Preliminary Analysis on Cellulose-based Gas Sensor by Means of Aerosol Jet Printing and Photonic Sintering

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Abstract: In this paper, we present a preliminary analysis on the possibility to realize low-cost and eco-friendly cellulose-based gas sensors by means of Aerosol Jet Printing (AJP) and flash lamp annealing (FLA). To the authors knowledge, it is the first time that these two techniques are combined in the realization of such a device. The intrinsic hygroscopic properties are the key element of this device: cellulose contains substantial amount of moisture, adsorbed from the environment, enabling the use of wet chemical methods for sensing without manually adding water to the substrate. The sensors were tested in terms of electrical resistance. The penetration of the carbon ink in the cellulose network was stated thanks to cross-sections captured at the microscope. Once placed in a damp environment, all the sensors showed a comparable behavior settling at an asymptotic value of 3.68 MΩ (relative standard deviation of 8%). In presence of different concentration of NH4OH, the sensors showed a resistance proportional to the amount of analyte present in the working volume, showing 25.6% increase compared to the 0.5 M concentration, while 34.1% compared to the 1M.

1 INTRODUCTION

Printed electronics represents an attractive and innovative field of application of Additive Manufacturing (AM) with flexible electronic devices, paper electronics and wearable devices that are becoming widespread coming with the need for advanced printing processes, reducing the number of processing steps, material waste and production cost (Gandhiraman *et al.*, 2014).

3D printing is considered a valid candidate for the new generation of biosensors (Ragones *et al.*, 2015; Yang *et al.*, 2016), and Aerosol Jet Printing (AJP) is belonging to this technological family. AJP is a direct-write printing technique which deposits a continuous aerosol beam of liquid droplets, generating specific surface features without masks(Hoey *et al.*, 2012; Lee, An and Chua, 2017). The liquid inks typically have a viscosity between 1 and 1000 cP, in case of pneumatic atomization, and 1 to 5 cP, in case of ultrasonic atomization (Tan, Tran and Chua, 2016). Once the mist is generated, it passes through the virtual impactor to regulate droplets dimensions and then it is focused on the substrate thanks to a coaxial sheath gas flow(Binder, Glatthaar and Rädlein, 2014; Wadhwa, 2015; Optomec, 2017).

This 3D printing technique is adopted in different fields: high-efficiency solar and fuel cells, thin-film transistors, resistors, antennae, MEMS, photodetectors, thermistors, etc. But it is also used in applications for the biological field like high-density assays for drug discovery, lab-on-chip devices, protein and glucose sensing (OPTOMEC, no date; Coupland *et al.*, 2010; Yang *et al.*, 2016; Wang *et al.*, 2017; Bolse *et al.*, 2017; Liu *et al.*, 2017; Agarwala *et al.*, 2018; Baldwin *et al.*, 2018; Cantù *et al.*, 2018; Gupta *et al.*, 2018; Khorramdel, Torkkeli and Mäntysalo, 2018; Di Novo *et al.*, 2019).

In the printed electronics field, the sintering step is mandatory to ensure a proper conductivity in the printed layer, which is usually performed by means of hoven for high temperature processes. One of the emerging topics in printed electronics is the usage of paper due to its unique capabilities (high Young's modulus, biodegradability, biocompatibility, renewability, low-cost, lightweight) (Kim *et al.*, 2014). This material needs great attention in sintering printed inks due to low melting/ignition point. Therefore, only low-temperature processes are allowed, which do not ensure high conductivities. Photonic sintering, also known as flash lamp annealing (FLA) or intense pulsed light (IPL), is a

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technology, developed by Novacentrix, in which the heating process is accomplished thanks to a couple of xenon flash lamps and mirrors which radiate energy toward the sample that must be sintered, following phenomenon of melting/sintering point depression in nanoparticles described by the Gibbs-Thomson equation.

The advantages are remarkable: this is a fast sintering technique (heat treatment in the milliseconds scale), it produces minimal damage to low temperature substrates and avoids unwanted processes (diffusion). Possible applications range from thin-film transistors, solar cells, RFID components to wearable devices, using conductive, bioresorbable or ceramic inks (silicon, silver, zinc) on different kind of substrates (wood, cardboard, textiles, glass, kapton (West *et al.*, no date; Akbari *et al.*, 2017; Mahajan *et al.*, 2017; Mudgal *et al.*, 2017; Rizwan *et al.*, 2017; Bobinger *et al.*, 2018; Cronin *et al.*, 2018; Danaei *et al.*, 2018; Li *et al.*, 2018; Schube *et al.*, 2018).

