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DEVELOPMENT, FABRICATION, AND ELECTROCHEMICAL CHARACTERIZATION OF SCREEN-PRINTED CARBON ELECTRODES: A COMPREHENSIVE STUDY ON REPEATABILITY AND PERFORMANCE OPTIMIZATION

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ABSTRACT

Screen-printed carbon electrodes (SPCEs) have shown great promise as electrochemical sensing and analytical platforms because of their cost effectiveness, disposability and compatibility with hand-held instrumentation. This work reveals an in-depth investigation of the development, fabrication, optimization, and repeatability analysis of the screen-printed carbon electrodes fabricated using a controlled screen-printing process on different substrates. A systematic procedure was followed to characterize the electrochemical behavior of SPCEs using several electrochemical techniques such as cyclic voltammetry (CV), differential pulse voltammetry (DPV) and square wave voltammetry (SWV). The study involved material choices, developing ink formulation, substrate preparation, optimization of electrode geometry, and to evaluate intra-electrode and inter-electrode repeatability, strict protocols of electrochemical testing were carried out. Results show that optimized SPCEs have good electrochemical performance including low charge transfer resistance, high peak current executing and good reproducibility in several batches. The batch-to-batch consistency of the developed electrodes was evaluated and the results showed that the co-efficient of variation (CV) in peak current measurements obtained from 10 replicate electrodes was below 8%. Electrochemical impedance spectroscopy (EIS) analysis has shown well defined semicircular plots related to the diffusion limited mechanism of electron transfer. The potential for these screen-printed electrodes in point-of-care applications for diagnostics, environmental monitoring and electrochemical sensing applications in the laboratory is discussed. This work defines high standards of quality control and performance benchmarking requirements for SPCE fabrication which will lead to the development of projections of electroanalytical methodologies in research and industrial research.

Keywords: screen-printed carbon electrodes, electrochemical characterization, repeatability, cyclic voltammetry, electrochemical impedance spectroscopy, analytical performance

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1. INTRODUCTION

1.1 Background and Significance of Electrochemical Sensors

Electrochemical sensors are one of the fastest growing areas in analytical chemistry and bioanalytical sciences and promise rapid response time, high sensitivity and good selectivity in quantifying different kind of analytes ranging from small organic molecules to biomacromolecules. The growth of small portable electrochemical platforms to replace the conventional laboratory-based electroanalytical instrumentation has transformed point-of-

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care diagnostics, environmental assumptions, and sensing applications that can be utilized in the field. Among the several electrochemical sensing platforms, screen-printed electrode (SPE) has become a result of great attention because of its particular set of characteristic advantages that includes low price, ease-of-mass manufacturing, low-sample-and reagent consumption and compatibility with microfluidic devices and multiplexed array formats.

1.2 Screen-Printed Electrodes: Advantages and Applications

Screen-printing technology is used to explain a robust, scalable and economical approach to electrode fabrication, which has been derived from the printing and electronic industries. Screen printed electrodes may be mass produced very quickly onto either flexible or rigid substrates with a specific geometrical dimension that is ideal for single use disposable electrochemical sensors. The versatility of screen printing means a range of functional materials can be incorporated into the printing process; these include carbon blacks, metal nanoparticles, conductive polymers, and biocompatible coatings to increase the number of applications it could have. Screen-printed carbon electrodes (SPCEs, in part) have been widely adopted in electroanalytical laboratories and commercial diagnostic platforms because of the great electrochemical properties of carbon-based materials, chemical stability, large potential window and known protocols for surface modification and functionalization.

1.3 Electrochemical Characterization Techniques

A good understanding of electrode performance requires the use of multiple, complementary, electrochemical techniques. Cyclic voltammetry (CV) is the gold standard for the fast screening of electrochemical systems that deliver information about the redox processes, kinetics of electrodes, and the reversibility of charge transfer reactions. Differential pulse voltammetry (DPV) helps to increase the sensitivity by subtracting background current and pulse modulation, which helps in obtaining trace-level analyte signals with low noise and better signal-to-noise ratios. Square wave voltammetry (SWV): In addition to amping the current pulse advantage of the pulse techniques, SWV offers improved peak current responses and faster scan rates, making it especially valuable for use in high throughput screening applications. Electrochemical impedance spectroscopy (EIS) can give non-faradaic and faradaic impedance measurements over a wide frequency range, and opens the door for analysis of the detailed properties of interface properties, resistance to charge transfer, and the electrode-electrolyte interactions.

1.4 Repeatability and Reproducibility in Electrochemical Electrode Fabrication

One of the critical factors that plays a key role in the limited use of electrochemical sensors for clinical diagnostics and regulated environments is the consistency in electrode performance caused by the inconsistencies in electrofabrication processes lot-to-lot. Repeatability (intra batch consistency) and reproducibility (inter batch consistency) are basic quality measures that have to be thoroughly characterised in order to ensure reliable analytical performance and regulatory compliance. The electrochemical performance of screen printed electrodes is affected by many factors such as the composition of the carbon ink used, the properties of the substrate material used, the

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printing pressure and speed, the curing temperature and duration, the mesh properties, and the environmental storage conditions. It is important to understand and be able to control these variables in order to introduce standardized fabrication protocols which will produce electrodes with predictable and consistent electrochemical properties.

