

**FABRICATION OF HIGH SURFACE AREA AMORPHOUS CARBON BASED
ELECTRODES FOR EDLC APPLICATIONS**

Dissertation submitted to Department of physics,

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(Affiliated to University of Calicut)



In partial fulfilment for the award of the degree of

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IN

PHYSICS

Submitted by

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JULY 2023

DECLARATION

I hereby declare that this submission “ **Fabrication of high surface area amorphous carbon based electrodes for EDLC applications**” is a record of my project work carried out under the guidance of **Dr. Dijo Damien Assistant Professor, Department of chemistry, Christ college, Irinjalakkuda**. I also declare that this project work has not been submitted previously for the award of any degree or diploma earlier. The findings in the report are based on the information collected by me and not copied from elsewhere.

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CERTIFICATE

This is to certify that the dissertation entitled “ **FABRICATION OF HIGH SURFACE AREA AMORPHOUS CARBON BASED ELECTRODES FOR EDLC APPLICATIONS** “ is a bonafide record work done by **MAIMUNATH P.S(AIAVMPH007)** at Christ college, Irinjalakkuda. Under the guidance of Dr. Dijo Damien in partial fulfilment of the requirements for the award of Master of science in Physics of Department Physics, MES Asmabi college , Calicut University during the period of March (2021) – July (2022).

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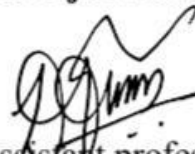
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CERTIFICATE

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ABSTRACT

Due to the rising demand for efficient energy storage devices, extensive research has been done on super capacitors as a potential alternative for conventional batteries. In this study, we show how to make high surface area amorphous carbon-based electrodes that are specifically made for applications in electrochemical double layer capacitors. The amorphous carbon structure allows for a greatly enlarged surface area, permitting more extensive charge storage at the electrode-electrolyte interface,

and the suggested electrodes are made using a cost-effective process. The performance of the manufactured electrodes is evaluated by electrochemical tests, such as cyclic voltammetry , Galvanostatic charge discharging and electrochemical impedance spectroscopy. The current study aims at demonstrating the electrochemical double layer capacitance features of porous carbon-based symmetrical supercapacitors in 6M aq. KOH. Fabricated supercapacitors exhibit an impressive specific capacitance of 95 F/g.

Contents

Chapter 1 : INTRODUCTION

- 1.1 Components of a Supercapacitor**
- 1.2 Types of Supercapacitors**
- 1.3 Operating Principles of EDLCs**
- 1.4 Applications of Supercapacitors**
- 1.5 Advantages of Supercapacitors**

Chapter 2: MATERIALS AND METHODS

2.1 Materials

- **Activated carbons**
- **Acetylene black**
- **PTFE**
- **KOH**

Hydraulic press

2.2 Fabrication of electrodes

CHAPTER 3: CHARACTERIZATION TECHNIQUES

3.1 Electrochemical methods

- **Cyclic voltammetry (CV)**
- **Electrochemical impedance spectroscopy (EIS)**
- **Galvanostatic charge discharge (GCD)**

3.2 Material characterization

- **Sem**
- **EDX**
- **XRD**

CHAPTER: 4 RESULT AND DISCUSSIONS

4.1 From cv

4.2 From EIS

4.3 From GCD

4.4 Xrd analysis

4.5 Sem analysis

4.6 EDX analysis

CHAPTER : 5 SUMMARY AND CONCLUSIONS

- **REFERENCE**

Chapter 1

INTRODUCTION

Introduction

Due to the excessive use of fossil fuels, renewable resources and environmentally friendly energy storage technologies have gained a lot of attention globally as solutions to the environmental problems. Improved electrical energy conversion and storage technologies are needed to address these problems. Batteries and supercapacitors are currently the most widely utilised electrochemical energy storage devices. Batteries use the faradaic reactions of electrode materials with electrolytes to store energy, which typically also involve chemical interconversions and phase shifts and result in high energy densities. These battery-type faradaic reactions, however, experience slow kinetics and material irreversible processes, which limits their power output and durability. In contrast, supercapacitors store the charge at the electrode/electrolyte interface via fast and non-diffusion limited faradaic reactions for pseudo-capacitive materials and physical ion adsorption/desorption processes for electrochemical double-layer capacitors (EDLCs).

Super capacitors are promising candidates for energy storage devices with high power density and a long cycling life due to their quick and highly reversible storage mechanisms. These devices are currently used in a variety of applications where high power delivery and/or uptake are required, such as energy harvesting. Supercapacitors, as opposed to batteries and conventional capacitors, utilise electrochemical porous materials like activated carbons or carbon fibres. The electrochemical double-layer capacitors (EDLC), which rely on ions entering the pores of the carbon electrodes to store energy, frequently use these electrode materials. To increase a useful capacitance, many researchers are using more effective carbon conducting materials as carbon nanotubes or carbon aerogel graphene. Because of their high surface area, reasonably high conductivity, and strong electrochemical stability. Graphene and carbon nanotubes are two examples of carbon nanomaterials that also have a number of other advantageous characteristics, including a large surface area, excellent thermal and electrical conductivity, high flexibility and tensile strength, chemical sensitivity, and low weight. The most common electrode for industrial supercapacitors is activated carbon (AC), which has a mostly microporous structure. Even if the surface area of AC ranges from 1000 to 2500 m² g⁻¹, a portion of the tiny micropores are inaccessible to electrolyte ions, which minimally affects the effective capacitance. The capacitance contribution of micropores declines at low temperatures more than that of mesopores. Large micro- or mesopore carbon is expected to facilitate charge storage at a range of temperatures.

1.1 COMPONENTS OF A SUPERCAPACITOR

Supercapacitors are made up of three basic parts: two electrodes, an electrolyte, and a separator that is similar to that of a regular capacitor. In the electric field of an electrochemical double layer, supercapacitors (SCs) are electrochemical capacitors (ECs) that store charge. They are a good contender for energy storage due to their remarkable electrochemical characteristics. Electric double-layer capacitors (EDLCs), redox electrochemical capacitors (RECs), and hybrid electrochemical capacitors (HECs) are the three types of SCs that can be distinguished based on their charge-storage mechanisms. Various kinds of carbon metal oxides and conductive polymers can be used to make supercapacitor electrodes. Activated carbons and graphene—both of which are based on carbon—as well as carbon nanotubes—are the main materials that are the focus of EDLCs, which operate on the idea of energy storage via charge separation at the electrode/electrolyte interface. RECs, on the other hand, are based on doped carbon, conductive polymers, and metal oxides. HECs combine the characteristics and operating Principles of the materials discussed above.

1.1.1 Electrode material

Among the several parts of the supercapacitor device, the electrode materials play the largest importance. High electrical conductivity, a bigger electrochemically active surface area, enhanced electrochemical and thermal stability, and increased surface wettability are all qualities that an ideal electrode material should have. It should also be economical and recyclable. In general, a number of elements, such as shape, porosity, and accessibility to electrolytes, determine how well electrode materials conduct electrochemistry. Notably, the performance of the supercapacitors has significantly enhanced because to the introduction of nanomaterials.

