Electrochemical Double Layer Capacitor (EDLC)

by

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(Chemical Engineering)

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Chemical Engineering Programme
Universiti Teknologi PETRONAS
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CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

MARLENE KERESHANAN

ABSTRACT

This project investigated the performance of the supercacitor using carbon electrodes with an aprotic electrolyte Lithium Perchlorate ($LiClO_4$) salt in Acetonitrile solvent. The experiments were done to study the life cycle, the voltage profiles and the capacitance profiles of this new assembled supercapacitor test cell. The samples used in these experiments are VGAF SHOW A DENKO and TIMREX HSAG 100 CAT Special Graphite M292. The methodology used in this experiment was the identification of equipments and items needed, material testing, running the experiments and conclusion of findings. The width of the electrochemical stability window of organic solvents is wide often exceeding 2 V that will be used in this experiment. The test cells assembled in these experiments were tested within the range of applied currents of 0.001A, 0.005A, 0.01A and 0.05A. In conclusion, this experiment showed that the specific capacities of the samples tested are highest when the current applied is at the lowest value of 0.001A. In the case of the first experiment, the specific capacity was 3.51mAh/g and in the second experiment, it was 5.83mAh/g .The voltage profile also shows that the samples are more stable at a lower applied current value of 0.001A.

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CHAPTER 1

INTRODUCTION

1.1 Project Background

There are three types of capacitor. They are the conventional capacitor that works based on electrostatic charges, the electrolytic capacitor which functions due to the ionic liquid that acts an electrolyte and of course the EDLC. However, this experiment is about carbon based Supercapacitor or Electrochemical Double Layer Capacitor (EDLC). The main objective of this experiment is to test the performance of the supercapacitor using carbon electrodes in an aprotic electrolyte Lithium Perchlorate ($LiClO_4$) salt in Acetonitrile solvent. Acetonitrile (ACN) is the chemical compound with formula CH_3CN. This colourless liquid is the simplest organic nitrile and is widely used as a solvent [1]. In this dual carbon cell, the cation, (Li^+) intercalates into the negative electrode and the anion, (ClO^-) intercalate into the positive electrode. A capacitor is simpler in the way that its only function is to store electrons as it can't produce them [2] .In this experiment. The negative ions are due to the electric field were gathered and the relatively positive and negative plate interface to form the dual-layer. The specific capacity of such devices is of the order of 0.1 mF/kg at an AC voltage of 230 V and 50 Hz frequency [5]. In this experiment, we would be testing the test cell just like a supercacitor. The concept of the supercapacitor is the same with the capacitor just difference in terms of materials used and performance. A large commercial EDLC would have capacity as high as 5000 farads as compared to electric capacitor which has the capacity of several farads and the same sized electrolytic capacitor will have a capacitance in the range of tens of millifarads [27].

The electrolyte may be aqueous or organic. Aqueous electrolytes offer low internal resistances but limit the voltage to one volt. In contrast, the organic electrolyte allows 2.5 volts of charge, but the internal resistance is higher [3]. Aqueous electrolytes such as acid (e.g. Sulfuric Acid) and Alkalis (e.g. Potassium Hydroxide) have the advantage of high ionic conductivity. These electrolytes have low cost and wide acceptance [4]. Electrolytes commonly exist as solutions of acids, bases, or salts. Sodium is attractive because of its high reduction potential of -2.71 volts, its low weight, its non toxic nature, its relative abundance and ready availability and its low cost. To construct batteries the sodium must be used in liquid form. Some examples of organic electrolyte used in experimentation are aprotic $(C_2H_5)_4NBF_4$ in acetonitrile, Organic salts (tetraethyl ammonium cation with anions: BF_4^-, PF_6^-, $CF_3SO_3^-$, and ClO_4^-) dissolved in non-aqueous solvents (acetonitrile, propylene carbonate, sulpholan)

1.2 Problem Statement

To study the life cycle and also the voltage profiles and capacitance profiles of this new assembled supercapacitor test cell to further analyze the life cycle of the cell using specific samples; TIMREX HSAG 100 CAT Special Graphite M-292 and VGAF SHOW A DENKO. The experiments conducted in the laboratory will be using equipments such as test cell, battery cycler tester, SEM (Scanning Electron Microscope) and X-RD (X-Ray Diffraction)

1.3 Objective

This final year project plans to investigate and study the performance of the supercacitor or better known as Electrochemical Double-Layer Capacitors using graphite and carbon electrodes with an aprotic electrolyte Lithium Perchlorate ($LiClO_4$) salt in Acetonitrile solvent. The data obtained will be the voltage versus the testing time using the software called MTIS Pro-ArBin and the capacity will be calculated.

1.4 Scope of Study

The focus of this study is based on the concept of electrochemistry. The study of the concept of capacitance and also the storage capacity of the project will be done. Non-aqueous aprotic electrolyte Lithium Perchlorate ($LiClO_4$) salt in Acetonitrile solvent will be used in this experiment to analyze the capacity of the supercacitor. This results and findings are then compared based on the voltage, capacity profiles and also on the life cycle of the cell. In this experiment, various equipments will be used to test and determine the crystalline structures of the various different carbons used in this study as electrodes. Such are X-Ray Diffraction and SEM.

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1 Fundamentals

Energy storage devices are classified according to their energy and power densities. Supercapacitors, ultracapacitors and electrochemical double layer capacitors (EDLCs) are commonly used names for a class of electrochemical energy-storage devices that are ideally suited to the rapid storage and release of energy [4]. Supercapacitors are intermediate systems between dielectric capacitors and batteries. Because of their highly reversible charge storage process, supercapacitors have longer cycle-lives and can be both rapidly charged and discharged at power densities exceeding 1 $kWkg^{-1}$ [4]. Even though are batteries able to store higher energy density than supercapacitors, they deliver less power; as compared to dielectric capacitors, supercapacitors can store higher energy density with less delivered power. This particular property makes them suitable for numerous applications such as power electronics and military field equipment.

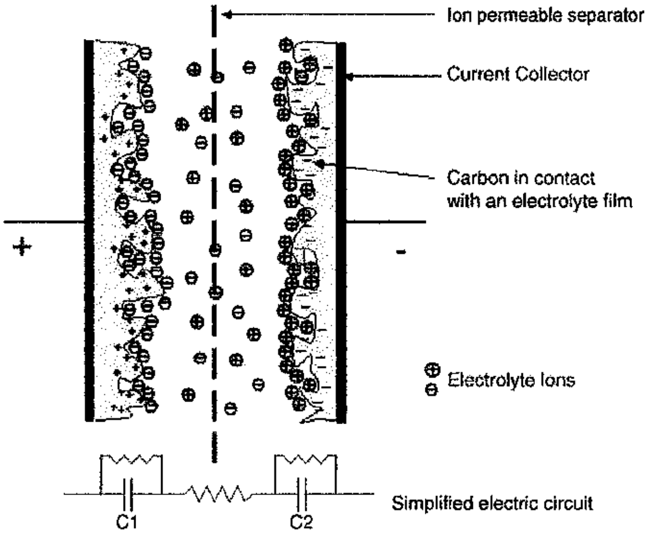


Figure 2.1.1: Representation of an electrochemical double layer capacitor (In its charged state).

There are 2 modes of energy for the supercapacitors; redox supercapacitors and ELDC (Electrochemical Double-Layer Capacitors). Redox supercapcitor undergoes reversible Faradaic-type charge transfer and the resulting capacitance, often large, is not electrostatic in origin. EDLC can store substantially more energy per unit mass or volume than a conventional capacitor because charge separation takes place across a very small distance between an electrode and the adjacent electrolyte and an increased amount of charge can be stored on the highly extended electrode surface-area [4].

Carbon/Carbon supercapacitors have been largely investigated because of their low-cost, high cycling-life and high capacitance. Variety of sizes are available from small (few farads) up to large-size (5000 F) devices are commercially available. Highly-porous carbons are used as electrode material due to their high surface area, good electronic conductivity and high electrochemical stability; the most frequently used is activated carbon ($1500–2000m^2g^{-1}$). Charge storage is performed through the reversible adsorption of the ions at the active material electrolyte interface; no faradic reactions occur during the charge–discharge of the supercapacitor [11].

The capacitance of the system is directly proportional to the electrode surface; therefore carbons are very efficient for this application because of the various possibilities of their modification and creation of a controlled pore size distribution. The electrostatic attraction of ions takes place in micropores although mesopores is needed for efficient charge propagation [12].

When the system of conductive electrodes is wetted by liquid electrolyte, the double electric layer forms at the electrode/electrolyte interface on the charge and this layer discharges during the operation thus, making it possible for electrical energy to be stored and delivered to the load on time. Since only the electrolyte-wetted surface-area contributes to capacitance, the carbon processing is required to generate predominantly 'open' pores that are connected to the bulk pore network [4]. The electrolyte can be aqueous or organic aprotic electrolyte [13]. In this experiment, we will be using an organic aprotic electrolyte as the aqueous electrolyte does not wet or impregnate the sample electrodes used.

Below are some of the advantages and limitation of a supercapacitor [17].

Advantages

- Unlimited cycle life – Hundreds of cycle at a time.
- Low impedance - enhances load handling when put in paralleled with a battery.
- Rapid charging, simple charge method

Limitations

- Low energy density - typically holds one-fifth to one-tenth the energy of an electrochemical battery.
- Cells have low voltages - serial connections are needed to obtain higher voltages. Voltage balancing is required if more than three capacitors are connected in series.
- High self-discharge - the rate is considerably higher than that of an electrochemical battery.

2.2 Specific energy, specific power and capacity

The specific energy stored and the specific power that can be delivered to the load are no doubt the characteristics of a supercapacitor besides the other factors like cycling life, self discharge current and efficiency [13]. The vast increases in capacitance achieved by supercapacitors are due to the combination of:

(i) an extremely small distance that separates the opposite charges, as defined by the electric double-layer;

(ii) Highly porous electrodes that embody very high surface-area [4].

Specific energy is as mentioned proportional to the capacitance and also voltage squared and both parameters are close to their limit in the supercapacitors devices compromising two carbonaceous electrodes and their organic electrolyte. Double layer capacitance is 12 (+/-2) μF/sq.cm for porous carbon materials [13]. Supercapacitors also have a higher specific power than most batteries, but their specific energy is somewhat lower [4]. Specific energy is defined as the energy per unit mass. Specific power can refer to power either per unit of mass, volume or area.

There are several factors which governs the capacity of a particular battery. They are physical size, discharge rate, temperature and cut off voltage. The ability to absorb energy is, to a large extent, limited by the size of the charger. The self-discharge of the supercapacitor is substantially higher than that of the electro-chemical battery. Supercapacitors with an organic electrolyte are affected the most. In 30 to 40 days, the capacity decreases from full charge to 50 percent [16].

2.3 Specific Capacitance, Energy and Power, Specific Capacity and Capacity Calculations

The cell capacitance is deduced from the slope of the discharge curve whereby

$$C = \frac{I}{\frac{\mathrm{d}V}{\mathrm{d}t}} \qquad [11]$$

Where C is the cell capacitance in Farad (F),

I the discharge current in Ampere (A) and $\mathrm{d}V/\mathrm{d}t$ is the slope of the discharge curve in volts per second ($\mathrm{V\ s^{-1}}$).

The specific capacitance $C_{m\mathrm{AM}}$in Farad per gram of active material ($\mathrm{F\ g^{-1}}$) is related to the capacitance of the cell C by

$$C_{m\mathrm{AM}} = \frac{2C}{m_{\mathrm{AM}}} \qquad [11]$$

Where mAM is the weight (g) per electrode of the active material.

The energy (E) and power (Pmax) of a supercapcitor can be obtained according to;

$$E = \frac{1}{2}CV^2$$ [4]

$$P_{\max} = \frac{V^2}{4R}$$ [4]

Where C is the capacitance in Farads, V is the nominal Voltage and R is the Resistance.

