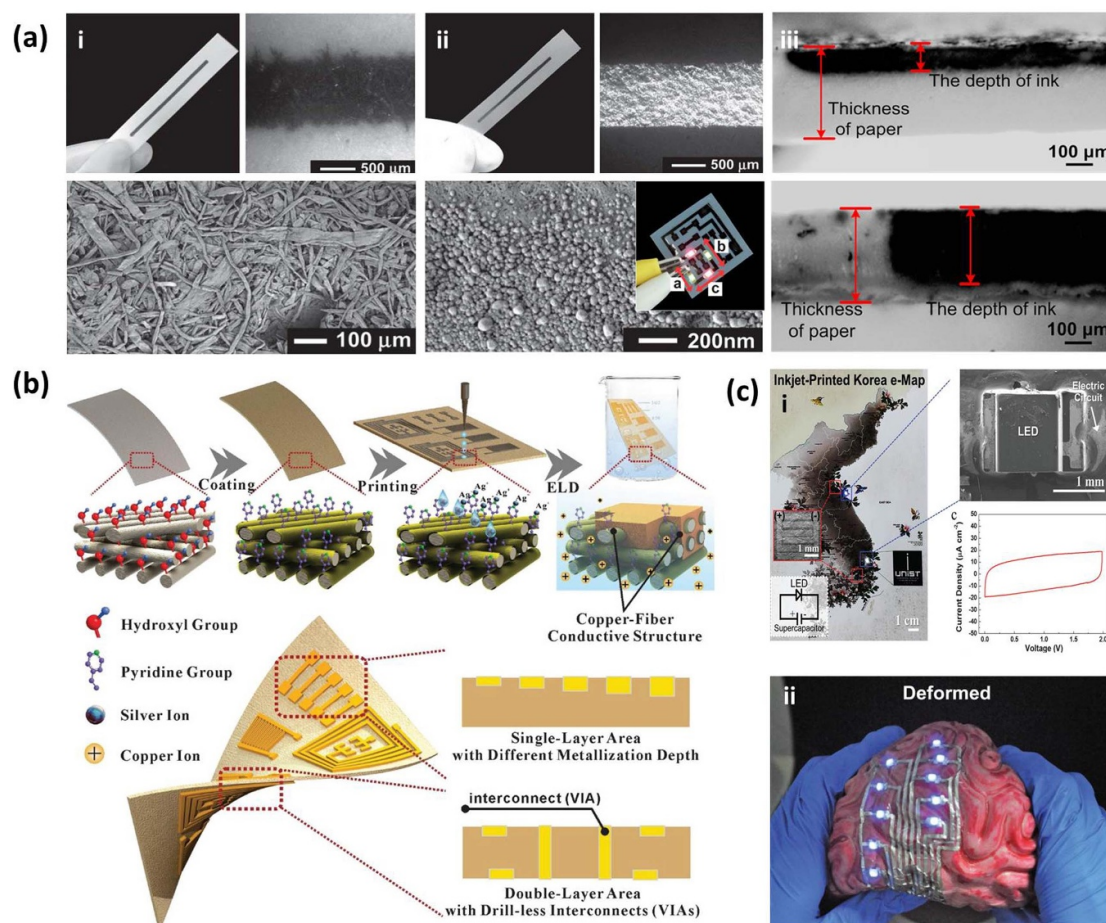


Figure 4. (a) (i) Top panel: silver lines on pulp paper, bottom panel: scanning electron micrograph of silver lines on pulp paper. (ii) Top panel: photograph of silver lines on nanopaper, bottom panel: (SEM) images of silver lines on nanopaper connecting LEDs. Reproduced with permission from [100]. Copyright 2013 Royal Chemical Society. (iii) Top: optical image of ink penetration on surface-treated paper. Bottom: optical image of ink penetration on paper without any pretreatment. Reproduced with permission from [75]. Copyright 2018 American Chemical Society. (b) Schematic diagram of a fabrication process for single/double-layered flexible circuits on cellulose paper. Reproduced with permission from [103]. Copyright 2018 Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim. (c) (i) Left panel: photograph of an inkjet-printed map of Korea, wherein the inkjet-printed SCs (marked by red boxes) were seamlessly connected to LEDs (marked by blue boxes) via inkjet-printed leads. Right, top panel: SEM of LEDs connected to inkjet-printed electric circuits. Bottom panel: current-voltage-profile (scan rate = 1.0 mV s^{-1}) of the inkjet-printed SC in the map. Reproduced with permission from [76]. Copyright 2016 Royal Chemical Society. (ii) Functionally connected LEDs on a soft brain-shaped toy after deformation. Reproduced with permission from [14]. Copyright 2018 Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim.