1.1.2 Electrolyte

The electrolyte, a further essential component of energy storage devices, is also greatly influenced by charge and discharge cycles. Several highly conducting aqueous and non-aqueous gel electrolytes have been developed for flexible super capacitors. In order to improve the capacitance of the super capacitors, redox additives have recently been added to the polymer electrolyte through a redox

interaction between the electrode/electrolyte interface. Charges organise themselves randomly at the electrode surface and in the electrolyte facing the electrode when an electronic conductor is submerged in an electrolyte solution. A layer of this EDL forms at the electrode-electrolyte interface, one layer on the conductor's surface and the other layer within the electrolyte. Along with the electrode material having a higher capacitance, high-performance electrolytes that work in a larger potential window are also necessary. A given electrolyte may often function without experiencing significant breakdown at a specific voltage range known as the "electrochemical stable potential window (ESW)". The value of ESW is based on the electrochemical stability of the component materials, such as salt and solvent. Due to the water breakdown reaction occurring around 1.23 V, the voltage for aqueous electrolytes is typically in the range of 1 to 1.2 V. For non-aqueous (organic solvents-based) and ionic liquid-based electrolytes, it is in the range of 2.5 to 2.7 V and 3.5 to 4.0 V, respectively. Furthermore, the performance of the electrolytes is influenced by their viscosity, freezing point, ionic conductivity, and ion size. The common types of aqueous electrolytes include salt (Li_2SO_4 , Na_2SO_4 , and K_2SO_4), alkaline (KOH , NaOH , and LiOH), and acidic (H_2SO_4 , HCl , and H_3PO_4) solutions. A typical range for the concentration of an aqueous electrolyte solution is 0.5 to 6 M. Compared to nonaqueous or unusual electrolytes, aqueous electrolytes have a higher ionic conductivity. The concentration of the electrolyte solution should be optimal to get the highest possible ionic conductivity; otherwise, ionic conductivity will often be reduced. Additionally, the molar ionic conductivity of hydrated cations and anions affects the ionic conductivity of a certain aqueous electrolyte.

1.1.3 Separator

Any physical barrier that exists between the two positive and negative electrodes to avoid electrical shorting due to physical contact with the electrodes can serve as a separator, including filter paper, polymeric microporous sheets, and even gel polymer electrolytes. Separators must be made of an inert material and be able to pass through the ions in the electrolyte.

1.1.4 Current collectors

Because they offer physical support, especially for the powdered active components, the current collectors are essential in the fabrication of electrode materials. They also function as an interface for

the transmission of electrons between the electrode materials and the outside circuit. The electrical resistance of the current collector as well as its interfacial resistance with the electrode materials are both partially explained by the total internal resistance of the manufactured supercapacitor. Higher mechanical strength, thermal and electrochemical stability, electrical conductivity, surface area, decreased contact resistance, light weight, and low cost are all desirable qualities in a current collector. Supercapacitors frequently use materials made of metal and carbon as its current collectors. Foil, mesh, and foam are utilised as metal-based current collectors made of nickel, stainless steel, titanium, copper, and aluminium. In general, the electrical conductivity and interfacial resistance of metal-based current collectors are higher. However, because of the corrosion and dissolution reactions, the use of these metals as current collectors depends on the kind of electrolyte systems and the operational potential window.

1.2 Types of Supercapacitors

Super capacitors are mainly classified in to three types :

1. Double layer capacitors
2. Pseudo capacitors
3. Hybrid capacitors

▪ **Double layer capacitors**

EDLCs are made up of an electrolyte, two electrodes made of carbon-based materials, and a separator. EDLC can either store the charges electrostatically or by a non-Faradic mechanism that eliminates the requirement for charge transfers between the electrode and electrolyte. The energy storage theory that EDLCs employ is the electrochemical double layer. When voltage is applied, there is no buildup of charges on the electrode's surface because the opposing charge attracts due to the difference in potential, which causes electrolyte ions to diffuse over the separator and onto the oppositely charged electrode pores. To stop ions from recombining in electrodes, a double layer of charge was formed. EDLCs are made possible by the double layer for achieving high energy density along with the increase in specific surface area and the decrease in distance between electrodes. Furthermore, due to the way EDLCs store energy, this permits quick energy absorption, efficient energy transfer, and good power output. Batteries and EDLCs may differ in some ways due to the non-Faradic phase, which doesn't involve a chemical reaction. This is because EDLCs have the capacity to sustain millions of cycles while batteries can only withstand a maximum of a few thousand. In Li-ion batteries, using high

potential cathodes or graphite anodes results in the solid electrolyte inter-phase; nonetheless, charging does not require an electrolyte solvent.

- **Pseudo capacitors**

A supercapacitor type having conducting polymer or metal oxide electrodes and a high electrochemical pseudocapacitance component. The Faradaic mechanism for storing charge involves the transfer of charge from the electrolyte to the electrode, just like in oxidation-reduction reactions. When a potential is applied to a pseudo-capacitor, oxidation and reduction take place on the electrode material, which also involves the charge's passage over a double layer, causing the Faradic current to pass through a SC cell. Pseudo-capacitors can also achieve higher specific capacitance and energy densities than EDLCs thanks to the Faradic mechanism involved.

- **Hybrid capacitors**

Supercapacitor of a certain type having asymmetric electrodes, one of which mostly exhibits electro-static capacitance and the other electro-chemical capacitance. The hybrid capacitors also combined performance characteristics that were previously impossible. Additionally, they are integrating the best aspects of EDLCs and pseudo-capacitors into a single SC. Even though hybrid capacitors were less studied than pseudo-capacitors or EDLCs, efforts to improve hybrid capacitors and to produce more accurate quantitative models of the hybrid capacitors were growing. In addition to the increased emphasis on developing high energy and high cycle life SCs, the enormous simplicity with which hybrid capacitors' performance and design can be tuned has enabled them to overtake EDLCs as the dominant SCs class.categories. The electrode structure of hybrid capacitors was used to classify them into three groups: asymmetric, composite, and battery-type.

1.3 OPERATING PRINCIPLES OF EDLCs

A non-faradaic method is used to store energy in EDLCs, which are capacitive energy storage devices. A supercapacitor device, like batteries, consists of two electrodes submerged in an electrolyte and a porous, ionically conducting separator in the middle to prevent electrical short circuits. In essence, when an external voltage is applied between the EDLC electrodes, the adsorption of the ions with the opposite ionic charge from the electrolyte balances the electronic charge that builds up at the

electrode surfaces. According to the electrostatic charge separation equation, the capacitance C produced by this charge separation at the electrode/electrolyte contact is given by,

$C/A = \epsilon_r \epsilon_0 / d$ Where ϵ_r is the electrolyte dielectric constant , ϵ_0 is the dielectric constant of the vacuum , d is the effective thickness of the double layer and A is the electrode surface area.

