“CYCLIC VOLTAMMETRY”

Dissertation Report Submitted in partial fulfillment of the
requirements for the award of the Degree of

BACHELOR OF SCIENCE
OF
DAVANGERE UNIVERSITY

By

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S.J.M. ARTS, SCIENCE AND COMMERCE COLLEGE

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DEPARTMENT OF CHEMISTRY

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DECLARATION

I hereby declare that the Dissertation report entitled **"CYCLIC VOLTAMMETRY"** submitted to the Department of Chemistry, Davangere University, Davangere is a record of an original work done by me under the guidance of **Dr. Harshavardhan.A,** Dept. of Chemistry, SJM Arts, Science and Commerce College, Chandravalli, Chitradurga. Associated with the Davangere University and this Dissertation Report is submitted in the partial fulfillment for the award of Bachelor Degree in Chemisry by Davangere University.

I also declare that, this Report is the outcome of my own efforts and that it has not been submitted to any other university or institute for the award of any Degree or Diploma or Certificate.

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CERTIFICATE OF ORIGINALITY

Date:

This is to certify that the Dissertation report titled **"CYCLIC VOLTAMMETRY"** is an original work of **ASHA LAKSHMI.A, SRUSHTI.S, JAYALAKSHMI.G NEHA.D.C;** bearing University Register Number **Reg. No. U13SJ21S0051, Reg. No.U13SJ21S0035, Reg. No.U13SJ21S0058, Reg. No.U13SJ21S0028** and is being submitted in partial fulfillment for the award of the Bachelor Degree in Chemistry of Davangere University. The report has not been submitted earlier either to this university/ Institution for the fulfillment of the requirement of a course of study.

Signature of the Guide

Date:

Signature of HOD

Date:

ACKNOWLEDGEMENT

With immense pleasure, We take this opportunity to acknowledge the many revered people behind the success of our Dissertation report, which was a rewarding and memorable experience.

We express our gratitude and sincere thanks to Principal of our college and extend our earned reverence to **Panchakshari,** S.J.M. Arts, Science and Commerce College, Chandravalli, Chitradurga who has given an opportunity to do this Dissertation work.

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Our sincere thanks to go out to our beloved parents and friends and faculty members of our college for standing behind us by providing moral support and helping us in every possible way.

Place: Chitradurga
Date:

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CONTENTS

Introduction

This chapter describes the instrumentation, devices, the electrode systems with special emphasis on carbon paste electrodes, preparation and standardization of carbon paste electrodes and wax impregnated carbon paste electrodes, experimental techniques and procedures used in the present work. The basic equipment needed for electrochemistry is a potentiostat, a recording device, and an electrochemical cell. Everything inside the cell is chemistry and everything outside of it is electronics.

Potentiostat

The conventional three-electrode potentiostat is connected to the working, reference, and auxiliary electrodes immersed in the test solution placed in the cell. It controls the potential of the working electrode with respect to the reference electrode while simultaneously measuring the current flowing between the working electrode and the auxiliary electrode. The potentiostat performs three functions:

a. it controls the applied potential, which is potential difference between the WE and RE (the applied potential controls what half reactions occur at the WE);
b. it allows to pass current between the WE and AE without passing current through the RE (which would change its potential if current did pass through it) and
c. it converts the cell current to a voltage for recording devices.

A potentiostat must be able to bring the potential of the WE (with respect to the RE) to the desired level in a short enough time. The time taken by the potentiostat for controlling the working electrode potential is called the rise time. The potentiostat’s internal feedback circuits prevent all but a very small current from flowing between the working and reference electrodes. Because the very basis of voltammetry is the control of electrode potential, a function generator is required to provide the potential sweep or pulse sequence to be applied to the working electrode. Most modern potentiostats include a built-in sweep and / or pulse generator, and those which are interfaced to a computer usually rely on the computer to generate the desired waveform. The inputs to the potentiostat are the connections to the electrodes in the cell. The outputs from the potentiostat are signal lines reflecting the current and potential of the working electrode(s).

(a). Potentiostat employed in the present work for cyclic voltammetery and DPV experiments

The electrochemical experiments were carried out using potentiostat provided with the Data Acquisition PC interface Card fabricated at Analytical Chemistry Division, Bhabha Atomic Research Centre, Trombay, Mumbai, India; compatible with an IBM PC and coupled to a printer. This instrument is capable of performing more than six electroanalytical techniques. The instrument incorporates a high speed, high accuracy and an electrolysis mode that consists of high-gain operational amplifier with circuits for controlled potential.

The working electrode current signal is handled a bit differently. This signal line is also presented as a voltage signal, but the voltage level is actually proportional to the current flowing at the working electrode. The potentiostat has an internal ‘current converter’ circuit that performs the necessary current-to-voltage conversion automatically. The current converter has a number of ranges, and the operator is expected to choose the range most appropriate for the experiment being performed. Each range is associated with a particular proportionality constant, such as ‘100 mA/V’ or ‘1 mA/V.’

Electrochemistry

- Electron transfer plays a fundamental role in governing the pathways of chemical reactions.
- Electrochemistry is a powerful and sensitive analytical tool used for both qualitative and quantitative analysis over a wide range of concentrations.
- Numerous standard electrochemical methods exist that can be categorized into three general classes.
- Potentiometry, Coulometry, Voltammetry.

Cyclic Voltammetry

- Cyclic Voltammetry is the most versatile Electro analytical techniques for the study of electro active species.
- Cyclic Voltammetry (CV) means cyclysing the potential of an electrode from starting potential to end potential and end potential to starting potential.
- CV involves the cycling of potential of an electrode between two designated values called switching potential s in an unstirred solution & measuring the resultant current.

Fundamentals of cyclic Voltammetry

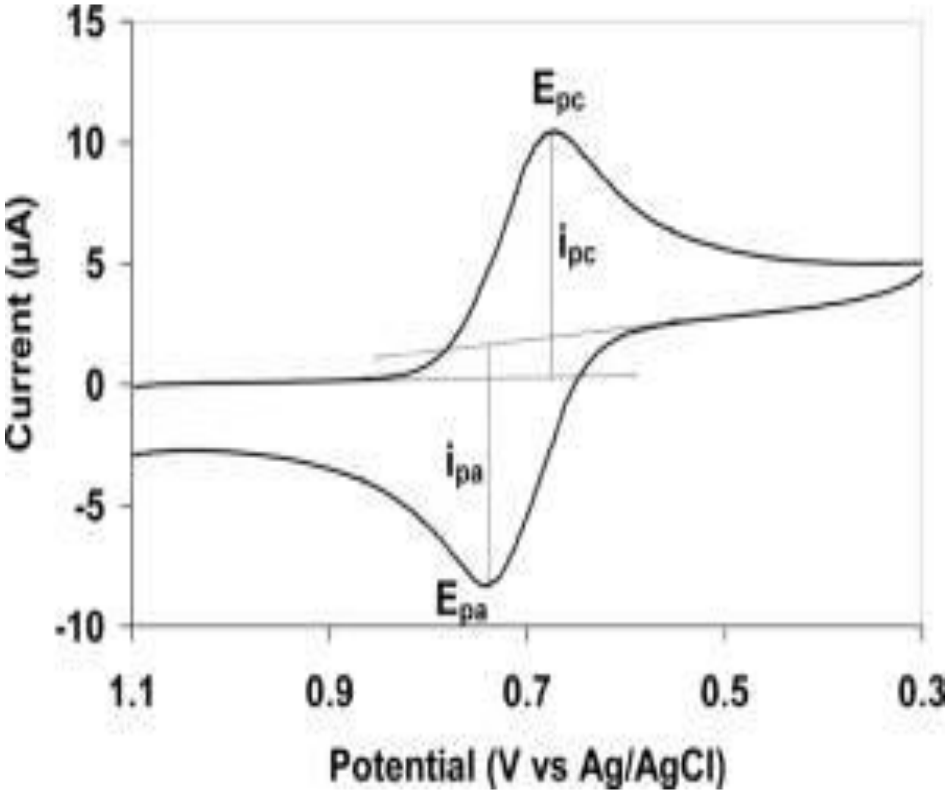
- Circuit
- Scan Rate
- Residual current
- Charging or non-Faradaic Current
- Faradaic Current

Electrodes used in CV

Three electrode system is used in CV are as follows.

