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Silver Printed Electrode on Poly-Imide (Pi) Film And Polyethylene Naphthalate (PEN) Film For ECG Monitoring Device: A Case Study

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Abstract. According to World Health Organization (WHO), cardiovascular disease (CVD) is among the tenth of primary global cause of death in 2017. However, this problem could be alleviated via early detections, especially for patients with a history of heart-related problems. In addition, ECG monitoring could reduce the cause of CVD if diagnose earlier thus reduce the risk of sudden death. The measurement of ECG signals is important to assist physicians about people heart and health conditions. The sophisticated characteristics of ECG signals could be acquired by using the good electrodes. Thus, electrodes play a vital role in ECG recording. The commercially available electrodes are the conventional wet electrode (gel) using silver/silver chloride (Ag/AgCl). The gel usage tends to dry gradually and could cause skin problems like dermal inflammation and skin irritation. Due to the limitations associated with conventional gelled electrode, dry electrode is expected to overcome the main limit of Ag/AgCl electrode. By fabricating the silver ink onto the polyimide (PI) and polyethylene naphthalate (PEN) substrates to produce a dry ECG sensor, which could withstand high temperature, reusable, and has been shown to be biocompatible. This study is a case study to measure the performance of dry electrode based by silver printed electrode on PI and PEN films in ECG application.

1. Introduction

Body sensor networks will be a key driving force for the wireless health revolution by allowing patients access to their physiological state at any time in their daily life. Brain and cardiac biopotential signals that are electroencephalography (EEG) and electrocardiography (ECG) signals are two critical health indicators that are directly suited for long-term monitoring using body sensor networks. Yet despite advancements in wireless technology and electronics miniaturization, the use of ECG has still been largely limited by the inconvenience and discomfort of conventional wet contact electrodes.

There are many challenges associated with ECG measurements, and past approaches have included optical and microwave doppler sensing, optical interferometry and the de-facto standard of sensing the ECG currents with electrode positioned on the skin together with an electrolytic gel (namely, the conventional wet electrode; Ag/AgCl electrode). As the sensor positioning and site preparation is critical to achieving the ECG measurement, this is performed by a medical professional. However, the ECG sensor could be based either upon an Ag/AgCl electrolytic sensor cell with wireless transmission or dry electrode; polyethylene terephthalate (PET), polyurethane (PU) nanoweb, textile (e.g. woven cotton, nylon and polyester), polydimethylsiloxane (PDMS), foam and rubber. The preferred approach which shall be studied is a printed capacitive electrode sensor, this being less positional tolerant, consumer friendly and not requiring a medical professional to setup.

There are two types of capacitive electrode which are contact and non-contact electrode to the human skin interface. The non-contact electrode might led to strong signals' artifact since the changes capacity between the electrode and the skin are difficult to control [1]. Recently, dry-contact electrodes are widely discovered since the direct coupling electrode-skin interface could be improved by the existence of sweat or moisture [2, 3]. Inkjet printing method is one of the common methods used for fabricating conductive ink on the flexible substrate. This technique allow personalization of electrode sizes and shapes, rapid prototyping and additive manufacturing approach with low cost and little waste of fabricated materials [4]. Momota and Morshed applied silver nanoparticle ink on polyimide substrate using inkjet printing technique to fabricate flexible electronic dry ECG electrode by comparing two different shape which are circular and pentagonal [5]. Other than that, Batchelor and Casson compared the performance of two type of inkjet electrodes printed on tattoo paper using silver ink and copper printed capacitive electrodes on plastic substrate, and compared the performance with traditional Ag/AgCl electrode [4].

In this project, the technology of printed electronics to develop a dry contact electrode is further explored. The material used is silver ink which is printed onto two types of flexible platform substrates; Poly-Imide (PI) and Polyethylene Naphthalate (PEN) films. PI reveals exceptional combination of thermal stability (>500°C), mechanical toughness, chemical resistance, excellent dielectric properties and inherently low coefficient of thermal expansion [6]. PEN is good and less expensive alternative to PI and usually surpasses Polyethylene Terephthalate (PET) in top end demanding applications. PEN has lower oxygen permeability, improved hydrolytic stability, higher tensile strength and service temperature, as well as reduced elongation and shrinkage due to its higher glass transition temperature (120°C vs 75°C) when compared to the performance of PET [7].

