WORKSHOP ON BIOSENSORS & ELECTROANALYTICAL TECHNIQUES

Electrochemical Impedance Spectroscopy:

Experiments using Rotating Disc Electrode (RDE)

Date :27th June 2023

Simple electron Transfer Reactions

$$\left[Fe(CN)_6\right]^{4-} \rightleftarrows \left[Fe(CN)_6\right]^{3-} + e^-$$

$$rate = k_f\left[Fe^{2+}\right] - k_r\left[Fe^{3+}\right]$$

$$k_f = k_f^0 e^{b_f E_{dc}} \qquad k_r = k_r^0 e^{b_r E_{dc}}$$

$$b_f = \frac{nF\alpha}{RT} \qquad b_r = \frac{-(1-\alpha)nF}{RT}$$

Any electrochemical reaction rate is

$$rate = f\left(T, E_{dc}, Conc.\right)$$

Working Electrode: Gold Disc

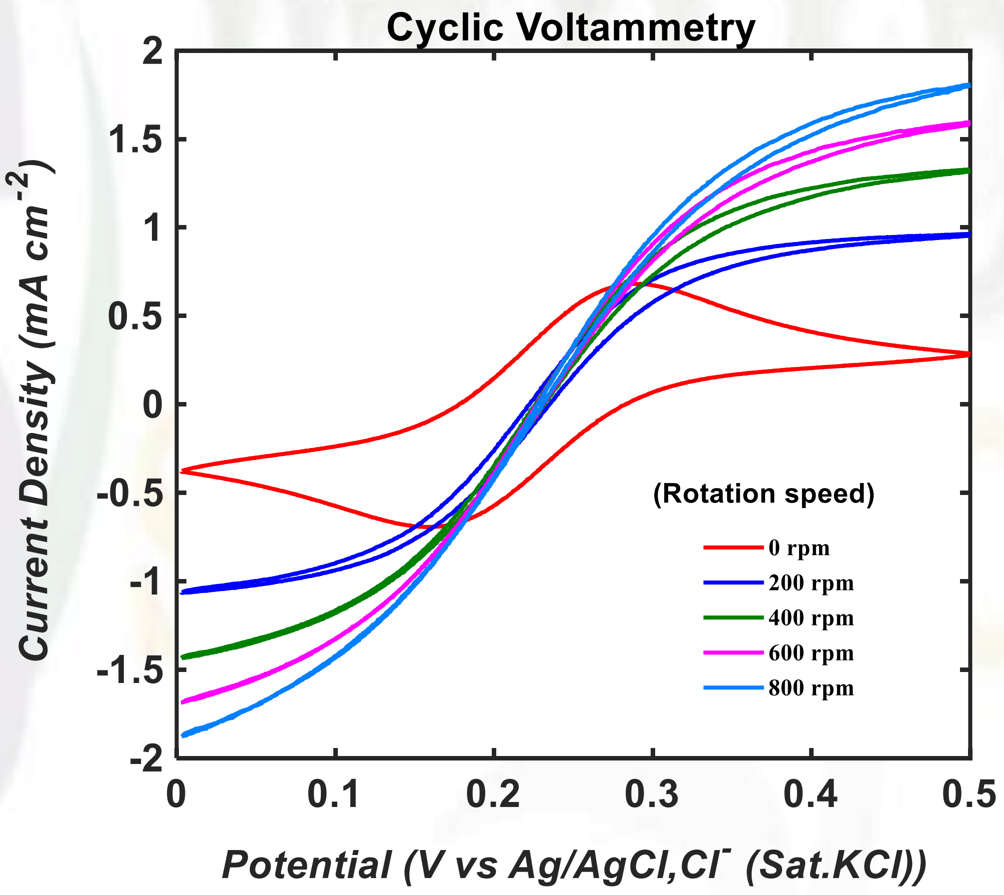
Reference Electrode: Ag/AgCl, Cl^- (Sat. KCl)

Counter electrode: Pt Mesh

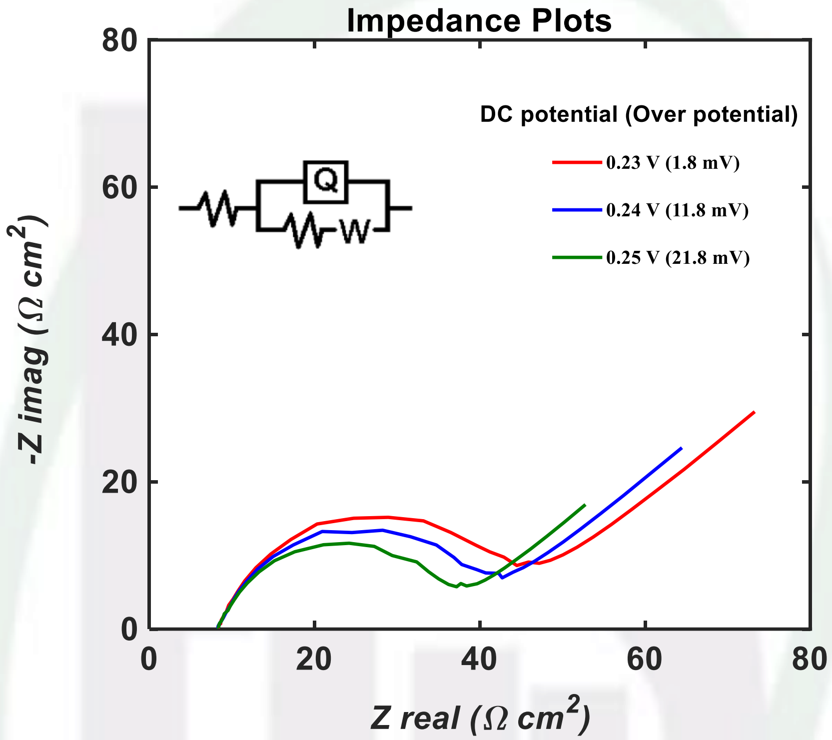
Electrolyte : 5 mM potassium Ferrocyanide + 5 mM potassium Ferricyanide

