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A NEW GOLD NANOPARTICLES MODIFIED SCREEN PRINTED PAPER CARD BASED MICROFLUIDIC DEVICE

(Nanopartikel Emas Baru Diubahsuai Skrin Dicetak Kad Kertas Berasaskan Peranti Mikrobendalir)

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Abstract

This work offers a novel platform for the straightforward and low-cost manufacture of paper microfluidic devices known as gold nanoparticles modified screen printed paper electrode AuNPs/SPPE. The component can function as an electrochemical cell as well as a microfluidic device. Different paper cards were evaluated for hydrophobicity, and metallic gold card was selected due to its superior hydrophobic properties. Thus, waxing and other pretreatment steps are eliminated. The AuNPs/SPPE was created by screen printing on metallic gold card using commercial silver ink. Working electrode was modified using synthesized gold nanoparticles (AuNPs). The presence and widespread distribution of Au particles are shown by SEM and EDX study. The conductivity and resistivity study shows good conductance. The device was applied in tramadol detection, tramadol was studied electrochemically using cyclic voltammetry technique, where it was oxidized at 0.1 to 0.3 volts. Positive results obtained from electrochemical investigation suggest good and promising future applications.

Keywords: microfluidic, screen printing, paper device

Abstrak

Kerja ini menawarkan platform baru untuk pembuatan peranti mikrofluidik kertas yang mudah dan kos rendah yang dikenali sebagai nanopartikel emas yang diubah suai skrin dicetak kertas elektrod AuNPs/SPPE. Komponen ini boleh berfungsi sebagai sel elektrokimia serta peranti mikrofluidik. Kad kertas yang berbeza dinilai untuk hidrofobisiti, dan kad emas logam dipilih kerana sifat hidrofobik yang unggul. Oleh itu, waxing dan langkah-langkah pretreatment lain dihapuskan. AuNPs/SPPE dicipta melalui percetakan skrin pada kad emas logam menggunakan dakwat perak komersial. Elektrod kerja diubah suai menggunakan nanopartikel emas yang disintesis (AuNPs). Kehadiran dan pengedaran zarah Au yang meluas ditunjukkan oleh kajian SEM dan

EDX. Kajian kekonduksian dan rintangan menunjukkan kelakuan yang baik. Peranti ini digunakan dalam pengesanan tramadol, tramadol dikaji secara elektrokimia menggunakan teknik voltammetrik kitaran, di mana ia teroksida pada 0.1 hingga 0.3 volt. Hasil positif yang diperolehi daripada penyiasatan elektrokimia menunjukkan aplikasi masa depan yang baik dan menjanjikan.

Kata kunci: mikrobendalir, percetakan skrin, peranti kertas

Introduction

Microfluidic is a portable device that requires a small volume of sample and can easily be integrated into a point of care diagnostics equipment, that can be used in remote settings, with poor infrastructure [1-2]. Because of its intriguing benefits and cutting-edge platform that is still being researched, microfluidics has garnered a lot of attention. The development of fabrication techniques, novel substrates, and detection techniques is ongoing. As a result, low-cost materials and low-cost fabrication and detection designs are emerging [1-6] in response to the growing demand for low-cost devices that will greatly simplify lives all over the world. Paper substrates are now included in microfluidic systems [5-10], which is a novel development that researchers are exploring since it is portable, inexpensive, and requires a small amount of sample [7-9]. Hydrophobic and hydrophilic patterns are typically formed on the paper to create microfluidic paper devices. The hydrophilic region is produced by screen printing the ink onto the patterned paper, and the hydrophobic portion is produced by melting photoresist or wax onto the paper [1,5]. Dungchai et al. used photoresist to form the hydrophobic barrier on a filter paper [5]. The wax technique is the most popular. The wax has been applied in a variety of ways, according to reports. A fabrication technique utilizing chromatographic paper was created by Songjaroen et al. [3]. The paper was dipped into melted wax, and the wax penetrating the area not covered by the iron template created the hydrophobic region. While, Zhang and Zha employed filter paper with a paraffin copper sheet attached. When heated, the paraffin melted through the filter paper and formed the hydrophobic barriers [4].