2. Design and Working Principle

The fundamental method of this project is printing method to transfer conductive nanomaterials, which is Silver, onto the flexible platform; Poly-Imide (PI) and Polyethylene Naphthalate (PEN). The printed conductive nanomaterials transformed the flexible platform into a capacitive electrode sensor for ECG monitoring device, namely ECG electrode.

The pattern for the capacitive electrode is designed by using autoCAD. The shape is a circle with diameter of 3.4 cm, containing hexagons with diameter of 2 mm and distance between hexagon is 1 mm. Then, the images are saved into file of .pdf format to fit into the requirement of material printer for printing technique. Figure 1 shows the capacitive electrode design. The material printer for printing the silver onto flexible substrates are inkjet deposition and analysis printer. The ink droplet is set to be twice (2x) and thrice (3x) for both flexible substrates (PI and PEN). The details of the printing parameter is included in Table 1. The process flow is shown in Figure 2. After the printing process, the samples are cured at 135 °C for 10 minutes. The results are shown in Figure 3.

This study was performed in accordance with the Declaration of Helsinki. Collection of human tissue samples for this study was approved as part of the study protocol. This human study was approved by IJN Research Ethics Committee. All adult participants provided written informed consent to participate in this study. All adult participants provided written informed consent for publishing identifying details in this study.

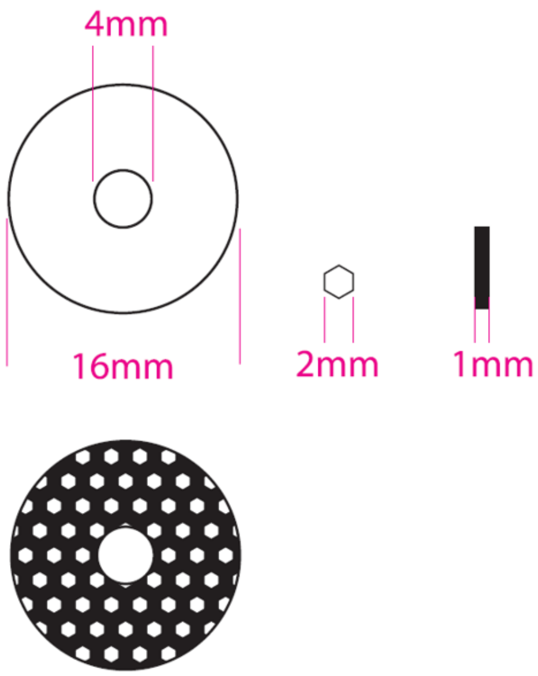


Figure 1. Design for capacitive ECG electrode by using autoCAD

Table 1. Printing parameter

Printing Parameter	Features
Printhead	DMC-11610 Fujifilm
Ink dot size	10PL
Print resolution	500 DPI
Print pass number	2 to 3
Ink	Silver
Print height	100 µm