- Working Electrode
- Reference Electrode
- Counter/Auxiliary Electrode
- Bare carbon-paste electrode and Modified carbon-paste electrode

Characteristic parameters of a cyclic Voltammogramz



Anodic peak potential (Epa)

Cathodic peak potential (Epc)

Anodic peak current (i_{pa})

Cathodic peak current (i_{pc})

1. INTRODUCTION

- Dopamine and serotonin are two important catecholamine neurotransmitters, present in the mammalian central nervous system.
- Dopamine (DA), a typical neurotransmitter, plays a significant role in the function of central nervous, renal, and hormonal system. A serious disease such as Schizophrenia, Huntington’s disease, and Parkinsonism may result from decrease in the concentration of Dopamine.
- Ascorbic acid (AA) is one of the monoamine neurotransmitters found in the central nervous system. The physiological functions such as sleep,thermoregulation,food intake, sexual activity, as well as psychopathological states such as depression, anxiety, alcoholism, and drug dependency were directly related to the concentration of serotonin. Therefore it is significant to develop sensitive and simple methods for the determination of Dopamine and Serotonin,

such as spectroscopy, chromatography and electrochemistry. Since dopamine and serotonin is an oxidizable compound it can be easily detectable by electrochemistry methods based on anodic oxidation.

- Carbon paste electrode was used widely in electrochemistry because it is attractive towards biologically active molecules , easy to preparation of modified electrode , renewability, low background current, and fast response.
- A number of modified carbon paste electrode were developed for the determination of Dopamine and Serotonin by using voltammetry technique.
- Modification of carbon paste electrode can be prepared by adding different types of modifiers. Modification can be done by grinding n agate mortar, by electro polymrisation. , and also immobilizations method
- The modified electrode has good electro catalytic activity such as sensitivity, selectivity, and also low detection limit when compared to traditional carbon paste electrode.

2. OBJECTIVES OF THE WORK

- Selectivity and specificity i.e. probing of specification, resulting from the applied potential.
- Selectivity from the choice of electrode material.
- High sensitivity and low detection limit resulting from the use of complex applied potential programmers.
- Possibility of giving results in real time or close to real time particularly in flow systems for on line monitoring.
- Study of interference bio-molecules such as uric acid, ascorbic acid and other neurotransmitters.
- Stability and reproducible studies of as prepared electrode materials for selected analytes.
- Application as miniaturized sensors, in situation where other sensors may not be usable.

3. METHODOLOGY

- Cyclic voltammetry (CV) was performed on Model EA-201 Electroanalyser (EA-201,Chemilink System). All the experiments were carried out in a conventional electrochemical cell.
- The conventional three-electrode potentiostat is connected to the working, reference, and auxiliary electrodes immersed in the test solution placed in the cell.
- It controls the potential of the working electrode (WE) with respect to the reference electrode (RE) while simultaneously measuring the current flowing between the WE and the auxiliary electrode (AE).
- A potentiostat must be able to bring the potential of the WE (with respect to the RE) to the desired level in a short enough time hence the time taken by the potentiostat for controling the WE potential is called the rise time
- Most modern potentiostats include a built-in sweep and / or pulse generator, and those which are interfaced to a computer usually rely on the computer to generate the desired waveform
- The inputs to the potentiostat are the connections to the electrodes in the cell. The outputs from the potentiostat are signal lines reflecting the current and potential of the WE(s).
- The electrode system contained a carbon paste working electrode (3.0 mm in diameter), a platinum wire counter electrode and a saturated calomel reference electrode (SCE).
- The carbon paste electrode was prepared as fallows 70 % graphite powder (particle size 50 mm and density is 20 mg/100 ml) 30 % silicone oil were mixed by hand to produce a homogeneous carbon paste electrode
- The carbon paste was then packed into the cavity of a homemade carbon paste electrode and smoothened on a weighing paper.

WORK CARRIED OUT SO FAR

Simultaneous electrochemical detection of dopamine in the presence of ascorbic acid using curcumin modified carbon paste electrode

1. Introduction

Ascorbic acid (AA) usually (Vitamin C) is present in both animal and plant kingdoms it is a significant vitamin in the diet of humans [1]. It is believed to be the primary antioxidant in human blood plasma. It is present in mammalian brain in the presence of DA and other neurotransmitters [2, 3]. Curcumin (CC) commonly known as turmeric and chemically it is diferuloylmethane, it is a polyphenol derived from the Curcuma longa plant [13]. CC has been used extensively in Ayurvedic medicine in India and also used as food supplement over many centurie. As it is nontoxic and has a variety of therapeutic properties including anti-oxidant, analgesic, anti-inflammatory and antiseptic activity.

Present work describes the modification of the carbon paste electrode for the detection of DA by using curcumin as modifier using cyclic voltammeric technique. Curcumin modified carbon paste electrode (CC/MCPE) displayed good selectivity for the detection of dopamine in presence of ascorbic acid. No examinations has been accounted for the detection of DA in the presence of AA using Curcumin as modifier.

2. Experiment

2.1 Reagents and chemicals

Curcumin was purchased from Sigma Aldrich. DA and AA were obtained from Himedia chemical company with analytical grade used without further purification. Dopamine stock solution was prepared in 0.1 M Perchloric acid. Potassium ferrocyanide and KCl solutions were prepared in double distilled water. Graphite powder purchased from Loba and silicon oil was purchased from Spectrochem. The chemicals for preparation of buffer solution were purchased from Merck. 0.2 M Phosphate buffer was used as supporting electrolyte.

2.2 Preparation of modified carbon paste electrode

The modified carbon paste electrode was prepared by using 4 mg curcumin powder, graphite powder and silicon oil at a ratio of 70:30 (w/w) an agate mortar by hand mixing to get homogenous paste. The modified carbon paste was tightly packed into the cavity of a homemade carbon paste electrode then polished the surface by rubbing on a butter paper.

3. Result and discussion

Effect of modifier concentration on the surface of carbon paste electrode

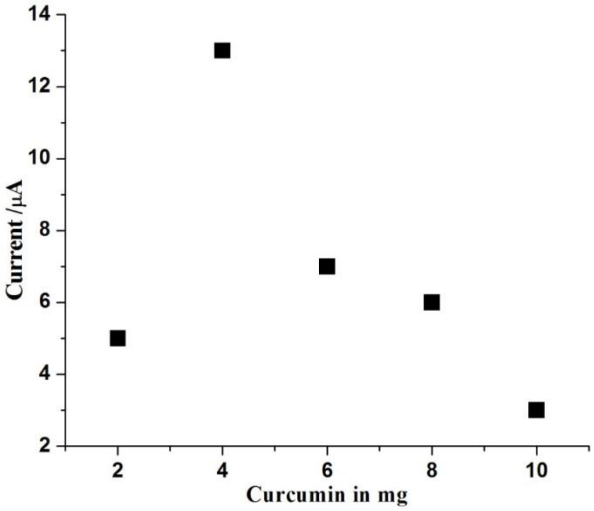


Figure 1: Graph of current v/s quantity of Curcumin in carbon paste electrode.

Characterization of [K4Fe(CN)6] at CC/MCPE

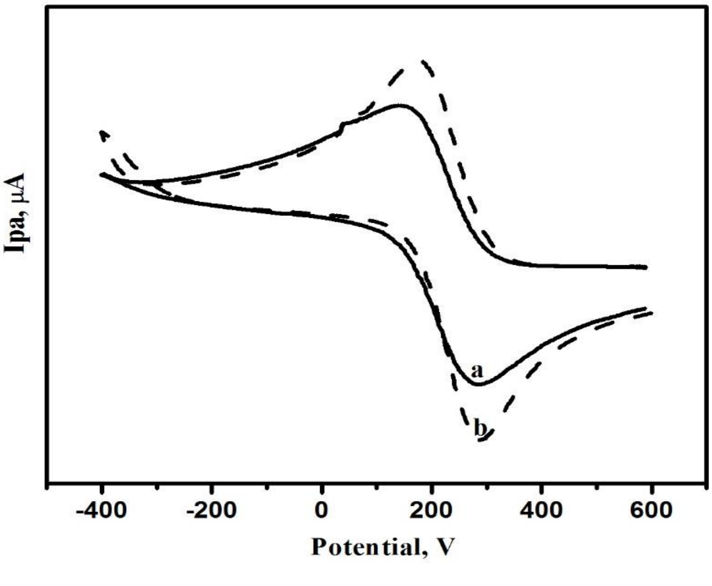


Figure-2. Cyclic voltammograms of 0.1mM [K4Fe(CN)6] at BCPE(solid line, a) and at curcumin modified carbon paste electrode (dashed line, b) Supporting electrolyte 1 M KCl at scan rate 50 mVs^{-1}