[119]. They spray-coated woven cotton fabrics with a mixture of CNFs and glycerol (as plasticizer) in water and characterized the film-forming properties and its influence on the printing using various ink droplet volume (figure 5(a)). The sheet resistance on pure fabric substrate was above $83.3 \text{ k}\Omega \text{ sq}^{-1}$; the CNF coating reduced it to $2.9 \pm 0.3 \text{ }\Omega \text{ sq}^{-1}$ but introduced the brittleness of CNF to the textile that was only partially ameliorated by glycerol that acted as plasticizer.

Particle-free metal precursor inks using the modified Tollens reaction have been suggested for e-textiles in order to obviate or simplify sintering. Stempien *et al* prepared silver traces via a modified Tollens' process with sintering temperatures below 90°C and reached sheet resistances of $0.155\text{--}0.235 \text{ }\Omega \text{ sq}^{-1}$ on synthetic textiles and $0.389\text{--}0.622 \text{ }\Omega \text{ sq}^{-1}$ on natural fibers and their blends with synthetic fibers [52]. They tested the resistance

to bending, washing, and dry-cleaning and found ink-textile combinations suitable for capacitors, textile heating actuators, and textile patch antennas. Shahariar and Kim *et al* reported on a reactive Tollens-type Ag ink (dispersed in polar protic solvents) printable on uncoated polyester textile knit, woven, and nonwoven fabrics [83] (figure 5(b(i))). The resulting metal films coated individual fibers to form a conducting network on the textile structure without degrading the comfort and mechanical properties of the textile. The conductivity and attainable resolution were related to the packing and the tightness of the fabric structures, fiber sizes of the fabrics, porosity, and surface energy. Electrical conductivities in the range of $0.2 \pm 0.025 \text{ }\Omega \text{ sq}^{-1}$ and $0.9 \pm 0.02 \text{ }\Omega \text{ sq}^{-1}$ were reached on woven (tight structure, low porosity) and knit (open structure, high porosity) polyester fabrics, respectively. In