The increasing demand for point of care testing has prompt fabrication of rapid and quantitative microfluidic paper devices [1,8-13,16] for hard drugs detection [11-13]. Rapid and quantitative microfluidic paper devices [1,8-13,16] for the detection of hard drugs [11–13] have been made as a result of the rising need for point-of-care testing. The availability of microfluidic devices can reduce the short- and long-term negative impacts of hard drugs, such as overdose, addiction, fatalities, and consumption, as the victim can be quickly identified in the general population [12-13]. Screen printing was employed due to its miniaturization ability, low cost, versatility, and ease of mass production [11,14]. This technique has been employed in the production of electrodes used in commercial electrochemical test strips [11,15-16]. The distribution of wax on the surface of the material might not be uniform depending on the application method. Thus, complete hydrophobicity of the paper cannot be assured. In order to address this problem, many paper cards were examined. And we report a novel substrate metallic gold card that has a hydrophobic nature. The fabrication is greatly facilitated by the removal of a key wax application step. Without any additional card pretreatment, the gold card can be screen-printed with Ag ink (silver ink) directly. When used as an ion selective electrode, silver ink exhibits greater sensitivity to various ions [17].

Gold nanoparticles (AuNPs) are considered to be highly stable metal nanoparticles that display a number of exceptional qualities, such as facial synthesis and surface modification, excellent biocompatibility, and strong Surface Plasmon Resonance (SPR) in the visible region of the spectrum, which make them the ideal candidate for detection of analytes with the naked eye [18]. Additionally, they have special optical and electrochemical properties that make them useful for sensing various analytes [19]. Due to the special characteristics of gold nanoparticles, which include their ability to withstand surface oxidation and chemical inertness, modification of electrode with gold nanoparticles is garnering considerable interest [20]. The sensitivity of the sensing platform can be improved by modifying the working electrode with AuNPs. In this study, a microfluidic paper device was created for tramadol detection by screen printing silver ink onto a card and modifying the working electrode with AuNPs. Tramadol can bind to the surface of these particles through the amino group by electrostatic contact, because oxygen-containing rings of hybrid aromatics display noticeably better binding to AuNPs [18]. By coordinating interactions with the electron-deficient surfaces of metal nanoparticles, hydroxyl groups containing electron-rich oxygen atoms are more likely to be bonded to the surface of metal nanoparticles [19]. This newly fabricated microfluidic device may function as both a microfluidic system and an electrochemical sensor.

Materials and Methods

All chemicals used were of analytical grade. Chloro auric acid ($HAuCl_4$), silver acetate, carbon conducting ink and silver conductive ink from Sigma Aldrich (USA), sodium citrate dihydrate, sodium acetate, ammonia, ethylamine, acetic acid and formic acid from Kermel chemicals (China), hydroxyl ethyl cellulose (HEC) from Koch-Light laboratories (England), potassium ferricyanide, potassium chloride from Harris reagent (England).

Synthesis of silver ink

In a glass vial, 2.0 g of silver acetate and 5 mL of ethylamine were combined, and 5 mL of ammonia was then added to the mixture to create a transparent solution. Followed by dropwise addition of 0.4 mL formic acid to the clear solution, which caused it to turn from light brown to black to gray. Lastly, 0.2 g of HEC was added to the solution while being continuously stirred [21].

Synthesis of AuNPs (gold nanoparticles)

In 200 mL of deionized water, 7.0 mg of $HAuCl_4$ was dissolved to create a pale-yellow solution. 50 mL of the solution were heated to a boil while being vigorously stirred, and 5 mL of 40 mM sodium citrate were added dropwise. Till a steady deep red color was obtained after 15 minutes of continuous heating and stirring [22]. The solution was kept at or below 7 ºC.

Fabrication of device

Two distinct patterns were created in Corel Draw. The first pattern, which is made up of circles with 1.6 cm in diameter, was printed using a printer on the card. The second pattern, the electrode layout, was printed on an A4 laser sheet using an HP laser jet 5100M. The electrode layout consists of the working electrode (WE), reference electrode (RE), and counter electrode (CE). The design on the laser sheet was exposed to a mesh, and the mesh was used to screen print silver ink onto the card as displayed in figure 1 [15]. The card was cured for 30 minutes at 120°C. Individual electrochemical cells or sensor strips were cut off. The working electrode was modified using gold nanoparticles. Gold nanoparticles modified screen printed paper electrode was prepared by drop coating the working electrode with 10 µL of AuNPs and the solution was allowed to dry at room temperature. For microfluidic system, 10 µL of AuNPs was drop coated onto the working electrode, with subsequent addition of 10 µL of 1 µM tramadol onto the AuNPs solution.