Electrochemical characterization of modified electrode

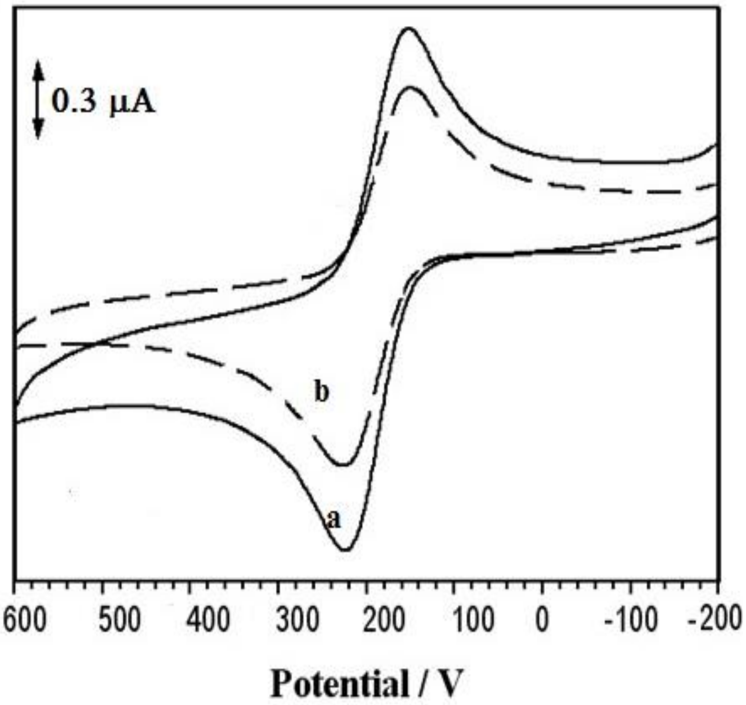


Figure 3: Cyclic Voltmmograms of 1x10^{-5}mM DA in 0.2 M phosphate buffer solution pH 7.0 at BCPE (dashed line,b) and CC/MCPE (solid line,a); scan rate of 50 mVs^{-1}.

Effect of pH

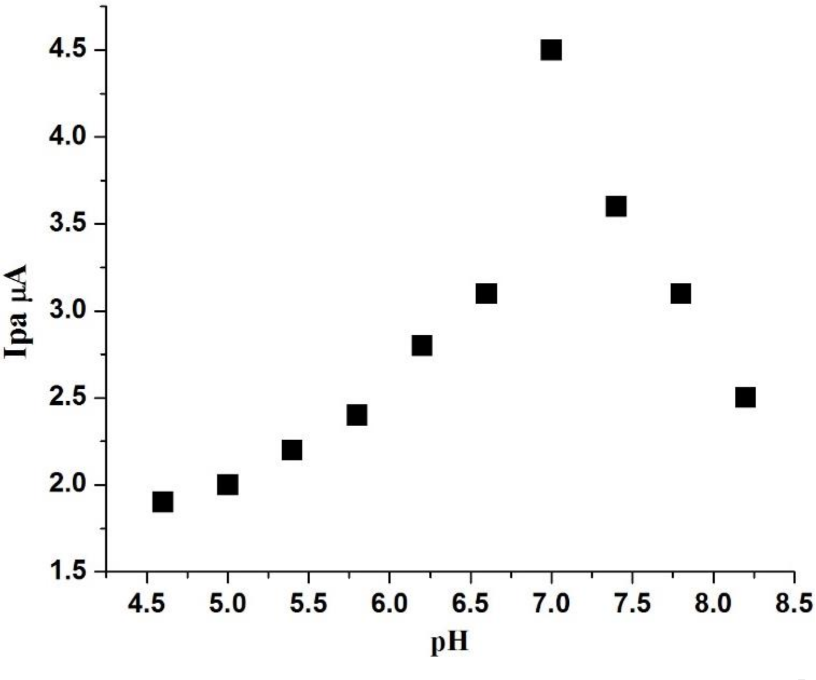


Figure 4: Plot of anodic peak current vs. pH (4.5 – 8.5) of at the 1X10 $^{-5}$ M DA at CC/MCPE.

Effect of Scan rate

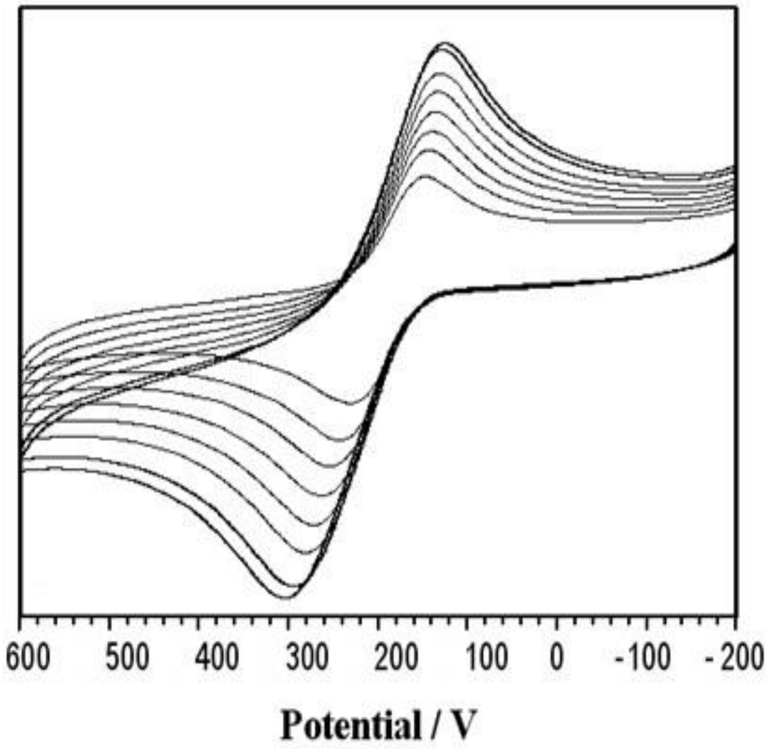


Fig-5: Cyclic Voltammograms of DA at CC/MCPE with different scan rate were 50 to 400 mVs^{-1}