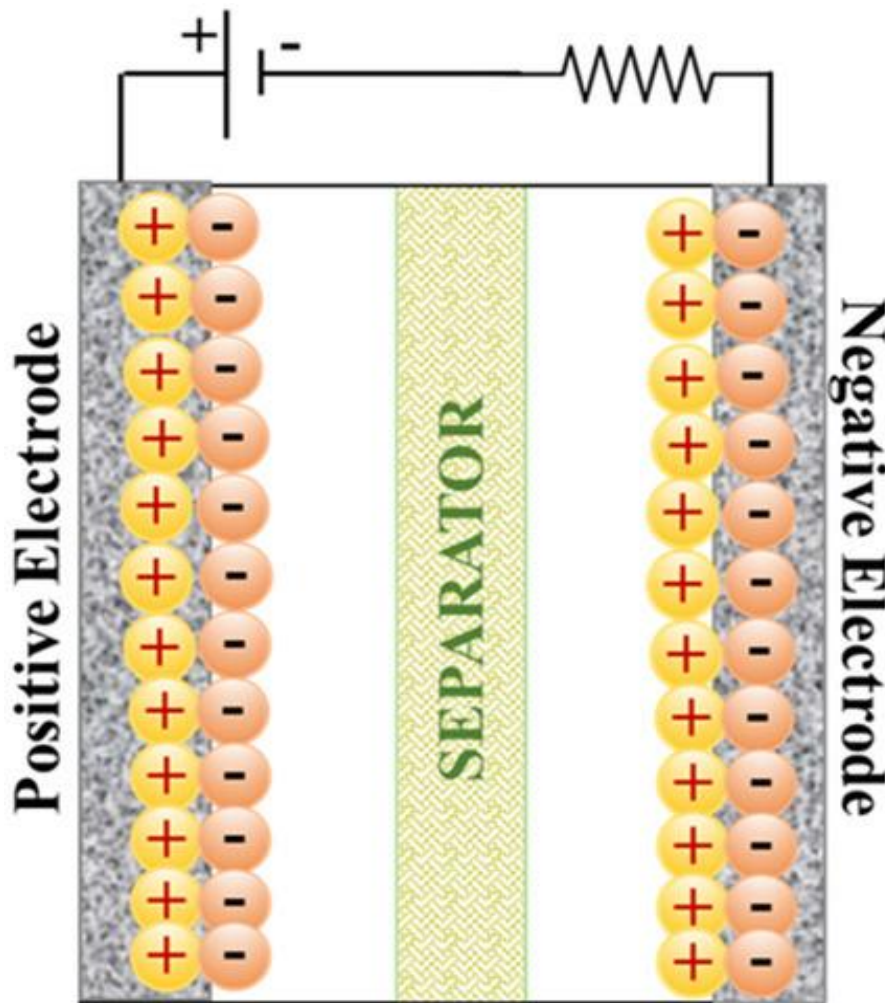


fig. 1.1 schematic illustration of Supercapacitors

This EDL develops at the electrode-electrolyte interface, with one layer on the conductor's surface and the other in the electrolyte. With the charges in the solution and the conductor separated by a distance of the order of molecular diameters, the two charged layers are thought to behave as a physical capacitor. The electrode surface structure, the electrolyte's composition, and the potential field between the charges at the interface all affect the EDL's properties. At the interface between the electrode and the electrolyte, positive or negative ions from the electrolyte form the solution part of the EDL, depending on the surface charge of the electrode materials.

In accordance with the Helmholtz model, the charges are concentrated on either side of the electrode surface. The various ion sizes and their surface reactivity are considered in a more detailed model of the EDL structure. The outer Helmholtz plane (OHP) is the point at which non-specifically adsorbed ions (cations) in solution, come closest to one another. The OHP's cations are often solvated, making them bigger than the anions that are less solvated. The coulombic interaction characterises the interactions of the OHP ions with the surface charge. In particular, deposited ions (often anions) and/or adsorbed solvent molecules' closest approach to the electrode surface is referred to as the inner Helmholtz plane (IHP). Chemical affinities of the ions to the electrode surface and the EDL field strength control these adsorption processes. The EDL's structure is significantly more complex in practice. At the electrode surface, a double layer forms and relaxes nearly instantly. Its formation time, or time constant, is 10^{-8} s. Because of this, the double layer's structure has the ability to react quickly to any changes that may occur at the same moment. The only chemical reaction that occurs during the process is a charge rearrangement. This quick sensitivity to change contrasts with the redox electrode reactions that occur in batteries and fuel cells. Redox reactions have a much slower time constant that is correlated with the impedance of the reaction and is in the range of 10^{-2} - 10^{-4} s. When carbons are dissolved in an electrolyte, they often adopt a voltage that is close to the EDL's zero point of charge. This is quite close to 0 V vs hydrogen in aqueous solutions. The capacitance C can be raised by adding a lot more ions and electrons to the double layer by using an external voltage. In general, the double-layer capacitance of carbons and metals ranges from 10 to 40 F/cm². The exact values mostly depend on the voltage and the degree of IHP involvement in the electrical double layer. A carbon electrode with a large surface area can provide a capacitance of around 4 F/g.

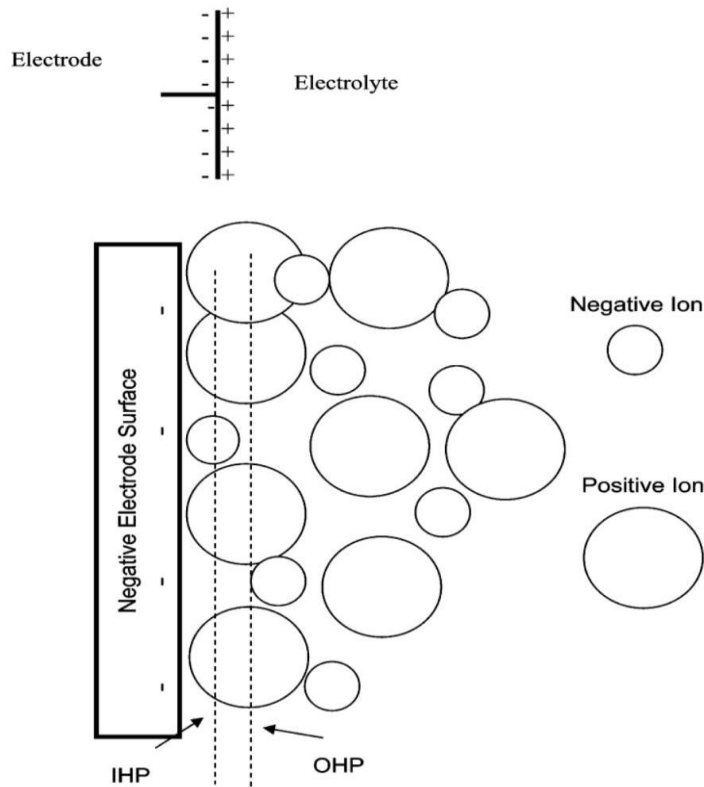


fig . 1.2 Helmholtz model of EDLC

1.4 Applications of Supercapacitors

Supercapacitors are widely employed in many different applications, including electric drives, UPS, traction, electric vehicles, SSDs, and LED torches, due to their special storage capacity.

- **Hybrid buses**

Supercapacitors largest market is in transportation. October 2017 BEST (Brihan Electric Supply and Transport) introduced emission-free electric buses in India towards the end of the month. The process of charging a battery takes time. The motors produce a back emf when braking. The supercapacitors are topped off with this back emf's regenerative energy. Battery life is increased and battery size is decreased when supercapacitors are used in combination with the battery. The sole power source for buses in Shanghai is a supercapacitor, which is refilled every third stop in 1-2 minutes using regenerative braking energy. The ability to use this technology on a wide scale has been proved to the world by China. In terms of the total running performance of buses, however, the technology utilising a supercapacitor and battery is more favourable.



Fig. 1.3 Hybrid bus in Mumbai , India

- **Automotive**

In an electric vehicle, the back emf generated by the motors is used to charge the supercapacitor while braking. Supercapacitors have a quick charge time. The vehicle's electrical system, engine, and battery are all powered by the stored energy. It is also utilised to recharge the battery. The first batch of Tigor EVs produced by Tata Motors for the government-run company EESL (Energy Efficiency Services) have just been released in India.