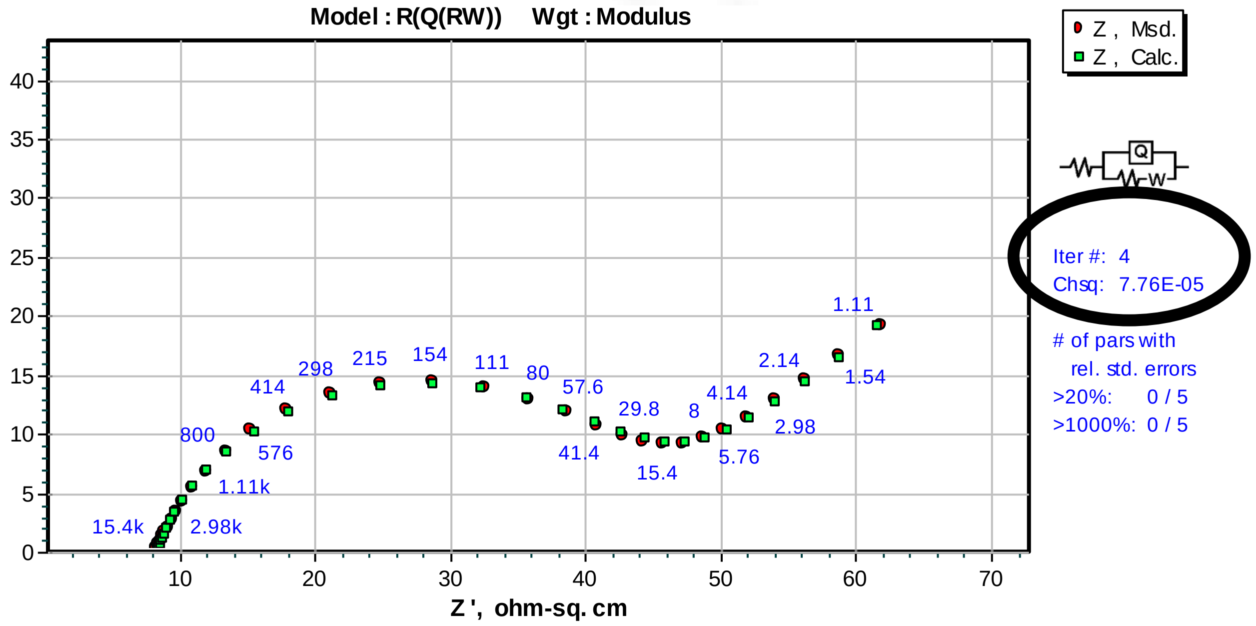
Supporting Electrolyte: 0.1 M Na_2SO_4

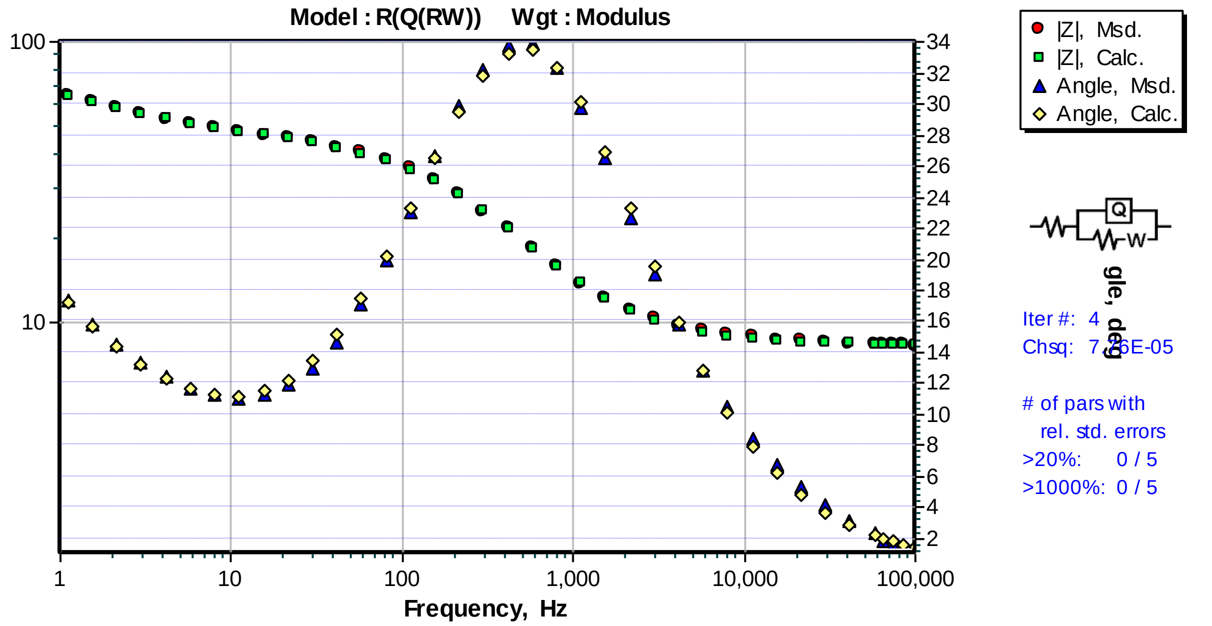
Electrochemical Studies using Rotating Disc Electrode