1.5 Research Objectives and Scope

The main goal of this extensive research project was the development, fabrication and thorough characterisation of screen printed carbon electrodes with a special focus on the optimisation of fabrication parameters, to obtain a very good electrochemical performance with very good repeatability over several batches and electrode units. Specific aims consisted in: (i) carbon ink formulation optimization and choice of suitable substrate materials; (ii) systematic study of the parameters of the printing method (pressure, speed, temperature) of optimal fab conditions; (iii) rigorous electrochemical characterization based on a variety of techniques with voltammetry; (iv) quantitative evaluation of intra-electrode and inter-electrode repetitiveness; (v) elaboration of correlation between fabrication parameters and electrochemical performances metrics; (vi) elaboration protocols of quality control for the framework of production of SPCE. The findings of this investigation are aimed to be a contribution toward screen printed electrode technology for the betterment of electrochemical sensors and the establishment of best practices for reproducible fabrication of high-performance electrochemical sensors.

2. Literature Survey and State-of-the-Art

2.1 Historical Development of Screen-Printed Electrodes

Screen printed electrodes appear to have emerged in the late 1980s and early 1990s with the confluence of progress in printing technology, materials science as well as analytical chemistry. The novel investigations on the feasibility of thermally curing conductive inks onto insulating substrates to create functional electrodes provided by the pioneer work on SPEs. Early applications were focused in glucose monitoring, drug and environment (contaminant) detection. Over the past three decades, the field has become much more developed with advances in ink chemistry, substrate materials, surface modification techniques, and electrode geometries serving to increase the application scope of screen-printed electrodes for immunoassays, DNA detection, enzyme-based biosensors, and electrochemiluminescence applications.

2.2 Carbon Materials in Electrochemistry

Carbon-based materials have very remarkable electrochemical properties making them perfect choices for electrode applications. The electrochemical behaviour of carbon materials is affected by the crystalline structure, surface chemistry, porosity, and hardness defect density of the carbon material. Graphite particles, graphene oxide, carbon nanotubes and glassy carbon are all used for electrochemical sensing and have their own unique advantages regarding the electron transfer kinetics, surface area and chemical inertness. Given their balance between electrochemical activity, mechanical robustness, and price, carbon black particles are the ideal choice for bulk electrode fabrication materials that are widely used in the commercial utilization of screen printed inks.

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2.3 Ink Formulation and Optimization

The electrochemical performance of screen printed electrodes is, in principle, determined by the composition and properties of the printing ink. The ink used in commercial screen print is typically made up of conductive particles (either a carbon black ink, graphite, or metals which are actually powders) that are dispersed in an organic binder system which is composed of resins, solvents, and other additives. The relative proportions of these components have a critical effect on viscosity, wettability, electrical conductivity, mechanical adhesion to substrate or electrochemical response. Previous studies have shown that ink particle size distribution, solvent choice and binder polymer chemical characteristics play a vital role in determining final electrode performance.

2.4 Substrate Materials and Their Impact on Performance

The substrate material on which the conductive ink is printed has an equal role on which the electrode performance and longevity will be determined. Common types of substrate material include polyester films, polyethylene terephthalate (PET), ceramic tiles and glass. The choice of substrate affects the adhesion of the ink, chemical compatibility, thermal stability, and mechanical flexibility. The surface characteristics of the substrate such as roughness, wettability and porosity influence the ink penetration and the formation of the final conductive layer.

2.5 Electrochemical Characterization Methodologies

Comprehensive electrochemical characterization of screen-printed electrodes usually consists of the battery of complementary techniques. Cyclic voltammetry is the main screening tool which offers fast screening of electrode reversibility and background current properties. Differential pulse and square wave voltammetry improves the detection limits and selectivity by subtracting the background current. Electrochemical impedance spectroscopy gives mechanistic information on electrode-electrolyte interfacial properties as well as charge transfer kinetics. Chronoamperometry and chronopotentiometry are used to measure the temporal response features and the kind of electrochemical processes.

2.6 Current Challenges and Research Gaps

Despite great advancement, there are still many challenges in the screen printed electrode technology field. The cause of batch-to batch variability of electrode performance is a critical drawback correlated with both clinical applications in medical practice and regulatory requirements for ablative procedures throughout the body, where quality control is necessary to a nice degree. The mechanisms of the variation of electrochemical performance as a function of the fabrication parameters requires more systematic investigation. Added to that, the long-term stability of the screen-printed electrodes when stored under a variety of different conditions and during repeated periods of use needs further characterisation. The creation of standardised protocols to evaluate electrode quality and validate their functions in different laboratories and across varying research groups would go a long way toward enabling comparison of outcomes and for accelerating technology adoption.

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3. Materials and Methods

3.1 Materials and Chemicals

3.1.1 Substrate Materials

Polyester films (PET, thickness 125 μm) and ceramic tiles (96% alumina, dimensions 25 mm × 75 mm × 1.5 mm) were employed as substrate materials for electrode deposition. PET films were selected for their flexibility, transparency, and compatibility with conventional printing processes, while ceramic substrates were chosen for their superior thermal stability and chemical inertness.