The Capacity is calculated using;

$$\mathbf{Q\ (A.s) = IT}$$

Specific Capacity is obtained when the Charge/Discharge Capacity is divided by mass;

$$\mathbf{Q_m\ (mAh/g) = \frac{Q}{m}}$$

Where m is the weight (g) per electrode of the active material

2.4 Materials

2.4.1 Electrode and Carbon

There are three types of electrode materials suitable for the supercapacitors which have high surface area. They are activated carbons, metal oxide and conducting polymers [17]. In the case of carbon electrodes, there are two contributions to the supercapacitor capacitance: the double layer capacitance and the pseudocapacitance. The latter is attributed to functional groups mainly located on carbon edges on the surface. The double layer capacitance value is built proportionally to the accessible carbon surface [19].

Typical capacitance of the double layer amounts to approximately 10 lF/cm^3. In order to obtain an electrochemical capacitor of high capacitance, its electrodes should be made of a material of high electric conduction and specific surface. Such features are characteristic for activated carbons. Activated carbons are produced by carbonization of natural organic materials (wood, coconut shell, hard coal, fruit stones) or synthetic ones (polymers, resins). The final formation of the surface of activated carbons occurs during the activation process. The surface of activated carbon contains oxygen-containing functional groups, able to enter into chemical reactions [4].

The high surface electrode material, also called Double Layer Capacitor (DLC), is least costly to manufacture and is the most common Carbon-paste electrode (CPE) is made from a mixture of conducting graphite powder and a pasting liquid. These electrodes are simple to make and offer an easily renewable surface for exchange. Carbon paste electrodes belong to a special group of heterogeneous carbon electrodes. These electrodes are widely used mainly for voltammetric measurements [14]. A variety of porous forms of carbon are currently preferred as the electrode materials because they have exceptionally high surface areas, relatively high electronic conductivity, and acceptable cost [4].

The factor of energy/charge storage into carbon is affected by the accommodation of carbon which highly depends on the crystallinity, microstructure and micromorphology of the carbonaceous material [15]. The important carbon parameter to optimize is the volumetric capacitance density. This is achieved by choosing the correct pore size which must be tuned on the ions size. Typical commercial carbons are in the range of 50 F/cm^3 while their mass capacitance density is in the range of 100 F/g [19]. The table below indicates the double layer capacitance for some carbonaceous materials along with the electrolytes used [4].

Table 2.4.1: Values for Electrochemical Double Layer Capacitance of Carbonaceous Materials

Carbonaceous Materials	Electrolyte	Double Layer Capacitance ($\mu F\ cm^{-2}$)	Remarks (m^2g^{-1})
Activated Carbon	10% NaCl	19	Surface area 1200
Graphite Powder	10% NaCl	35	Surface area 4
Carbon Black	1M H_2SO_4	8	Surface area 80-230
Carbon Black	31 wt.% KOH	10	-
Carbon Aerogel	4M KOH	23	Surface area 650

The electrode properties are also determined by some geometric factors. They are the result of a compromise between the required energy and power densities. To get high power, the ionic and the electronic paths must be minimized in the system. The electrode thickness is made as small as possible to reduce the path length for the ions. The electrode width is made as short as possible to reduce the path length for the electrons [19]. The direct consequence of these two measures is that in a given volume the number of parallel layers which are stacked or rolled is increased. It follows that the cross section for the current is increased while the distance to be crossed is decreased. These geometric consideration leads to low series resistance and to high power capabilities.

The carbon purity also plays an important role as the key property which must be taken into consideration. This will condition the potential window in which the supercapacitor is stable during its life. The impurity redox reactions will be the root of the pressure build up in the supercapacitor cell. A too big amount may lead to the opening of the cell with the consequence of electrolyte leakage. They will also accelerate the self discharge and the aging of the component. The capacitance drop and the series resistance increase are accelerated [19].

The attraction of supercapacitor carbon electrode as arises from factors such as [2];

- High conductivity
- High surface area
- Good corrosion resistance

- High temperature stability
- Low cost
- Processability and compatibility in composite materials
- Relatively low cost
- Controlled pore structure

2.4.2 Electrolyte

The electrolyte may be aqueous or organic. The aqueous variety offers low internal resistance but limits the voltage to one volt. In contrast, the organic electrolyte allows 2.5 volts or more of charge, but the internal resistance is higher [17]. In this experiment, we will be using an organic aprotic electrolyte as the aqueous electrolyte does not wet or impregnate the sample electrodes used. The electrolyte used will be a Lithium Perchlorate ($LiClO_4$) salt in Acetonitrile solvent. A suitable electrolyte for supercapacitors must have the following characteristics [18]:

- High ionic conductivity to minimize the cell resistance and resistive heating of the device.
- High melting point to provide sufficient conductivity at sub-ambient temperatures and prevent solidification and phase separation.
- Low cost
- Non toxicity to be environmentally accepted for ease handling and waste treatment.

Operating voltage of supercapacitors usually depends on the electrolyte stability. In electrochemical capacitors aqueous solvents are commonly used. The width of the electrochemical stability window of such solvents amounts to approximately 0.8–1.0 V, due to water decomposition. Organic solvents on the other hand have a wider electrochemical stability window, often exceeding 2 V [20]. The electrochemical stability window is a potential range within which the Faradaic current does not flow. The width of the electrochemical stability window depends on the electrode material, the solvent, and the type of electrolyte [20].

Some of the reportedly used electrolytes are 2M H2SO4 aqueous solution and 1M (C2H5)$_4$NBF$_4$ in acetonitrile. The charge storage properties were tested by galvanostatic charge–discharge voltage cycles at current densities, j, between 1 and 100mAcm^{-2} in the range of 0–0.8V for the aqueous electrolyte and 0–2V in the aprotic solution [21]. The limiting capacitances measured at 1mAcm^{-2} are respectively *C*o-aprotic and *C*o-acidic. Table 1 indicates the electrochemical properties for the carbide-derived carbons using these electrolytes [21].

Table 2.4.2: Electrochemical properties for the carbide-derived carbons

Carbon	$(C_2H_5)_4NBF_4$/acetonitrile				H_2SO_4				$C_{o\text{-aprotic}}/C_{o\text{-acidic}}$
	$C_{o\text{-aprotic}}$			C_{50}/C_o	$C_{o\text{-acidic}}$			C_{50}/C_o	
	Fg^{-1}	Fm^{-2}	Fcm^{-3}		Fg^{-1}	Fm^{-2}	Fcm^{-3}		
SiC	129	0.096	51.4	0.90	153	0.114	61.0	0.80	0.84
TiC/SiC	130	0.100	57.7	0.89	153	0.118	67.9	0.86	0.85
TiC/TiO_2	128	0.087	52.8	0.84	217	0.148	89.5	0.77	0.59
TiC	152	0.107	62.7	0.85	196	0.138	80.9	0.81	0.78

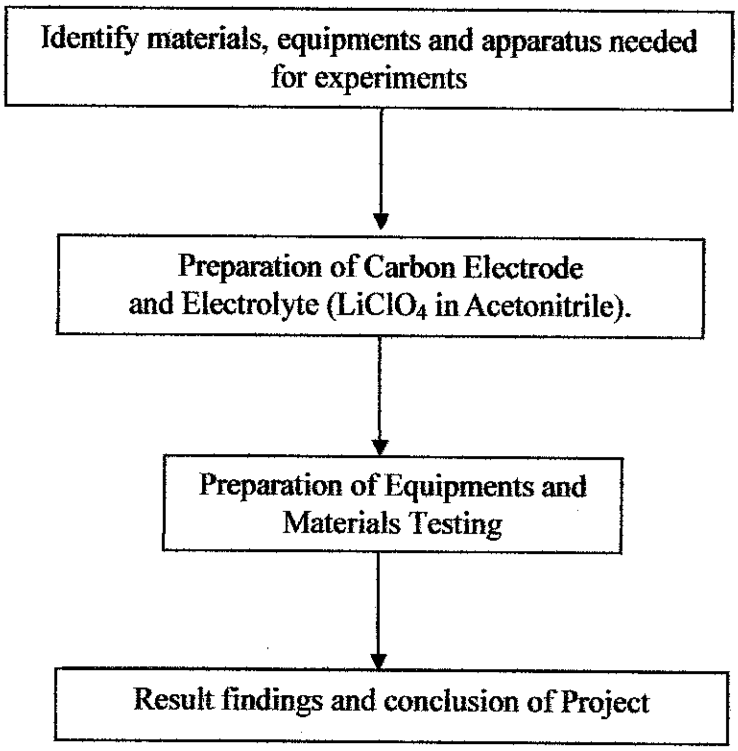
Mesoporous carbons exhibit electric capacity of 75-158 Fg^{-1} in 2.0 M H2SO4 electrolyte solution at a scan rate of 1-50 mVs^{-1}. The mesoporous carbons possess a high surface area of 780 – 1500m^{2}g^{-1} and large pore sizes of 2.6 to 13.7mm [22]. The higher capacitance values correlates with its higher surface area [23].

CHAPTER 3

METHODOLOGY & PROJECT WORK

This part focuses on the material preparation and material testing. This experiment will be done with carbon samples Timrex HSAG 100 CAT Special Graphite M-292 as the cathode and VGAF SHOW A DENKO carbon as the anode. The material analysis that will be done involves X-ray Diffraction and Scanning Electron Microscopy. The preparation of samples and equipments will be done followed by the testing of the test cells and coin cells with varying currents. The data obtained will be analyzed in terms of its efficiency, capacity and voltage-test time profile.

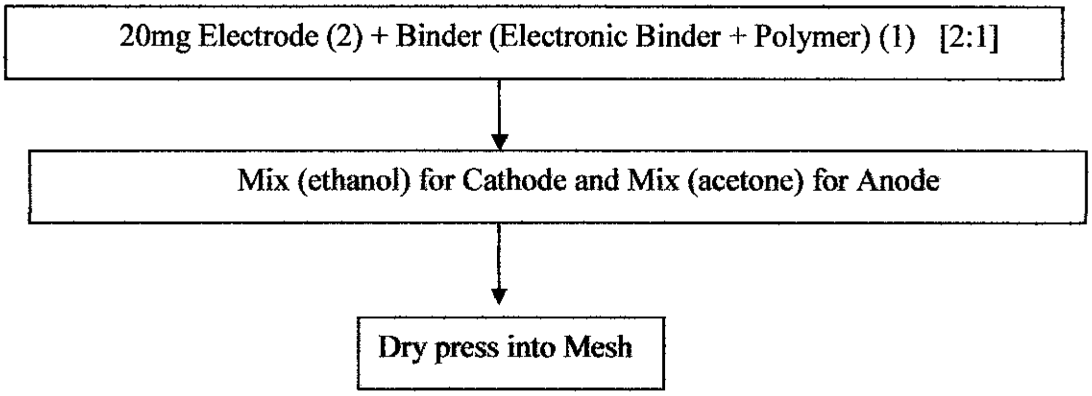
3.1 Process Flow of Experiment



3.2 Experimental Preparation

3.2.1 Electrode Preparation

In this experiment, both cathode and anode will be tested as a whole cell. Carbon samples will be used as cathode and anode for test cell in this intercalation research. Its two principal properties are adhesion and cohesion. Electrode preparation will include carbon sample, binder and current collector. A binder is an ingredient used to bind together two or more other materials in mixtures. The binder is made from Teflon and carbon mixture. The samples of carbon and binder will be mixed. In this experiment for the anode, an acetone solvent will be added to the mixture of carbon and binder and it will be allowed to dissolve. As for the cathode, the mixture will be added with solvent, ethanol and left to be dissolved too. The sample will be of 20mg of carbon added to 10mg of binder. The assumption is that the mixture is homogenous. Before both mixtures are dried up due to evaporation, the mixture is transferred to a current collector and it will be meshed manually against flat surface glassware. The electrode will be dried for a 2 to 3 hours at 90^{0}C in the dryer.



