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Development of silver/carbon screen-printed electrode for rapid determination of vitamin C from fruit juices

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ABSTRACT

A silver/carbon screen-printed electrode (S/C-SPE) was fabricated using polymer-based conductive inks blended with in-house synthesized nanoparticles and investigated for its application in electrochemical analysis. Silver nanoparticles (~59 nm) and carbon nanoparticles (~76 nm) were synthesized having zeta potential (<±60 mV) and particle morphology were investigated for both the nanoparticles. These nanoparticles were blended with a thermoset epoxy resin to formulate different conductive inks *viz.* silver (Ag) ink, carbon (C) ink and silver/silver chloride (Ag/AgCl) ink. The formulated inks were characterized for their viscosity, which ranged from 1500 to 4500 cP making them ideal for screen printing. Post printing and thermal curing, the sensing area of the S/C-SPE was characterized for its elemental content by EDX, electrode thickness ranged from 0.3 to 0.8 μm and bulk resistance (ρ) (4.7 -13.4×10^{-4} Ω-cm). The level of vitamin C was determined in different fruit juices at the S/C-SPE using a developed voltammetric method and compared with that of standard biochemical method. The vitamin C content of different juices ranged from 5 to 60 mg/100 ml. The analytical characteristics of the S/C SPE (linear range, reproducibility, stability, matrix effect, electrode saturation and response time) were investigated. The results obtained by the two methods were in good agreement.

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1. Introduction

In the last few years with the recent advancement in development of printed electronics, there has been a growing interest in replacing traditional silicon techniques with new and robust methodologies to develop printed electronics as they offer low cost, less tedious processing and better results. Development in the field of analytical chemistry is aimed to meet the need of fast, easy and on site analysis as well as improved analytical methods which offer better result outputs with lesser environmental impact. A field detection system for real time analysis is highly desirable to overcome time consuming process such as sample collection, its transfer to a laboratory as well as tedious conventional testing methods. Such real time testing system put forward a rapid return of the results with minimized errors and costs as compared to the offsite laboratory-based analyses (Hayat & Marty, 2014; Wang, 2002). Current progress in analytical chemistry is to develop electrochemical devices which include possibility of miniaturization and portability, sensitivity, selectivity, a wide linear range, minimal space and power requirement and cost effective instrumentation (Honeychurch & Hart, 2003; Thiyagarajan, Chang, Senthilkumar, & Zen, 2014). Such devices can be accomplished with printed electronics (PE) with various functional inks containing nanomaterials such as metallic nanoparticles, organic electronics molecules/polymers and carbon based nanomaterials, employed to bulk production process for advanced electronic equipments (Lee, Jun, Kim, & Joung, 2006). Use of silver nanoparticles based conductive inks, has been found to be a very powerful tool for direct patterning of desired schematic conductive tracks in electronic devices (Kim, Jeong, Park, & Moon, 2006). When coupling such nanoparticles with advanced polymer composites, printed schematics can be annealed or cured at moderate temperatures to form conductive films of low resistance with potential suitability for use in printed electronics (Allen, Bayles, Gile, & Jesser, 1986; Buffat & Borel, 1976; Jiang, Zhang, & Zhao, 2003).

Vitamin is a nutrient that does not provide energy to the body but it is essential nutrients. It is necessary for the growth of the body and also associated with the enzyme function in the body (Okiei et al., 2009). Vitamin C minimum daily requirement for

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adults is 60 mg. Vitamin C is potent anti-oxidant and free radical scavenger, thus, helps in body tissue growth and repair (Cathcart, 1991). It is essential in synthesizing collagen, ligaments and blood vessels, iron absorption, and immune response activation and is involved in wound healing and osteogenesis. Furthermore, Vitamin C has the properties of reducing agent and weak acid (Valente, Sanches-Silva, Albuquerque, & Costa, 2014).

Electrochemical analysis such as voltammetry is an increasingly popular methodology applied to the determination of vitamin C in real samples (Wawrzyniak, Ryniecki, & Zembrzuski, 2005), because it offers low detection limits with little or no sample preparation, against more expensive techniques. Voltammetry offers an attractive alternative to the titrimetric or instrumental methods such as spectroscopy and liquid chromatography, in particular in food quality control due to its low cost as well as simplicity of the employed procedures necessary to determine vitamin C. It is quick, easy and does not require complicated, expensive equipment and well-qualified personnel like the previously mentioned instrumental techniques.

Present work comprises synthesizing stable silver and carbon nanoparticles and blending them to a thermoset clear epoxy resin (Oldenzijl, Chen and Gunther 2015) to formulate different conductive inks. The solid content of the said conductive inks was maintained to make ink screen printable with viscosity in the range of 1000–10,000 cP. A screen-printed three electrode electrochemical cell was manually screen-printed on to a polyester (PET) sheet and was heat cured, which annealed nanoparticles with epoxy polymer so as to get printed tracks. The S/C-SPE was characterized for its electrochemical performance by cyclic voltammetry analysis of ferro-ferri couple at different scan rate followed by the S/C-SPE was used to estimate ascorbic acid (vitamin C) from different fruit juices.