In light of this, the present work aims to propose the combined use of AJP and FLA, to realize a cellulose gas sensor, designed for food industry. Food waste is one of the hardest challenges for humanity as it impacts on society not only in food terms (direct food loss and waste), but also in environmental terms (exploitation of water and land, disposal, emissions) (Springmann *et al.*, 2018) For example, the use by-date is an estimation of the date on which a perishable product may no longer be edible, and this number cannot clearly state the condition of the food (Labuza and Taoukis, 1990).

Ammonia and its compounds can be considered toxic, capable of generating damages and unwanted pathologies to human body if ingested (nausea, migraine, vomiting, allergies) (Douny *et al.*, 2019; Filipe-Ribeiro *et al.*, 2019). The research is working on wireless and automated systems capable to continuously give a feedback of the sample (Bellitti *et al.*, 2017; Tonello *et al.*, 2017). The possibility to include a sensor inside the packaging appears to be a solution which can monitor the current state of the food defining its freshness (Yam, 2012; Liu *et al.*, 2015).

Paper-based sensor with carbon electrodes exploits the intrinsic hygroscopic characteristics of cellulose paper. Cellulose fibers contain a certain amount of moisture, even if paper looks dry. This aspect is mandatory in evaluating the presence of water-soluble gases. In presence of a water-soluble gas (due to food degradation), electrical impedance or conductance of paper can be probed thanks to carbon electrodes realized on the surface of the paper sheet. The feasibility of these sensors has been recently reported in literature using a low-cost cutter plotter for preparation (Barandun *et al.*, 2019). Our goal is to develop this class of devices by combining the AJP and FLA methods, investigating the improvement we can achieve thanks to the more sophisticated preparation technique. Chapter two will present methods followed during tests, while chapter three shows the results obtained during validation and characterization tests. Chapter four will summarize the preliminary results achieved, with future perspective of this application.

2 SENSOR FABRICATION

2.1 Sensor Design

Optomec's Aerosol Jet Printing AJ300 system was employed in the fabrication of the proposed gas sensor: this 3D printing machine is designed to interface with AutoCAD drawings. Specific attention was put in the definition of printing parameters, considering inks viscosity, in order to achieve a homogeneous filling of the interdigitated geometry, also thanks to the use of a crossed pattern. The sensors were designed thanks to AutoCAD software (Autodesk), a devoted Optomec's gadget for AutoCAD ensured a proper ink filling of closed shapes. The lines were designed as polylines to improve the timing and the homogeneity of printed lines.

Surface area of the interdigitated electrodes and spacing in between, are the two main factors that must be considered during the design phase, thus determining the overall impedance of the sensors, which have a critical effect on its sensitivity. More specifically, a total sensing area of 150 mm^2 and a spacing of about 1.5 mm were selected (fig. 1).

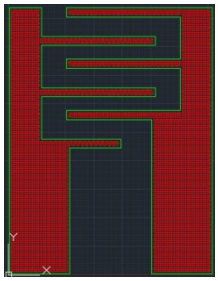

Figure 1: AutoCAD layout of the sensor.

2.2 Materials and Methods

Whatman™ chromatography 1 cellulose paper is the selected substrate on which we printed our sensors, due to the capability of this kind of paper to capillary handling liquid samples. The second reason we employed this material is the high degree of purity respect to other commercial grades which include chemical additives that could influence the functioning principle of the proposed sensors. Thanks to its highly hygroscopic properties, cellulose fibers within paper contain a substantial amount of moisture, even if it looks dry (at a relative humidity of 50%, paper contains ~5% water by weight). Additional ions coming from the dissociation of water-soluble gases on the surface of paper increases its ionic conductance.

Carbon ink (EXP 2652-28) was purchased by Creative Materials Inc., characterized by a starting viscosity of 15-20 mPa·s. The ink was properly atomized together with its own thinner to obtain a better printability and adhesion to the substrate. Carbon ink specific process parameters are reported in Table 1, while figure 2 presents a prototype and our AJ300 during printing.

Table 1: Printing process parameters for C ink.

Process Parameters	
Sheath gas flow (SCCM)	110
Atomizer flow (SCCM)	770
Exhaust flow (SCCM)	750
Process speed (mms-1)	4
Plate temperature (°C)	70

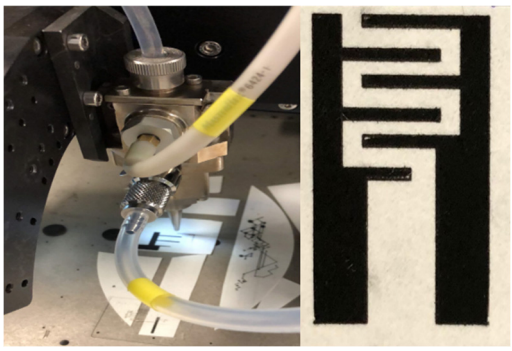

Figure 2: AJ printer printing a first concept (left); a printed sensor in its final layout (right).