Impedance Experiments with stationary Electrode

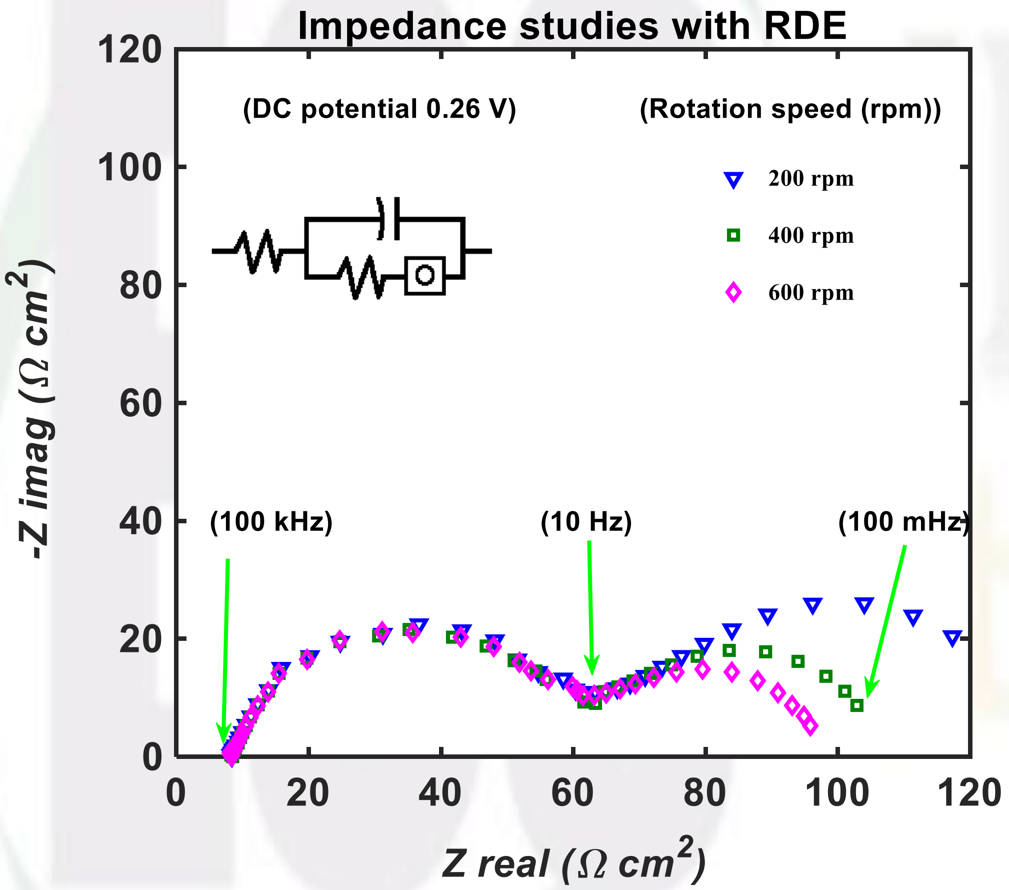


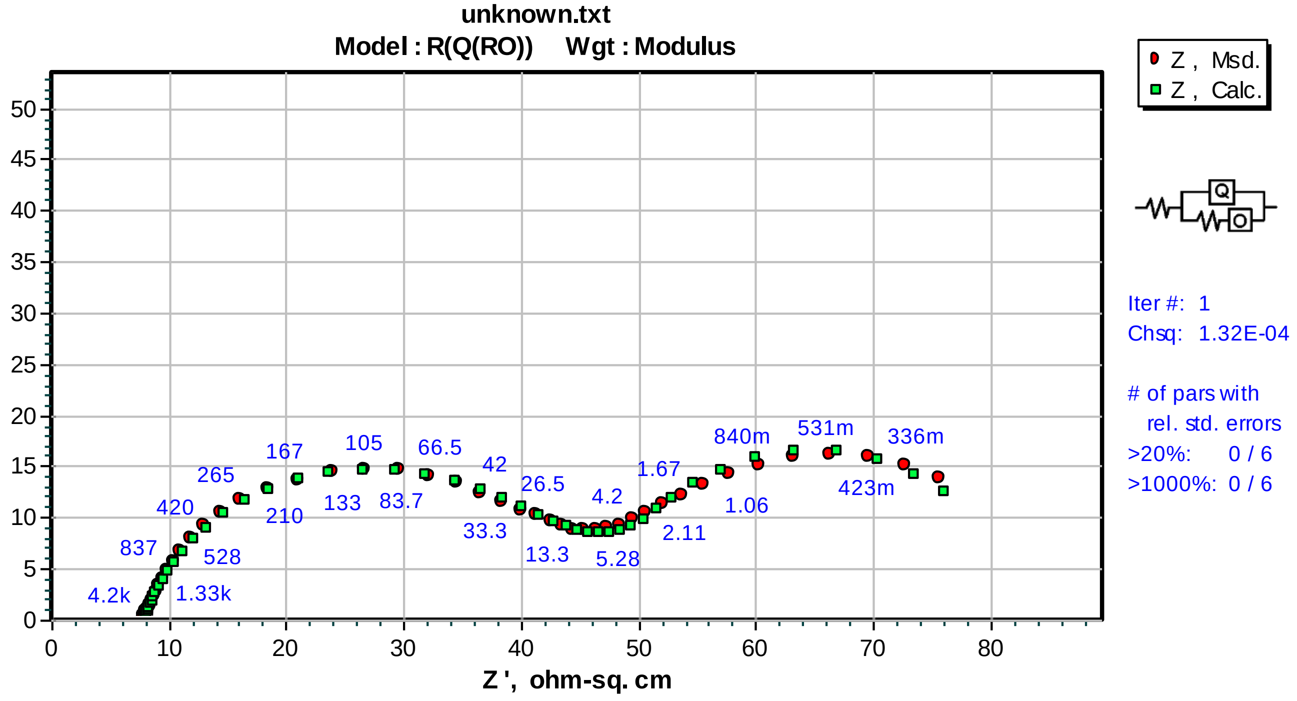




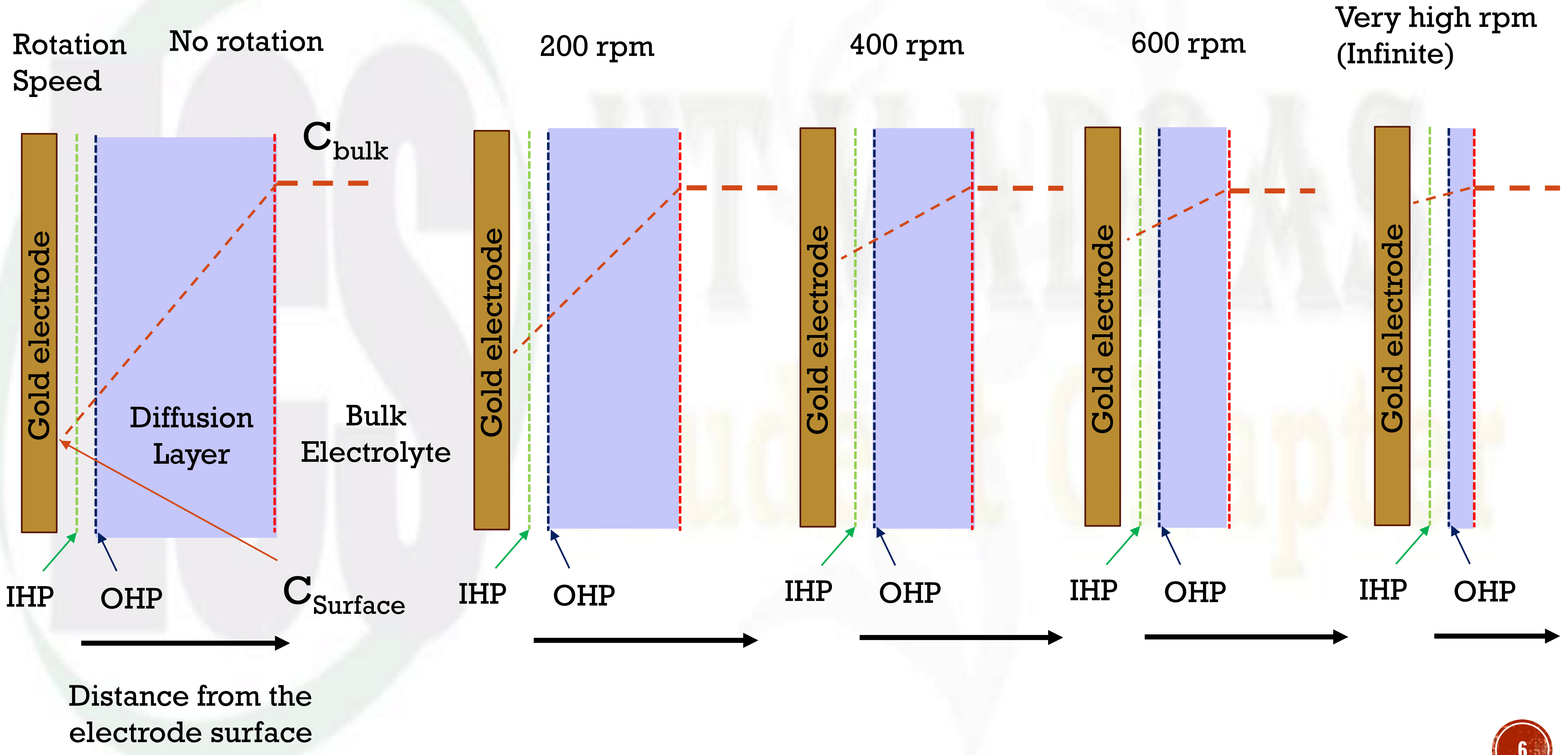
Parameters(0.24V)	Values
R	8.33
Q-Y_o	7.96E-5
Q-n	0.82
R	35.94
W	1.45E-2