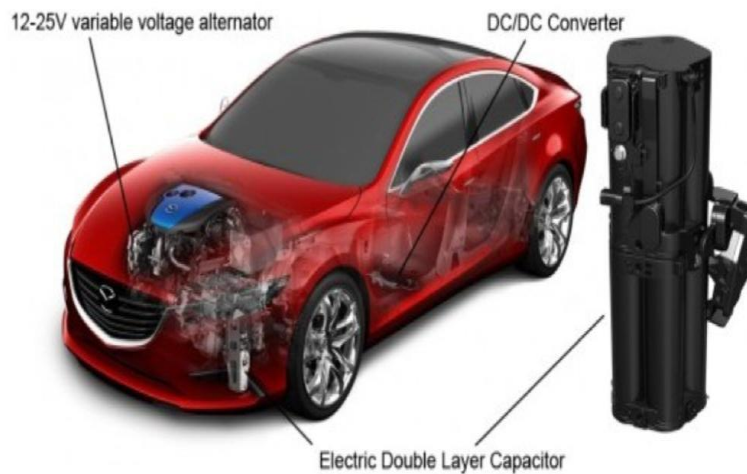


Fig. 1.4 Electric vehicles using Supercapacitor

- **Traction**

The three different phases that the traction vehicle must go through are the acceleration phase, the cruising phase (driving at a constant speed), and the deceleration phase. The power can be both immensely positive and negative throughout these stages. Serious problems are brought about by this change in power, including supply voltage fluctuations, losses in the primary power supply, etc. Super capacitors are integrated with a dc-dc converter as a short-term storage device to solve the problem mentioned above.

- **Electronic and low power application**

Supercapacitors are utilised in radio tuners, mobile phones, laptops, and other memory-related components. Wherever a brief power burst is needed, such as with LED flash units, they are used.

- **Renewable energy**

Three-bladed advanced variable speed turbines are employed today. Depending on the turbine's operating point and wind speed, the blades' angle can be changed. The blades must be promptly forced to the 90° position in the event of a turbine or power converter failure in order to protect them from mechanical harm. Wind energy uses supercapacitors to supply power for regulating blade pitch.

1.5 Advantages of Supercapacitors

- Super capacitors have a large energy storage capacity compared to electrolyte capacitors and batteries because they use activated carbon material, which improves capacitance value.
- A longer shelf life than batteries. In batteries, energy is stored and released by a chemical reaction that degrades the electrode material.
- Super capacitors can supply high and frequent power demand peaks and can recharge quickly.
- Super capacitors are able to deliver significant power bursts for a brief period of time and have a high power density.

Chapter 2

Materials and Methods

2.1 Materials

- High surface area amorphous carbon (AC)
- Conductive additives :Acetylene black (AB)
- Binder :poly tetrafluoroethylene (PTFE)
- Pottasium hydroxide (KOH)
- - Activated carbon



Fig. 2.1 Amorphous carbon

Because of its very porous nature, carbon is capable of being converted into many other forms with a very high specific surface area. This is a primary justification for the use of carbon as an electrode material. Energy storage has seen successful use of activated carbons (ACs). The physical (thermal) and chemical activation of carbon, which is plentiful in the environment, are both possible. When a structure is physically activated (thermally), hot gases are employed to transform the structure into ACs. A temperature range of 500 to 1100 °C is typically used for carbonization. Despite the fact that chemical activation requires lower temperatures (between 400 and 800 °C), its pyrolysis and activation are carried out in the presence of dehydrating agents. Carbon has several allotropes depending on the hybridization; graphitic carbon has layers of graphene whereas non-graphitic carbon lacks the long-range 3-D network. The structural characteristics of ACs are similar to those of pure graphite. Micropores (nanopores), mesopores, and macropores are three different types of porous structures that can be used to make ACs. Although they do not increase the adsorption capacity, these pores are crucial to the kinetics of adsorption. ACs with various porosity structural regions can result by varying the conditions during carbonization and activation.

- Acetylene black

For the purpose of improving the prepared electrodes' electrical conductivity and lowering charge transfer resistance, conductive chemicals are added to the electrode slurry. In order to prepare electrodes, carbon black is a commonly used conductive addition. Carbon black comes in various forms, including acetylene black. It is made through the thermal breakdown of acetylene. This kind of carbon black is very pure and exceedingly conductive. As a result, it plays a crucial role in the manufacturing of silicon goods, electrical wires, and dry cells, among other things. Acetylene black can be used as a replacement for carbon black.



fig. 2.2 Acetylene black

➤ PTFE

The polymeric binders improve the mechanical durability of the electrodes by aiding in the electrode material's strong adhesion to the current collector. The only components of the PTFE binder are perfluoro ethylene units, which give the electrodes hydrophobicity and insulator behaviour. This leads to poor electrolyte wettability and greater contact resistance.

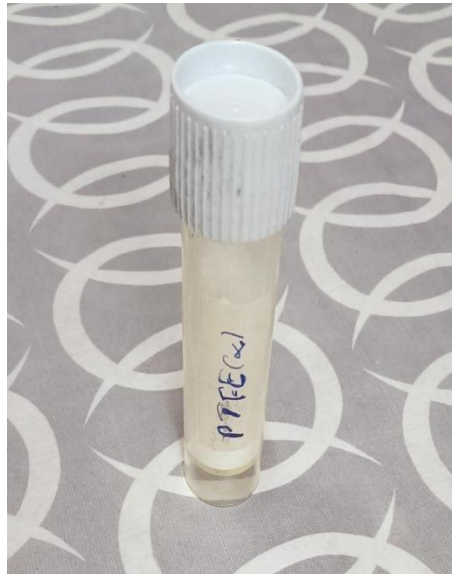


Fig 2.3 PTFE

➤ **Potassium hydroxide (KOH)**

Potassium hydroxide (KOH), an alkaline aqueous electrolyte, is frequently employed in carbon-based supercapacitors due to its non-toxicity, low cost, and good conductivity. The KOH electrolyte has a 1.0 V potential window. When compared to 1 M KOH, 6 M KOH has a higher specific conductivity (0.6266 S cm⁻¹) at room temperature, making it better suited for boosting electrochemical characteristics. Thus, the electrolyte for systems with three and two electrodes is typically 6M KOH.

Hydraulic press

To effectively complete heavy processing operations, hydraulic press machines are frequently utilised in industrial applications. The closed liquid in the hydraulic cylinder produces a significant amount of compression force with only a little amount of applied force. It has a movable die space, stroke length, and applied pressure. Metal, plastic, wood, rubber, and other materials can all be processed using these devices. Applications include powder metallurgy, metal forming, moulding, bending, forging, sheet

drawing, cold extrusion, straightening, flanging, and stamping. The hydraulic press machine is frequently used in the production of electrical goods, auto parts, rails, knives, and aircraft.



Fig. 2.4 Hydraulic press

2.2 Fabrication of electrodes

Activated carbons are typically blended with acetylene black and poly tetrafluoroethylene in an 8:8:1 weight ratio, homogenised, and distributed in an ethanol solution to create electrodes. To ensure complete evaporation of the water content, the slurry was then uniformly deposited on a nickel mesh current collector and dried at 60°C for 8 hours in an oven. In order to assess the electrochemical behaviour, the two electrode tests were carried out using activated carbon as the working electrode in electrolyte solutions containing 6 M KOH.