3.2.2 Electrolyte Preparation

The electrolyte used in this study will be Lithium Perchlorate ($LiClO_4$) salt in Acetonitrile solvent with a concentration of 1 molar. The preparation is done at room condition. Lithium Perchlorate ($LiClO_4$) salt is measured and added in the Acentonitrile solvent to form I molar of aprotic electrolyte. The electrodes are then assembles into a test cell and the electrolyte is then dripped onto the separator between the cathode and anode. This is to ensure that the electrodes are wetted with electrolyte to enable transference of ions. After completing the test cell, it is then attached to the battery cycler to obtain voltage, capacity and current profile of the test cell.

3.3 Material Analysis

3.3.1 X-Ray Diffraction

X-ray diffraction finds the geometry or shape of a molecule using X-rays. X-ray diffraction techniques are based on the elastic scattering of X-rays from structures that have long range order. The most comprehensive description of scattering from crystals is given by the dynamical theory of diffraction [19]. X-ray techniques are a family of non-destructive analytical techniques which reveal information about the crystallographic structure, chemical composition, and physical properties of materials and thin films. These techniques are based on observing the scattered intensity of an X-ray beam hitting a sample as a function of incident and scattered angle, polarization, and wavelength or energy [19].

- Single-crystal X-ray diffraction is a technique used to solve the complete structure of crystalline materials, ranging from simple inorganic solids to complex macromolecules, such as proteins.

- High-resolution X-ray diffraction is used to characterize thickness, crystallographic structure, and strain in thin epitaxial films. It employs parallel-beam optics.
- X-ray pole figure analysis enables one to analyze and determine the distribution of crystalline orientations within a crystalline thin-film sample.
- X-ray rocking curve analysis is used to quantify grain size and mosaic spread in crystalline materials.

A crystal lattice is a regular three-dimensional distribution (cubic, rhombic, etc.) of atoms in space. These are arranged so that they form a series of parallel planes separated from one another by a distance d, which varies according to the nature of the material. For any crystal, planes exist in a number of different orientations - each with its own specific d-spacing [24].

3.3.2 Scanning Microscope Electrons (SEM)

The SEM is a microscope that uses electrons instead of light to form an image. The SEM also has much higher resolution, so closely spaced specimens can be magnified at much higher levels. Because the SEM uses electromagnets rather than lenses, the researcher has much more control in the degree of magnification. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition and other properties such as electrical conductivity [25]. All of these advantages, as well as the actual strikingly clear images, make the scanning electron microscope one of the most useful instruments in research today [24].

A beam of electrons is produced at the top of the microscope by an electron gun. The electron beam follows a vertical path through the microscope, which is held within a vacuum. The beam travels through electromagnetic fields and lenses, which focus the beam down toward the sample. Once the beam hits the sample, electrons and X-rays are ejected from the sample [24].

The types of signals produced by an SEM include secondary electrons; back scattered electrons (BSE), characteristic x-rays, light (cathodoluminescence), specimen current and transmitted electrons. These types of signal all require specialized detectors for their detection that are not usually all present on a single machine. The signals result from interactions of the electron beam with atoms at or near the surface of the sample. In the most common or standard detection mode, secondary electron imaging or SEI, the SEM can produce very high-resolution images of a sample surface, revealing details about 1 to 5 nm in size. Due to the way these images are created, SEM micrographs have a very large depth of field yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample [25].

3.3.3 Equipment Preparation

The equipment preparation in this part focuses on the assembling of the 4 test cells and coin cells. Wash all the 4 test cells with water and then rinse with ethanol or acetone and finally dry it initially with air and then place it into the oven. A replacement for the argate mortar is then needed to be found as the existing one was missing or not available. As a replacement a small sized pestle and mortar.

The current collectors and the separators-polymer filters (PTFE) are next cut into the 10 cent and 1 cent coin sizes. Preparation for the other tools needed to run the experiment was also obtained and kept. These includes items such as spatula, glass pellets (flat wide ones), glass containers, gloves, scissors, transparency paper (as working base for the mixing). The electronic weigh machine is also obtained to measure the electrode content of each test cell. All the necessary equipments were then placed into the oven such as the test cells, bikers, spatula, stirrer rod, glass pellets, and separators.

3.3.4 Sample Preparation

The sample preparation begins with the preparation of the electrode which is a mixture of the carbon and Teflon binder. The carbon samples are as below:

- TIMREX HSAG 100 CAT Special Graphite M-292
- VGAF SHOW A DENKO carbon

The preparation of the electrode is done by mixing the carbon with the binder which is the Teflon. The quantity of carbon is approximately 20mg and binder is 10mg. For the preparation the weighed is slightly increased as there will be losses in future transfer and assembling. The electronic weighing machine is used to measure the weight (mg) of these materials.

The following are the quantity of carbon and binder for each 4 samples for both the carbon samples:

Table 3.3.4.1: Weight of TIMREX HSAG 100 CAT Special Graphite M-292

Sample	Carbon	Binder
A	0.026	0.014
B	0.0246	0.0122
C	0.0233	0.0109
D	0.0235	0.0118
E* (Cathode)	0.0222	0.0117
F* (Anode)	0.021	0.0118

Table 3.3.4.2: Weight of VGAF SHOW A DENKO Carbon

Sample	Carbon	Binder
A	0.0225	0.0138
B	0.0214	0.0124
C	0.0216	0.0122
D	0.021	0.0105
E* (Cathode)	0.023	0.0108
F* (Anode)	0.0235	0.0113

These samples of the carbon and Teflon binder are then mixed in a small glass container and meshed well so that it will be even. This is then followed by the preparation of the current collector. The current collector which is the small wire cut into the size of a 1 cent coin is then pressed with the carbon and Teflon binder. Samples of TIMREX HSAG 100 CAT Special Graphite M-292 were used as cathode and VGAF SHOW A DENKO was used the anode. Sample A for cathode was paired with sample A for anode to form a complete cell except for samples E and F. Samples E and F for TIMREX HSAG 100 CAT Special Graphite M-292 was paired up to form one complete cell under one testing. The same method was applied to samples E and F of VGAF SHOW A DENKO.

Table 3.3.4.3: Weight of TIMREX HSAG 100 CAT Special Graphite M-292

Sample	Carbon	Binder
Cathode	0.0228	0.0113
Anode	0.0228	0.0108

Another experiment was run using TIMREX HSAG 100 CAT Special Graphite M-292 as both the cathode and anode as tabulated above. Results are discussed in the following chapter. The same method of preparation of the current collector were also applied this experiment.

The preparation method for the current collector is as the following:

1. The carbon and Teflon binder is mixed in a small biker and then mixed evenly using a glass rod.
2. A drop of acetone is added and then mixed evenly for the cathode electrode and ethanol is added for the anode electrode.
3. The mixture is then transferred to a current collector.
4. Transparency paper is then placed on both sides of the current collectors.
5. Mesh the paper by using a glass rod.
6. All the steps above are repeated for all the 6 samples.

3.3.5 Battery Cycler Test

The battery cycler is also important equipment in this study. This device will help obtain the various data needed such as the voltage, current and capacity. To begin, the first sample of each of the 4 main test cells are placed into a small plastic container and filled with silica to absorb the moisture. Poke 2 holes on each ends of the plastic container and pass a wire through. The first wire connects to the top of the test cell and the second to the body of the test cell. The software used for the battery cycler is the MITS Pro-Arbin Instrumentation.

Battery Cycler Test - MITS Pro-Arbin Instrumentation

1. All 6 samples using the test cell were tested with the battery cycler
2. The voltage was set at 2 V.
3. Testing started by taking data for 0.001 A, 0.005 A, 0.01 A and 0.05 A.
4. Obtain the results of the data and plot a graph for Voltage (V) vs Time Tested (s)

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Material Analysis

Test samples of graphite carbons were analyzed using SEM (Scanning Electron Microscopy). These samples were analyzed at the mechanical laboratory. The results and images were obtained and reported.

The test samples include:

1. TIMREX HSAG 100 CAT Special Graphite M292
2. VGAF SHOW A DENKO

4.2 Results of Material Analysis

4.2.1 Scanning Electron Microscopy (SEM)

TIMREX HSAG 100 CAT Special Graphite M292

Chemical Composition:

- Graphite : 41-1487 (I)
- Crystal Structure is Hexagonal with the following value:
  - a = 2.47040
  - b = 2.47040
  - c = 7.2440

Image/Size of particle is as follow:

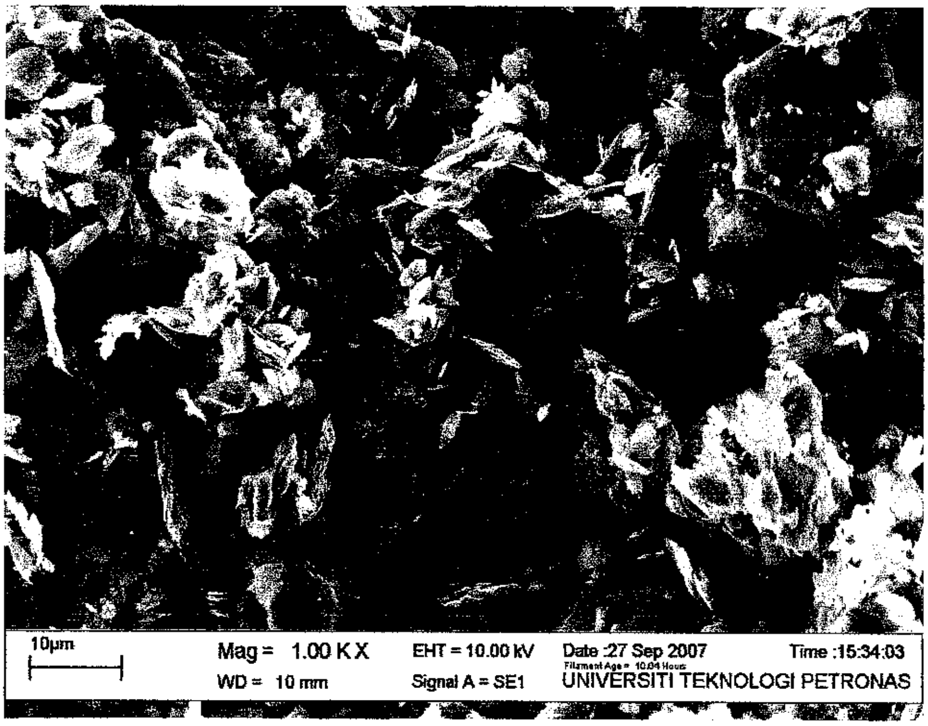


FIGURE 4.2.1.1: IMAGE SIZE OF TIMREX HSAG 100 CAT Special Graphite M292 under 1000 Magnification