Characterization

Scanning electron microscopy (SEM) was used to examine the morphology of the AuNPs and Ag ink, and energy dispersive X-ray spectroscopy was used to examine the elemental spectra (EDX). Using a digital multimeter (DT9205A), the resistance of the manufactured devices was measured, and resistivity was computed using the formula.

$$\rho = RA/l \quad (1)$$

Where, ρ is Resistivity, R is the resistance, A is the cross-sectional area and L is the length, while conductivity was calculated using the formula

$$\sigma = 1/\rho \quad (2)$$

Where σ is the conductivity, and ρ is the resistivity [23].

Electroanalytical measurement

DropSens potentiostat (United Kingdom) coupled to a computer and the sensor strip, which has three electrodes: a working electrode, counter electrode, and reference electrode, were used to perform electrochemical measurements.

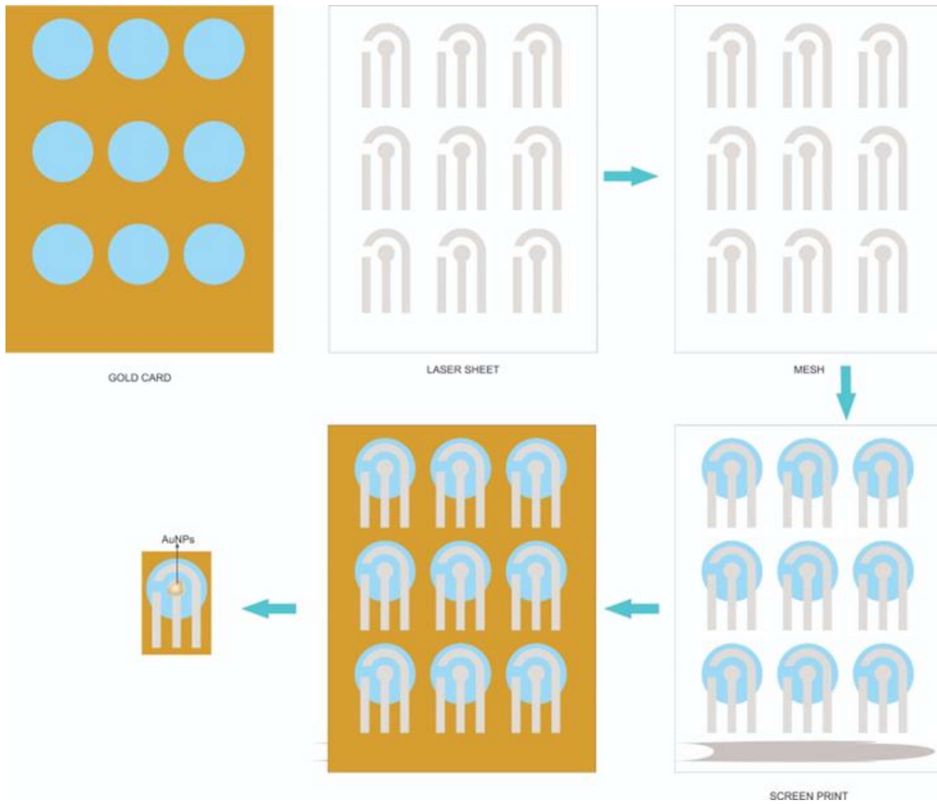


Figure 1. Schematic illustration of the fabrication steps

Results and Discussion

Scanning electron microscope

SEM Phenom Prox was used to capture the SEM imaging at 500x magnification, 100 µm interval, and 15 kV. The sample holder was created using aluminum foil as the blank. The images in Figure 2a have no particles at all dispersed across the surface. Figure 2b, on the other hand, shows an image of Ag ink with a white deposit covering the surface. Figure 2c displays image of surface without any particles at all. Figure 2d shows an image of gold nanoparticles, which are depicted as dazzling white deposits covering the surface. It has a highly distinct distribution of AuNPs dispersed widely throughout the surface.