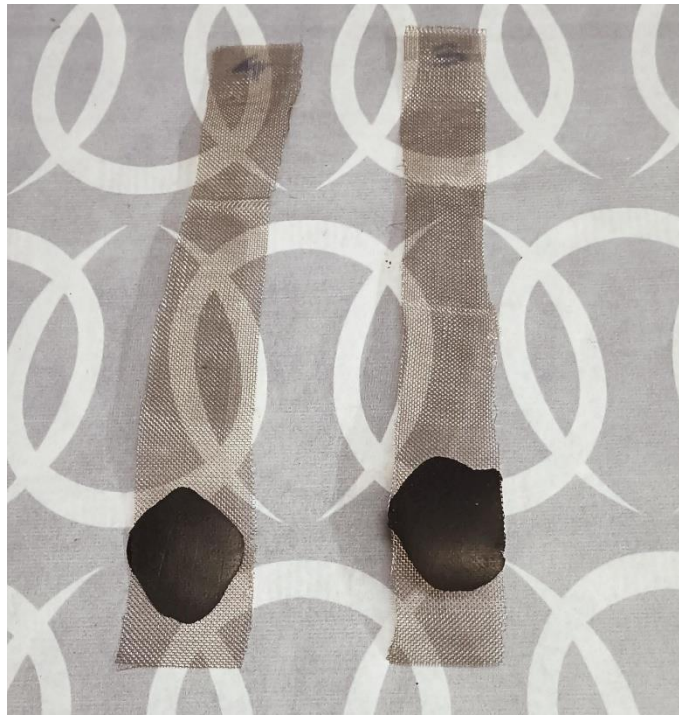


fig. 2.5 electrodes coated with material

Chapter 3

Characterization techniques

CHARACTERIZATION TECHNIQUES

3.1 Electrochemical methods

- **Cyclic voltammetry (CV)**

Regarding renewable energy, cyclic voltammetry (CV), an effective method for assessing electrochemical processes, has received a lot of attention. The charge-transfer kinetics of electrodes, the concentration of redox couples, chemical reaction mechanisms, the diffusion coefficient of redox couples, and other topics are covered in CV studies. The formal potential of redox couples and its close relationship to their standard reduction potential are also covered. When the capacitance is simply derived from the EDL, the CV curves are rectangular, and the capacitance is approximated from the measured current density at the middle of the potential range, I , and the potential scan rate, r , that is, $C = I/r$. The electrolyte solution determines the potential range, V . Because the CV curve is not always rectangular, it is important to carefully select the point of the potential for the capacitance calculation when the pseudo-capacitance is included. The CV curves get twisted and even unbalanced as r increases. If the pseudo-capacitance is included, this is indicated. As a result, $C = Q/(2V)$, the total electric charge determined by integrating a CV curve, is frequently utilised. The value of r should be sufficiently low to generate strong performance because, generally, the value of C diminishes as r increases. By using potentiostatic polarisation, the value of Q can also be determined from a current decay curve. Again, at constant current density, I , when the capacitance is simply derived from EDL, the potential of chronopotentiograms, V , changes linearly with time, t . Then, the capacitance is determined by the linear relationship's slope, dV/dt , which is represented by the equation $C = I/(dV/dt)$. The chronopotentiogram departs from the linear connection, similar to CV, when factors other than EDL contribute to the capacitance.

- **Electrochemical impedance spectroscopy (EIS)**

EIS, another electrochemical method used in conjunction with CV, measures impedance. The foundation of EIS is a constant DC bias, small-amplitude AC signal. Equivalent circuits made of passive components like resistance, capacitance, and Warburg impedance have been used to analyse EIS data. The charge-transfer resistance, double-layer capacitance, and diffusion impedance are among the elements that are given a physical significance. For nonfaradic processes like double-layer capacitance, experimental data have consistently deviated from a straightforward description. By providing a tiny amplitude of an alternate potential over a broad range of frequency, f (for example, 1 mHz to 1 MHz), EIS is typically conducted on capacitor electrodes at the open-circuit potential. The Bode plot, which uses a linear section of the $\log |Z|$ vs. $\log f$ curve, provides impedance, $|Z|$ versus f , and capacitance is given by $C = 1/(2\pi f|Z|)$. Of course, capacitance depends on frequency. In a Nyquist diagram, where the imaginary part of impedance, $Z(f)''$, is plotted against the real part of impedance, $Z(f)'$, a charge-transfer resistance can also be determined from the diameter of a semicircle. The numerical values of the resistance determined by a Nyquist diagram, however, are not exact since complete semicircles are rarely attained.

- **Galvanostatic charge discharge(GCD)**

Two-electrode cells are used to measure the galvanostatic charge/discharge (GCD). Similar to GCP, the value of C is obtained, except that it applies to the entire cell. Since the electrode materials can occasionally be harmed by high potentials, consideration must also be given to the collector and counter electrode materials when establishing V in non-aqueous systems, in addition to the electrolytes and solvents. It is advised to conduct measurements using two electrode cells and three electrode cells on the same electrode material. The device's power and energy densities as determined using GCD curves.

3.2 Material characterization

- **Scanning electron microscopy (SEM)**

The SEM is a type of electron microscopy that allows for the direct viewing of a specimen's surface features at the micro and submicron scales by scanning the surface with an electron beam. The SEM has been employed in many fields of science and industry because of the enormous magnifications and outstanding resolutions it is capable of, particularly in materials engineering, biological, and medical sciences for the investigation and characterization of the micro-structure of substances. Its application offers a way to address some of the activated carbon porosity generation and characterization problems that have not yet been answered. The SEM operates in the same way as its optical predecessors, but to "image" a specimen and learn more about its structure and composition, it employs a concentrated electron beam rather than light. The SEM can provide data on an object's topography (surface characteristics), morphology (particle shape and size), composition (the elements present in the object and their relative amounts), and crystallographic information (the arrangement of atoms within the object). Because of this capability, the SEM is a very valuable tool for studying activated carbons. The topographic data obtained from the SEM enables the study and measurement of surface aspects such as pore characteristics, the description of which has been a primary focus of activated carbon chemists. Additionally, SEM is an effective tool for researching the surface chemistry of activated carbons due to its capacity to expose a specimen's compositional features. The SEM also has other characteristics that make it a special tool for investigating activated carbons, such as its capacity to reveal information about a sample that is less than 1 nm in size. Thus, even microscopic pores might theoretically be made visible for SEM micrograph analysis. SEM micrographs, once more, have a wide depth of field that produces a nearly three-dimensional appearance that is helpful for analysing the surface structure of a sample. This is owing to the extremely narrow electron beam used in the process.

- **Energy dispersive x-ray analysis (EDX)**

In order to create a specimen map, energy-dispersive X-ray analysis (EDX) is used in conjunction with scanning electron microscopy (SEM) to analyse the types and quantities of elements at the nanomaterial surface or close to the surface. Since X-rays only develop in a 2- μ m-deep region, EDX is not a reliable method for surface characterisation. The image of the elements in the sample is created during EDX analysis as the electron beam is moved across the sample. In most cases, this procedure takes many hours. While elements with atomic numbers below 11 are difficult to detect by EDX, it is

possible to measure the composition and quantity of heavy metal ions in nanoparticles that are placed close or at the surface of sample.

- **X-ray diffraction**

Utilising an X-ray diffractometer, the structural characteristics of the activated carbon black were examined. A two-dimensional semiconductor X-ray detector was used in conjunction with an 18 kilowatt X-ray generator. Within the scanning range of 10-60 and at a scanning speed of 1 m/s, XRD diffraction peaks were observed in a 2θ continuous scanning mode. The collected data were then subjected to a 2θ -calibration procedure utilising a standard silicon specimen that had been annealed. The diffraction intensity ratio $I_{\text{crystalline}}/I_{\text{amorphous}}$ is proportional to the corresponding mass ratios for determining the degree of crystallinity using X-ray diffraction analysis, provided that the crystalline and amorphous sections have the same chemical make-up. In this case, the crystalline portion is represented by the diffraction intensity $I_{\text{crystalline}}$, whereas the amorphous portion is represented by $I_{\text{amorphous}}$. Based on the maximum intensity of the 002 peak, the relative fractions of the crystalline and amorphous sections were computed in the current investigation. Using the equations shown below, the crystalline and amorphous fractions were computed.

$$\text{crystalline carbon proportion (\%)} = (I_{\text{crystalline}} / I_{\text{total}}) * 100$$

$$\text{amorphous carbon proportion (\%)} = (I_{\text{amorphous}} / I_{\text{total}}) * 100$$

Chapter 4

RESULT AND DISCUSSIONS

4.1 From cv

When capacitance C is fixed with sweeping voltage, current will remain constant because $I(t) = C \frac{dv}{dt} = C \cdot S$. Therefore, it is predicted that the resulting cyclic voltammetry curves of perfect capacitors will display rectangular current response if the same scan rate of dv/dt is used in both the forward and reverse directions of the sweep.