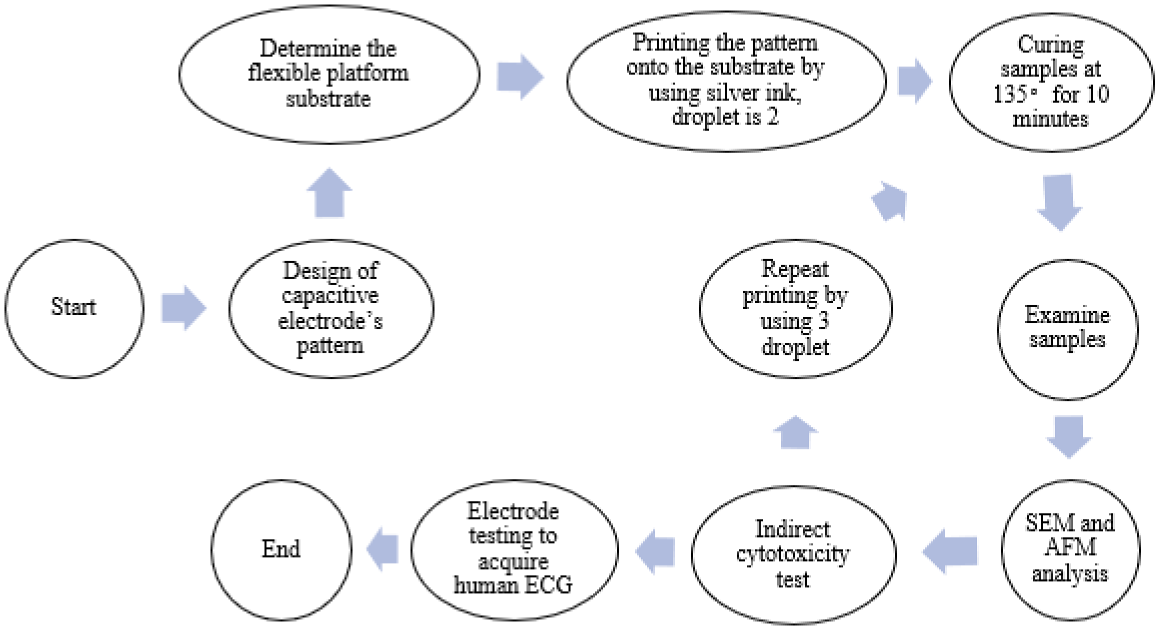


Figure 2. Process flow

(a) Electrode on PEN substrate (b) Electrode on PI substrate

Figure 3. Printed capacitive electrode on PEN and PI substrates

The film was subjected to scanning electron microscopy, SEM (Crossbeam 340, Zeiss, Germany) and atomic force microscope, AFM (Axio Observer D1 Nanowizard3, Zeiss, Germany) analyses. The surface morphology of the sample was observed at 30,000× magnifications with an accelerating voltage of 5 kV. Prior, an ultrathin gold film was deposited on the film using an automated platinum sputter coater (Q150RS, Quorum Technologies Ltd., England) which acted as a conductive layer for better resolution image. While the surface topography was performed using a silicon cantilever with a tip of 10 nm radius and 10–15 µm height. The resonance frequency was set at 370 kHz and the scan rate was set at 0.5 Hz. Post-analysis of root mean square roughness (R_{rms}) was then quantified using JPK data processing software (spm-5.1.8, JPK Instruments AG, Germany). Then, indirect cytotoxicity test on human skin fibroblast was performed to ensure the printed capacitive electrode is non-toxic and safe for human skin contact.

Prior, human skin fibroblast cells (HSF 1184, ECACC, UK) were cultured in Dulbecco's Modified Eagle's Medium (DMEM) supplemented with 10 % fetal bovine serum (FBS) and 1 % (v/v) penicillin/streptomycin. The cells were incubated for 24 hours at 37 °C in a humidified atmosphere of 95 % air and 5 % CO_2. For sub-culturing purposes, 0.25 % trypsin-EDTA solution was used to detach the cells. Prior to the *in vitro* cell studies, all samples were UV-sterilised for at least 30 minutes on each side. All analyses were performed in triplicate, and the non-treated cells were used as a control. For the indirect approach, the sterilised samples were incubated in fresh DMEM solution for 1, 3 and 5 days to obtain the extraction medium according to the ASTM F813 standard protocol. In a 96-well plate, 2 × 10^5 HSF cells/mL were growth by incubation at 24 hours in a CO_2 incubator. The media were discarded, replaced with 200 μL of the extracted medium and incubated for another 24 hours. Subsequently, 100 μL of 5 mg/mL 3-(4, 5-Dimethylthiazol-2-yl)-2, 5-diphenyltetrazoliumbromide (MTT) solution (Invitrogen, USA) was added, and the suspension was incubated for another 4 hours. The crystallised formazan was dissolved by pipetting 200 μL of dimethylsulfoxide (DMSO) solution into each well. The absorbance of the purple formazan product produced by the viable cells was spectrophotometrically measured with a microplate reader (BioTek808, USA) at 540 nm. The obtained data were then calculated as a percentage of cells viability based on equation (1).

