

Towards inkjet printable conducting polymer artificial muscles



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ABSTRACT

Inkjet printing is a key technology in the field of defined polymer deposition as well as in fabrication of strain sensors. It is also one of the most promising alternatives to prevalent fabrication of conducting polymer actuators. Nevertheless, inkjet printed actuators were not yet realised due to rheological properties of conducting polymer solutions that challenge jetting and the complex solution–membrane interactions, that lead to poor adhesion or uncontrolled infiltration. In order to enable this fabrication method, hybrid hydrophilic–hydrophobic polyvinylidene fluoride (PVDF) ultrafiltration membranes were used. They ensure good adhesion with printed poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) layer. Furthermore, PEDOT:PSS compositions that have liquid properties suitable for printing but do not cause uncontrolled infiltration through the membrane were identified. This allowed the realisation of the first inkjet printed PEDOT:PSS based ionic actuators working in air.

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1. Introduction

Ionic electroactive polymer based artificial muscles are promising alternative to prevalent actuators, especially where compliant muscle-like response is desirable [1–3]. Among them, conducting polymer actuators (CPAs) are most promising for biomedical applications, where biocompatibility, compactness and accurate positioning is essential [4,5]. The primary factor leading to the actuation in CPAs is a volume change of the polymer caused by the ion ingress and egress into and out of the polymer during its redox reactions [3,6,7]. Despite their promising advantages, applications of CPAs are hold down by their low efficiency and fast performance deterioration [8–11]. The absence of a tactile, force or position feed-back is another feature limiting the development of functional devices. Therefore a fabrication technique that is up-scalable and enables facile integration of sensory feedback is needed.

Inkjet printing (Fig. 1a) is a key technology in the field of defined polymer deposition and has many promising applications (reviewed by [12]). It is also a low cost, high speed and high precision technique and it could be used in linear arrays for high

throughput fabrication. Furthermore, it allows the use of several functional inks in parallel that gives way to material and shape patterning and could be employed in one step fabrication of the actuator and a strain sensor. Inkjet printed strain sensors were already reported [13–18].

Nevertheless, inkjet printed actuators were not yet realised, mainly because of two challenges: (1) rheological properties of conducting polymer solutions that challenge jetting [19–21] and (2) complex solution–membrane interactions, that lead to poor adhesion or uncontrolled infiltration [20,22–24].

First of all, physical properties of the ink, especially its viscosity, density and surface tension are crucial for inkjet printing [19,20]. In general, viscosity determines the velocity and the amount of the fluid ejected and it should be below 20 mPas. On the other hand, surface tension determines the shape of the droplet and in order to obtain spheroids it should range from 30 mN/m to 350 mN/m [19]. In addition, inkjet printing of conductive polymers is challenging due to their small solubility and miscibility. PEDOT:PSS is the only commercially available conducting polymer that is extensively used in flexible and printed electronics [25–27]. Nevertheless, various additives are often needed in order to adjust rheological properties of the ink as well as the electrical and mechanical properties of the printed films.

Solvent casted or spin coated PEDOT:PSS films are known to poorly adhere to many other materials [28–30]. When PEDOT:PSS

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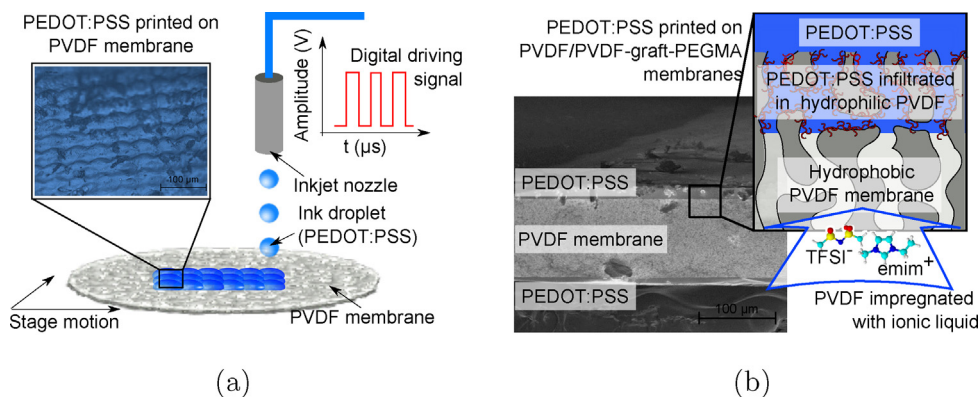


Fig. 1. (a) Schematic illustration of a piezoelectric drop-on-demand (DoD) inkjet printing: a voltage wave is used to deform piezoelectric material and generate a pressure pulse in the fluid, which subsequently ejects a droplet of ink (PEDOT:PSS) from the nozzle; a pattern is generated by moving the stage with the substrate (PVDF membrane) in two dimensions. *Inset:* Picture taken with optical microscope after 20 layers of PEDOT:PSS were printed. (b) SEM image of the cross-section of the inkjet printed actuator and schematic illustration of its structure.