With the aim to define the sintering parameters for our carbon sensors, a sample geometry was considered in order to define FLA sintering parameters. These samples were tested in terms of electrical resistance, evaluated thorough the digital bench-top multimeter Hewlett-Packard 34401a, applying testing probes to the extremities of each path, in standardized and repeatable points, thus measuring the resistance offered by all its length.

An optical microscope by Orma Scientific NB50T (trinocular zoom 0.8x–5x–LED), with its devoted software and HDMI MDH5 camera model, was used to acquire the images and to evaluate the features of the printed elements (Orma Scientific, Sesto San Giovanni, Milan, Italy).

After the definition of the abovementioned parameters, four sensors were put in four different vials to examine their capability to absorb ions present in a humidified atmosphere and their response was simultaneously studied in terms of resistance. 5 ml was the selected working volume and distilled water was employed for the stabilization step (used to reach a relative humidity of 100 % inside the vial to let sensors work properly in presence of water-soluble gases), aiming to state the repeatability of this kind of measurement principle with these AJ printed sensors. After this step, a qualitative study was performed by means of distilled water/NH_4OH solutions in two different concentrations, 1 M and 0.5 M, which will develop a gas-phase of ammonia in a humidity saturated air background. Additional tests have also been carried out with ethanol in an ambient air background to test the working mechanism of this class of devices. Four digital bench-top multimeter Hewlett-Packard 34401a were connected thanks to a GPIB-USB cable to a personal computer and the behaviour of the sensors monitored thanks to a program written in LabVIEW.

3 PRELIMINARY RESULTS

Once printed, carbon samples must be sintered in order to ensure the realization of a unique conductive track. Impulse duration was studied keeping it fixed, initially at 1000 µs, increasing the lamp voltage from 200 up to 300 V. The same approach was replicated for other three pulse time-lengths, specifically 1250, 1500 and 1750 µs. At the end of this step the best sintering parameters which gave the lowest resistance were selected. As evidenced in figure 3, 1000 µs seems to be a too short impulse duration, being not able to completely penetrate the whole thickness of our chromatographic paper (paper thickness is about 180 µm). 1250 and 1500 µs showed a similar behavior, with the second one presenting a lower resistance.1750 µs and 250 V are the best sintering parameters, as they allow to reach a final resistance of 2.8 kΩ (relative standard deviation of 7%). So, the

complete pool of production parameters for our sensors are the ones reported in table 1, with six consecutive depositions, sintered at 250 V, 1750 µs.

Figure 4 displays a magnification of an interdigitated printed element, suggesting the need of a proper wavelength to fully penetrate the cellulose sheet to sinter carbon ink. The effectiveness of the deposition process was initially tested evaluating the resistance of individual dry electrodes resistance, after both printing and sintering steps. It was found a mean value of 8.45 kΩ (relative standard deviation of 23%) after printing, while 7.44 kΩ (relative standard deviation of 21%) after sintering. It is interesting to compare these values with those reported in literature for a preparation method based on an ultra-low cost cutter plotter printer. In this case the average resistance is of an individual electrode is 6.40 kΩ with a relative standard deviation of 43% (Barandun *et al.*, 2019). The presented results demonstrate a difference in standard deviation of 22% so the combine use of AJP and FLA have improved the sensors realization procedure.

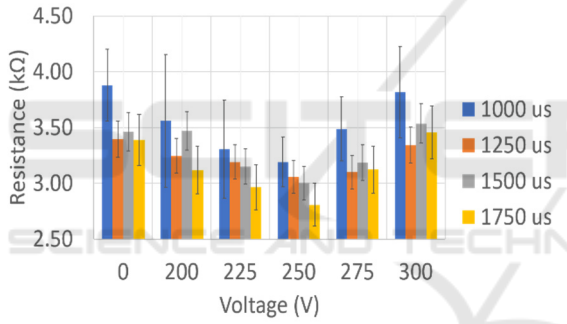

Figure 3: Resistance measurements as funciton of voltage at different impulse duration, for C samples.

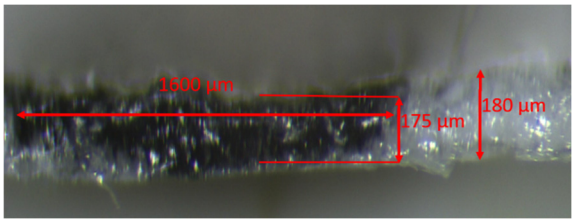

Figure 4: Magnification of an interdigitated element (bottom).

To validate and demonstrate the functioning of our paper-based sensors, they were tested in a damp environment. Four sensors were simultaneously put in four different vials in presence of a working volume of 5 ml of distilled water. Figure 5 shows the behaviour of the sensors reaching the stabilization at an asymptotic value of 3.68 MΩ (relative standard deviation of 8%). The condition step was 30 hours long in order to clearly evidence the stabilization of the sensors reaching a relative humidity inside the vial of 100 %. Since sensors resistance appeared to be stable before reaching the thirtieth hour, in future works the precondition duration will be performed taking into account only the strictly necessary time.