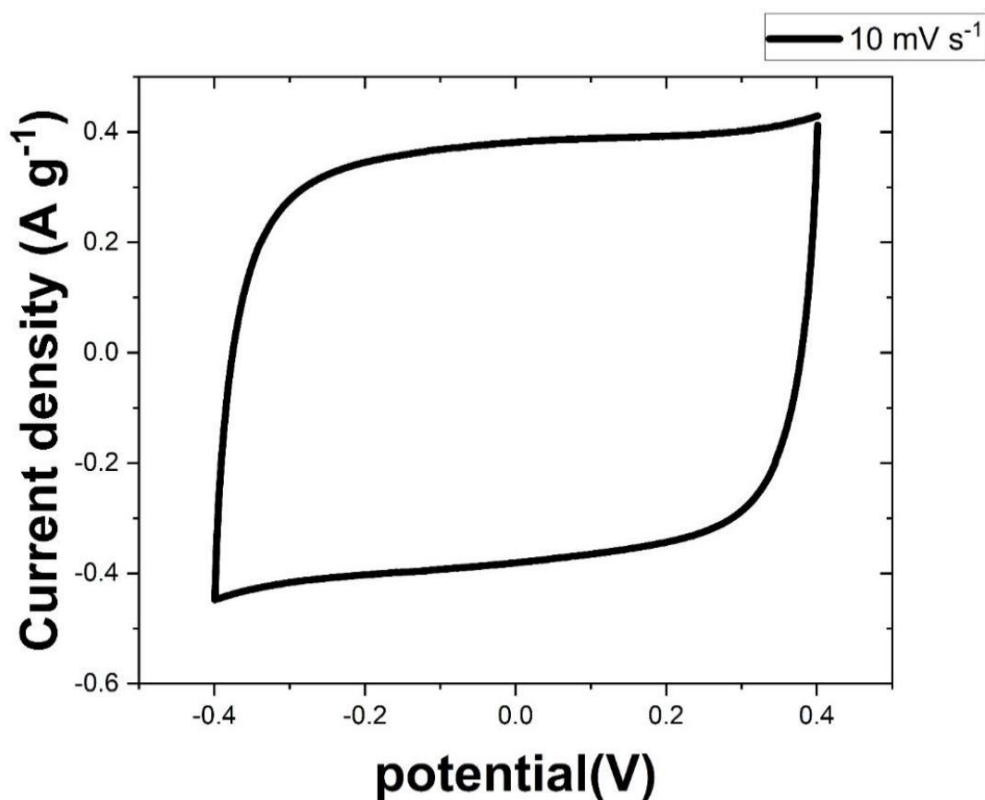


Fig. 4.1

This experiment's cv curve, which is virtually rectangular, indicates capacitive behaviour.

Scan rate= .01 V

Average mass of the electrode material=0.0323 g

From the curve,

Average current density=0.34783A/g

Capacitance= current density/ scan rate in V

$$C = 0.3478 / 0.01$$

$$= 34.78 \text{ F/g}$$

4.2 From EIS

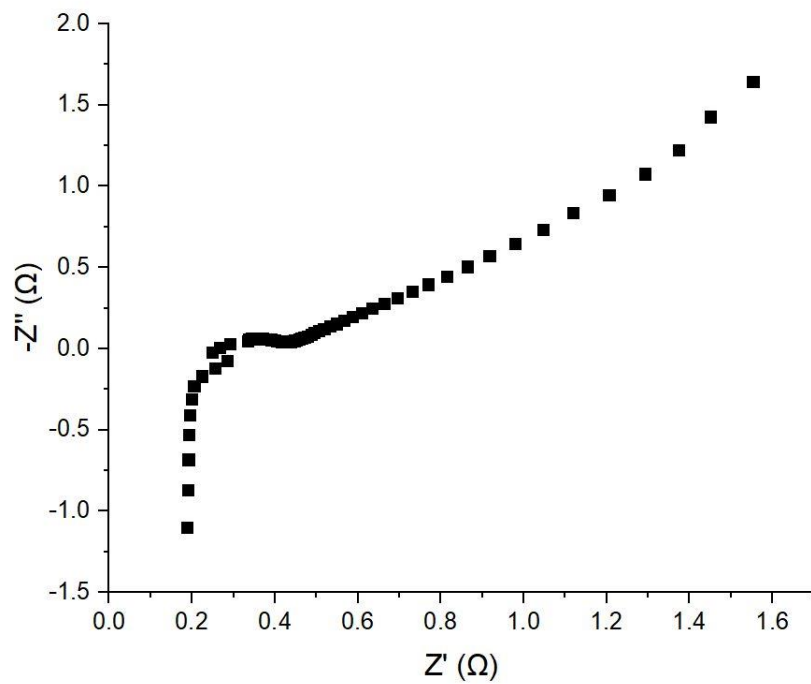


Fig. 4.2

This figure shows Nyquist plots of AC in the KOH electrolyte obtained from the EIS experiment.