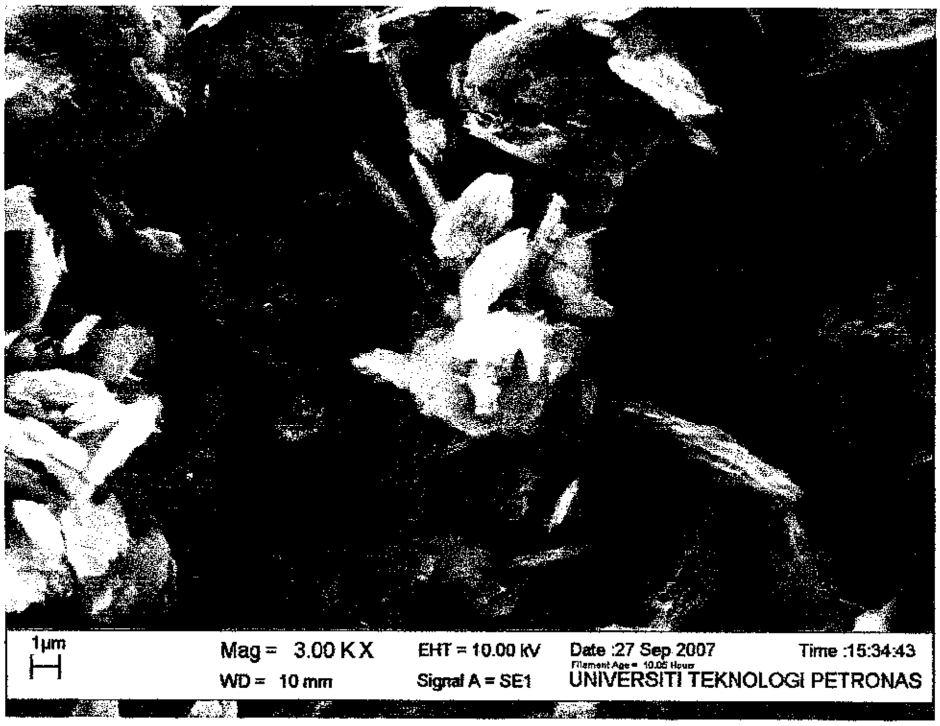


FIGURE 4.2.1.2: IMAGE SIZE OF TIMREX HSAG 100 CAT Special Graphite M292 under 3000 Magnification

VGAF SHOW A DENKO

Chemical Composition:

- Graphite : 26-1076
- Crystal Structure is Hexagonal with the following value:
    - a = 2.45600
    - b = 2.45600
    - c = 20.08800

Image/Size of particle is as follow:

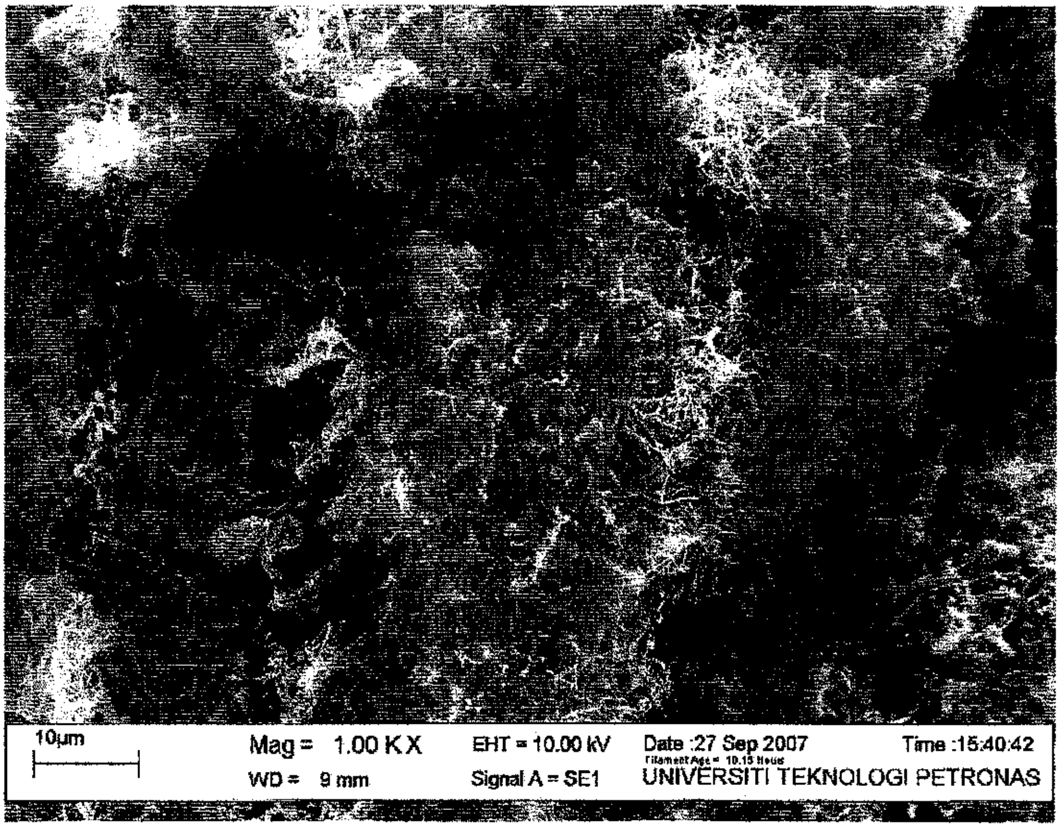


FIGURE 4.2.1.3: IMAGE SIZE OF VGAF SHOW A DENKO under 1000 Magnification

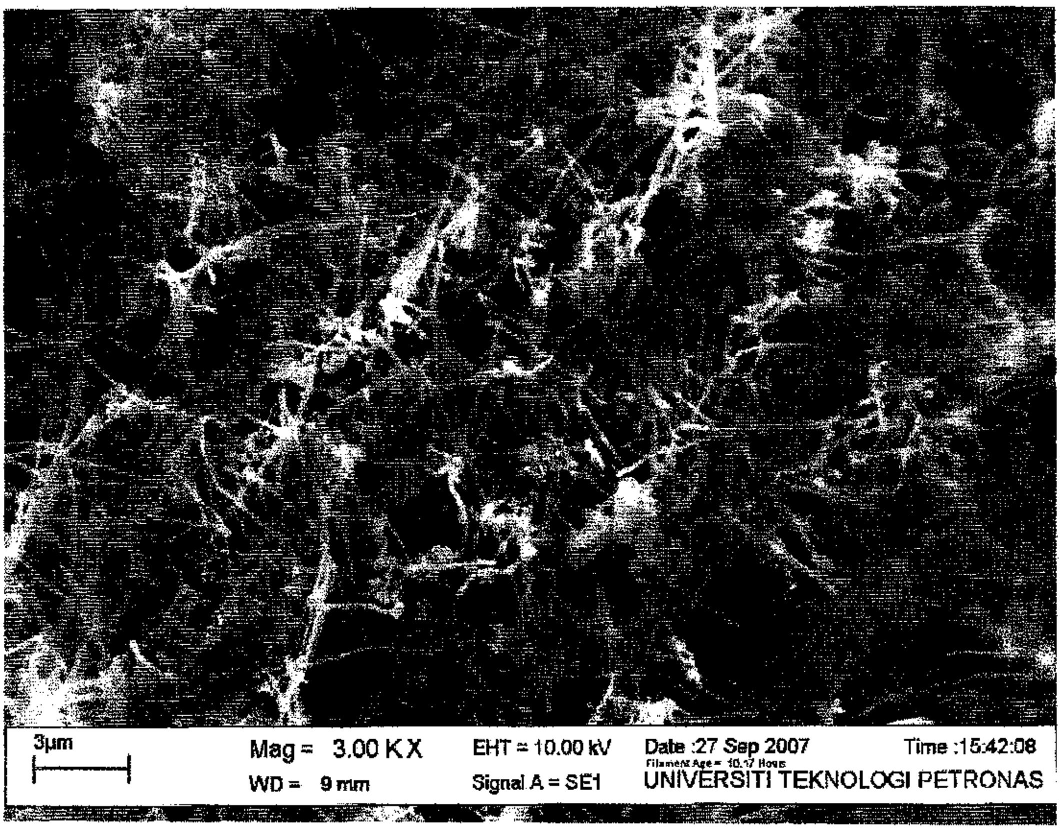


FIGURE 4.2.1.4: IMAGE SIZE OF VGAF SHOW A DENKO under 3000 Magnification

4.2.2 X-Ray Diffraction (X-RD)

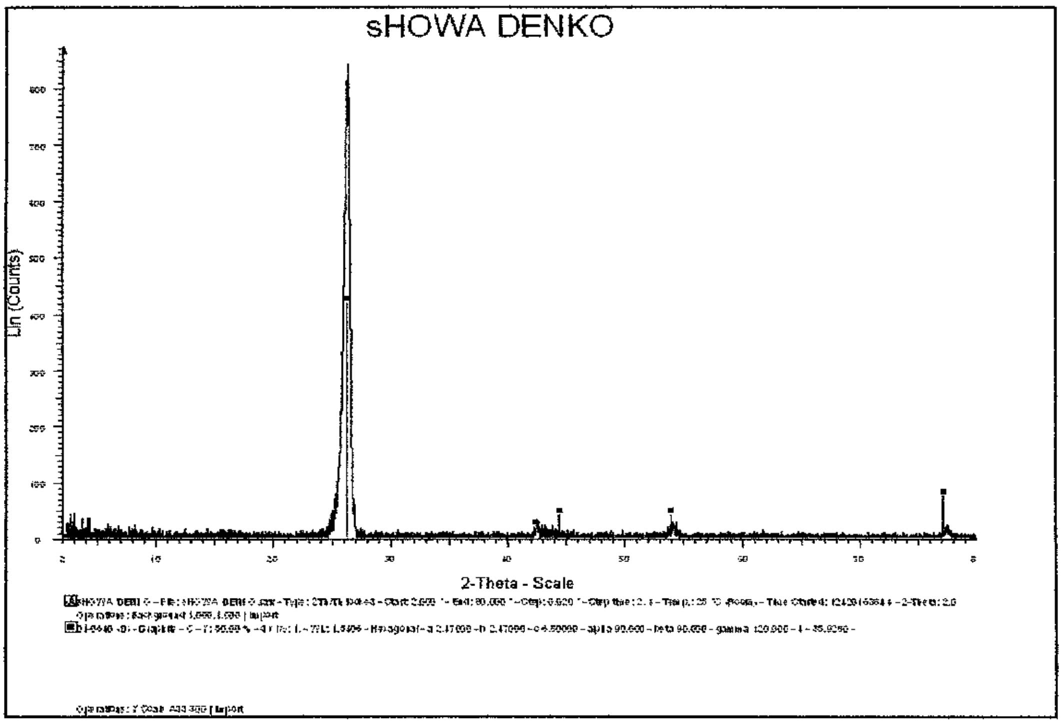


Figure 4.2.2.1: X-RD for VGAF SHOW A DENKO

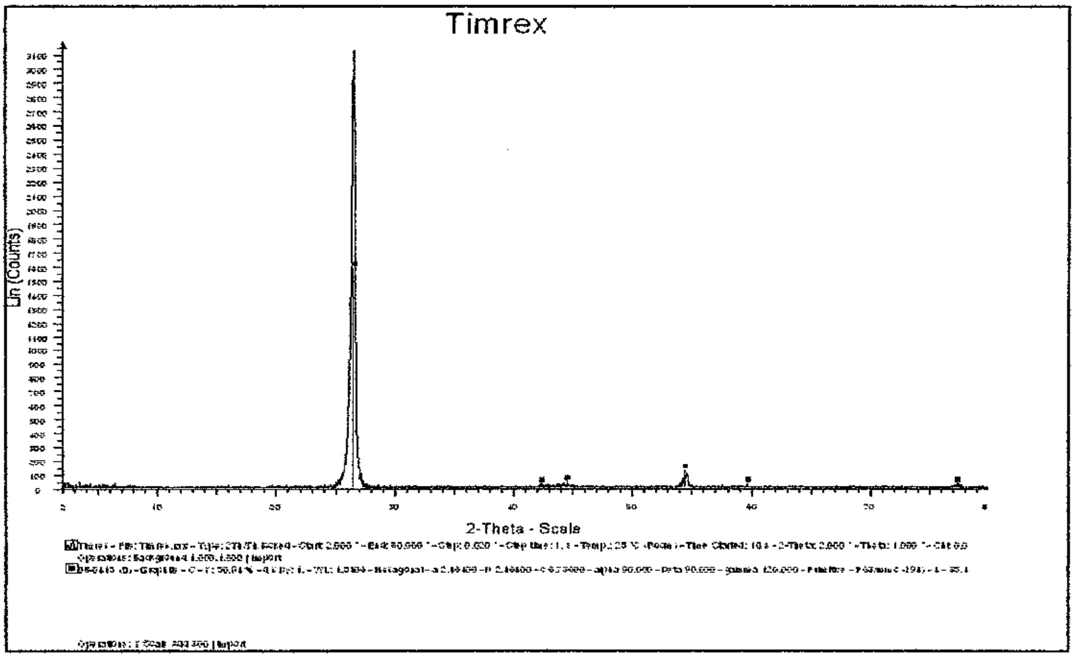


Figure 4.2.2.2: X-RD for TIMREX HSAG 100 CAT Special Graphite M292

4.3 Test Cell Analysis for Combined Carbon Electrode Samples

The results for the test cell analysis using the Battery Cycler Test - MITS Pro-Arbin Instrumentation is based on the two carbon samples whereby TIMREX HSAG 100 CAT Special Graphite M292 was used as the cathode and VGAF SHOW A DENKO was used as the anode material. For this analysis, only sample C was able to produce results.