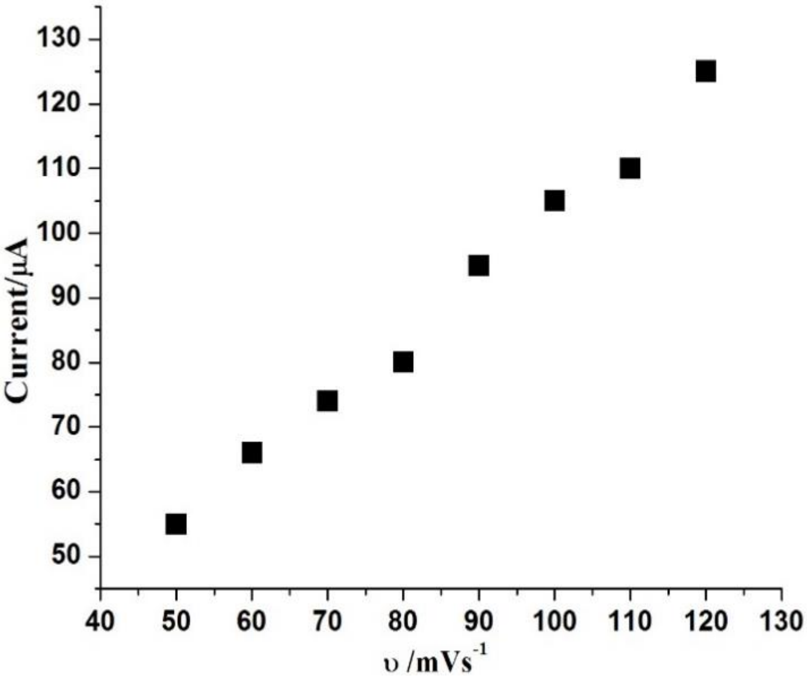


Figure 6: The plot of oxidation peak current vs. scan rates of DA at CC/MCPE

Effect of Concentration

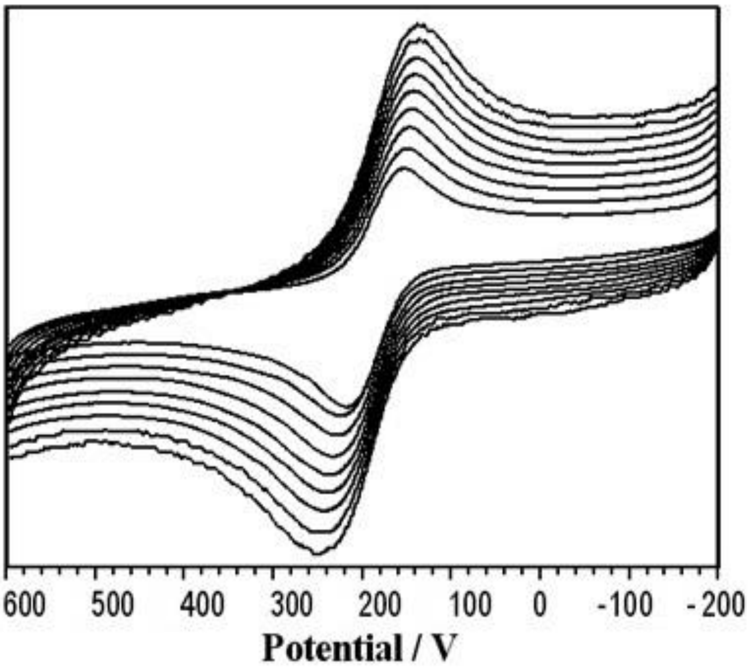


Figure-7: Effect of variation of concentration of DA (a) 1 X 10^{-5}M, (b) 2 X 10^{-5}M, (c) 3 X 10^{-5}M, (d) 4 X 10^{-5}M, (e)5 X 10^{-5}M, (f) 6X10^{-5}M, (g) 7 X 10^{-5}M, (h) 8 X 10^{-5}M, (j) 9 X10^{-5} M on anodic peak current at CC/MCPE; scan rate 50 mVs^{-1}.

Simultaneous detection of DA and AA

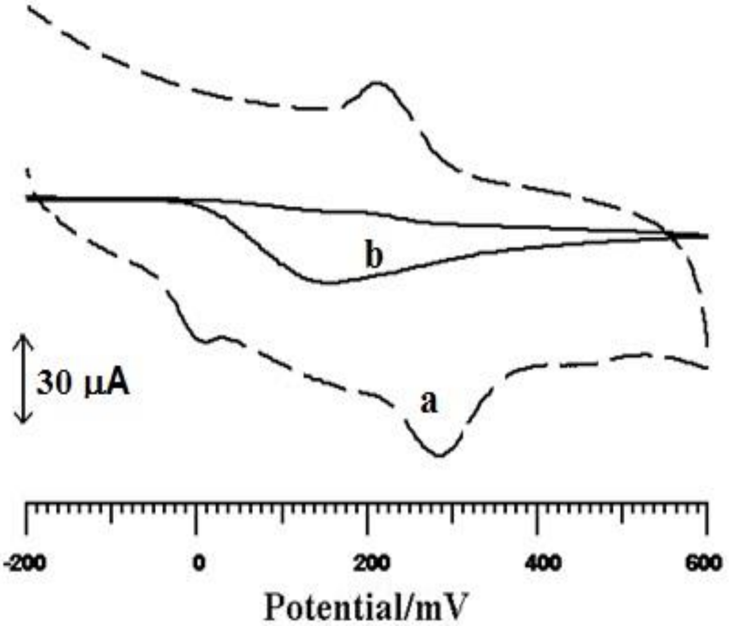


Figure-8. Cyclic voltammograms for simultaneous determination of 1×10^{-5} M DA and 4×10^{-5} M AA at BCPE (Solid line,b) and CC/MCPE (dashed line,a) in 0.2 M phosphate buffer;scan rate: 50 mVs^{-1}

Further work to be Carried Out

- The detailed work has to be carried out based on variety of chemicals by which electrodes have to be prepared.
- The better chemically modified electrode is chosen for the determination of neurotransmitters such as dopamine and serotonin.

Chapter-1

Nano science and technology:

Nanotechnology is the manipulation of matter on an atomic and molecular scale. Nanotechnology as defined by size is naturally very broad, including fields of science as diverse as surface science, organic chemistry, molecular biology, semiconductor physics, micro fabrication, etc.

Nanomaterials

The nanomaterials field includes subfields which develop or study materials having unique properties arising from their nanoscale dimensions.

Nano metaloxide

Metaloxide nanoparticles can exhibit unique physical and chemical properties due to their limited size and a high density of corner or edge surface sites. Particle size is expected to influence three important groups of basic properties in any material. Examples: MgO, Al_2O_3, ZnO etc. various type we have for our convenience we select .

- **Mechanochemical method**

Mechanochemical processing is a novel method for the production of nanosized materials, where separated nanoparticles can be prepared. The method has been widely applied to synthesize a large variety of nanoparticles, including ZnS, CdS, ZnO, $LiMn_2O_4$, SiO_4 and CeO_2.

Powder X-ray diffraction(PXRD)

Powder X-ray diffraction (PXRD) is a versatile and non-destructive analytical technique, which reveals detailed information about the chemical composition and crystallographic structure of materials.

The crystalline size of the obtained powders can be determined from X-ray line broadening using the Scherer's equation,

$t = k\lambda/(\beta\cos\theta)$

UV - Vis Spectroscopy

UV - Vis Spectroscopy is a technique which measures the intensity of light passing through a sample (I) and compares it to the intensity of the light before it passes through the sample (I_o).

The ratio I/I_o is called the transmittance, and is usually expressed as a percentage (*%T*). The absorbance (*A*), is based on the transmittance.

$A = -\log(\%T)$

Electrochemistry

- Electron transfer plays a fundamental role in governing the pathways of chemical reactions.
- Electrochemistry is a powerful and sensitive analytical tool used for both qualitative and quantitative analysis over a wide range of concentrations.
- Numerous standard electrochemical methods exist that can be categorized into three general classes.
- Potentiometry, Coulometry, Voltammetry.

Cyclic Voltammetry

- Cyclic Voltammetry is the most versatile Electro analytical techniques for the study of electro active species.
- Cyclic Voltammetry (CV) means cyclysing the potential of an electrode from starting potential to end potential and end potential to starting potential.
- CV involves the cycling of potential of an electrode between two designated values called switching potential s in an unstirred solution & measuring the resultant current.

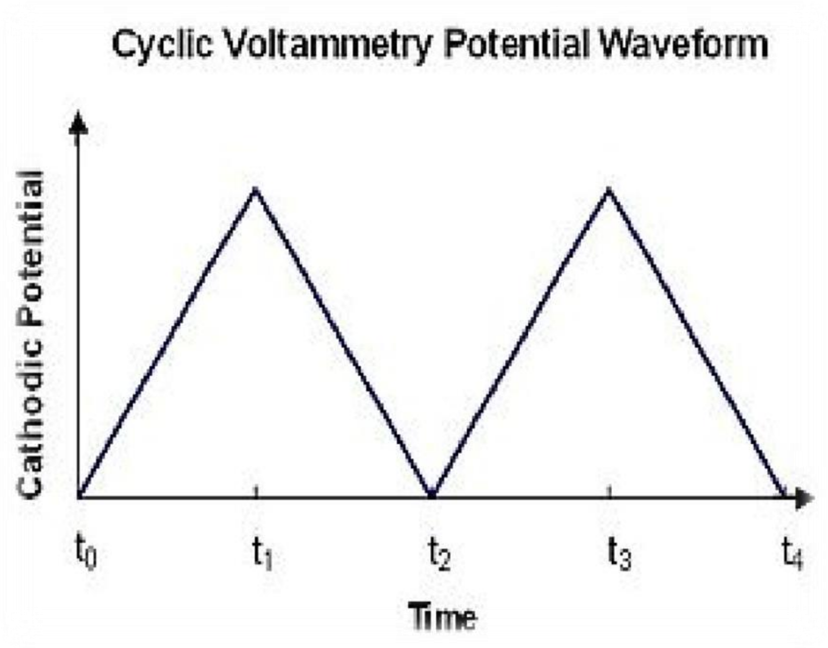


Fig.1. Cyclic voltammetry cyclic form

In CV, the potential is changed in a triangular form.The peak current of the forward potential scan is given by Randles - Sevecks equation.