3.1.2 Printing Inks

Commercial screen-printing carbon ink (model: SE100, composition approximately 70% carbon black particles, 20% resin binder, 10% organic solvents) was obtained from specialized suppliers. Carbon black particles within the ink exhibited mean particle size of approximately 25-50 nm as determined by transmission electron microscopy. Alternative ink formulations with varying carbon loading (60%, 70%, 80%) were prepared by controlled addition of carbon black particles and polymer binders.

3.1.3 Electrochemical Reagents

Potassium ferrocyanide $K_3[Fe(CN)_6]$ (analytical grade, ≥98%), potassium ferricyanide $K_4[Fe(CN)_6]$ (analytical grade, ≥98%), potassium chloride (KCl, ≥99%), and phosphate buffered saline (PBS, pH 7.4) were obtained from commercial chemical suppliers. Ultrapure water (18.2 MΩ·cm resistivity) was obtained from a Milli-Q water purification system. All chemicals were used without further purification.

3.1.4 Supporting Electrolytes

Potassium phosphate buffers at pH 7.4 and pH 4.0 were prepared by appropriate mixing of monobasic and dibasic potassium phosphate solutions. Acetate buffers at pH 5.0 were prepared from acetic acid and sodium acetate. All buffer solutions were filtered through 0.22 μm syringe filters before use to remove particulate impurities.

3.2 Electrode Fabrication and Screen-Printing Protocol

3.2.1 Substrate Preparation

PET film substrates were cleaned sequentially with deionized water, ethanol, and acetone, then dried under a stream of compressed nitrogen. Ceramic substrates were subjected to ultrasonic cleaning in acetone for 15 minutes, followed by rinsing with deionized water and drying at 120°C for 30 minutes. Prior to printing, all substrates were treated with a corona discharge surface treatment unit to enhance wettability and improve ink adhesion.

3.2.2 Screen-Printing Process

Screen-printing was performed using a semi-automatic screen-printing apparatus equipped with a squeegee system, adjustable printing angle, and programmable print pressure and speed controls. Stainless steel mesh screens (mesh size 200×200 threads per inch, stencil thickness 20 μm) were employed for all printing operations. The printing process parameters are detailed below:

- **Printing Pressure:** 5-8 $kg \cdot cm^{-2}$ (adjusted to ensure complete ink transfer while minimizing mesh deformation)
- **Squeegee Speed:** 50-80 $mm \cdot s^{-1}$ (controlled to allow adequate time for ink release from mesh onto substrate)
- **Number of Print Passes:** 1-3 passes (to achieve appropriate ink film thickness and electrical conductivity)
- **Printing Angle:** 45-60° (to optimize ink transfer and substrate contact)

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Geometric definition of the working electrode area was achieved through careful masking and selective screen design. The final electrode design incorporated a circular working electrode (diameter 2-5 mm), auxiliary electrode, and reference electrode connection pads, all fabricated in a single printing operation.

3.2.3 Thermal Curing and Post-Processing

Immediately following screen-printing, electrode samples were transferred to a programmable furnace for thermal curing. The curing protocol consisted of:

- **Initial temperature ramp:** 25°C to 120°C at 2°C·min^{-1} (volatilization of residual solvents)
- **Isothermal hold:** 120°C for 15 minutes (evaporation of volatile components)
- **Secondary temperature ramp:** 120°C to 250°C at 3°C·min^{-1} (resin crosslinking and binder polymerization)
- **Final isothermal hold:** 250°C for 30 minutes (complete thermal stabilization)
- **Cooling ramp:** 250°C to 25°C at 5°C·min^{-1} (controlled cooling to prevent thermal stress)

Post-curing, electrode samples were allowed to equilibrate to room temperature before electrochemical testing.

3.3 Electrode Characterization Methods

3.3.1 Morphological Characterization

Scanning electron microscopy (SEM) was employed to evaluate the surface morphology and microstructure of fabricated screen-printed electrodes. Samples were mounted on aluminum stubs using double-sided conductive tape and sputter-coated with a thin layer of gold to enhance image quality. SEM images were acquired at 5-20 kV accelerating voltage with magnifications ranging from 500× to 10,000×. Energy dispersive X-ray spectroscopy (EDS) was performed simultaneously to confirm elemental composition and identify the presence of residual organic materials or contaminants.

3.3.2 Surface Roughness and Profilometry

Three-dimensional surface topography was assessed using optical profilometry. A white light interferometer was employed to generate high-resolution topographical maps of the electrode surface over a scan area of 1.28 mm × 0.96 mm. Surface roughness parameters including arithmetic mean roughness (Ra), root mean square roughness (Rq), maximum profile height (Rz), and surface skewness (Rsk) were determined from triplicate measurements at different locations on each electrode sample.

3.3.3 Contact Angle Measurement

Surface hydrophilicity of the fabricated electrodes was assessed through contact angle measurements using a dynamic contact angle analyzer. Ultrapure water droplets (5 μL) were deposited on the electrode surface, and the contact angle was measured at 1-second intervals for 30 seconds. Advancing and receding contact angles were recorded to provide information about surface wettability and interfacial properties.

3.4 Electrochemical Characterization Protocols

3.4.1 Instrumentation and Setup

All electrochemical measurements were performed using a three-electrode system connected to a computer-controlled potentiostat/galvanostat (model: Ivium CompactStat, resolution ±0.1 mV, potential range ±5 V). The screen-printed electrode served as the working electrode, a platinum wire (0.5 mm diameter, 2 cm length) functioned as the auxiliary electrode, and a Ag/AgCl (3 M KCl) electrode was employed

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as the reference electrode. All electrodes were immersed in a glass electrochemical cell containing 10 mL of the selected electrolyte solution.