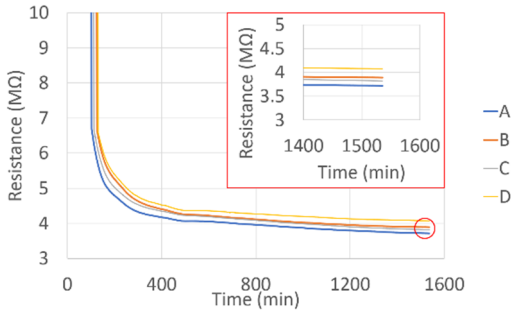

Figure 5: Resistance values during stabilization of four sensors in distilled water.

After this, a simple qualitative study was performed aiming to state the capability of the sensor to recognize different concentrations of the same analyte in a humidity saturated background, which optimize the sensing mechanism of this kind of devices (Barandun *et al.*, 2019). Once run a precondition phase in presence of 5 ml of water, one sensor was exposed to the vapours generated by a 1M solution of NH_4OH in water, one sensor with a 0.5M solution of NH_4OH and one with a new vial with distilled water (blank sample). To further test the effect of the working environment, an additional experiment has been carried with a fourth sensor exposed to vapors generated from an ethanol solution. This allows to test the effects of extreme working conditions (saturated ethanol, vapors).

Figure 6 evidences these experiments.

The data regarding NH_4OH vials are reported as $\Delta G/G_0$ in percentage in table 2. ΔG values are calculated as the difference between the maximum and minimum resistance values when the analyte is recognized after vial change, while G_0 is the minimum resistance value in the same time interval. The sensors show a 25.6% increase compared to the 0.5 M concentration, while 34.1% compared to the 1M. Thus, with an 8.5% difference between the highest and the lowest concentration.

The exposure to saturated ethanol vapours in the ambient air background cause a sudden and large increase of the sensor resistance. This extreme behaviour is likely to arise from the large ethanol concentration (saturated vapours), which modifies the condition of the cellulose fibres making them

anhydrous, hence much less suitable for ionic conduction. The result further supports the working mechanism proposed in literature, based on the intrinsic ionic conductivity of the water layer covering cellulose in ambient conditions. To better clarify this point, we have planned additional tests with lower ethanol concentrations.

Table 2: ΔG comparison between 0.5M and 1M solutions.

Concentration (M)	$\Delta G/G_0$ (%)
0.5	25.6
1	34.1

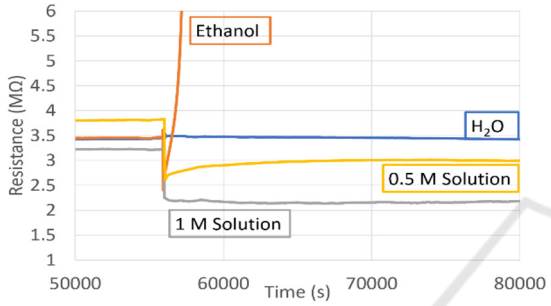

Figure 6: Behaviour of the sensors in presence of different analytes (water, ethanol, NH_4OH 1 M and 0.5 M).

4 CONCLUSIONS

We present a new kind of disposable gas sensor, produced thanks to Aerosol Jet Printing and Flash Lamp Annealing on a cellulose-based material. The production method presents good reproducibility despite a porous substrate such as chromatographic paper. In this first research step, we want to verify the functioning of the sensors, considering a first design which is not optimized for our future perspectives. The following steps will regard the complete optimization of the production procedure, thus redefining the optimal sintering parameters for the new the design of the sensors. Once tested in presence of a damp environment, our device behaved in the same way, showing an asymptotic value of 3.68 MΩ, regardless of the specific starting conducting value. The sensors were also qualitative tested, showing a signal proportional to the amount of NH_4OH present in the sample volume. Future developments will regard the investigation of the limit of detection of these cellulose-based sensors and their selectivity. In particular, tests are planned to check the effects of ethanol vapours, which are likely to alter the hydrous status of the cellulose fibres, especially at high concentrations. More in general, considering the prospective to tune the sensing properties of this class of devices to a given gas-target, this possibility should be strictly checked against its working principle, whose simplicity represents at the same time the strength and the weakness of these devices. Indeed, if gases containing ammine groups can be easily sensed by this simple and extremely cost-effective device, any alteration of the hydrous status if the paper may deeply alter its sensing capability. Any functionalization of the paper aimed to tune its sensitivity to a given gas-compound, should be carried out in careful consideration of this aspect.

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