$i_p = Kn^{2/3}Ad_o^{1/2}v^{1/2}C_o^{*}$ …

Characteristic parameters of a cyclic Voltammogramz

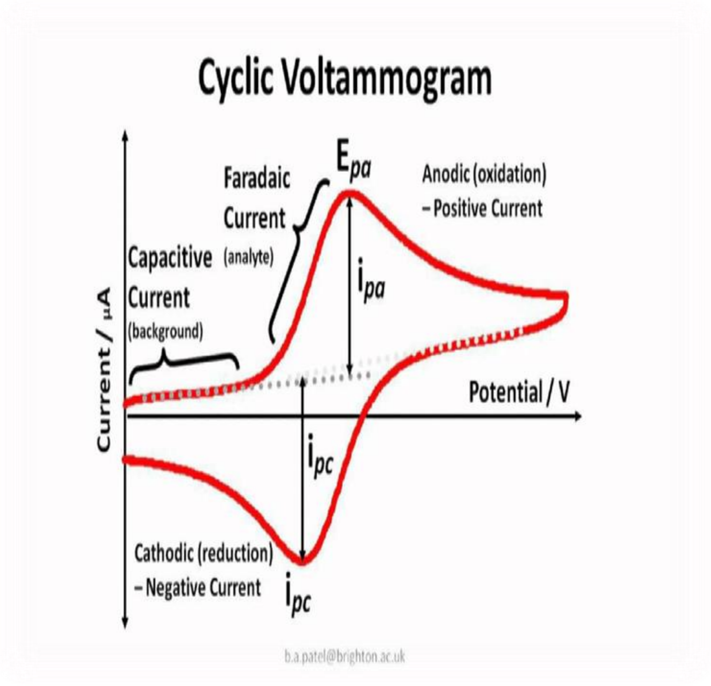


Fig.2. Cyclic voltammetry reversible form

Anodic peak potential (Epa)

Cathodic peak potential (Epc)

Anodic peak current (i_{pa})

Cathodic peak current (i_{pc})

Fundamentals of cyclic Voltammetry

- Circuit
- Scan Rate
- Residual current
- Charging or non-Faradaic Current
- Faradaic Current

Solvent used in CV

- A number of physicochemical properties must be considered while choosing a solvent for electrochemical work.
- It must be a liquid at room temperature. It must have sufficient solubility for ionic substances to form conducting electrolyte.
- It must have a wide enough potential region for the study of the redox process of interest, that is, solvent itself must not undergo oxidation or reduction in this potential region.
- It must possess the required acid-base properties.
- Ex ;- Acetonitrile ,Water ,Methylene chloride . etc,,

Supporting Electrolytes used in CV

- All ionic salts or ionisable compounds in a solvent are defined as the supporting electrolytes.
- H_2SO_4, $HClO_4$ and HCl are normally employed for studies in acidic aqueous solutions and NaOH or KOH are employed for alkaline media. In neutral region, if buffering is important, acetant, citrant and phosphate buffers are usually employed.
- If the redox process does not involve acid-base reactions, no buffers are needed and any electrolyte may be used.

Electrodes used in CV

Three electrode system is used in CV are as follows.

- Working Electrode
- Reference Electrode
- Counter/Auxiliary Electrode
- Bare carbon-paste electrode and Modified carbon-paste electrode

Electrode Processes

- **Mass Transfer**

Mass transfer is the movement of material from one location to another in solution.

1. Diffusion
2. Migration and
3. Convection

- **Reversible reactions**

For a reversible reaction, these values are independent of the voltage sweep rate is given by,

$E_p = E_o - RT/nF \ln(D_o/D_R) + 1.109RT/Nf$

- **Irreversible Systems**

For an irreversible electrode process, the peak current is given by,

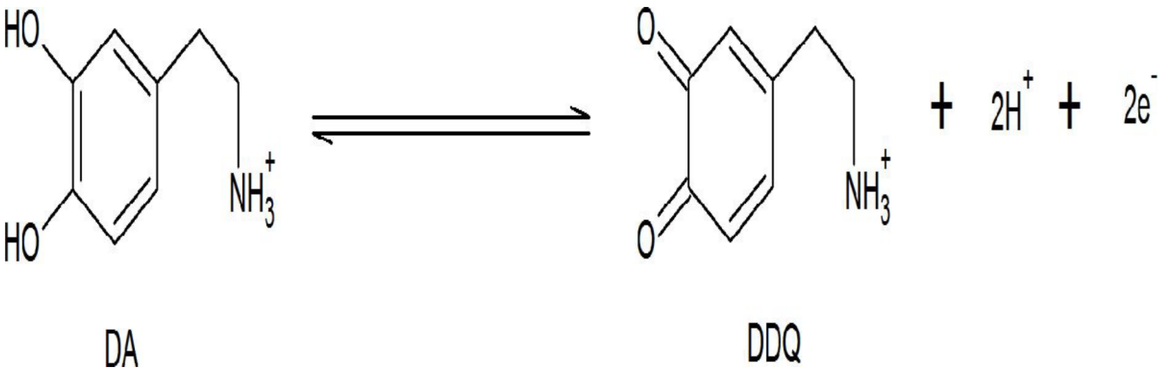
$Ip = 2.69 \times 10^5 n (\alpha n)^{1/2} A D_o^{1/2} \nu^{1/2} C_o$

- **Quasi Reversible Systems**

Chemistry of dopamine

Dopamine (DA), also known as “4-(2-amineoethyl) benzene-, 2-diol” belongs to a member of the catecholamine family.

- Dopamine plays an important role in the functions of the central nervous system, renal, hormonal, and cardiovascular systems. Dopamine was first synthesized by *George Barger* and *James Ewen* in 1910.
- Dopamine undergoes oxidation to form Dopaquinone as shown.


HO
HO
NH3+
O
O
NH3+
+ 2H+ + 2e-
DA
DDQ

CHAPTER II

EXPERIMENTAL METHOD

ZnO nanoparticle was synthesized by using zinc acetate, and the Oxalic acid and Ascorbic Acid (as reducing agents), by solution free mechanochemical method.

The particles were characterized by using X-ray diffraction (XRD), UV-visible absorption spectroscopy (UV-vis).

Preparation of ZnO nanoparticle

Apparatus

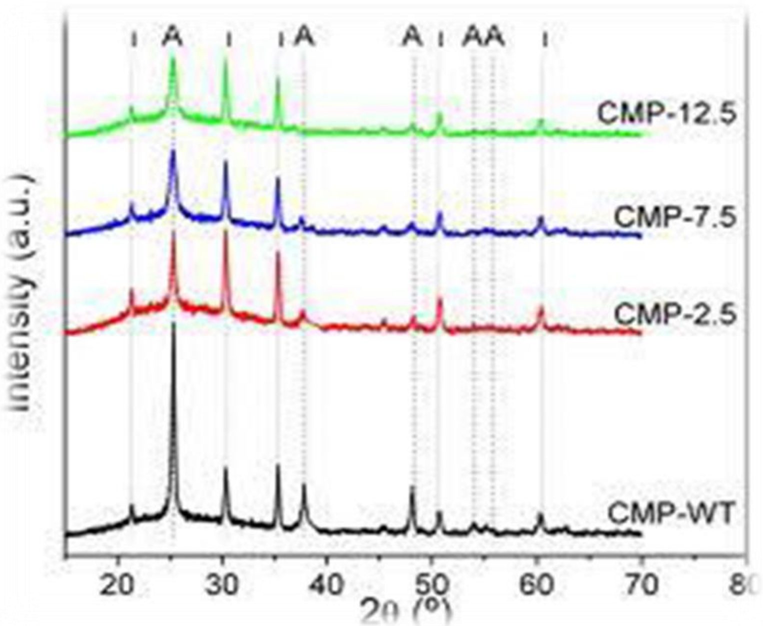
The prepared ZnO was characterized by powder X-ray diffraction (PXRD) using CuKα radiation ($\lambda = 0.1543$ nm)at 40 kv, at a scanning rate of 2^o min^{-1}, in PAN analytical 'X' PERT PRO X-ray diffractometer. UV- visible spectra were recorded on a UV-visible spectro photometer-shimdazo (1650) using sonicated ZnO in double distilled water.