4.3 From GCD

Charge discharge 1A/g

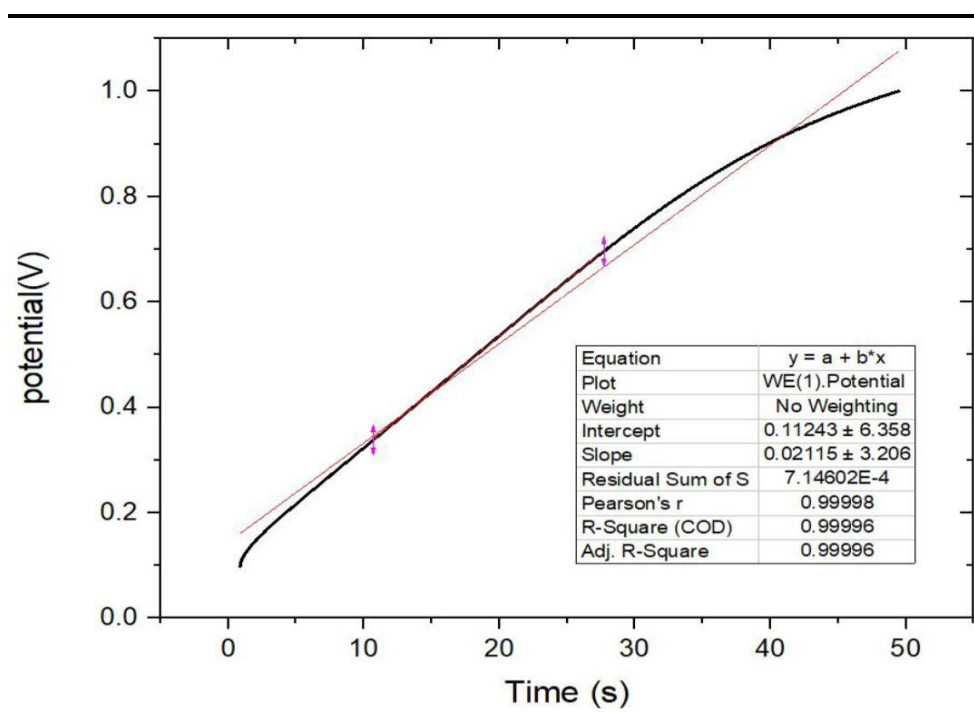


Fig. 4.3

Slope of discharge curve=0.02115

Capacitance $C = I/(V/t)$

$$=1/0.02115$$

$$=47.2813$$

Specific capacitance= 94.5626 F/g

Charge discharge 2 A/g

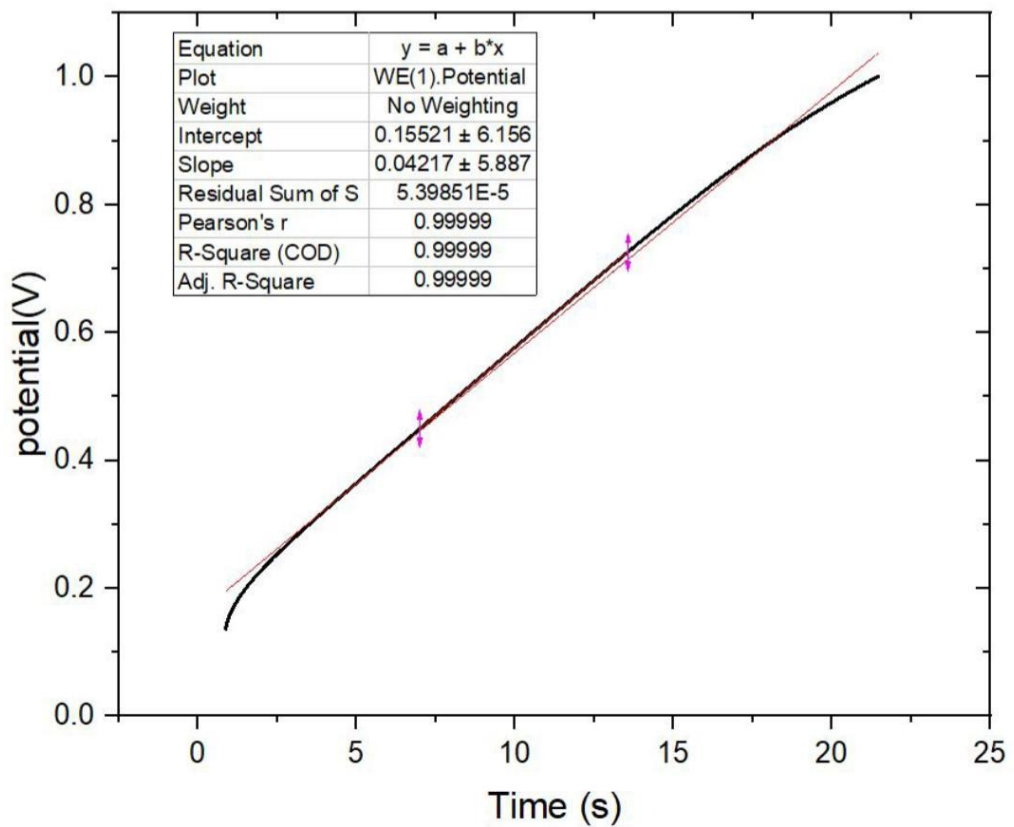


Fig. 4.4

Slope of the curve= 0.04217

$$\text{Capacitance } C = I/(V/t)$$

$$= 2/0.04217$$

$$= 47.4271$$

$$\text{Specific capacitance} = 94.8542 \text{ F/g}$$

Charge discharge 10 A/g

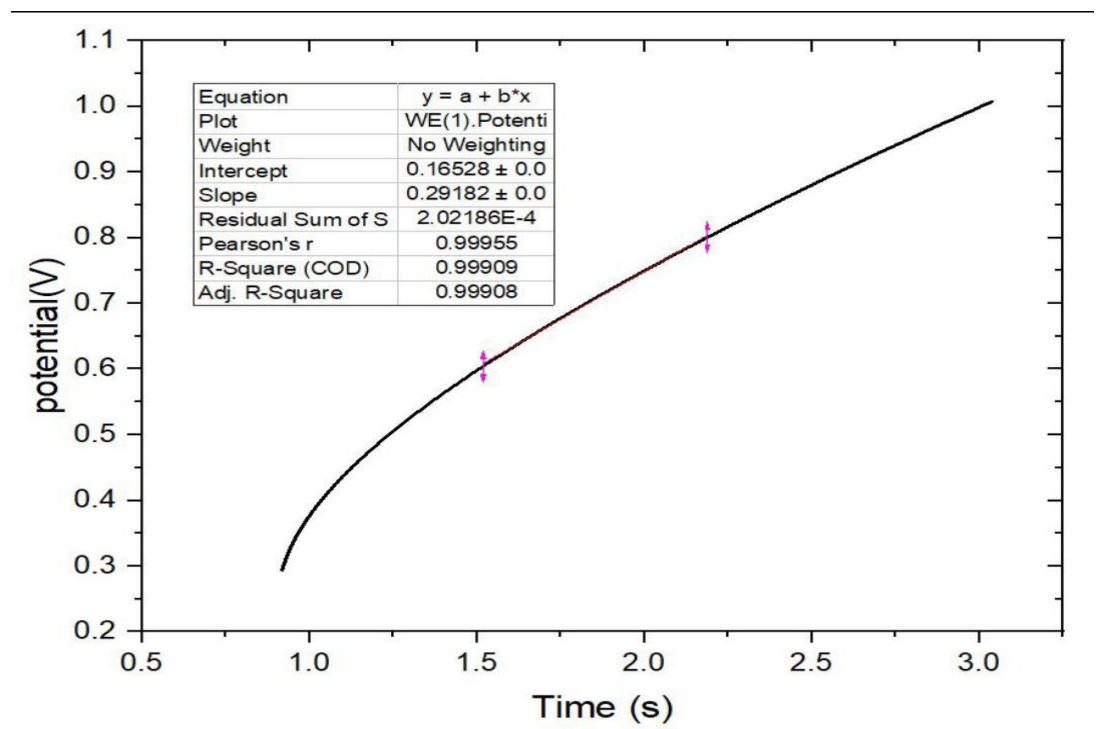


Fig. 4.5

$$\text{Slope of the curve} = 0.29182$$

$$\text{Capacitance } C = I/(v/t)$$

$$= 10/0.29182$$

$$= 34.2677$$

$$\text{Specific capacitance} = 68.5354 \text{ F/g}$$

The galvanostatic experiments were performed for more accurate capacitance assessment as the measurement from the CV are not sometimes reliable. They were measured at different current density range is 1 A/g, 2 A/g and 10 A/g. Highest capacitance value was obtained at 2A/g of KOH electrolyte.