Impedance Experiments with Rotating Electrode

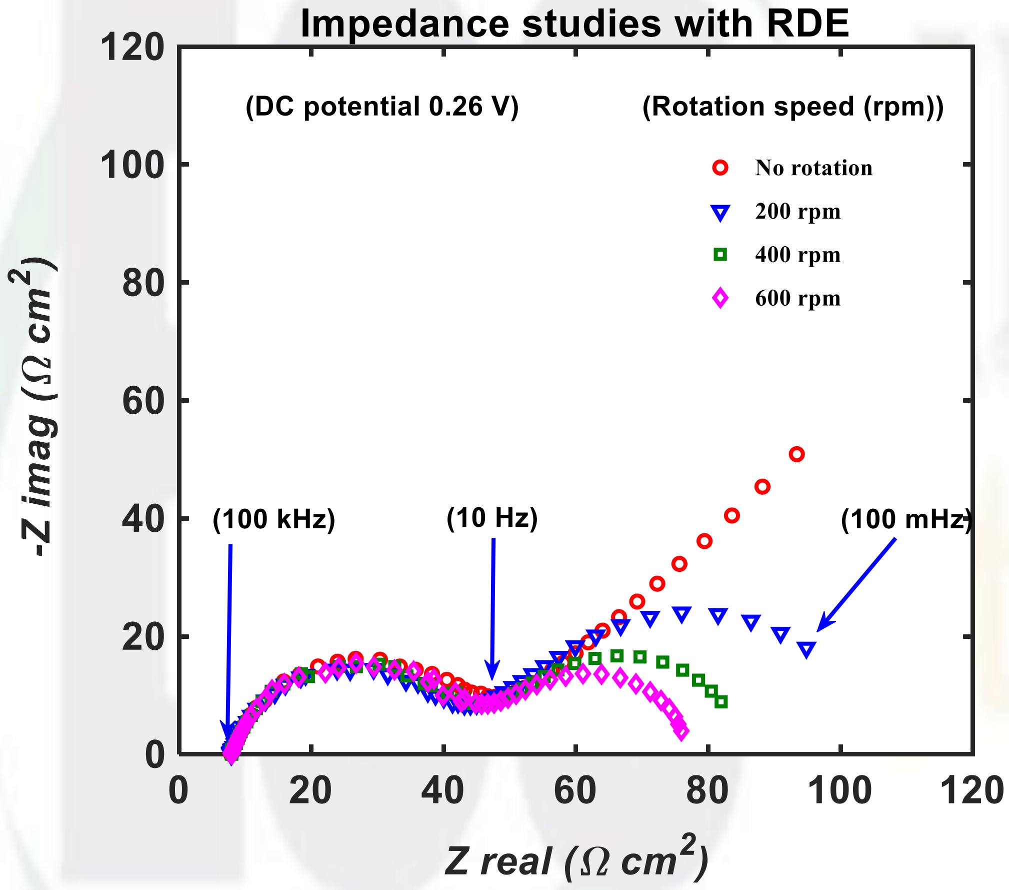




Boundary layer Thickness with rotation speed



INFINITE AND FINITE WARBURG



Infinite Warburg

$$Z_W = \frac{W}{\sqrt{j\omega}}$$

Finite Warburg

$$Z_{w-o} = \frac{W}{\sqrt{j\omega}} \tanh\left(B\sqrt{j\omega}\right)$$

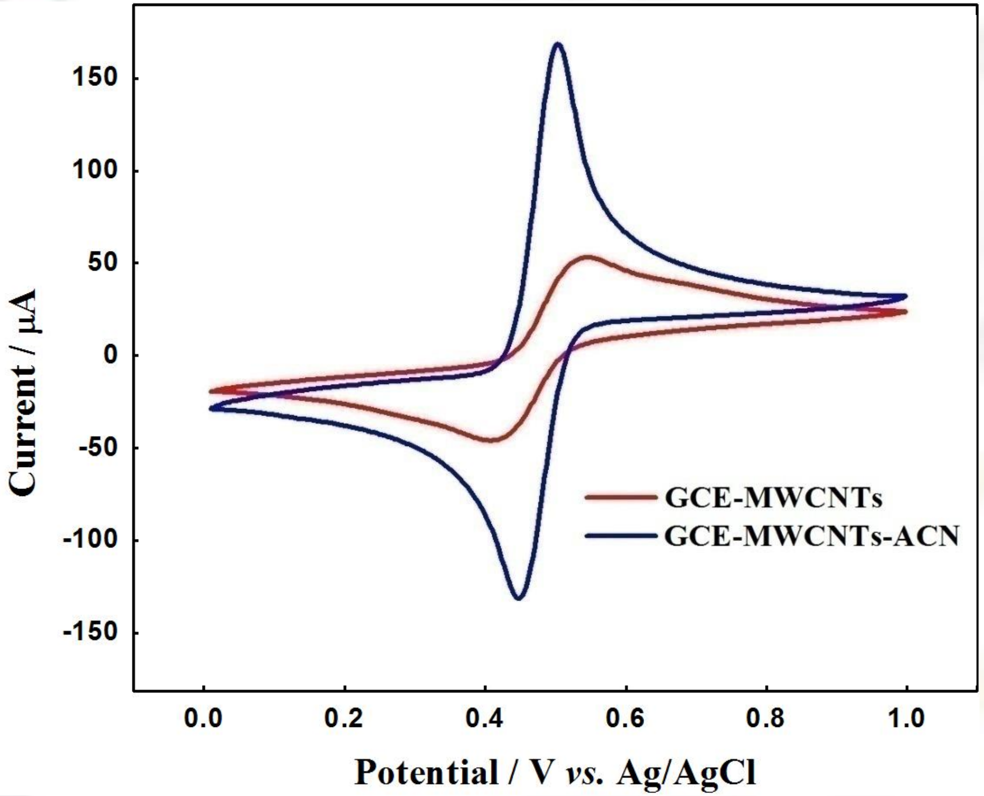
$$B = \frac{\delta}{\sqrt{D}}$$