Energy dispersive X-ray spectroscopy

The elemental spectra and value of Ag ink are shown in Figure 3a. Using the SEM image, several spectrums were captured at various distributions (Figure 2b). There is one prominent peak and several smaller peaks of silver in the spectral image, which indicates the presence of silver and the value confirms that silver is widely spread. The other elements were impurities discovered on the aluminum blank before the addition of silver, according to the elemental values obtained. The elemental spectra and value of AuNPs are shown in Figure 3b. Using the SEM image, several spectrums were captured at various distributions (Figure 2d). One sharp peak and several smaller peaks of AuNPs can be seen in the spectral image, which indicates the presence of AuNPs and the value confirms that AuNPs are widely spread. Due to the small quantity of contaminants present, the finding further supports the suitability of the process employed to create AuNPs and their purity. The aluminum blank included every element that has been seen with Ag ink and AuNPs. These are therefore impurities that are present on the blank.

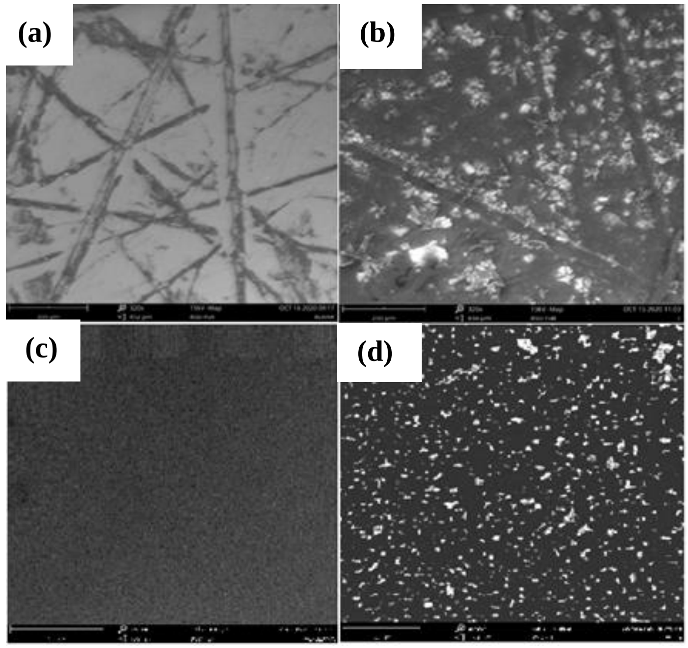
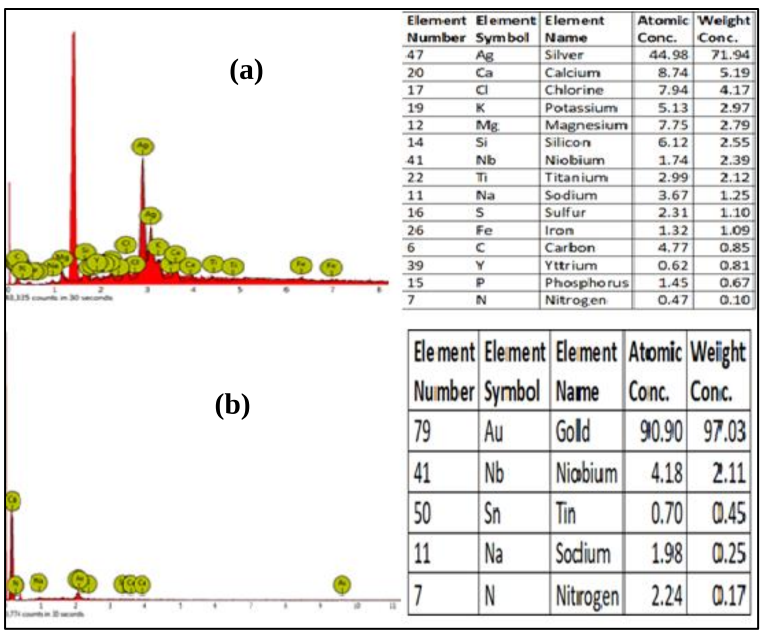


Figure 2. SEM image of (a) blank, (b) prepared Ag ink, (c) blank, and (d) AuNPs



Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
47	Ag	Silver	44.98	71.94
20	Ca	Calcium	8.74	5.19
17	Cl	Chlorine	7.94	4.17
19	K	Potassium	5.13	2.97
12	Mg	Magnesium	7.75	2.79
14	Si	Silicon	6.12	2.55
41	Nb	Niobium	1.74	2.39
22	Ti	Titanium	2.99	2.12
11	Na	Sodium	3.67	1.25
16	S	Sulfur	2.31	1.10
26	Fe	Iron	1.32	1.09
6	C	Carbon	4.77	0.85
39	Y	Yttrium	0.62	0.81
15	P	Phosphorus	1.45	0.67
7	N	Nitrogen	0.47	0.10