is deposited on hydrophobic PVDF membranes, nearly immediate separation of layers was reported [22,31]. On the other hand, PVDF membranes provide excellent mechanical properties, chemical and ageing resistance, large available free volume for ion storing and good ionic conductivity, therefore, they are more advantageous than other suggested alternatives [32–34]. In order to ensure good adhesion between inkjet printed PEDOT:PSS and PVDF membranes, we use hybrid PVDF membranes that were previously developed in our group by grafting polyethylene glycol methacrylate (PEGMA) on the upper surfaces of the membrane [22]. Hydrophilic nature and mobility of PEGMA allow proximate interaction with PEDOT:PSS and cause capillary spreading of its solution. That subsequently leads to infiltration of PEDOT:PSS aqueous solution up to the thickness of the hydrophilic layer, further strengthening the interface.

In addition to the established requirements for the ink that allow good jetting and a need of additives in order to ensure good electrical conductivity and mechanical properties of a printed film, composition of the ink is also restricted by membrane–ink interactions. It was previously shown that liquids having surface tension lower than 35 mN/m are infiltrated to the hydrophobic PVDF membranes [22,35]. Even when gravitation forces are negligible (as in case of a single droplet ($\ll 1$ nL) on the membrane), if surfactants are being used PEDOT:PSS ink penetrates through the membrane, leading to percolation between the electrodes. That might have adverse influence on the performance of PEDOT:PSS/PVDF/PEDOT:PSS actuators [22,36].

Understanding of the interactions between the polymer film and the porous membrane that is addressed in this article allowed us to set up limits to membrane preparation and polymer solution composition. Adhesion between layers was improved and the infiltration was limited to about 20 μm . This led to the realisation of the first inkjet printed PEDOT:PSS based ionic actuators.

2. Experimental section

2.1. Membrane preparation

Commercially available PVDF membrane filters (VVHP04700 Merck Milipore Corp.) with pore diameter of 0.1 μm , thickness of 125 μm and 47 mm in diameter were functionalised in order to obtain hydrophilic upper surfaces while retaining hydrophobic centre as previously reported by our group [22]. For that 2 ml of 10 vol% PEGMA with average M_n – 500 (409529 ALDRICH) solution in 10 vol% ethanol and water mixture was sprayed on the pristine PVDF membranes. Spraying with Mecafer Ag4 airbrush

(0.4 μm needle) and following parameters: valve opening – quarter turn, pressure – 2 kgf/cm^3 , distance between the target and the airbrush – 30 cm, were used for deposition of the precursor only on the surface. The membrane was dried at room temperature for few minutes and PEGMA was sprayed in the same conditions on the other side. Spraying with these parameters leads to deposition of about 1 ng/mm^2 of PEGMA (assuming no infiltration and a flat membrane surface).

Membranes coated with a dried PEGMA monomer layer were then treated by low-pressure plasma with argon flow rate of 5 ml/min, input power of 10 W and 1 W bias (measured power of 5 W and 0.2 W bias), controlled by a 13.56 MHz RF generator (Aviza Technology Inc. OMEGA201). The low-pressure plasma treatment was operated at 50 mTorr after 1 min given for gas stabilisation. Membranes were turned over and procedure repeated on the other side. After plasma treatment, the PEGylated PVDF membrane was kept in air for at least 72 h to neutralise radicals on PVDF backbone. The non-reacted PEGMA monomer was removed by washing membranes in ultrasonic bath in water, ethanol and again in water for 1 h each. The remaining solvent was removed by drying membranes in the oven at 70 $^\circ\text{C}$ for 2 h. About 0.2 ng/mm^2 of PEGMA were grafted (assuming flat membrane surface).

Atomic force microscopy (AFM) of membranes and PEDOT:PSS printed on them was performed with Veeco-Dimension Icon AFM (Veeco) in a tapping mode using RTESP-300 AFM probes (resonance frequency of about 300 kHz) and the averaged roughness was calculated using the NanoScope Analysis software (Veeco). SEM images of membranes and actuators were obtained using a Hitachi S-4800 field emission scanning electron microscope (acceleration voltage 800 V, working distance of about 5 mm).

2.2. Ink preparation and characterisation

PEDOT:PSS 1.3 wt% conductivity grade dispersion in water (483095 ALDRICH), having PEDOT and PSS ratio of 1:2.5 was bought from Sigma Aldrich. Glycerol (G9012 SIGMA-ALDRICH), ethylene glycol (324558 SIGMA-ALDRICH) as well as poly(ethylene glycol) of M_n – 400 (PEG400) (202398 ALDRICH) or Triton (X100 SIGMA-ALDRICH) were used as additives to PEDOT:PSS based inks. Inks of various compositions were prepared by mixing PEDOT:PSS with secondary dopants on mechanical shaker at 300 rpm for at least 3 h. Before printing solutions were filtered with 5 μm polytetrafluoroethylene (PTFE) syringe filter. Between printings, prepared inks were stored in the fridge at 4 $^\circ\text{C}$.

Surface tension of the ink was measured by pendant drop test using Digidrop GBX Contact Angle Meter. Viscosity was measured

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