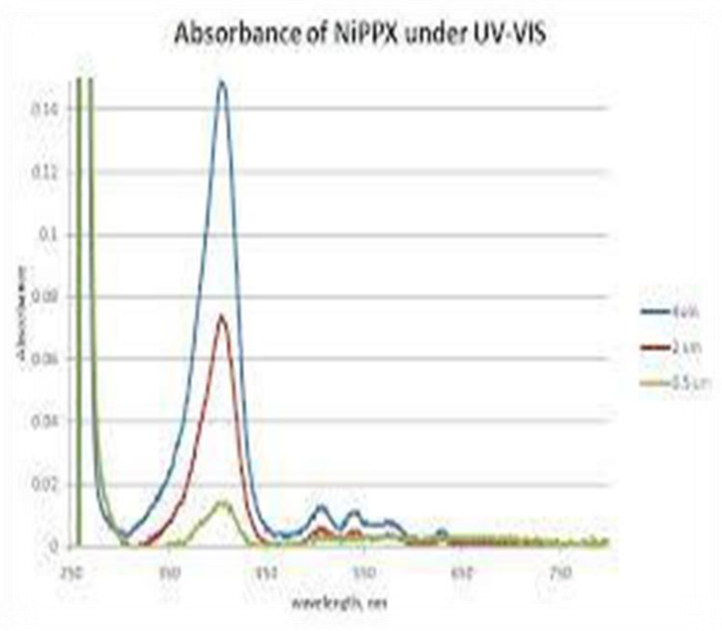
The cyclic voltammetric measurements (CV) AND Differential pulse voltammetry techniques (DPE) were performed on a model 660c (CH Instruments[4]).

Preparation of bare carbon paste electrode and modified carbon paste electrode

CHAPTER III

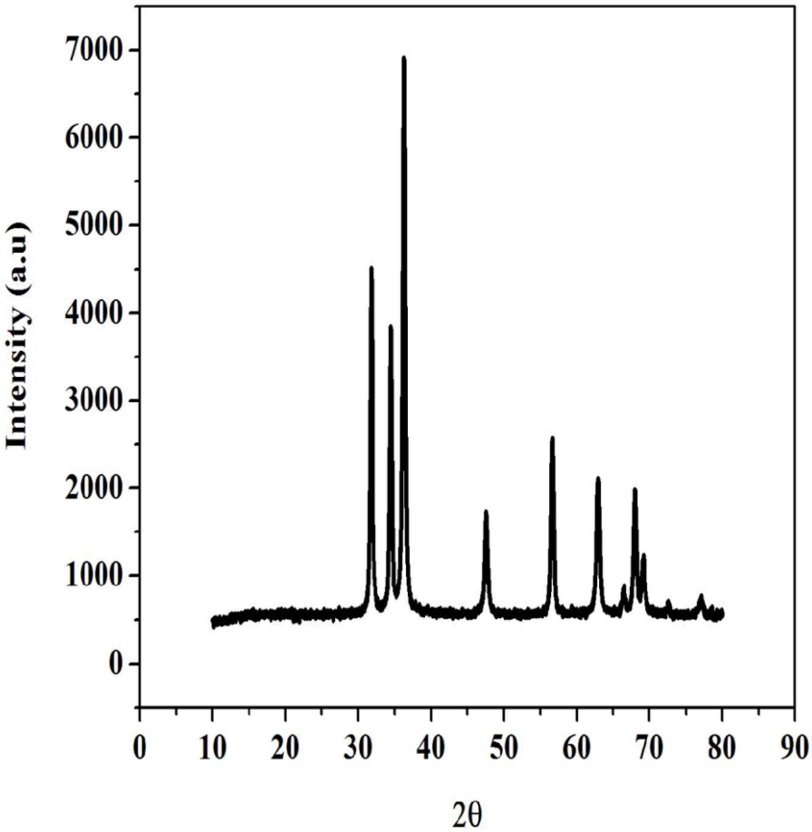
RESULT AND DISCUSSION





Characterization of Nano particle by special techniques

By powder x-ray diffraction

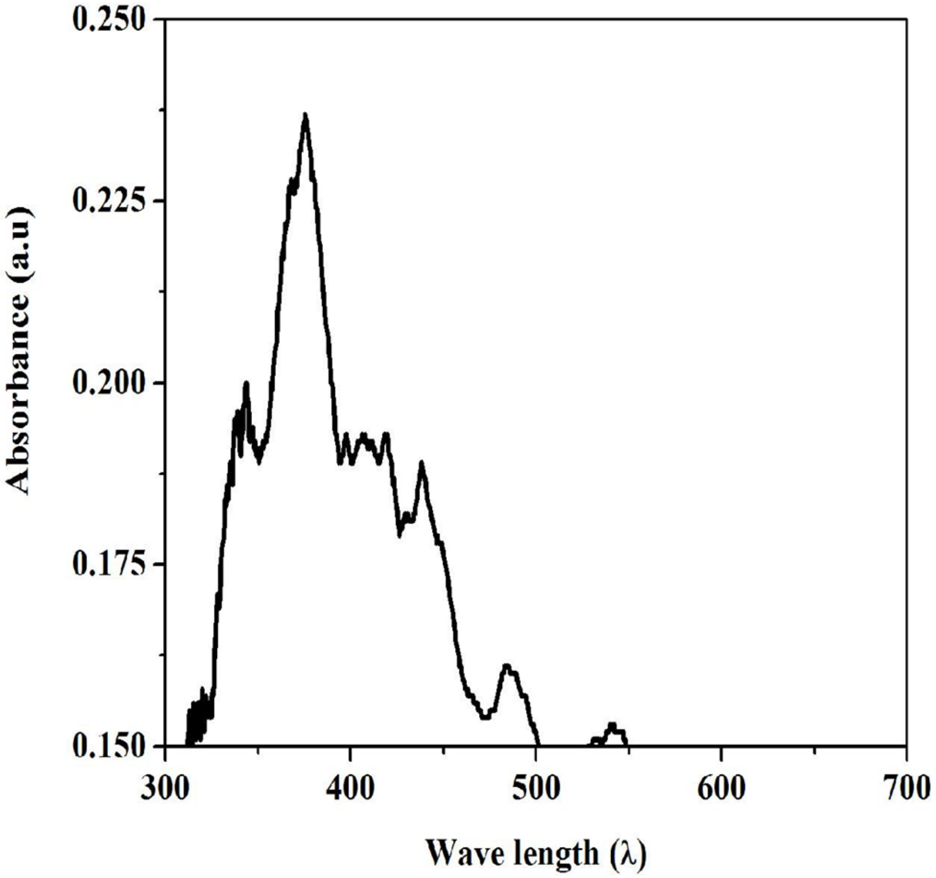


XRD patterens for ZnO nanoparticles

The average particle sizes for both nanoparticle was calculated using the Deby-Scherrer Equation.

ZnO nanoparticle has, the average particle size is **36.66nm**.nearly equal to 37**nm**

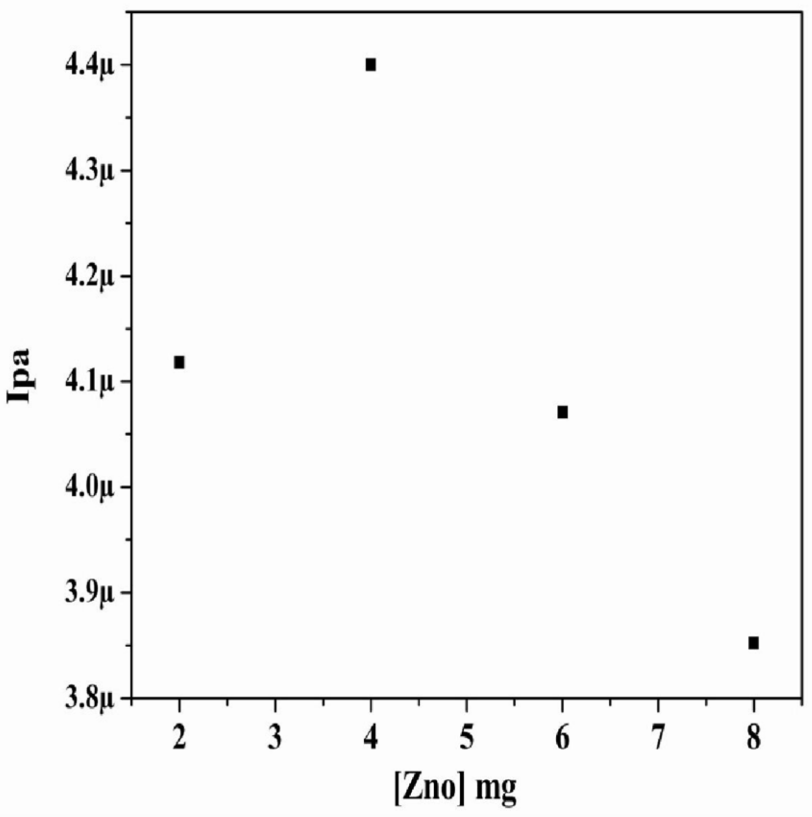
UV-Visible absorption Spectra



UV-Visible absorption Spectra of ZnO[OA] nanoparticle

It shows a sharp absorption peak at the λ_{max}375nm and the sample has a bandgap of 3.3306eV that is slightly lower than that of bulk ZnO (3.37 eV).