4.3.1 Voltage – Test Time Profile

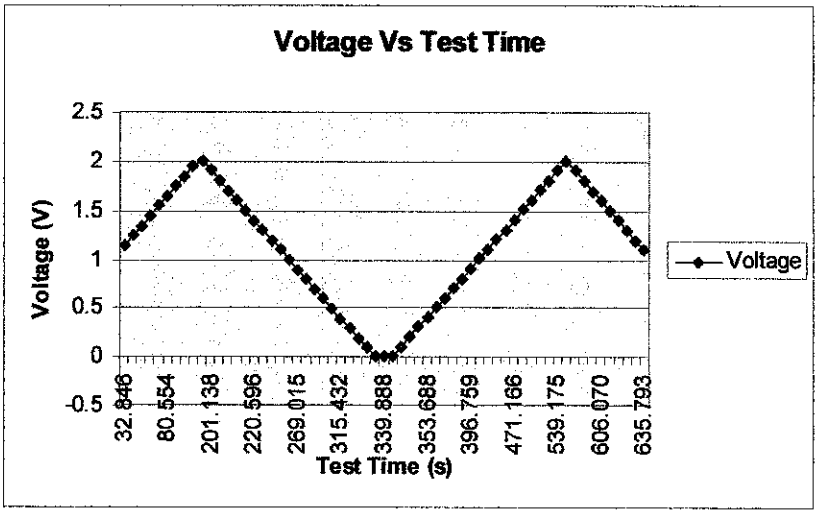


Figure 4.3.1.1: Result of Test Cell Analysis for 0.001 Ampere

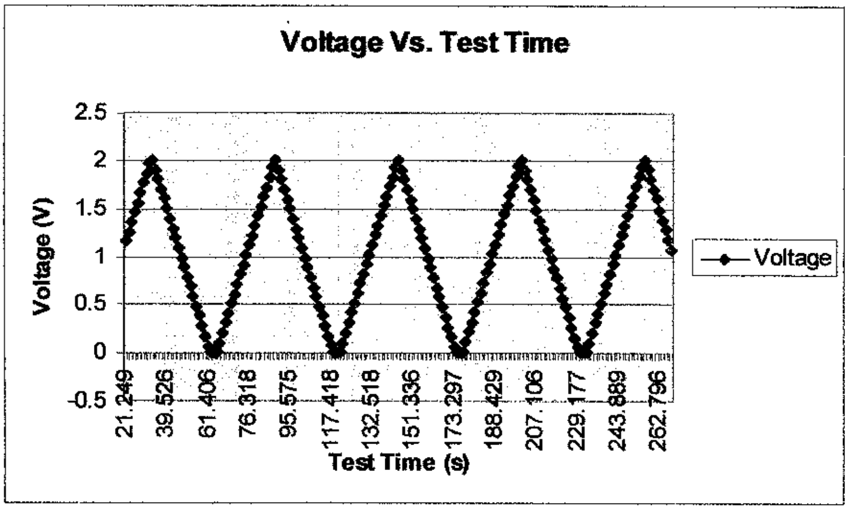


Figure 4.3.1.2: Result of Test Cell Analysis for 0.005 Ampere

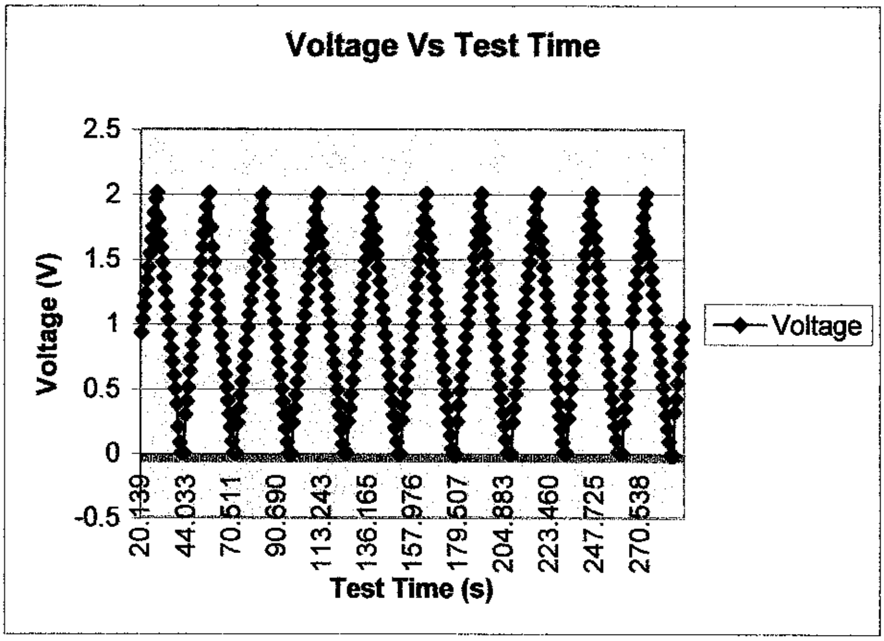


Figure 4.3.1.3: Result of Test Cell Analysis for 0.01 Ampere

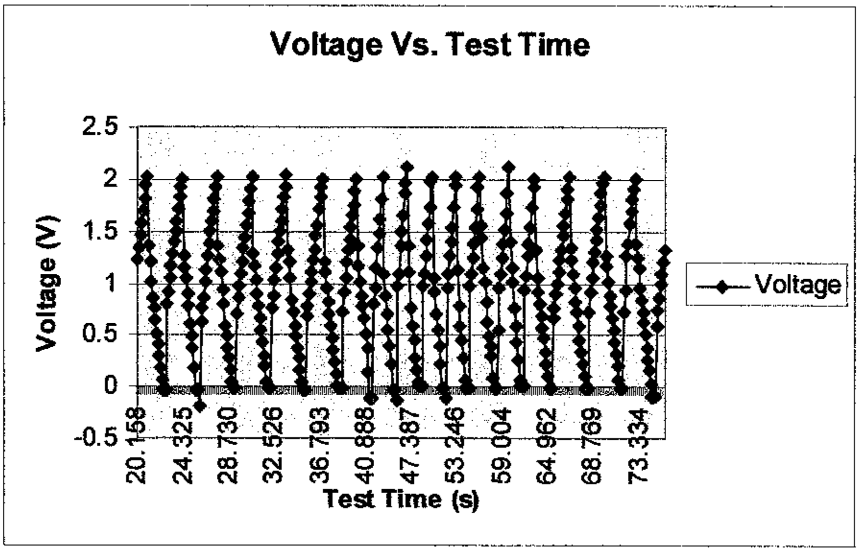


Figure 4.3.1.4: Result of Test Cell Analysis for 0.05 Ampere

Overall the results showed a variation in cycles at different applied current under the voltage potential of 2V. It can be seen that the voltage-test time profile for 0.05A show erratic behavior where else the voltage-test time profile for 0.001A shows stability. The time taken for testing the cell under 0.001A is longer. The higher the applied current value is, the shorter the test time for a trend to develop. This is probably due to the diffusion limit.

The diffusion limit or diffusion limit aggregation (DLA) is process where particles are randomly moving because of the Brownian motion. DLA is applicable to aggregation in any system where diffusion is the primary means of transport in the system [29]. Particles are allowed to freely random walk until it gets within a certain critical range at which time it is pulled onto the cluster [29].In this case, these particles cluster together when there is a high amount of current applied to the electrode's surface that causes the displacement of the ions.