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
79	Au	Gold	90.90	97.03
41	Nb	Niobium	4.18	2.11
50	Sn	Tin	0.70	0.45
11	Na	Sodium	1.98	0.25
7	N	Nitrogen	2.24	0.17

Figure 3. EDX image (a) spectral image of Ag ink with elemental values (b) spectral image of AuNPs with elemental values

Microfluidic paper device

The typical substrate for paper-based printed electronics is a paper with a hydrophobic resin coating on the surface. Hydrophilic and evenly absorbent characteristics, thus, are incompatible with the needs of printed electronics [24]. A variety of paper cards were examined to find the one with the highest hydrophobicity. When a drug sample was dropped, art card figure 4A gave a good print, but it absorbed and flowed. Figure 4B Teslin card, which is typically used to create I.D. cards, could not produce a nice print right away. Moreover, the laser sheet figure 4C did not print well. Metallic silver and gold card figure 4D and E offered a nice print, did not absorb, and could be bent without harming the conducting part. Metallic silver and gold card were selected as the best substrates for additional examination. The image of the single sensor strip printed with commercial silver ink is shown in Figure 4F.

Conductivity and resistivity

Ag ink that had been synthesized had a conductivity of 23.1 mS. (millisiemens). The resistivity of the printed ink was calculated using the formula in equation 1. After printing with carbon ink, synthesized silver ink, and commercial silver ink on a paper card, the resistance of each ink was measured using a multimeter. The depiction of data recorded is shown in Table 1; commercial silver ink had a lower resistance. Hence, for more investigation, commercial silver ink was chosen. A lack of information from the synthesis process prevented the successful synthesis of Ag ink.

Gold and silver card printed with commercial silver ink were heated at 60 ºc. As can be seen in table 2, the resistance measurement decreased after 30 minutes, but not after an hour. As a result, 30 minutes was determined to be the ideal heating time. Resistance values obtained in table 2 were utilized in calculating the resistivity with the above resistivity formula. Gold card had a higher conductivity than silver card because it has a lower resistance than silver card. Due to the high conductivity that metal-coated papers typically display, they can perform in a variety of ways [25]

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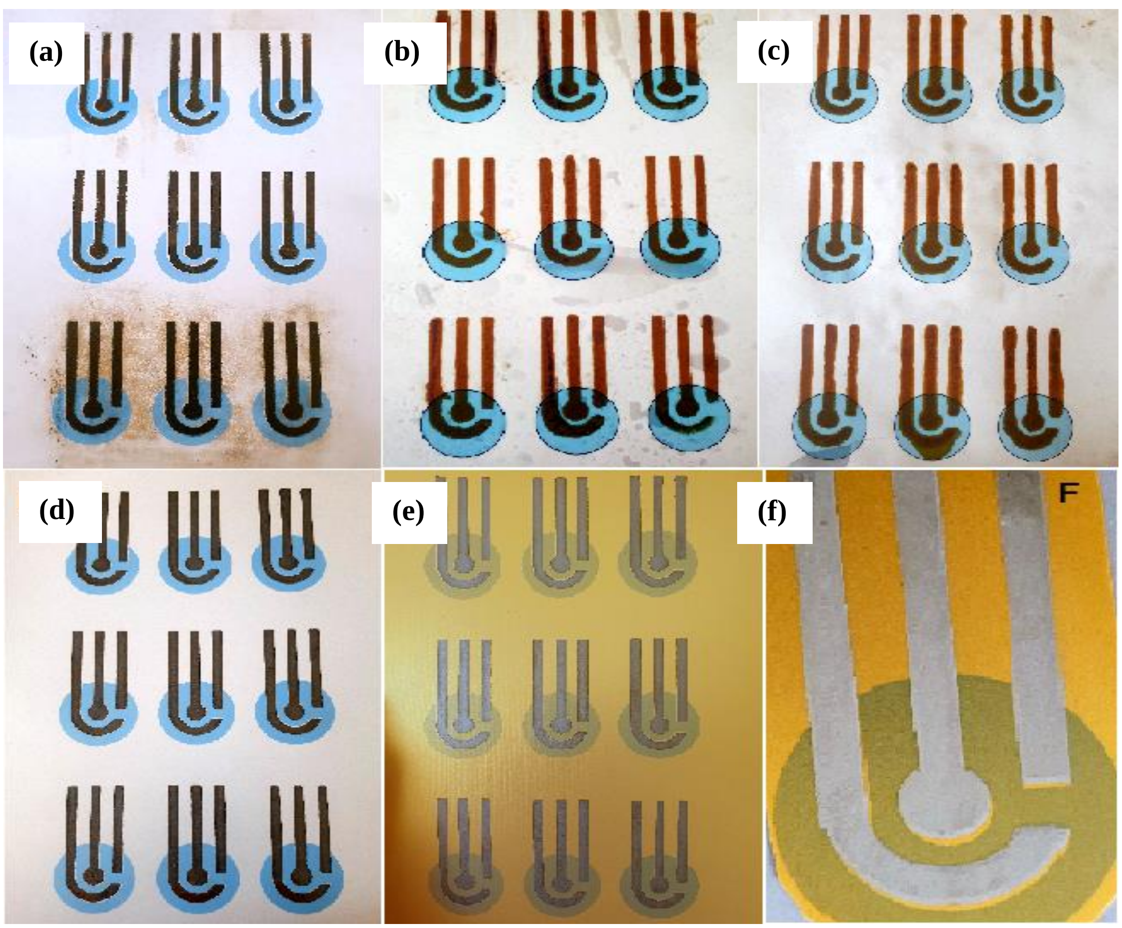