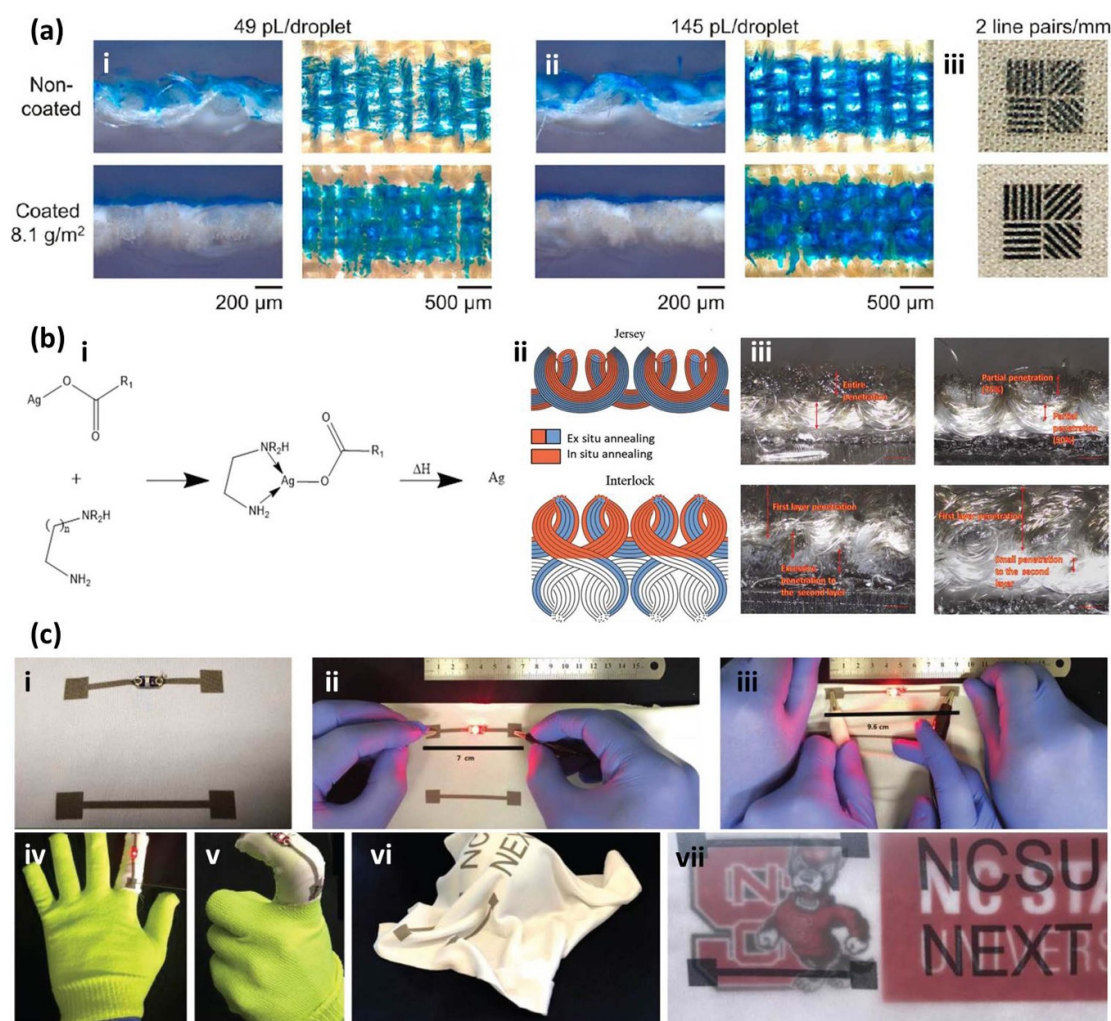


Figure 5. (a) Optical micrographs of uncoated fabrics (top panels) and fabrics coated (bottom panels) with 8.1 g m⁻² CNFs/glycerol (10/1 w/w). The cross sections (i, ii; left) and top views (i, ii; right) were inkjet-printed with cyan pigment at 49 pL/droplet (i) and 145 pL/droplet (ii). (iii) Fabrics with two printed line pairs per mm. Reproduced with permission from [115]. Copyright 2017 American Chemical Society. (b) (i) Chemical reaction mechanism of reactive Ag ink, (ii) ink penetration into jersey and interlock knit structures with *in situ*/*ex situ* annealing. (iii) Cross-sectional optical micrographs of samples with 15 print passes (10×, 200 μm scale bar). Top panel, left: jersey knit with *ex situ* annealing. Top panel, right: jersey knit with *in situ* annealing. Bottom panel, left: interlock knit with *ex situ* annealing. Bottom panel, right: interlock knit with *in situ* annealing. Reproduced with permission from [78]. Copyright 2017 American Chemical Society. (c) Demonstration of the stretchability and wearability of inkjet-printed e-textiles. (i) Electrical leads with and without LED. (ii) Electrical current powers the LED. (iii) Conductivity is maintained at >30% strain. (iv) Integration of a leads on a textile glove. (v) Conductivity is maintained upon finger joint flexion. (vi) Flexibility and drapability. (vii) Characteristic porosity. Reproduced with permission from [79]. Copyright 2019 Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim.

further work, the authors studied the effect of the number of print passes, annealing, and fiber structure on the conductivity of inkjet-printed Ag leads on PET knit fabrics [84]. Commercial single jersey and interlock knit fabrics were used as substrates (figure 5(b(ii))). The jersey knit is more tightly constructed with more filaments per yarn than the interlock knit fabric, resulting in lower resistivities for the same number of print passes. Increasing the number of passes decreased the sheet resistance on both textiles. Fabrics with printed patterns and an integrated light-emitting diode were subjected to more than 500 consecutive strain cycles (with a 2.1% increase compared to initial resistance and a 17% increase after 1000 cycles for the best jersey knit) and exhibited good washability and breathability for applications

in wearable technology. Finally, the authors successfully fabricated a demonstrator consisting of a glove with a conductive pattern integrated at the finger area. Its stretchability and wearability were shown by attaching a LED that remained lighted after finger joint flexion (figure 5(c)).

5. Outlook and conclusion

Inkjet printing of metal inks on porous substrates has been successfully used to create soft devices. The performance of the printed devices (where electrical conductivity was commonly reported, but mechanical stability only in a few cases) depended on the porosity and surface chemistry of the substrates, the type of inks and their solvents, and the printing, drying and

sintering processes. The complexity and the large number of possible combinations implies that functional printing on soft, porous substrates requires rational pairing of materials and process parameters, even more so than in graphical printing. It is not yet clear whether a 'generic' conductive ink can be formulated that works with a broad range of porous substrates.

Porous substrates can be used to control the spreading and absorption of ink and to overcome the coffee ring effect. The effects of porosity are well-documented, although detailed models of the kinetics are still lacking. Some reports indicate that the mechanical durability of inkjet-printed electrodes is improved by porous substrates, but comparative studies are scarce. Important challenges remain in the systematic design of porosity in paper, textiles, and polymers. Today, there is little knowledge on which pore size distribution and porosity is suitable for which conductive ink. Optimal pairings of inks and substrates will require a deeper understanding of the interactions between metal inks and substrates, a formidable task for multi-component materials such as paper.

Future research should identify measurable characteristics of substrate and ink that indicate printability and predict later performance. Mechanistic understanding is required to delineate for which combinations such parameters will provide reliable predictions. Sinter-free and low-cost inks based on copper and other metals will probably require additional process steps, and it will be interesting to see whether porous substrates can protect them from oxidation and aging. The potential in the field of porous substrates based flexible electronics will provide opportunities for future research in materials and methods in materials science and several relevant engineering disciplines.

Data availability statement

No new data were created or analyzed in this study.

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