$$\%\ Cell\ viability = \frac{Experimental\ OD_{540}}{Control\ OD_{540}} \times 100 \qquad (1)$$

3. Results and Discussions

In this case study, the results are separated into three categories, which are, scanning electron microscopy (SEM) and atomic force microscopy (AFM) analysis, indirect cytotoxicity test, and electrode testing on capturing the human heart electrical signals. The results are discuss in the following subsections.

3.1. SEM and AFM analysis

SEM images of PI and PEN films were shown in Figure 4. For silver-printed PI and PEN, small particles were observed on the film when compared to control. Silver-printed PEN shows more prominent particles compared to silver-printed PI. This surface morphology indicates that silver particles distribute on the PI and PEN film confirming the formation of silver-printed film.

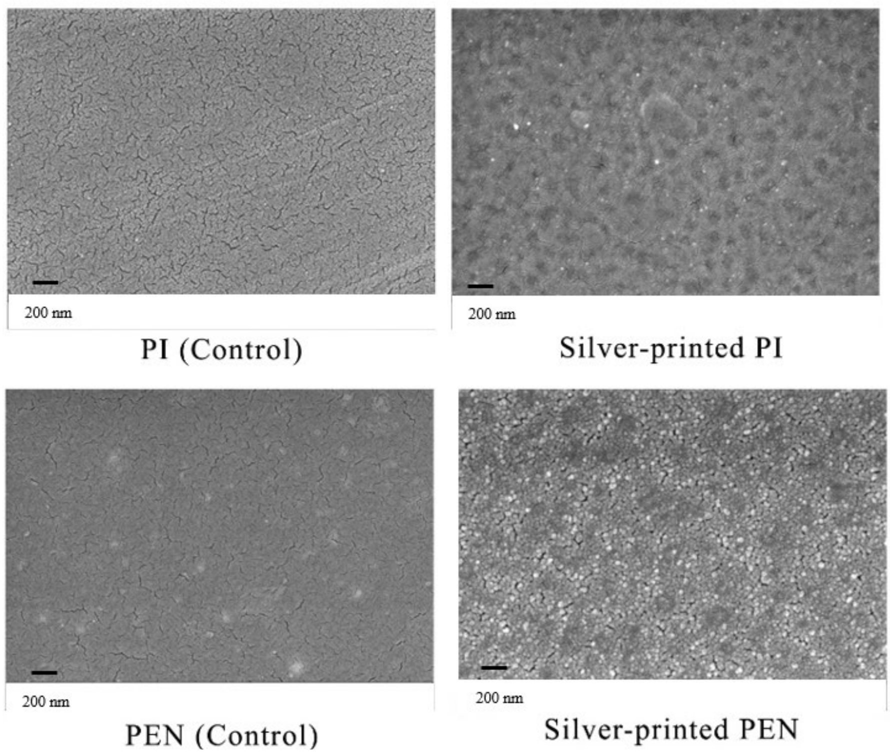


Figure 4. SEM morphology for PI (control), silver-printed PI, PEN (control) and silver-printed PEN under 30k magnification.

Figure 5 shows AFM images for PI and PEN film. After silver-printed, surface roughness of PI film was increased to 312.6 nm and higher than PEN film. However, the conductivity of silver-printed PI is lower than silver-printed PEN. The formation and propagation of microcracks on the film surface might contribute to the loss of electrical conductivity.