Figure 4. (a) Art card, (b) Teslin card, (c) laser sheet, (d) silver card, (e) gold card, (f) and single sensor strip

Electrochemical analysis

The electrochemical analysis employed the cyclic voltammetric approach, which provides data on the investigation of a reversible redox reaction. With the three-electrode sensor attached to a potentiostat, cyclic voltammetry was performed. The freshly created AuNPs/SPPE were studied using the cyclic voltammetric technique to determine their oxidation and reduction peak currents, which also shows an electron transfer reaction has taken place. Thus, indicating the feasible application of the sensor. With gold card AuNPs/SPPE in 2 mM $K_3Fe(CN)_6$ with 0.1 M KCl, a broad oxidation peak was seen at around 0.4 V to 0.55 V, whereas a minor reduction peak was seen at 0.15 V to 0.3 V in figure 5A. With the silver card AuNPs/SPPE, a broad oxidation peak was also seen between 0.3 and 0.45 V, whereas the reduction peak was shown between 0.2 and 0.45 V in figure 5B. When ferricyanide is subjected to cyclic voltammetry, ferricyanide is reduced to ferrocyanide and vice versa, demonstrating that the reaction is reversible and that a one electron transfer diffusion reaction has occurred [26]. These findings reveal that the oxidation peaks, which signify the formation of oxide, exhibit a greater current for gold card AuNPs/SPPE than for silver card AuNPs/SPPE, further demonstrating the superior conductivity of gold card.

The gold card AuNPs/SPPE was selected for further analysis. Figure 6 shows the results of the AuNPs/SPPE study in acetate buffer and tramadol. The cyclic voltammogram showed no oxidation peak but a small reduction peak in acetate buffer, while an oxidation peak current was obtained at around 0.1V to 0.3V and a reduction peak current at around 0.00V to -0.15V for acetate buffer with tramadol. As a result, AuNPs enhance detection capabilities and provide evidence of an oxidation reaction between AuNPs and tramadol.

Table 1. Resistance Measurement of different inks printed on gold and silver cards

Gold Card Electrodes	Working Electrode	Reference Electrode	Counter Electrode
Carbon ink	516	465	785
Prepared Silver ink	42.5	70.5	135.6
Commercial Silver ink	3.1	2.6	4.1
Silver card Electrodes			
Carbon ink	292	263	414
Prepared Silver ink	43.1	24.1	51.6
Commercial Silver ink	4.3	3.6	6.6

Table 2. Resistance measurement, resistivity and conductivity of gold and silver card with commercial silver ink after heating

Electrode	W.E	R.E	C.E
Gold card/Resistance	1.9	1.7	2.4
Resistivity (Ω cm)	3.4×10^{-5}	3.5×10^{-5}	2.5×10^{-5}
Conductivity (mS)	29367.54	28487.43	39479.54
Silver card/Resistance	2.43	2.2	2.9
Resistivity (Ω cm)	4.35×10^{-5}	4.5×10^{-5}	3.06×10^{-5}
Conductivity (mS)	22962.27	22013.05	32673.012