2. Material and methods

Solid chemicals such as Silver Nitrate ($AgNO_3$), Silver Chloride ($AgCl_2$), Tri-sodium citrate were purchased from Merck-KGaA (Germany), the epoxy resin - Thermoset E-105 Epoxy resin and Circalok 6055B Hardener were procured from LORD Corporation (USA). Polymer dielectric (D2081009D6) was purchased from Gwent Electronic Materials Ltd. (United Kingdom). Sodium dihydrogen *ortho*-phosphate and ascorbic acid were procured from Merck Chemical (Germany). 2,4-Dinitrophenylhydrazine (DNPH) dye purchased from Sigma-Aldrich (Madrid, Spain). The deionized (DI) water (resistivity = 18.2 MΩ.cm, and conductivity = 0.058 at 25 °C) for synthesis of nanoparticles was collected from Merck Millipore water purification system (USA) installed in our laboratory.

2.1. Synthesis of nanoparticles for conductive inks formulation

2.1.1. Synthesis of silver nanoparticles by bottom-up technique

Silver nanoparticles (SNPs) were synthesized by bottom-up techniques using chemical reduction of 100 ml 1 mM aqueous solution of $AgNO_3$ with quick addition of 10 ml 1.5% tri-sodium citrate as reducing agent at 80–85 °C under continuous stirring (Sodha, Jadav, Gajera, & Rathod, 2015). The unfavorable agglomeration was avoided with the $AgNO_3$/PVP mass ratio of 1: 4 (Xiliang et al., 2014). Following addition of reducing agent the temperature was raised to boiling and maintained for 10 min. The reaction was given a cold temperature shock by immediately placing it in ice bath. Gryeish black powder of SNPs was obtained by high speed centrifugation at 12,000 rpm with twice intermediate washing with DI water for 15 min and drying under vacuum at 70 °C for 30 min. These dried SNP powder was further used for formulation of conductive inks.

2.1.2. Synthesis of carbon nanoparticles by top-down technique

Wooden charcoal pieces were burnt in fireplace for 3–4 h. The small pieces of black mass so obtained with innate resistance between 50 and 80 Ω using two-probe multimeter were used for synthesis of carbon nanoparticles (CNPs). These pieces were socked in DI water overnight and blended using high speed homogenizer as mentioned by (Yadav, Yadav, & Singh, 2012). The aqueous black slurry so obtained was filtered under high vacuum through 0.2 μm PTFE membrane filter. The filtrate containing the carbon nanoparticles was dried overnight at 60 °C.

2.2. Formulation of conductive inks

2.2.1. Formulation of silver conductive ink

Silver conductive ink was prepared by dispersing SNP powder in Thermoset E-105 clear epoxy resin at 10,000 rpm using high speed homogenizer at 25 °C in a temperature controlled water bath. The percentage (%) silver content was kept as 90–94% w/v. Once the nanoparticles and resin were homogeneously mixed, Circalok 6055B Hardener was added in 0.84:1 w/w ratio. The nanoparticles dispersed epoxy resin and hardener was mixed at 25 °C under high vacuum to avoid formation of air bubbles. The final solid content in the silver paste was 45–47% w/v (Jewell, Hamblyn, Claypole, & Gethin, 2015).

2.2.2. Formulation of carbon conductive ink

Carbon conductive ink was prepared by uniformly dispersing carbon nanoparticles in thermoset E-105 clear epoxy resin and Circalok 6055B Hardener by process similar to silver ink. The final solid content of the carbon ink was adjusted to 40–42% w/v (Calixto et al., 2007).

2.2.3. Formulation of silver/silver chloride conductive ink

Silver/silver chloride ($Ag/AgCl_2$) ink was prepared by initially dissolving 80% w/v silver chloride ink E-105 epoxy resin followed by dispersing SNPs by high speed homogenizer followed by addition of Circalok 6055B Hardener similar to that of silver ink formulation. The final solid content was adjusted to 40–45% w/v (Jewell et al., 2015).

2.3. Fabrication of screen-printed electrodes (SPEs)

Coral Draw X7 graphic suite was used to design the S/C-SPE. A flat bed mono-filament polyester screen with average pore size of 45 μm was used to prepare separate screen for the four printing steps as shown in Fig. 1(A). Manual screen printing process was

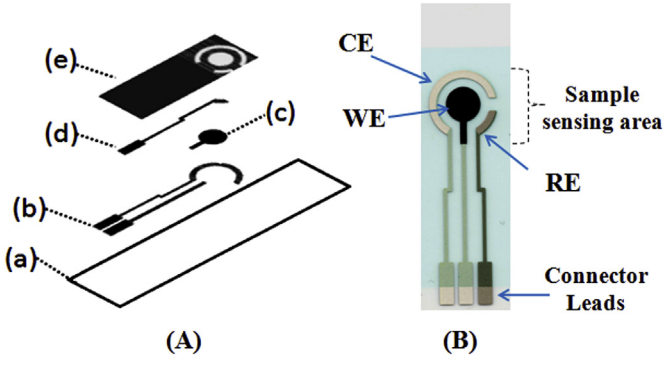

Fig. 1. A) Scheme for screen printing of silver/carbon screen-printed electrode (S/C-SPE). (a) polyester substrate, (b) silver track, (c) carbon layer, (d) silver/silver chloride track and (e) insulating layer; B) Screen printed silver/carbon screen-printed electrode (S/C-SPE).

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