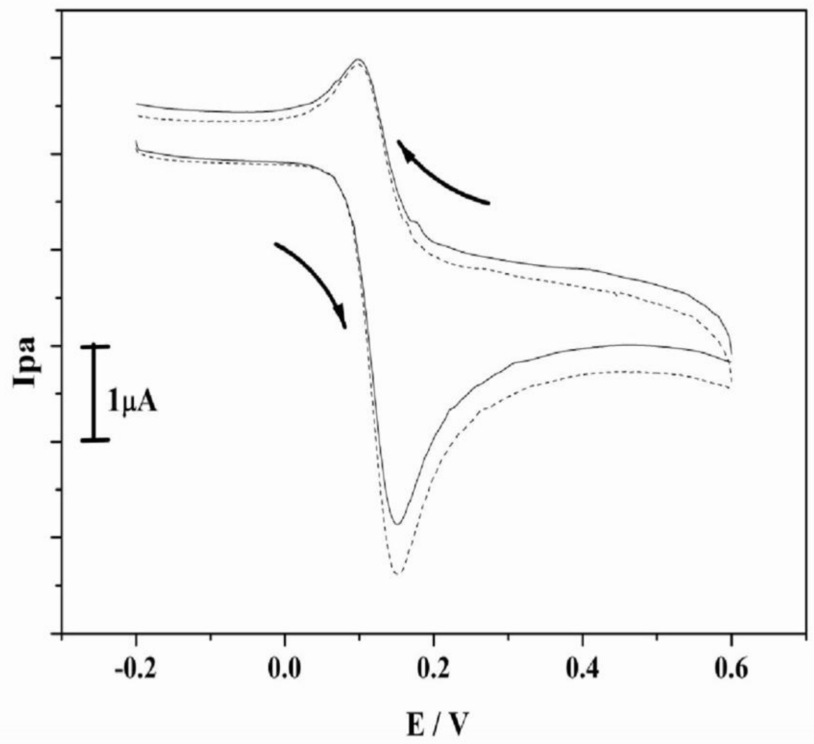
Effect of ZnO nanoparticle concentration in CPE on Dopamine



Plot of Anodic Peak Current (i_{pa}) v/s different concentrations of ZnO nanoparticles in Modified CPE.

The effect of ZnO nano particles concentration in the carbon paste electrode (CPE) on the peak current for the investigation of 25x 10^{-4} M DA in 0.2M disodium phosphate buffer solution of pH 7.0 was studied by CV method

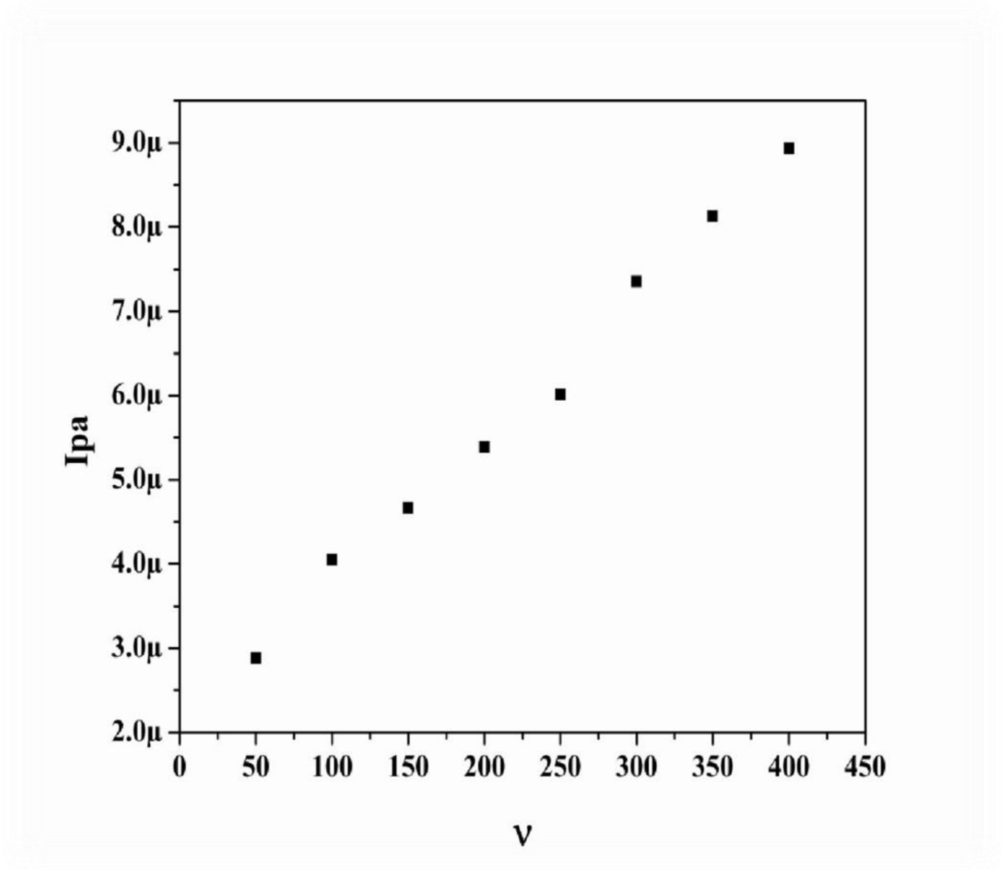
The electrocatalytic response of DA at the bare CPE, and ZnO nanoparticle MCPE



Cyclic Voltammogram of 25μM DA in PBS at pH 7.0 at (solid line) for bare CPE and (dotted line) for ZnO nanoparticles for MCPE

It shows the electrochemical responses of 25x 10^{-4} M DA in 0.2M phosphate buffer solution at pH 7.0 at the bare CPE (solid line)and the ZnO nanoparticle MCPE (dotted line) with scan rate 100mV/s