WORKSHOP ON BIOSENSORS
&
ELECTROANALYTICAL TECHNIQUES

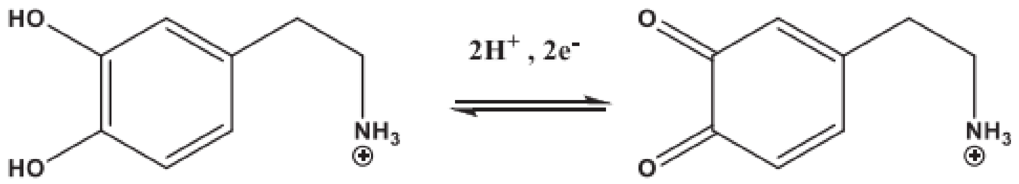
Cyclic Voltammetry and Amperometry

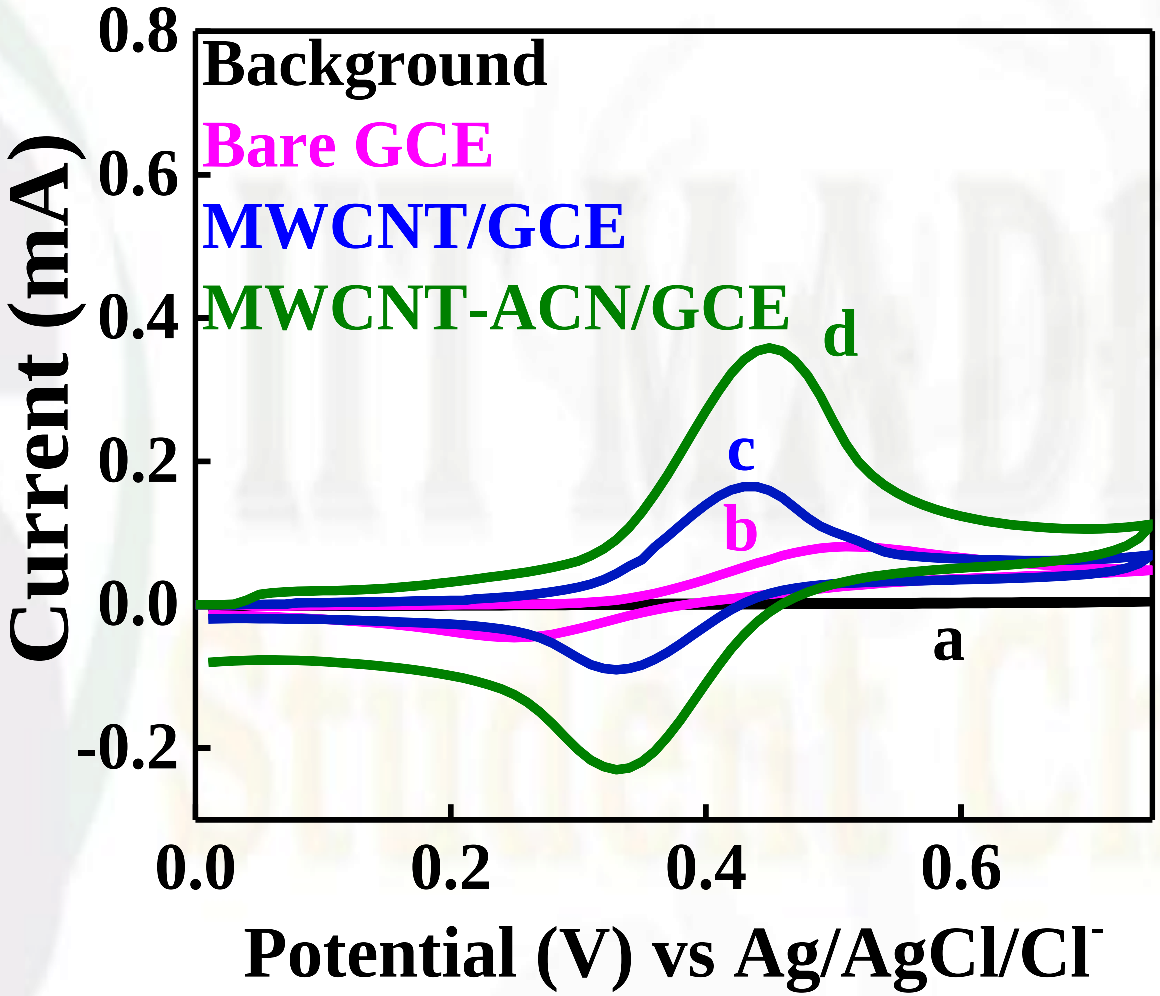
Date: 27th June 2023

Electrochemical response of dopamine (DA) on MWCNTs and MWCNTs filled with ACN



CV response of 1 mM DA in pH 4 solution at GCE, GCE-MWCNTs, and GCE-MWCNTs-ACN at a scan rate of 50 mV s^{-1}