3.4.2 Cyclic Voltammetry (CV)

Cyclic voltammetry was performed at scan rates varying from 10 to 500 $mV \cdot s^{-1}$ in a solution containing 5 mM $K_3[Fe(CN)_6]/K_4[Fe(CN)_6]$ (1:1 molar ratio) in 1 M KCl supporting electrolyte. The potential was scanned from -0.2 V to +0.6 V vs. Ag/AgCl reference electrode. Peak currents (Ipa, Ipc), peak potentials (Epa, Epc), and formal potential (E°') were extracted from the resulting cyclic voltammograms. The peak current separation (ΔEp = Epa - Epc) and peak current ratio (Ipa/Ipc) were calculated to assess the reversibility of the electrochemical system.

3.4.3 Differential Pulse Voltammetry (DPV)

DPV measurements were conducted under the following conditions:

- **Initial potential:** -0.2 V
- **Final potential:** +0.6 V
- **Step potential:** 5 mV
- **Pulse height:** 50 mV
- **Pulse width:** 20 ms
- **Scan rate:** 20 $mV \cdot s^{-1}$

Peak current and peak potential values were determined from the normalized DPV traces. The signal-to-noise ratio was quantified by measuring the baseline noise as the standard deviation of potential-current points in a region of the voltammogram devoid of faradaic current.

3.4.4 Square Wave Voltammetry (SWV)

Square wave voltammetry was performed with the following parameters:

- **Initial potential:** -0.2 V
- **Final potential:** +0.6 V
- **Step potential:** 5 mV
- **Square wave frequency:** 25 Hz
- **Square wave amplitude:** 25 mV
- **Scan rate:** 100 $mV \cdot s^{-1}$

Peak current and peak potential were determined from the square wave voltammograms. The peak current enhancement relative to CV was calculated for comparative analysis.

3.4.5 Electrochemical Impedance Spectroscopy (EIS)

EIS measurements were conducted at the formal potential of the ferrocyanide redox couple (E = E°', typically around +0.2 V vs. Ag/AgCl) using 5 mM $[Fe(CN)_6]^{3-/4-}$ in 1 M KCl. Frequency sweep was performed from 100 kHz to 0.1 Hz with an alternating current amplitude of 10 mV (peak-to-peak). Complex impedance data were fit to appropriate equivalent circuit models (typically Randles circuit) to determine charge transfer resistance (Rct), double layer capacitance (Cdl), and other kinetic parameters.

3.5 Repeatability and Reproducibility Assessment

3.5.1 Intra-Electrode Repeatability

Intra-electrode repeatability was evaluated by performing 5-10 consecutive cyclic voltammetric measurements on the same electrode without removal from the electrochemical cell. Peak current values were recorded for each measurement, and the coefficient of variation (CV%) was calculated as:

$$CV\% = \frac{\sigma}{\bar{x}} \times 100$$

where σ is the standard deviation and $\bar{x}$ is the mean peak current value.

3.5.2 Inter-Electrode Repeatability

Inter-electrode repeatability was assessed by fabricating a batch of 10-15 nominally identical screen-printed carbon electrodes under identical fabrication conditions. Each

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electrode was independently characterized by cyclic voltammetry using the same protocol and electrolyte solution. Peak current values, peak potential values, and formal potentials were determined for each electrode. Statistical analysis including calculation of mean, standard deviation, coefficient of variation, and 95% confidence intervals was performed on the collected data.

3.5.3 Batch-to-Batch Reproducibility

Multiple batches of screen-printed carbon electrodes were fabricated over an extended time period (spanning several weeks) using identical fabrication protocols. Electrochemical characterization of representative electrodes from each batch was performed to assess long-term reproducibility. Temporal trends in electrode performance were identified and analyzed.

4. Results and Data Analysis

4.1 Electrode Fabrication Optimization

4.1.1 Effect of Printing Parameters on Electrode Performance

Systematic variation of printing pressure (5-8 $kg \cdot cm^{-2}$), squeegee speed (50-80 $mm \cdot s^{-1}$), and number of print passes (1-3) revealed significant influences on the resulting electrode performance. Electrodes fabricated at 6.5 $kg \cdot cm^{-2}$ printing pressure with 65 $mm \cdot s^{-1}$ squeegee speed and two print passes exhibited optimal electrical conductivity and electrochemical response. Lower printing pressures resulted in incomplete ink transfer and higher electrode resistance, while excessive pressure caused mesh deformation and non-uniform ink distribution.

4.1.2 Thermal Curing Protocol Optimization

The developed two-stage thermal curing protocol (120°C for solvent volatilization followed by 250°C for binder crosslinking) resulted in superior electrode performance compared with single-stage curing protocols. Electrodes cured using the optimized protocol demonstrated peak current values approximately 15-20% higher than those from alternative curing regimens, along with significantly reduced background currents.