4.3.2 Charge & Discharge – Cycle Profile

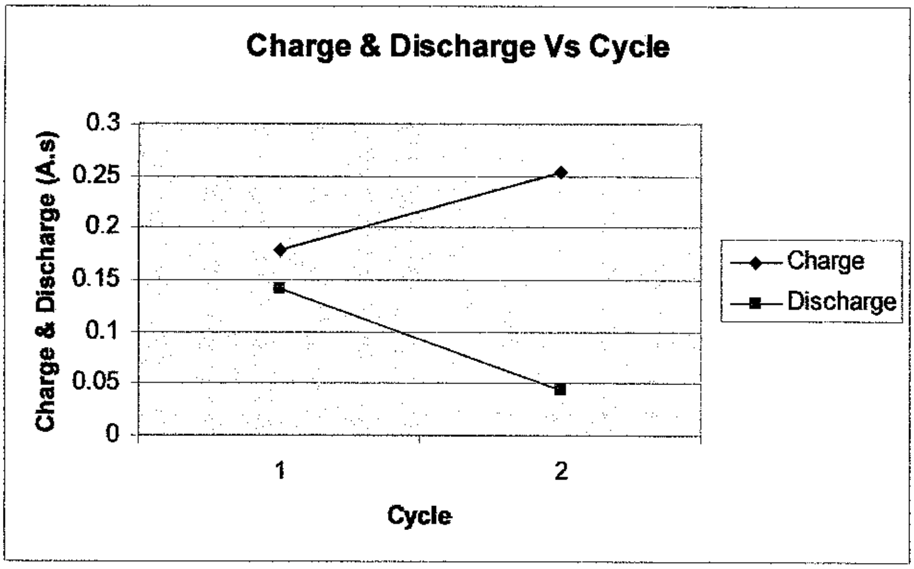


Figure 4.3.2.1: Result of Charge &Discharge Test Cell Analysis for 0.001 Ampere

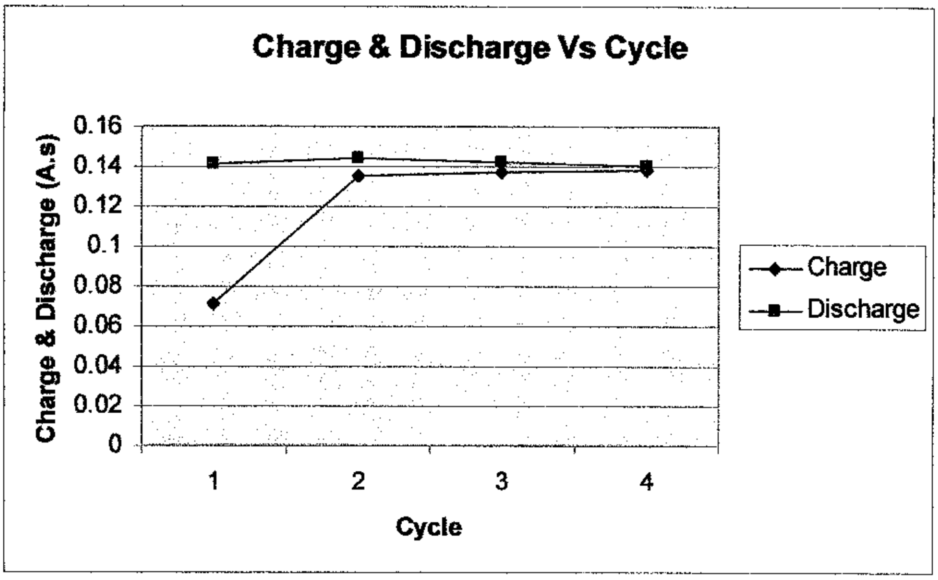


Figure 4.3.2.2: Result of Charge &Discharge Test Cell Analysis for 0.005 Ampere

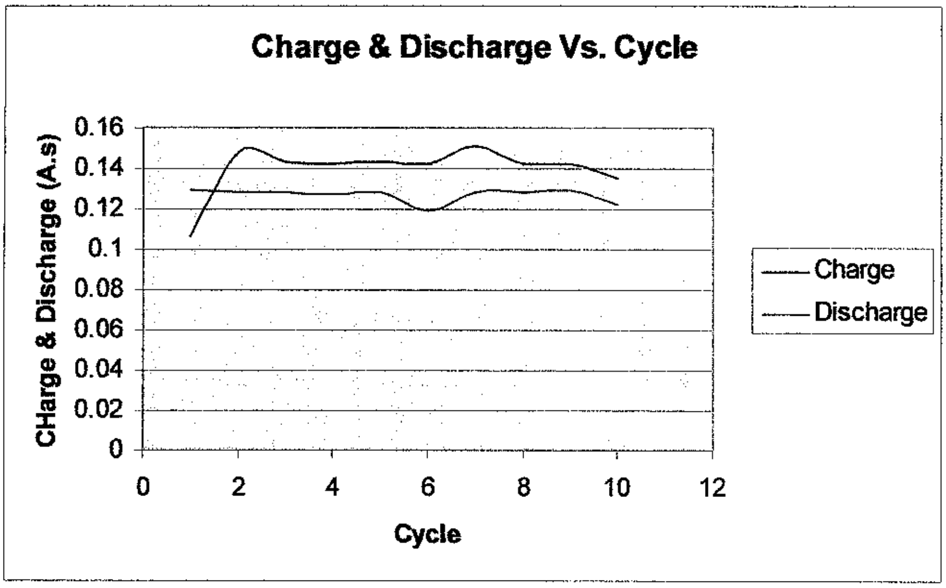


Figure 4.3.2.3: Result of Charge &Discharge Test Cell Analysis for 0.01 Ampere

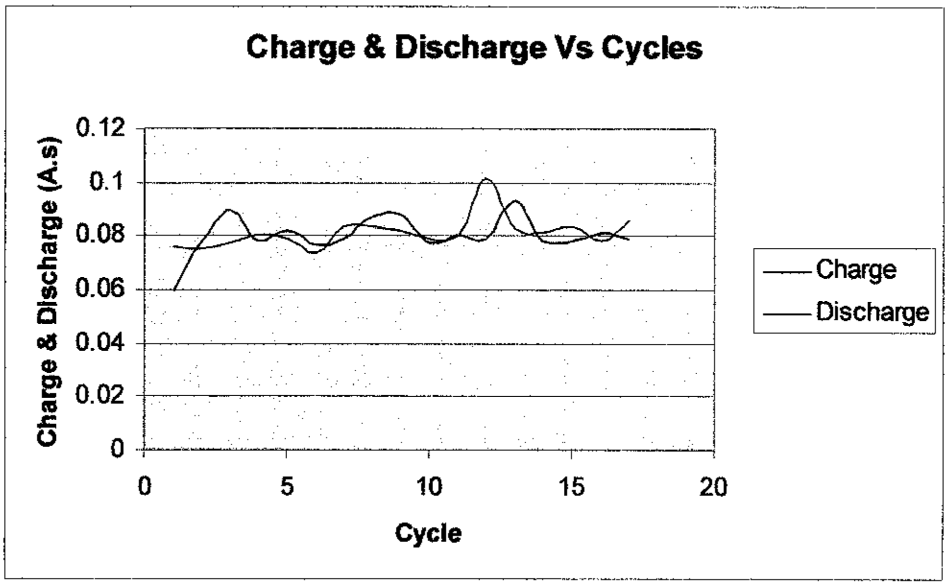


Figure 4.3.2.4: Result of Charge &Discharge Test Cell Analysis for 0.05 Ampere

Table 4.3.2.1: Average Charge and Discharge for Different Current

Current (A)	Average Charge	Average Discharge
0.001	0.215685	0.0921805
0.005	0.12043125	0.14184625
0.01	0.139416	0.126563
0.05	0.080055882	0.081129412

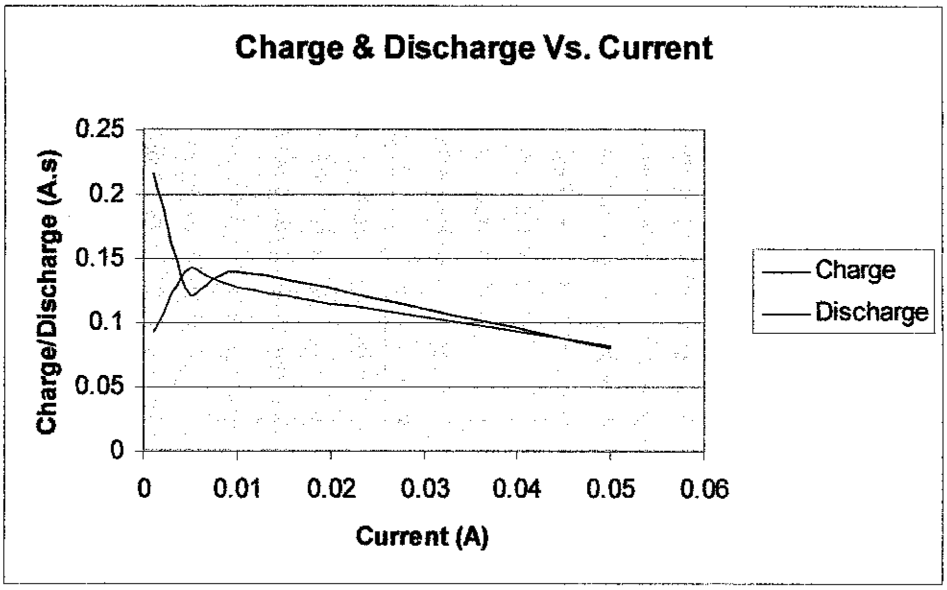


Figure 4.3.2.5: Average Charge and Discharge for Different Current

Table 4.3.2.2: The Specific Capacity of Test Cell with Different Applied Current

Current Applied (A)	Specific Capacity (mAh/g)
0.001	3.51
0.005	1.95
0.01	2.00
0.05	1.28

From the Charge & Discharge – Current graph, the charge-discharge trend increases up to a point where it can be seen that the capacity limit is maximum when the applied current is 0.005A. The specific capacity of this test cell is at maximum when it is applied with 0.001A. This is also due to the DLA (Diffusion Limit Aggregation). When the applied current is small, it able to displace the ions from the electrolyte evenly onto the electrode's surface as compared to when the surface of the electrode is bombarded with a high applied current. This will displace the ions in the electrolyte but it will not be scattered evenly on the electrode's surface as there will be overlapping in certain areas of the electrode's surface and some areas will contain or accommodate no ions.

As seen in the Charge & Discharge – Cycle profiles earlier on, the trend showed stability when the test cell was applied with 0.001A and 0.005A. When the applied current was increase to 0.01A and 0.05A, it begun to show erratic behavior due to DLA. Regarding the specific capacity of this sample, it showed a decline in trend as the current applied was increased. From the table above, when the sample was applied with the lowest current of 0.001A, it produced the highest specific capacity at 3.51mAh/g. While when the sample was applied with current 0.05A, the specific capacity was calculated to 1.28mAh/g.

4.4 Test Cell Analysis for TIMREX HSAG 100 CAT Special Graphite M292

The results for the test cell analysis using the Battery Cycler Test - MITS Pro-Arbin. The samples that were successfully used in this experiment are tabulated in table 3.3.4.3. This experiment is based on one carbon sample which is TIMREX HSAG 100 CAT Special Graphite M292 that was used as both the cathode and the anode material.

4.4.1 Voltage – Test Time Profile

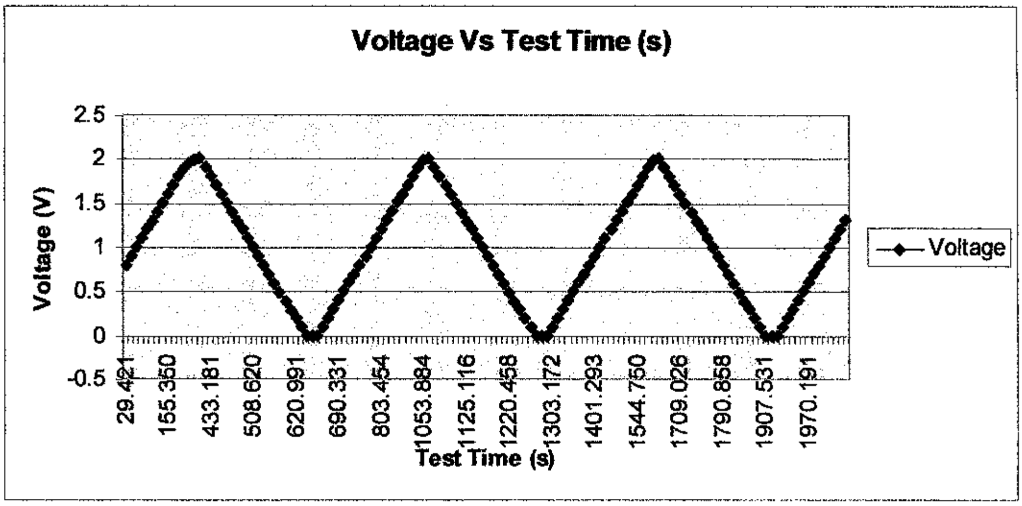


Figure 4.4.1.1: Result of Test Cell Analysis for 0.001 Ampere

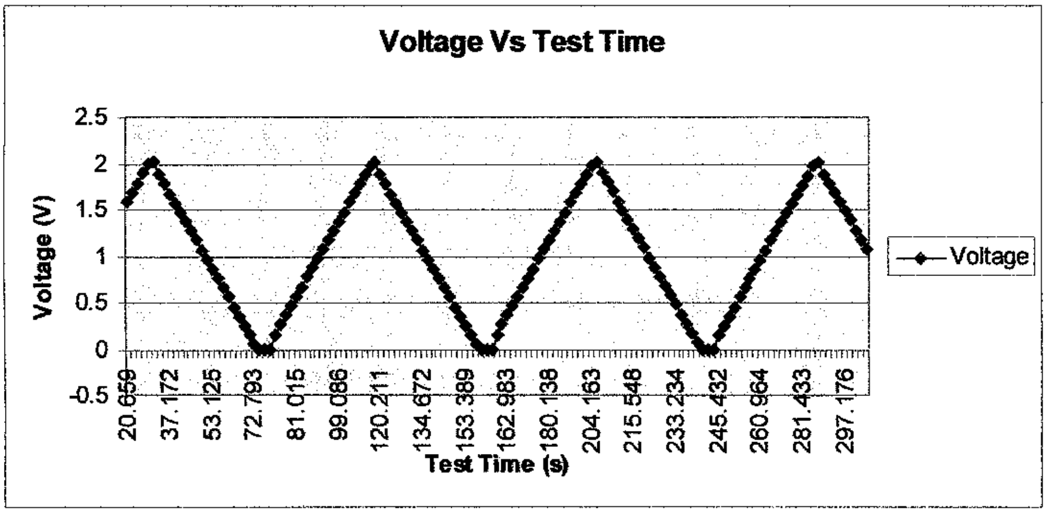


Figure 4.4.1.2: Result of Test Cell Analysis for 0.005 Ampere

Overall, It can be seen that the voltage-test time profile for 0.05A show erratic behavior where else the voltage-test time profile for 0.001A shows stability. The time taken for testing the cell under 0.001A is longer. The higher the applied current value is, the shorter the test time for a trend to develop. This is probably due to the diffusion limit whereby the particles are allowed to freely random move until it gets within a certain critical range at which time it is pulled onto the cluster [29].