CV response of 3 mM DA in pH 4 solution at GCE, GCE-MWCNTs, and GCE-MWCNTs-ACN at a scan rate of 50 mV s^{-1}

Kinetic study

Randles-Sevcik equation

$$i_{peak} = (2.69 \times 10^5)\, n^{3/2} A\, C\, D^{1/2}\, \nu^{1/2}$$

where:

A — Electrode surface area (**cm^2**)

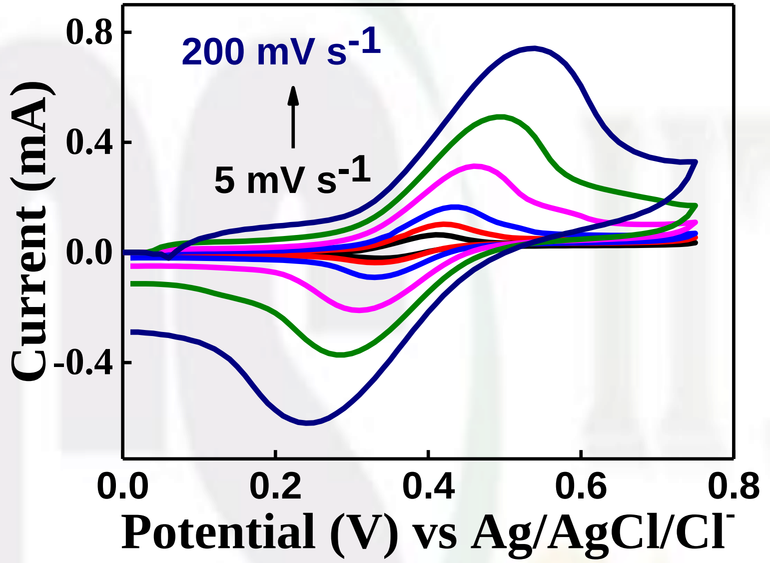
C — Bulk concentration of the analyte (**mol/cm^3**)

n — Number of electrons transferred in every species

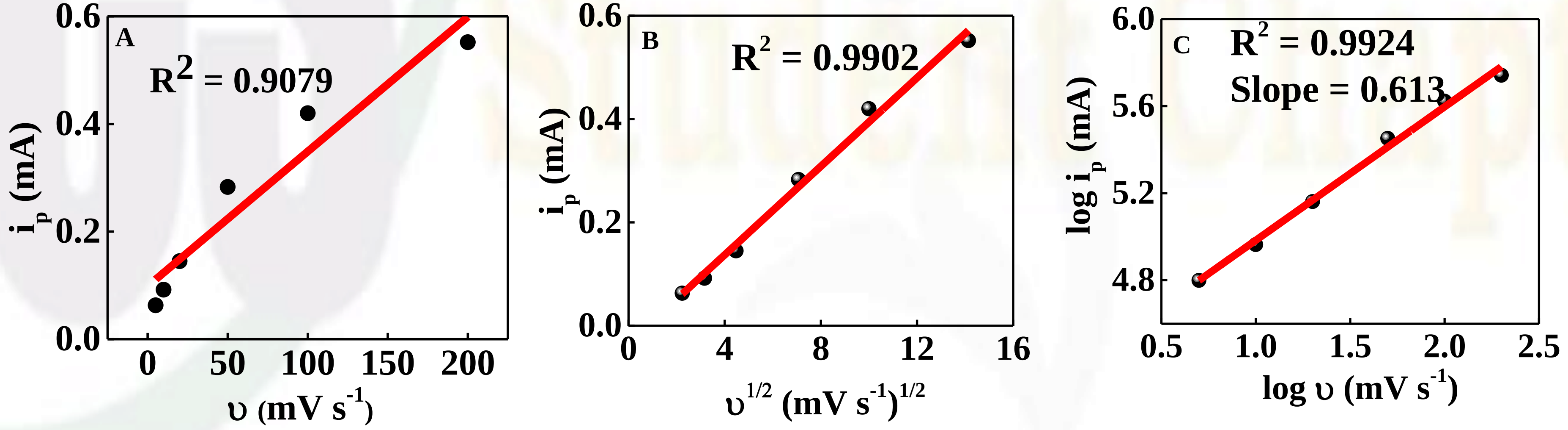
v — Scan rate (**V/s**)

D — Diffusion coefficient of the oxidized analyte (**cm^2/s**)

Effect of Scan rate towards electrochemical Sensing of Dopamine using GCE/MWCNTs

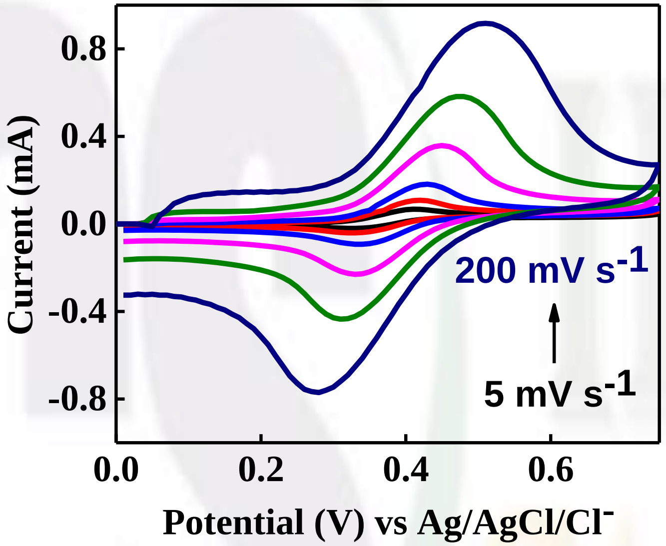


Cyclic voltametric responses of 3 mM DA at various scan rates (5 to 200 mV s^{-1})