4.2 Morphological and Surface Characterization

4.2.1 Scanning Electron Microscopy (SEM) Analysis

SEM imaging revealed that the screen-printed carbon electrode surface consisted of irregular carbon black particles (25-50 nm individual diameter) embedded within an organic polymer matrix. The resulting microstructure exhibited a tortuous network of conductive pathways interspersed with polymeric binder material. Representative SEM images are presented in Figure 1. Particle-to-particle contact was uniform across the electrode surface, with occasional small voids (< 1 μm diameter) attributable to incomplete resin infiltration during the curing process.

4.2.2 Surface Roughness Characterization

Surface roughness measurements (Table 1) indicated mean arithmetic roughness (Ra) values of 1.2 ± 0.3 μm for optimized screen-printed carbon electrodes. Root mean square roughness (Rq) values were approximately 1.5 ± 0.4 μm. Maximum profile height (Rz) ranged from 8 to 12 μm, reflecting the irregular character of the carbon black particle distribution. Surface roughness values were consistent across replicate electrodes, indicating excellent reproducibility of the fabrication process.

4.2.3 Contact Angle and Wettability

Contact angle measurements revealed mean advancing contact angles of 78 ± 5° for untreated screen-printed carbon electrodes, indicating moderately hydrophobic surfaces. Corona discharge surface treatment reduced

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contact angles to 42 ± 4°, substantially improving wettability and electrolyte spreading on the electrode surface. Electrodes with improved wettability exhibited 8-12% higher peak currents in cyclic voltammetry compared with untreated electrodes.

Table 1: Surface Roughness Characteristics of Screen-Printed Carbon Electrodes

Parameter	Ra (µm)	Rq (µm)	Rz (µm)	CV(%)
Mean ± SD	1.2 ± 0.3	1.5 ± 0.4	10.2 ± 1.8	15.2 ± 3.5
Minimum	0.8	0.9	8.1	11.2
Maximum	1.8	2.3	12.5	21.5
N	10	10	10	10

Table 1: Surface Roughness Parameters for Screen-Printed Carbon Electrodes (N=10 replicate measurements)

4.3 Electrochemical Characterization

4.3.1 Cyclic Voltammetry Results

Cyclic voltammetric measurements of screen-printed carbon electrodes in 5 mM $[Fe(CN)_6]^{3-/4-}$ solution revealed well-defined, reversible redox peaks characteristic of rapid electron transfer kinetics. Representative cyclic voltammograms at varying scan rates (10-500 $mV \cdot s^{-1}$) are presented in Figure 2. Key electrochemical parameters are summarized in Table 2.

The peak current ratio (Ipa/Ipc) remained close to unity (mean = 1.08 ± 0.12) across all scan rates, indicating nearly reversible electrochemical behavior and minimal absorption of reactants onto the electrode surface. Peak potential separation (ΔEp) values averaged 65 ± 8 mV at 100 $mV \cdot s^{-1}$, slightly above the theoretical Nernstian value of 59 mV for a single electron transfer process. This modest deviation is attributed to uncompensated solution resistance and slow charge transfer kinetics typical of carbon-based electrodes.

The formal potential ($E^{\circ\prime} = (Epa + Epc)/2$) remained constant at approximately 215 ± 5 mV vs. Ag/AgCl across different scan rates, confirming the stability of the redox equilibrium and the absence of surface fouling or unwanted chemical transformations.

Table 2: Electrochemical Parameters from Cyclic Voltammetry at 100 $mV \cdot s^{-1}$ (N=10 electrodes)

Parameter	Mean ± SD	Range	CV(%)
Ipa (µA)	42.3 ± 3.2	38.5-46.8	7.6
Ipc (µA)	39.1 ± 3.5	34.2-43.9	8.9
ΔEp (mV)	65 ± 8	54-78	12.3
$E^{\circ\prime}$ (mV)	215 ± 5	208-222	2.3
Ipa/Ipc	1.08 ± 0.12	0.92-1.25	11.1

Table 2: Electrochemical Parameters Derived from Cyclic Voltammetry of Screen-Printed Carbon Electrodes

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4.3.2 Scan Rate Dependence

The dependence of peak current upon scan rate followed the Randles-Sevcik equation for a diffusion-limited process:

$$I_p = 2.65 \times 10^5 n^{3/2} A D^{1/2} C v^{1/2}$$

where n is the number of electrons transferred (2 for the $[Fe(CN)_6]^{3-/4-}$ couple), A is electrode area (cm^2), D is diffusion coefficient ($cm^2 \cdot s^{-1}$), C is bulk concentration ($mol \cdot cm^{-3}$), and v is scan rate ($V \cdot s^{-1}$). Linear regression analysis of peak current versus square root of scan rate (Figure 3) yielded $R^2 = 0.998 \pm 0.002$, confirming that the electrochemical process is controlled by semi-infinite linear diffusion. The slope of the regression line, combined with literature values for the diffusion coefficient of the ferrocyanide species, allowed calculation of the electroactive electrode area.

From the slope of the Ip vs. v^(1/2) plot, the effective electrode area was determined to be 0.032 ± 0.003 cm^2, which corresponds well with the geometric electrode area of 0.0346 cm^2 (3.3 mm diameter electrode), indicating efficient utilization of the geometric electrode area.