Effect of scan rate for ZnO

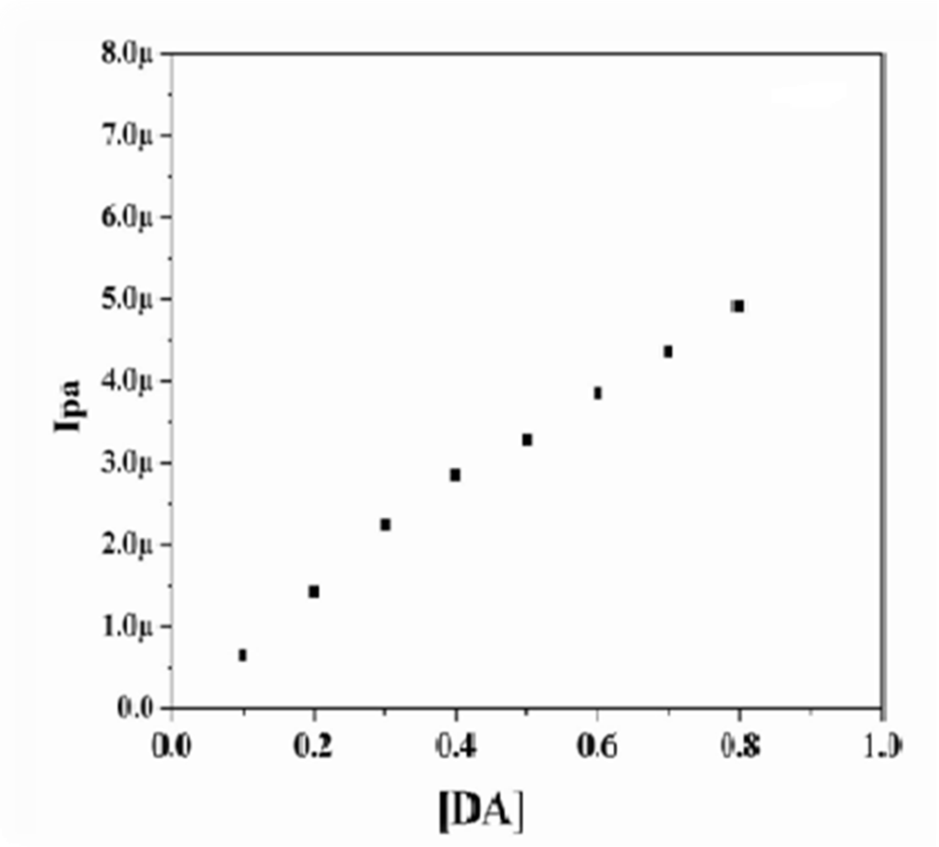


Plot of Anodic Peak Current vs the different Scan rates (50-400mVs^{-1}) in 0.2M PBS solution at pH 7.0

ZnO nanoparticles MCPE showed increase in the redox peak currents with increase in scan rate *(50-400mVs^{-1})*

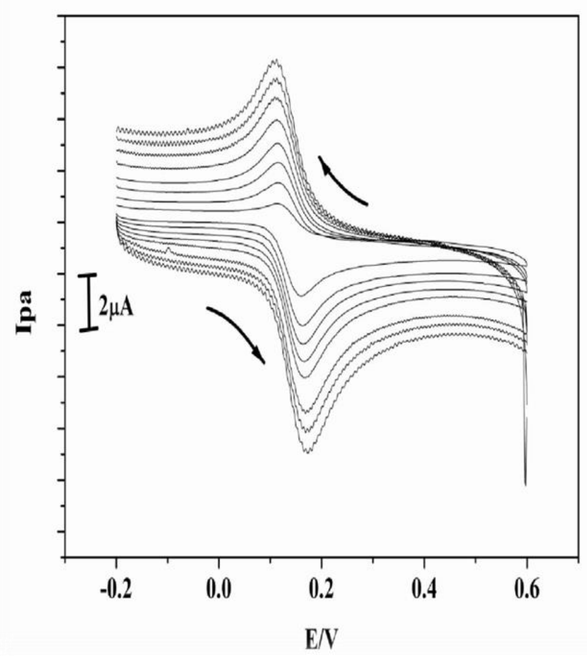
ZnO nanoparticles MCPE showed increase in the redox peak currents with increase in scan rate *(50-400mVs^{-1})*

Concentration effect of DA for ZnO MCPE

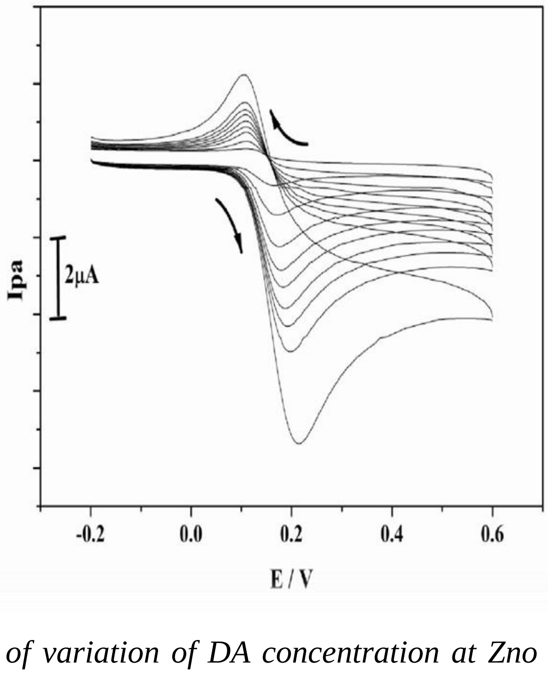


Plot of Anodic Peak Current (i_{pa}) v/s concentration of DA (0.1 - 0.9µM) we get linear peak

The correlation coefficient for the linearity was 0.99811 and the detection limit for DA in the linear range region was calculated according to recent reported and it was found to be 2.38320 x 10^{-7}M.



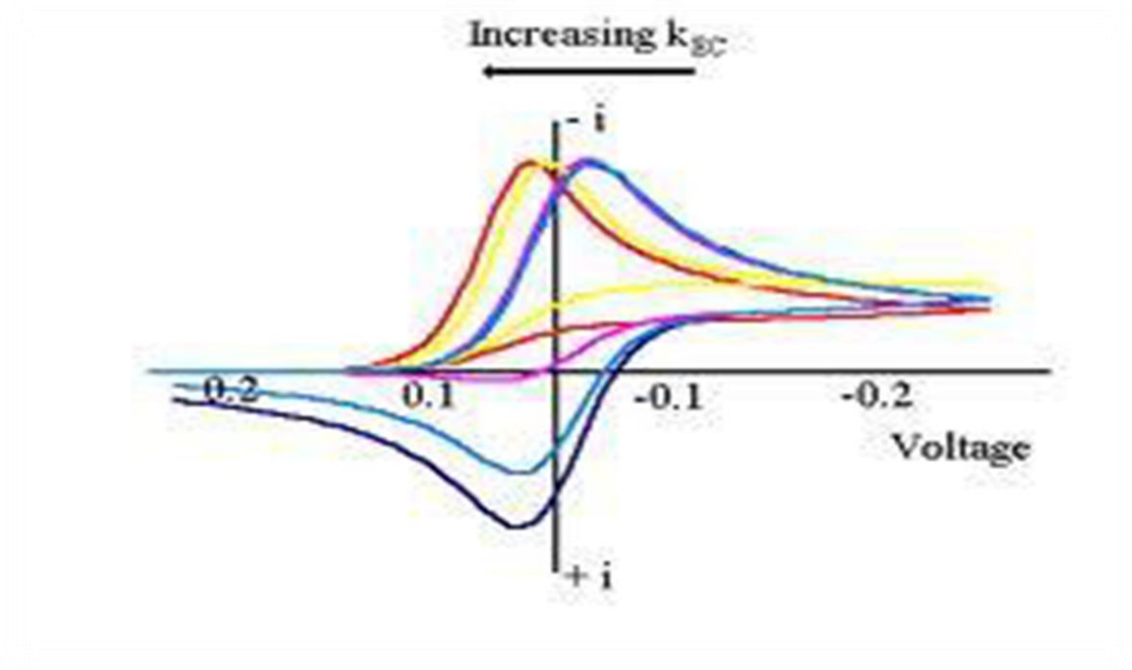
Cyclic Voltammogram of variation of scan rate for DA at ZnO modified CPE in 0.2M PBS at pH 7.0.



Cyclic Voltammogram of variation of DA concentration at Zno modified CPE in 0.2M PBS at pH 7.0

CHAPTER IV

CONCLUSION



- In this present study the ZnO nanoparticle mechanochemically synthesised by the chemical reaction between Zn(CH3COO)2 and H2C2O_4.2H_2O, via bottom-up process. Advantage of this method is convenient to synthesis ZnO nanoparticle in normal laboratory condition with high yield and low cost and also it an simple and efficient method.
- The average grain size of ZnO was 36.66nm with a significant slightly blue shift of 375nm as compare to bulk particle. And bandgap was obtained as 3.306eV. From the XRD the prepared particle have high purity and good crystalline structure.
- Thus the ZnO nanoparticle MCPE shows good electrocatalytic activity and low detection limit compare to the CPE.

- The different concentration of ZnO in CPE in presence DA at 0.2M PBS (pH 7) was studied. The ZnO nanoparticle MCPE shows good current enhancement compare to the bare CPE. And also the correlation coefficient 0.99915 from the different scan rate by using MCPE.

Hence this present synthetic method extended to many metal oxides, ferrites for synthesis and their modified electrode used as sensor for application of determination of some biological active molecules and other neurotransmitters.

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