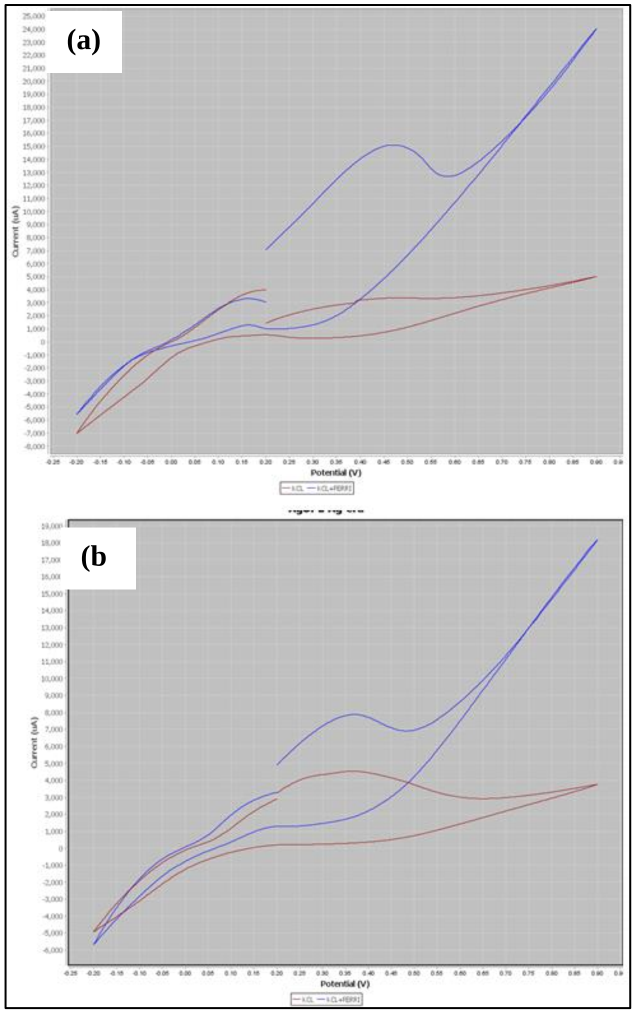

Figure 5. Cyclic voltammogram (a) Au card AuNPs/SPPE in 2 mM ferricyanide and 0.1 M KCl (blue line) 0.1 M KCl (red line) and (b) Ag card AuNPs/SPPE in 2 mM ferricyanide and 0.1 M KCl (blue line) 0.1 M KCl (red line)

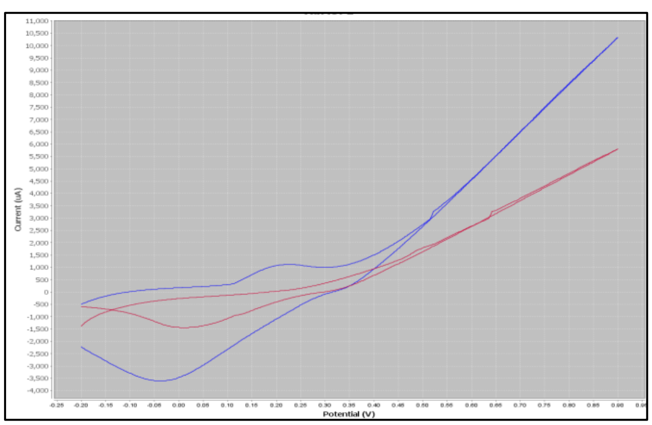

Figure 6. Cyclic Voltammograms of 0.1M Acetate Buffer (Red line), 1µM Tramadol (Blue line) for AuNPs/SPPE

Conclusion

Paper electrodes with gold nanoparticle modifications were successfully created using the screen-printing technique. The intriguing aspect of this research is that the metallic gold card we used as a substrate, which is also inexpensive, allowed us to forgo the wax phase. According to the conductivity value, the card exhibits very high conductance and produced a good print with the inks. Since the current was larger for the gold card, the electrochemical experiment further establishes the high conductivity of the manufactured sensor strip. Using cyclic voltammetry, the electrochemical method of detecting tramadol proved successful. We are certain that the manufactured sensor strip can be used in electrochemical detection based on this initial electrochemical measurement.

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