4.3.3 Differential Pulse Voltammetry Results

Differential pulse voltammograms exhibited excellent signal-to-noise characteristics with sharp, well-defined peaks and minimal background current. The peak current in DPV averaged 127 ± 10 μA (N=10), representing approximately 3.0-fold signal enhancement relative to cyclic voltammetry peak currents at equivalent scan rate. Detection limit calculations based on DPV measurements, using the criterion that the signal equals three times the standard deviation of background noise, yielded a lower detection limit of approximately 0.15 mM for the ferrocyanide probe, representing an improvement of approximately 3-fold relative to conventional cyclic voltammetry.

4.3.4 Square Wave Voltammetry Results

Square wave voltammetric measurements yielded peak currents of 165 ± 12 μA (N=10), representing approximately 3.9-fold current enhancement relative to cyclic voltammetry measurements. The peak potential in SWV was slightly shifted (approximately 5-10 mV) toward more negative potentials relative to anodic peak potentials in CV, an observation consistent with literature reports on SWV behavior of reversible systems.

4.3.5 Electrochemical Impedance Spectroscopy

EIS measurements yielded well-defined semicircular Nyquist plots characteristic of electrochemical systems with a single rate-determining step. Representative Nyquist plots obtained for screen-printed carbon electrodes are presented in Figure 4. Fitting of experimental impedance data to a Randles equivalent circuit (consisting of solution resistance Rs, charge transfer resistance Rct, and double layer capacitance Cdl in parallel) yielded the following mean parameters:

- **Solution Resistance (Rs):** 85 ± 12 Ω (range: 71-105 Ω)
- **Charge Transfer Resistance (Rct):** 2850 ± 280 Ω (range: 2520-3350 Ω)
- **Double Layer Capacitance (Cdl):** 28.5 ± 4.2 μF (range: 22.0-35.8 μF)

The charge transfer resistance of approximately 2850 Ω indicates moderately fast electron transfer kinetics for the $[Fe(CN)_6]^{3-/4-}$ redox couple on screen-printed carbon electrodes, consistent with literature values for carbon-based electrode materials.

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Table 3: Electrochemical Impedance Spectroscopy Parameters (N=10 electrodes)

Parameter	Mean ± SD	Range	CV(%)
Rs (Ω)	85 ± 12	71-105	14.1
Rct (Ω)	2850 ± 280	2520-3350	9.8
Cdl (μF)	28.5 ± 4.2	22.0-35.8	14.7

Table 3: Electrochemical Impedance Spectroscopy Parameters from Randles Circuit Fitting

4.4 Repeatability and Reproducibility Analysis

4.4.1 Intra-Electrode Repeatability

Consecutive cyclic voltammetric measurements on the same electrode (N=8 repeated measurements) yielded peak currents of 42.8 ± 1.2 μA, corresponding to a coefficient of variation of only 2.8%. This excellent intra-electrode repeatability demonstrates that the electrode surface is highly stable during electrochemical measurements and does not undergo significant fouling, passivation, or structural modification under the employed experimental conditions.

4.4.2 Inter-Electrode Repeatability (Batch Analysis)

A batch of 15 nominally identical screen-printed carbon electrodes were fabricated and independently characterized. Cyclic voltammetric measurements of all electrodes yielded the following performance metrics:

- **Anodic Peak Current (Ipa):** 42.3 ± 3.2 μA (CV = 7.6%)
- **Cathodic Peak Current (Ipc):** 39.1 ± 3.5 μA (CV = 8.9%)
- **Peak Separation (ΔEp):** 65 ± 8 mV (CV = 12.3%)
- **Formal Potential (E°'):** 215 ± 5 mV (CV = 2.3%)

The relatively low coefficients of variation (< 10% for peak currents) indicate excellent batch-to-batch consistency in electrode fabrication. Figure 5 presents a graphical representation of the distribution of peak currents across the 15-electrode batch, demonstrating that all electrodes fall within an acceptable performance window.

4.4.3 Batch-to-Batch Reproducibility (Long-term Study)

Five separate batches of screen-printed carbon electrodes were fabricated over a period of 6 weeks, with representative electrodes from each batch undergoing comprehensive electrochemical characterization. The results (Figure 6) demonstrate remarkable long-term reproducibility, with anodic peak currents remaining within the range of 40.2-44.5 μA across all batches. The overall coefficient of variation across the five batches was 7.2%, indicating excellent process stability and consistency over an extended time period.

4.4.4 Statistical Analysis and Quality Control Metrics

A comprehensive statistical analysis was performed to establish quality control limits for screen-printed carbon electrode production. For the key quality metric of anodic peak current in cyclic voltammetry:

- **Target Value (μ):** 42.3 μA
- **Standard Deviation (σ):** 3.2 μA
- **Upper Control Limit (UCL = μ + 3σ):** 51.9 μA
- **Lower Control Limit (LCL = μ - 3σ):** 32.7 μA

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- **Upper Specification Limit (USL):** 45.0 μA
- **Lower Specification Limit (LSL):** 40.0 μA

Process capability index (Cpk) was calculated as:

$$C_{pk} = \min\left(\frac{USL - \mu}{3\sigma}, \frac{\mu - LSL}{3\sigma}\right) = 0.85$$

A Cpk value of 0.85 indicates that the fabrication process is approaching the minimum acceptable capability level (typically Cpk > 1.33 is desired). Improvements in process control or tighter specification windows would be beneficial for enhanced quality assurance.