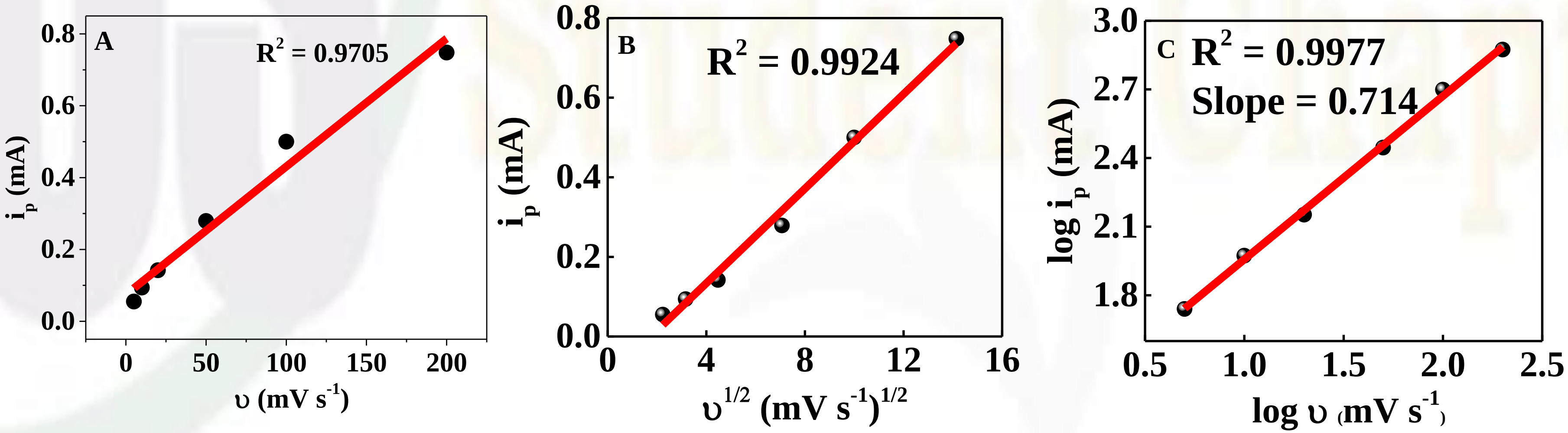
Linear plots: (A) i_p vs. υ; (B) i_p vs. $υ^{1/2}$; (C) log i_p vs. log υ

Effect of Scan rate towards electrochemical Sensing of Dopamine using GCE/MWCNTs-ACN



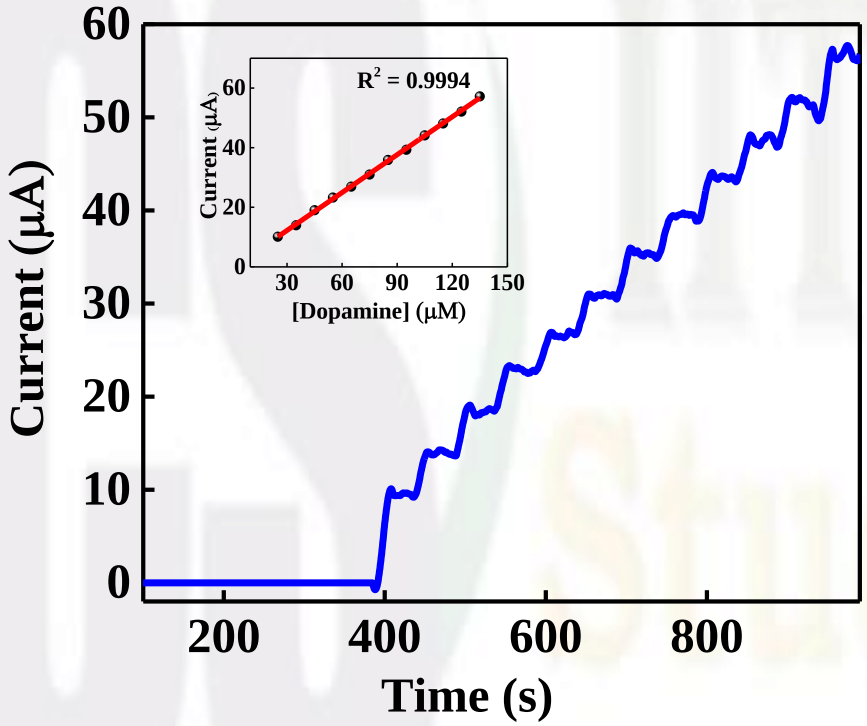
Cyclic voltametric responses of 3 mM DA at various scan rates (5 to 200 mV s^{-1})

$D = 6.3 \times 10^{-5}\ cm^2/s$



Linear plots: (A) i_p vs. υ; (B) i_p vs. $υ^{1/2}$; (C) log i_p vs. log υ

Sensitivity Study



$$\text{LOD} = \frac{3 \times SD_{blank}}{\text{Slope}}$$

Std. Deviation = 0.37265

Slope = 0.423

Sensitivity = 0.423 μA / μM

LOD = 2.6 μM

Amperometric responses of DA in pH 4 phosphate buffer solution using GCE/MWCNTs-ACN.
E = 0.55 V (Inset plot: calibration plot of current vs. concentration of DA).