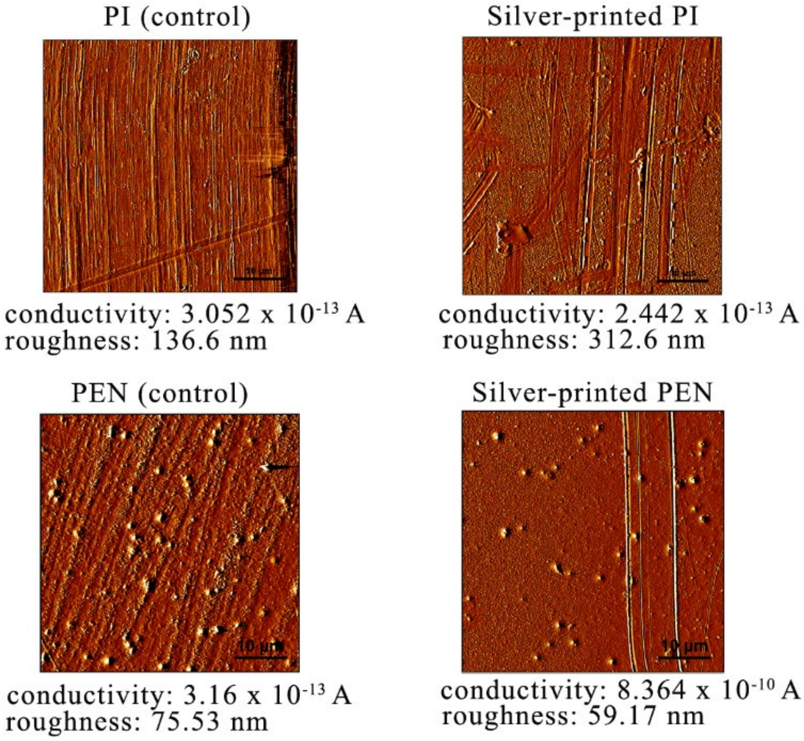


Figure 5. AFM images for PI (control), silver-printed PI, PEN (control) and silver-printed PEN.

3.2. Indirect cytotoxicity test

The indirect measurement is focused on the degradation and extraction of silver ions on every types of different materials, which evaluates cells toxicity by measuring the cells metabolic activity. The MTT assay determines the ability of viable cell's mitochondria to reduce the formazan indicates the decrease in mitochondrial metabolism of the cells [8]. Hence, the percentage of cell viability directly correlates to the number of cells whose mitochondrial metabolism is intact even after the exposure of Ag release from each sample. Figure 6 shows the percentage of viable cells tested on all the samples reached over 70% of viability indicated the safe range of cytocompatibility study of *in vitro* cells test. As day increased, the amounts of silver ions released were increased. This will give effect to the percentage of cells viability. Higher percentage of HSF cells viability were observed even after 5 days of extraction for materials from PEN 2x, PI 2x and PI 3x. As for PEN 3x, the percentage of cell viability slightly declined from day 3 to day 5 but the data were not significant which indicated that the silver printed (2x and 3x) on substrates (PI and PEN) is non-toxic to human cells. Higher concentrations of silver ions can give effects to cell viability as shown by the percentage of cell viability in PI 3x is slightly lower than PI 2x. However, the considerable range of silver toxicity and the exposure time in contact with living tissue should be considered for short term biomedical applications, for example biosensor or patch [9].