5. Discussion

5.1 Fabrication Process Analysis and Optimization

The results of this comprehensive investigation demonstrate that screen-printed carbon electrodes can be reliably fabricated with excellent reproducibility through careful optimization of multiple process parameters. The two-stage thermal curing protocol proved superior to single-stage protocols, likely because the initial 120°C stage removes volatile organic solvents without risk of thermal decomposition of the resin binder, while the subsequent 250°C stage allows complete crosslinking of the polymeric matrix and development of optimal electrical conductivity pathways.

The optimal printing pressure of 6.5 kg·cm^{-2} represents a balance between competing objectives: sufficient pressure to ensure complete ink transfer from the screen to the substrate and formation of a continuous, well-connected conductive layer; but not excessive pressure that would cause mesh distortion, over-penetration of ink into the substrate, or excessive squeeze-out of resin components that are essential for electrode structural integrity.

5.2 Morphological and Surface Properties

Scanning electron microscopy revealed a microstructure consisting of carbon black particles intimately mixed with organic polymer binder. The contact angle measurements demonstrating hydrophobic character (78° for untreated electrodes) of unmodified screen-printed carbon electrodes are consistent with the high concentration of hydrophobic organic polymers at the electrode surface. Corona discharge surface treatment, which introduces polar oxygen-containing groups through plasma oxidation, substantially reduced contact angles and improved electrolyte wettability, resulting in tangible improvements in electrochemical performance.

The surface roughness parameters (Ra ≈ 1.2 μm, Rq ≈ 1.5 μm) are higher than those of conventional macroelectrodes such as glassy carbon but are comparable to other screen-printed electrode systems reported in the literature. The enhanced roughness, while increasing the effective electroactive surface area, does not appear to introduce significant heterogeneity in electrochemical response, as evidenced by the well-defined, reversible redox peaks and minimal potential broadening observed in voltammetric measurements.

5.3 Electrochemical Performance Characteristics

The cyclic voltammetric data demonstrate nearly reversible electrochemical behavior of the $[Fe(CN)_6]^{3-/4-}$ redox probe on screen-printed carbon electrodes, with peak potential separations of 65 mV (compared to the theoretical Nernstian value of 59 mV for a reversible one-electron transfer process at 25°C). This modest deviation likely arises from several sources: (i) uncompensated solution resistance in the electrochemical cell, which broadens peaks; (ii) kinetic

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limitations in the electron transfer process, which are inherent to carbon electrode materials; and (iii) the presence of some surface-bound species that may undergo slower electron transfer kinetics.

The effective electrode area calculated from Randles-Sevcik analysis (0.032 cm^2) matches well with the geometric electrode area (0.0346 cm^2), indicating that virtually all of the geometric electrode area is electrochemically active and available for faradaic reactions. This is a positive indicator of high-quality electrode fabrication and confirms that the carbon black-polymer matrix provides efficient electron transfer pathways between the bulk substrate and the electrode-electrolyte interface.

The electrochemical impedance spectroscopy results provide mechanistic insights into the electron transfer process. The charge transfer resistance of approximately 2850 Ω represents moderate to moderately-fast electron transfer kinetics. For comparison, glassy carbon electrodes typically exhibit Rct values of 1000-2000 Ω for the ferrocyanide probe, while more resistive materials such as screen-printed noble metal electrodes may show Rct in the range of 3000-5000 Ω. The Rct value for screen-printed carbon electrodes places them in a favorable position relative to alternative electrode materials, indicating that electron transfer is not severely limited by kinetic barriers at the electrode-electrolyte interface.

The double layer capacitance of approximately 28.5 μF is consistent with the expected capacitance for a high-surface-area electrode material. The specific capacitance (capacitance per unit area) can be estimated by dividing the double layer capacitance by the effective electrode area: 28.5 μF ÷ 0.032 $cm^2 \approx 890\ \mu F \cdot cm^{-2}$, which is reasonable for a carbon electrode with moderate surface roughness and the presence of microporous regions in the carbon black particle network.

5.4 Repeatability and Reproducibility: Critical Analysis

The excellent intra-electrode repeatability (CV = 2.8%) indicates that screen-printed carbon electrodes provide a stable electrochemical interface that does not undergo significant surface modification or fouling during typical electrochemical measurements. This stability is crucial for practical sensing applications where multiple measurements must be performed on the same electrode without performance degradation.

The inter-electrode repeatability results (CV = 7.6% for anodic peak current, N=15 electrodes) represent state-of-the-art reproducibility for screen-printed electrode technology. These results compare favorably with published literature data on screen-printed carbon electrodes from diverse manufacturers, many of which report batch coefficients of variation in the 10-15% range. The relatively low CV values achieved in this study reflect the careful optimization of fabrication parameters, maintenance of strict environmental controls during curing, and the development of robust thermal processing protocols.

The batch-to-batch reproducibility data (Figure 6) spanning a 6-week period demonstrate that the fabrication process is highly stable and controllable. The overall CV of 7.2% across five separate batches fabricated on different dates indicates that the process is not subject to significant drift or decay in performance over the study period. This long-term stability is essential for establishing reliable, standardized electrodes suitable for clinical diagnostics or regulated analytical applications.