4.4 X Rd analysis

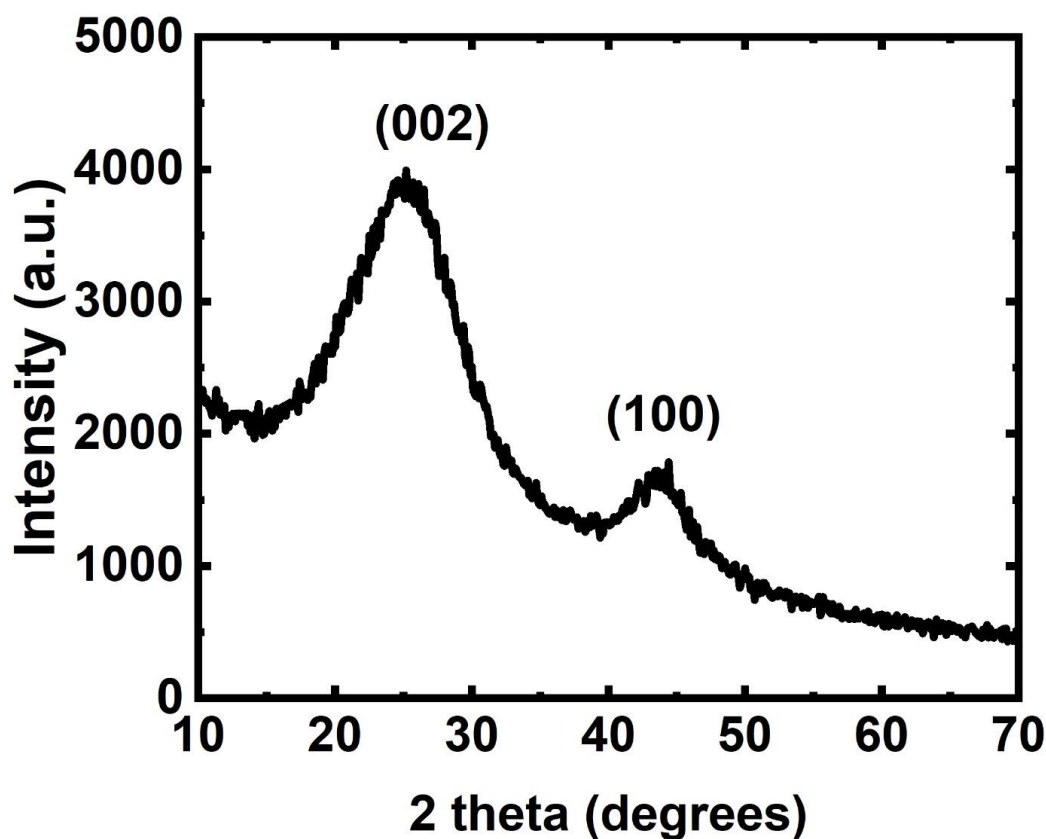


Fig. 4.6 xrd analysis

In order to learn more about the structure of crystalline and non-crystalline materials, XRD uses the dual wave/particle characteristics of x-rays. The amorphous or graphitic phases of carbon were the focus of the XRD analysis. Amorphous phase, which is of short order, exhibits a hump in broad-range XRD. Fig. 4.6 displays the XRD patterns of activated carbon. The presence of the 002 peak and the 100 peak suggests that our carbon is not completely amorphous but rather possesses crystallinity.

4.5 Sem analysis

We also used SEM to examine the structure of the activated carbon to establish that it is the source of the porosity, as shown in Fig. A highly porous structure that is typical of activated carbons was seen. In the course of the carbonization process, pores form in the carbon and encourage the diffusion of koh molecules into them, increasing the koh-carbon reactions and leading to the development of more pores in the activated carbon. This would increase the prepared activated carbon's surface area and pore volume.

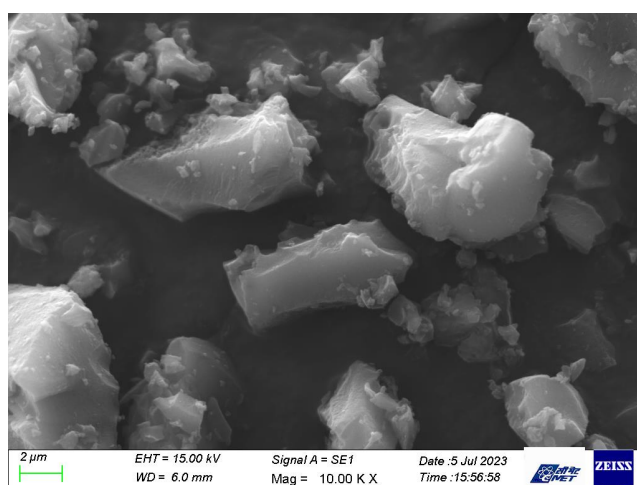
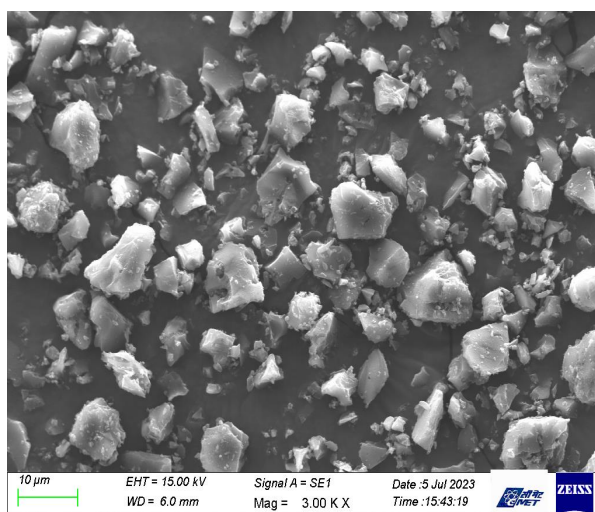


Fig . 4.7 sem analysis of carbon

4.6 EDX analysis

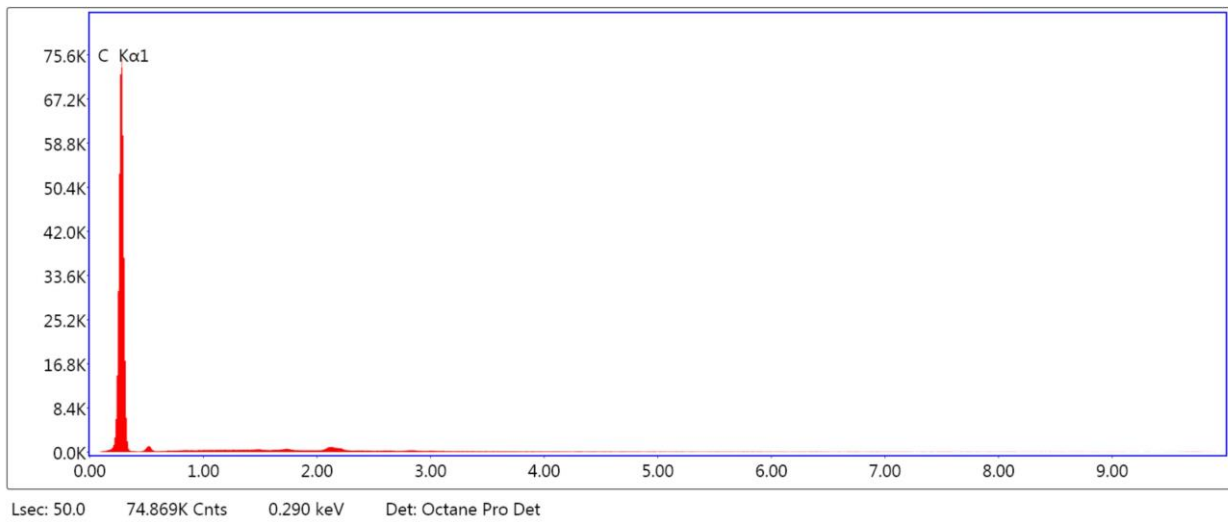


Fig. 4.8 EDX analysis

In the EDX analysis , the higher the peak in a spectrum , the more concentrated the element is in the specimen.

CHAPTER 5

SUMMARY AND CONCLUSIONS

Conclusion

The present work describes the fabrication of high surface area amorphous carbon based electrodes for EDLC Applications. Cyclic voltammetry, galvanostatic charge/discharge and electrochemical impedance spectroscopy have been used to compare the electrochemical behaviors for the supercapacitors electrode based on the mesoporous AC material in 6 M KOH, electrolyte solutions. AC electrode in 6 M KOH exhibited excellent electrochemical storage properties including a highest specific capacitance of 94.8542 F g^{-1} at 2 A g^{-1} . In conclusion, supercapacitors will be crucial for energy storage in the future. Supercapacitors are necessary in several applications that need for high power or great safety. Carbon materials will continue to draw attention from researchers because they are the most often used electrode materials for supercapacitors.