For the voltage-test time profile under applied current of 0.05A, the sample managed to undergo multiple cycles as compared to when it is applied with a lower current value.
As you shown, results can be obtained with less test time as the current applied increases.
This can factor in the diffusion limit which takes place within the sample.

4.4.2 Charge & Discharge – Cycle Profile

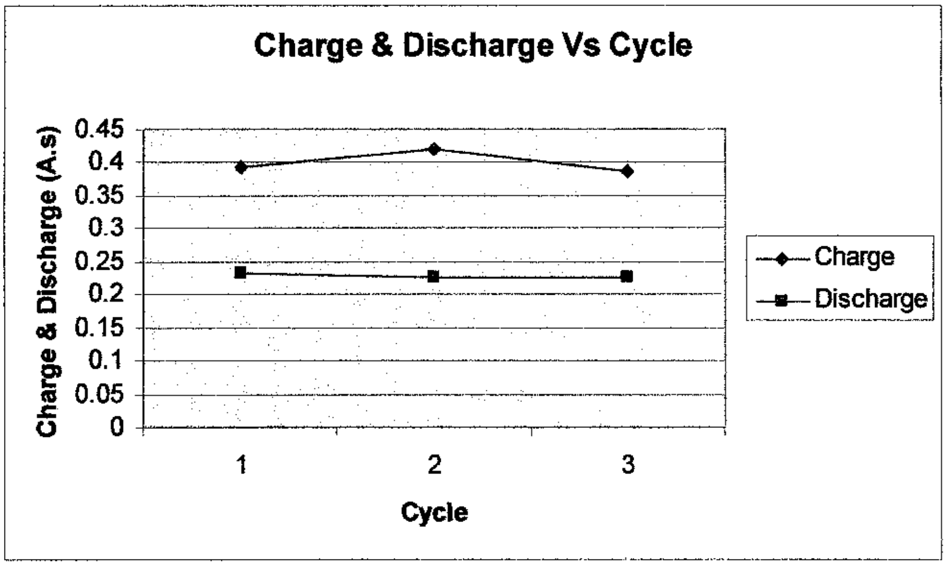


Figure 4.4.2.1: Result of Charge &Discharge Test Cell Analysis for 0.001 Ampere

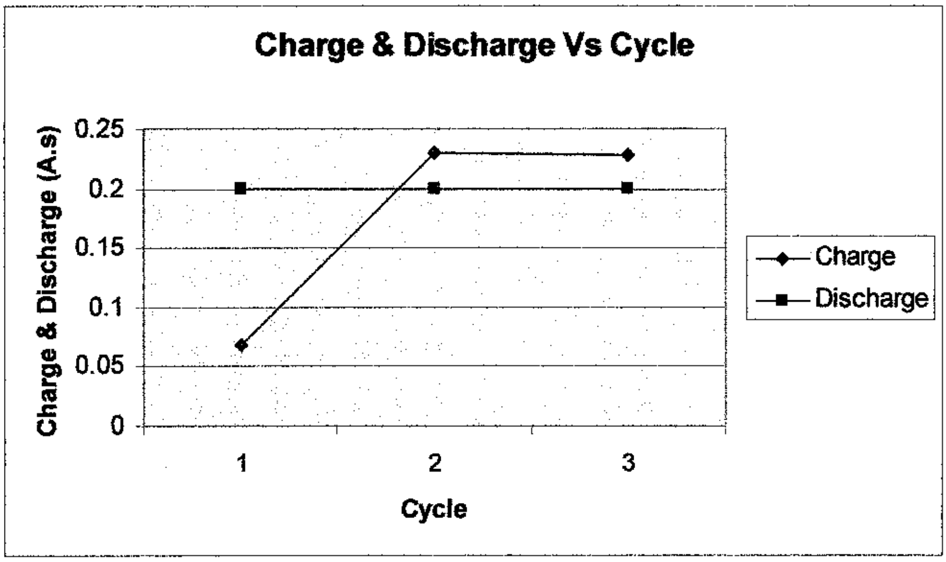


Figure 4.4.2.2: Result of Charge &Discharge Test Cell Analysis for 0.005 Ampere

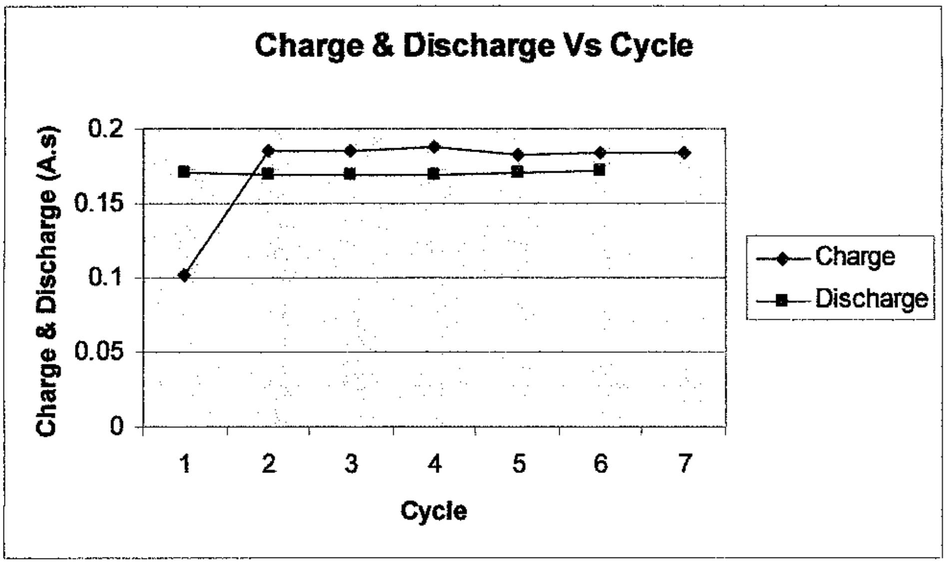


Figure 4.4.2.3: Result of Charge &Discharge Test Cell Analysis for 0.01 Ampere

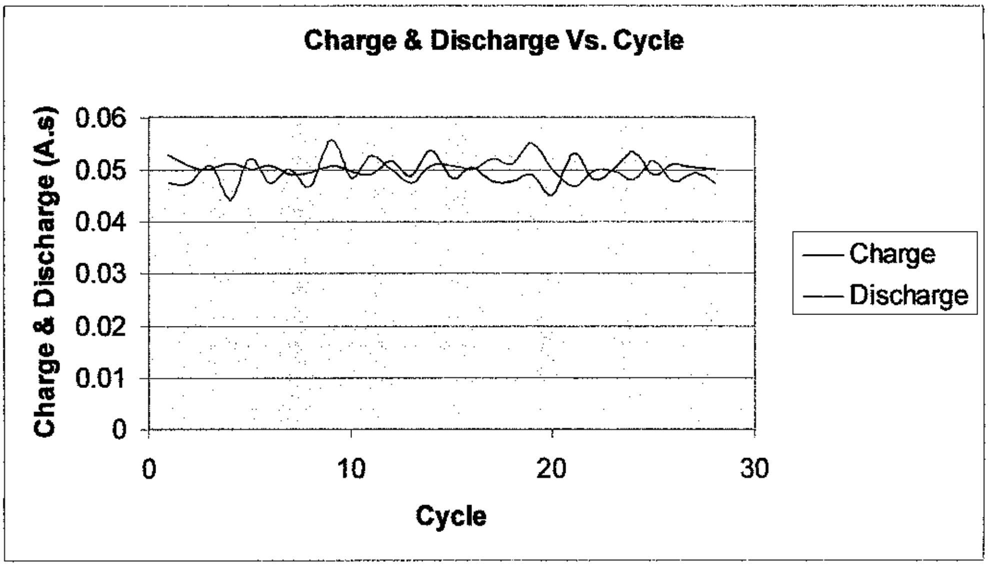


Figure 4.4.2.4: Result of Charge &Discharge Test Cell Analysis for 0.05 Ampere

Table 4.4.2.1: Average Charge and Discharge for Different Current

Current (A)	Average Charge	Average Discharge
0.001	0.399307	0.22673
0.005	0.18851625	0.167095
0.01	0.172852857	0.169952857
0.05	0.050005172	0.049653448

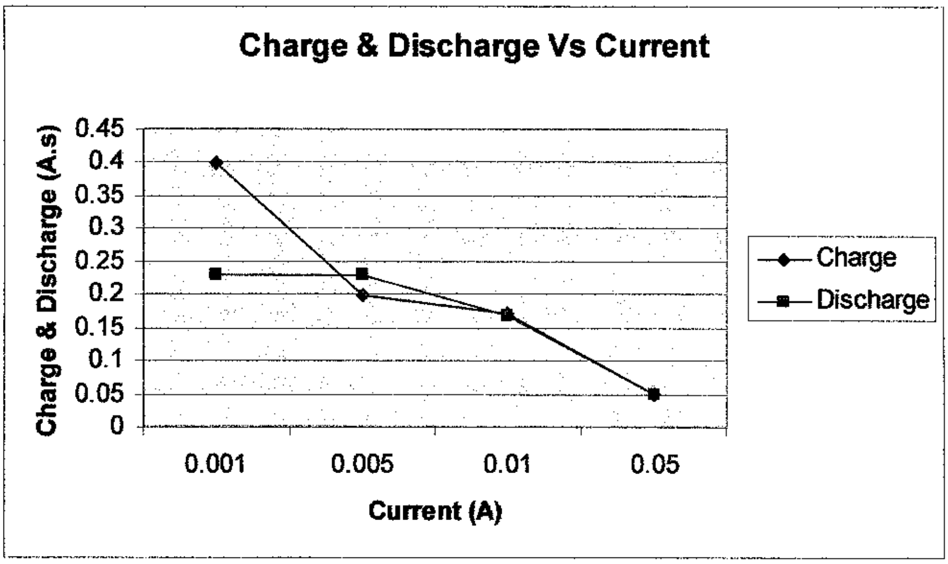


Figure 4.4.2.5: Average Charge and Discharge for Different Current

Table 4.4.2.1: The Specific Capacity of Test Cell with Different Applied Current

Current Applied (A)	Specific Capacity (mAh/g)
0.001	5.83
0.005	3.19
0.01	2.62
0.05	0.744

From the Charge & Discharge – Current graph, the charge-discharge trend decreases. It can be seen that the capacity limit is maximum when the applied current is 0.001A. The specific capacity of this test cell is at maximum when it is applied with 0.001A.
The specific capacity of this sample is 5.83mAh/g when applied with 0.001A. The specific capacity trend decreases as the current applied onto the sample increases.

This is also due to DLA (Diffusion Limit Aggregation) as mentioned above. When the current applied is 0.05A, it showed the lowest specific capacity at 0.744mAh/g. This happens due to the unevenly spread ions from the electrolyte onto the electrode's surface as compared to when the surface of the electrode is bombarded with a low applied current.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

For further studies and experimentation, it is recommended that these samples are put to test under different voltages specifically 3 V, 4 V and 5 V. It is also recommended to test complete cathode-anode samples of the same material to evaluate the potential of a singular particular material in this case; VGAF SHOW A DENKO as a complete test cell.