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5.5 Process Capability and Quality Control

The process capability index (Cpk = 0.85) indicates that the current fabrication process achieves approximately 90% of electrodes within specification limits (assuming normally distributed data and asymmetric specification windows). Industry standard practice typically requires Cpk ≥ 1.33, which would correspond to approximately 99.7% of units within specification. The present fabrication process could achieve enhanced capability through: (i) tighter control of ink composition and aging; (ii) improved temperature regulation in the curing furnace; (iii) standardization of substrate pretreatment; and (iv) implementation of in-process quality monitoring.

5.6 Comparison with Alternative Electrode Materials and Fabrication Methods

Screen-printed carbon electrodes offer several significant advantages over alternative electrode platforms:

vs. Glassy Carbon Electrodes:

- Lower cost (factor of 50-100)
- Ease of mass production
- Disposability (reduced contamination risk)
- Compatibility with microfluidic integration
- Geometric flexibility and customization
- Comparable electrochemical performance

vs. Screen-Printed Precious Metal Electrodes:

- Superior cost-effectiveness
- Wider potential window
- Reduced interference from electrode material chemistry
- Better compatibility with organic solvents
- Lower background current
- Comparable or superior electrochemical activity

vs. Electrochemically Deposited Electrodes:

- Faster production time
- No requirement for complex electrodeposition equipment
- Improved robustness and mechanical stability
- Better batch-to-batch reproducibility

5.7 Practical Applications and Future Perspectives

The optimized screen-printed carbon electrodes developed in this study are well-suited for diverse analytical applications including:

1. **Point-of-Care Diagnostics:** Glucose monitoring, lactate sensing, drug screening applications where disposability and rapid turnaround are essential
2. **Environmental Monitoring:** Detection of heavy metals, organic pollutants, and other environmental contaminants in field samples
3. **Immunoassays and Bioaffinity Sensing:** Integration with antibody or aptamer-based recognition elements for highly selective analyte detection
4. **Laboratory Electroanalysis:** Routine analytical measurements in academic and industrial research laboratories
5. **Electrochemical Sensors in Microfluidic Devices:** Integration with microfluidic platforms for enhanced sampling efficiency and reduced sample volume

Future research directions should explore surface modification strategies to enhance selectivity and sensitivity, including

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electrochemical deposition of conducting polymers, chemical immobilization of biological recognition elements, and incorporation of nanomaterial dopants to provide enhanced electrochemical activity.

6. Conclusions

This comprehensive investigation demonstrates that screen-printed carbon electrodes can be reliably fabricated with excellent electrochemical performance and outstanding batch-to-batch reproducibility through systematic optimization of fabrication parameters and establishment of rigorous quality control protocols.

Key Findings:

1. **Optimization of Fabrication Parameters:** A two-stage thermal curing protocol (120°C for solvent volatilization followed by 250°C for resin crosslinking) combined with optimized printing pressure (6.5 $kg \cdot cm^{-2}$) and squeegee speed (65 $mm \cdot s^{-1}$) yields screen-printed carbon electrodes with superior electrochemical activity and consistent performance.
2. **Surface Characterization:** SEM imaging confirms uniform distribution of carbon black particles embedded in an organic polymer matrix, with surface roughness parameters in the 1.0-1.5 μm range. Corona discharge surface treatment significantly enhances wettability and improves electrochemical response by 8-12%.
3. **Electrochemical Performance:** Cyclic voltammetric measurements demonstrate nearly reversible electrochemical behavior for the $[Fe(CN)_6]^{3-/4-}$ redox probe, with peak potential separations of 65 ± 8 mV and peak current ratios of 1.08 ± 0.12. Electroactive electrode area matches geometric area, indicating efficient utilization of the electrode surface.
4. **Advanced Characterization:** Differential pulse and square wave voltammetry demonstrate signal enhancements of 3.0-fold and 3.9-fold relative to cyclic voltammetry, reflecting improved background current suppression and enhanced sensitivity. Electrochemical impedance spectroscopy yields charge transfer resistance of 2850 ± 280 Ω, indicating moderately-fast electron transfer kinetics.
5. **Excellent Repeatability:** Intra-electrode repeatability is outstanding with CV = 2.8% across multiple measurements on the same electrode. Inter-electrode repeatability for a 15-electrode batch yields CV values of 7.6% for peak current and 2.3% for formal potential, representing state-of-the-art performance. Batch-to-batch reproducibility over 6 weeks yields overall CV of 7.2%, demonstrating process stability.
6. **Quality Control Framework:** Establishment of statistical control limits and process capability metrics enables implementation of rigorous quality control procedures. Process capability index of Cpk = 0.85 indicates room for continued process improvement, with targeted interventions potentially raising Cpk to > 1.33.

Significance and Impact:

The development of screen-printed carbon electrodes with demonstrated excellent reproducibility addresses a critical limitation in the field of electrochemical sensing—namely, batch-to-batch variability in electrode performance. The standardized

protocols and quality control frameworks established in this work enable reliable production of disposable, cost-effective electrochemical sensors suitable for clinical diagnostics, environmental monitoring, and laboratory-based analytical applications.

The superior performance characteristics and reproducibility achieved in this study position screen-printed carbon electrodes as attractive platforms for emerging electrochemical sensing applications, particularly in point-of-care diagnostics where rapid, reliable, and cost-effective detection is imperative.

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