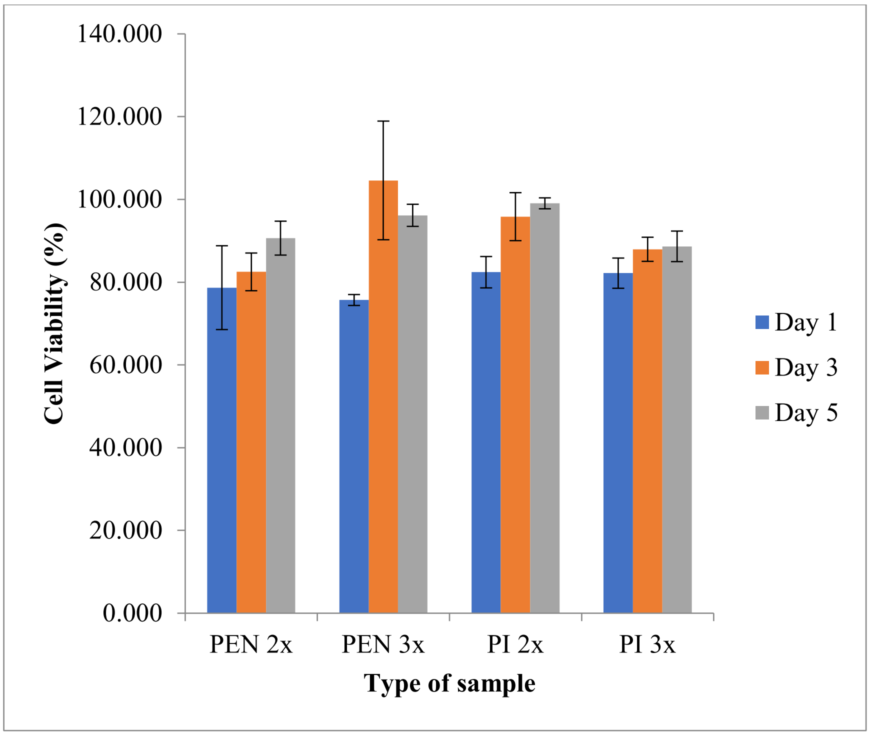
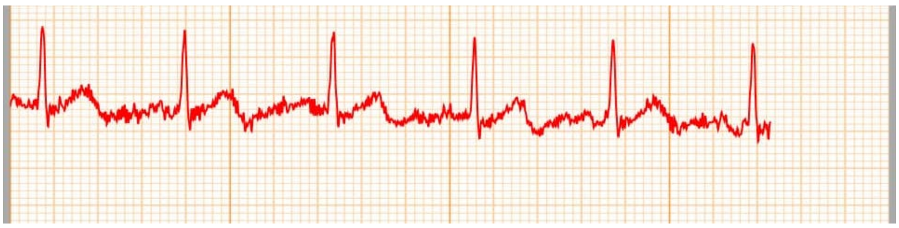


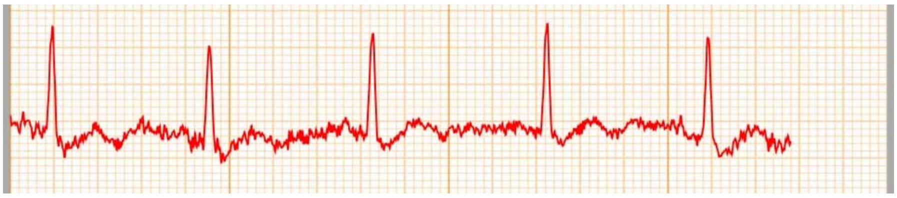
Figure 6. Cytotoxicity test

3.3. *ECG testing*

From section 3.2, it is shown that both inkjet droplet of 2x and 3x drops are non-toxic towards skin cell and suitable to be used with skin contact for up to 5 days. Thus, the printed capacitive electrodes are further testing to capture the human ECG signals. The electrodes are attached to the 2-channel ECG device which was attached to human body. Then, the ECG is recorded. Figure 7 shows the ECG signals acquired from the user by using both printed silver electrodes. To further the investigation could be done by using different surface area for printed capacitive electrode. Thus, the electrical properties can be further explored.



(a) The acquired ECG by using PEN substrate.



(b) The acquired ECG by using PI substrate.

Figure 7. ECG signals acquired from printed capacitive electrode which attached to human skin and connected to ECG device.

4. Conclusion

The fabrication of dry electrode by using inkjet printer and silver has shown a promising outcome. The substrate of PEN and PI, both are non toxic to human skin thus is consider to be safe and suitable for long-term ECG monitoring to up to five days. The conductivity of silver-printed PI is 2.4 A which is lower than silver-printed PEN, 8.3 A. In this study, both electrodes able to acquire the electrical signals or activity of the user in resting position. Further improvement could be done to reduce the noise proesence in the acquiring signals.

Acknowledgments

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