Based on the graphs, the voltage and capacity profile varied with different currents from 0.001 A to 0.05 A. This is important as the results shows that the cell is more stable and efficient when tested with lower currents. These samples are also best to be tested at a lower applied current as results show more stability when the current applied is low as compared to the higher ones. This is due to the DLA that can be reduced when the current applied is low to provide better end results. This can be observed as the specific capacitance data shows that both samples applied with higher current indicates a lower specific capacitance compares to when the sample was applied with 0.001 A. In the first experiment, the specific capacity at 0.001A was 3.51mAh/g and 5.83mAh/g in the second experiment under the same applied current.

From both experiments, it can be observed that the specific capacity of the sample of TIMREX HSAG 100 CAT Special Graphite M292 when it is used both as cathode and anode showed a higher value compared to the combine sample of TIMREX HSAG 100 CAT Special Graphite M292 as cathode and VGAF SHOW A DENKO as anode under the applied current 0.001A.

The Voltage and Capacity profiles also indicate that the capacity increases with more cycles which show that the cell does function. This shows that there is a displacement of electrolyte ions and that these ion managed to accommodate the carbon graphite host to store and deliver energy when there an applied load.

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APPENDICES

Combined Carbon Electrode Samples

Table 1: Calculations for Capacity under current 0.001 A

Cycles	Point	Time (s)	Delta (s)	Current (A)	Q=IT
Cycle 1	Point 1	20.008	178.897	0.001	0.178897
	Point 2	198.905	140.982	0.001	0.140982
Cycle 2	Point 3	339.887	252.473	0.001	0.252473
	Point 4	592.36	43.379	0.001	0.043379
	Point 5	635.739			

Table 2: Calculations for Capacity under current 0.005 A

Cycles	**Points**	**Time (s)**	**Delta (s)**	**Current (A)**	**Q=IT**
Cycle 1	Point 1	19.987	14.24	0.005	0.0712
	Point 2	34.227	28.111	0.005	0.140555
Cycle 2	Point 3	62.338	27.088	0.005	0.13544
	Point 4	89.426	28.893	0.005	0.144465
Cycle 3	Point 5	118.319	27.318	0.005	0.13659
	Point 6	145.637	28.442	0.005	0.14221
Cycle 4	Point 7	174.079	27.699	0.005	0.138495
	Point 8	201.778	28.031	0.005	0.140155
	Point 9	229.809			

Table 3: Calculations for Capacity under current 0.01 A

Cycles	**Points**	**Time (s)**	**Delta Time (s)**	**Current (A)**	**Q=IT**
1	1	20.008	10.645	0.01	0.10645
	2	30.653	12.88	0.01	0.1288
2	3	43.533	14.76	0.01	0.1476
	4	58.293	12.799	0.01	0.12799
3	5	71.092	14.321	0.01	0.14321
	6	85.413	12.757	0.01	0.12757
4	7	98.17	14.241	0.01	0.14241
	8	112.411	12.69	0.01	0.12689
5	9	125.10	14.27	0.01	0.14269
	10	139.369	12.769	0.01	0.12769
6	11	152.138	14.23	0.01	0.1423
	12	166.368	11.919	0.01	0.11919

7	13	178.287	15.111	0.01	0.15111
	14	193.398	12.808	0.01	0.12808
8	15	206.206	14.16	0.01	0.1416
	16	220.366	12.829	0.01	0.12829
9	17	233.195	14.21	0.01	0.1421
	18	247.405	12.869	0.01	0.12869
10	19	260.274	13.469	0.01	0.13469
	20	273.743	12.244	0.01	0.12244
	21	285.987			

Table 4: Calculations for Capacity under Current 0.05 A and the Efficiency

Cycles	Points	Time (s)	Delta Time (s)	Current (A)	Q=IT	Cycles	Efficiency (%)
1	1	20.008	1.191	0.05	0.05955	1	127.0361041
	2	21.199	1.513	0.05	0.07565		
2	3	22.712	1.552	0.05	0.0776	2	96.19845361
	4	24.264	1.493	0.05	0.07465		
3	5	25.757	1.782	0.05	0.0891	3	87.09315376
	6	27.539	1.552	0.05	0.0776		
4	7	29.091	1.563	0.05	0.07815	4	102.4952015
	8	30.654	1.602	0.05	0.0801		
5	9	32.256	1.632	0.05	0.0816	5	96.32352941
	10	33.888	1.572	0.05	0.0786		
6	11	35.46	1.533	0.05	0.07665	6	96.0208741
	12	36.993	1.472	0.05	0.0736		
7	13	38.465	1.581	0.05	0.07905	7	105.1865908
	14	40.046	1.663	0.05	0.08315		
8	15	41.709	1.744	0.05	0.0872	8	95.87155963
	16	43.453	1.672	0.05	0.0836		
9	17	45.125	1.752	0.05	0.0876	9	93.20776256
	18	46.877	1.633	0.05	0.08165		
10	19	48.51	1.552	0.05	0.0776	10	101.9329897
	20	50.062	1.582	0.05	0.0791		
11	21	51.644	1.602	0.05	0.0801	11	100.062422
	22	53.246	1.603	0.05	0.08015		
12	23	54.849	1.571	0.05	0.07855	12	128.7714831
	24	56.42	2.023	0.05	0.10115		
13	25	58.443	1.853	0.05	0.09265	13	89.15272531
	26	60.296	1.652	0.05	0.0826		
14	27	61.948	1.563	0.05	0.07815	14	103.7747921
	28	63.511	1.622	0.05	0.0811		
15	29	65.133	1.562	0.05	0.0781	15	106.4020487
	30	66.695	1.662	0.05	0.0831		
16	31	68.357	1.613	0.05	0.08065	16	96.83818971

	32	69.97	1.562	0.05	0.0781		
17	33	71.532	1.573	0.05	0.07865	17	108.4551812
	34	73.105	1.706	0.05	0.0853		
	35	74.811				Average Efficiency	102.0484154

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Table 5: Calculations for Capacity under current 0.001 A

Cycles	Points	Test Time (s)	Delta Test Time (s)	Current (A)	Q=IT	Efficiency (%)
1	1	29.421	393.235	0.001	0.393235	58.39536155
	2	422.656	229.631	0.001	0.229631	
2	3	652.287	419.422	0.001	0.419422	53.92087206
	4	1071.709	226.156	0.001	0.226156	
3	5	1297.865	385.264	0.001	0.385264	58.24655301
	6	1683.129	224.403	0.001	0.224403	
4	7	1907.532	163.384	0.001	0.163384	

Table 6: Calculations for Capacity under current 0.005 A

Cycles	Points	Test Time (s)	Delta Test Time (s)	Current (A)	Q=IT	Efficiency (%)
1	1	20.659	13.579	0.005	0.067895	293.8287061
	2	34.238	39.899	0.005	0.199495	
2	3	74.137	45.985	0.005	0.229925	115.0460748
	4	120.122	39.718	0.005	0.19859	
3	5	159.84	45.694	0.005	0.22847	114.6672372
	6	205.534	39.728	0.005	0.19864	
4	7	245.262	45.555	0.005	0.227775	31.45867633
	8	290.817	14.331	0.005	0.071655	
		305.148				

Table 7: Calculations for Capacity under current 0.01 A

Cycles	Points	Test Time(s)	Delta Test Time (s)	Current (A)	Q=IT	Efficiency (%)
1	1	20.136	10.175	0.01	0.10175	167.4103194
	2	30.311	17.034	0.01	0.17034	
2	3	47.345	18.516	0.01	0.18516	91.40743141
	4	65.861	16.925	0.01	0.16925	
3	5	82.786	18.456	0.01	0.18456	91.70459471

	6	101.242	16.925	0.01	0.16925	
4	7	118.167	18.836	0.01	0.18836	90.01380336
	8	137.003	16.955	0.01	0.16955	
5	9	153.958	18.286	0.01	0.18286	92.83058077
	10	172.244	16.975	0.01	0.16975	
6	11	189.219	18.372	0.01	0.18372	93.07097757
	12	207.591	17.099	0.01	0.17099	
7	13	224.69	18.356	0.01	0.18356	92.90695141
	14	243.046	17.054	0.01	0.17054	
8	15	260.1	5.85	0.01	0.0585	
	16	265.95				

Table 8: Calculations for Capacity under Current 0.05 A and the Efficiency

Cycles	Points	Time	Delta Time (s)	Current (A)	Q=IT	Efficiency
1	1	21.955	1.051	0.05	0.05255	90.48525
	2	23.006	0.951	0.05	0.04755	
2	3	23.957	1.012	0.05	0.0506	93.97233
	4	24.969	0.951	0.05	0.04755	
3	5	25.92	1.002	0.05	0.0501	100.998
	6	26.922	1.012	0.05	0.0506	
4	7	27.934	1.021	0.05	0.05105	86.28795
	8	28.955	0.881	0.05	0.04405	
5	9	29.836	1.001	0.05	0.05005	104.0959
	10	30.837	1.042	0.05	0.0521	
6	11	31.879	1.012	0.05	0.0506	93.97233
	12	32.891	0.951	0.05	0.04755	
7	13	33.842	0.982	0.05	0.0491	101.9348
	14	34.824	1.001	0.05	0.05005	
8	15	35.825	0.991	0.05	0.04955	94.04642
	16	36.816	0.932	0.05	0.0466	
9	17	37.748	1.017	0.05	0.05085	109.6362
	18	38.765	1.115	0.05	0.05575	
10	19	39.88	0.993	0.05	0.04965	97.78449
	20	40.873	0.971	0.05	0.04855	
11	21	41.844	0.981	0.05	0.04905	107.2375
	22	42.825	1.052	0.05	0.0526	
12	23	43.877	1.031	0.05	0.05155	97.0902
	24	44.908	1.001	0.05	0.05005	
13	25	45.909	0.972	0.05	0.0486	97.83951
	26	46.881	0.951	0.05	0.04755	
14	27	47.832	1.071	0.05	0.05355	94.49113
	28	48.903	1.012	0.05	0.0506	
15	29	49.915	0.971	0.05	0.04855	104.2225

	30	50.886	1.012	0.05	0.0506	
16	31	51.898	1.011	0.05	0.05055	99.01088
	32	52.909	1.001	0.05	0.05005	
17	33	53.91	0.953	0.05	0.04765	109.234
	34	54.863	1.041	0.05	0.05205	
18	35	55.904	0.952	0.05	0.0476	107.2479
	36	56.856	1.021	0.05	0.05105	
19	37	57.877	0.981	0.05	0.04905	112.2324
	38	58.858	1.101	0.05	0.05505	
20	39	59.959	0.902	0.05	0.0451	110.9756
	40	60.861	1.001	0.05	0.05005	
21	41	61.862	1.061	0.05	0.05305	87.84166
	42	62.923	0.932	0.05	0.0466	
22	43	63.855	0.961	0.05	0.04805	103.2258
	44	64.816	0.992	0.05	0.0496	
23	45	65.808	1.001	0.05	0.05005	99.5005
	46	66.809	0.996	0.05	0.0498	
24	47	67.805	1.067	0.05	0.05335	90.15933
	48	68.872	0.962	0.05	0.0481	
25	49	69.834	0.981	0.05	0.04905	105.1988
	50	70.815	1.032	0.05	0.0516	
26	51	71.847	1.021	0.05	0.05105	93.24192
	52	72.868	0.952	0.05	0.0476	
27	53	73.82	1.011	0.05	0.05055	98.02176
	54	74.831	0.991	0.05	0.04955	
28	55	75.822	1.002	0.05	0.0501	94.91018
	56	76.824	0.951	0.05	0.04755	
29	57	77.775	0.991	0.05	0.04955	100
	58	78.766	0.991	0.05	0.04955	