A workshop on bio-sensors, electro-analytical techniques and hands-on session

ECS IIT MADRAS Student Chapter

CONDUCTANCE VS CONDUCTIVITY

Effect of varying contact area for conductance and conductivity

With contact area= 56 cm² **With contact area= 43 cm²**

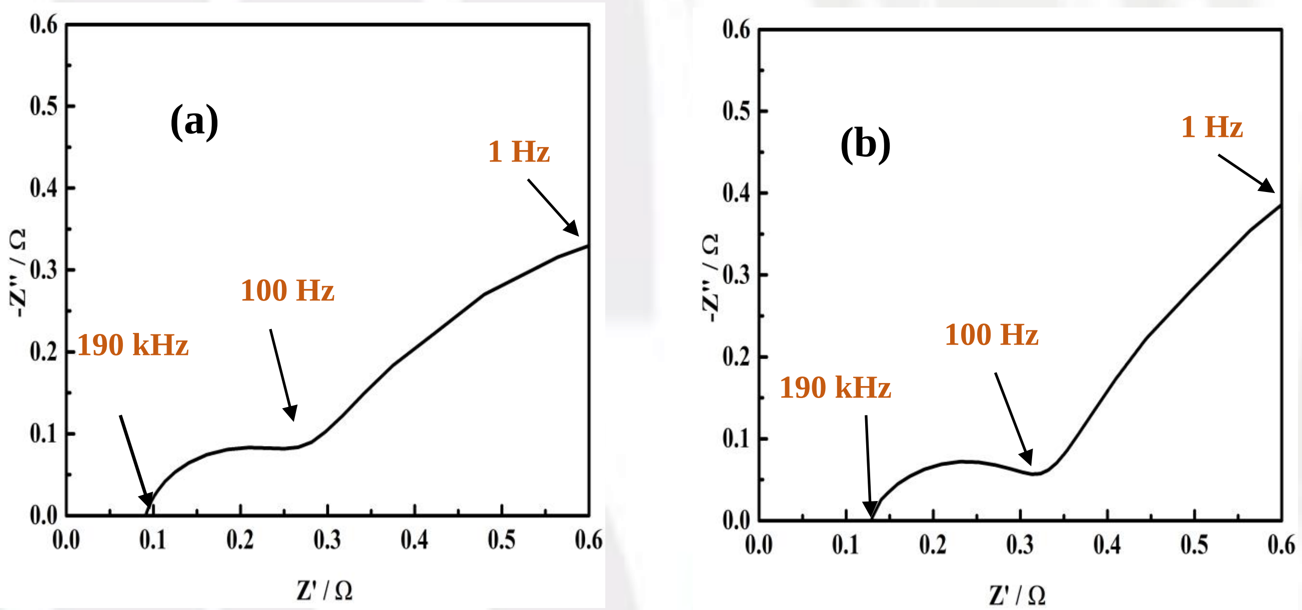


Fig. 1 Nyquist plots of Nafion ®-212 sandwiched between graphite blocks

Impedence for N-212 = 0.090 Ω

As RA = φL

0.090*56 = φ*0.0050

φ = 1012.5 Ω cm

Conductance (G) = 0.01 mS

Conductivity (k) $= 1\ mS\ cm^{-1}$

Impedence for N-212 = 0.129 Ω

As RA = φL

0.129*43 = φ*0.0050

φ = 1017.95 Ω cm

Conductance (G) = 0.007 mS

Conductivity (k) $= 1\ mS\ cm^{-1}$

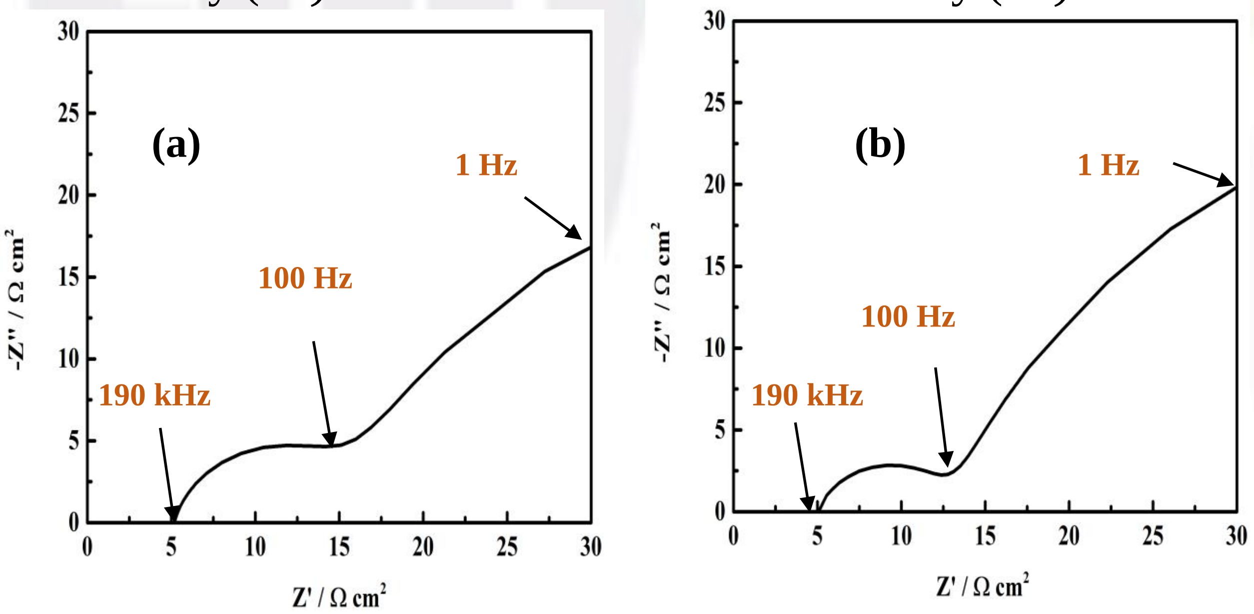


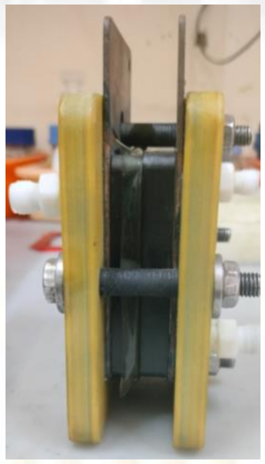
Fig. 2 Nyquist plots of Nafion ®-212 sandwiched between graphite blocks.

Resistance, **R = φL/A**

Where R= resistance of conductor, L= length of conductor, A=cross-sectional area of conductor, φ= resistivity of material.

Conductance (G) **G = 1/R**

Conductivity (k) **k = 1/φ**



(a)

Fig. 3 (a) Cell set-up to measure impendence of Nafion®-212.

Conclusion:

- In summary, conductance will vary with the change of length and cross-sectional area of the conductor, but conductivity is the intrinsic property of the material, will remain same.

THANK YOU