CERTIFICATION OF APPROVAL

Carbon-Carbon Supercapacitor

by

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Approved by,

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TRONOH, PERAK
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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.

MOHD SALLEHUDDIN BIN SAID

ACKNOWLEDGEMENT

First and foremost I would like to thank to Allah for constantly giving the strength and courage in completion of this project. Even in good and bad times of the semester where many obstacles were faced and overcome.

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ABSTRACT

Carbonaceous waste used in this project was taken from Ethylene Malaysia Sdn. Bhd. Normally this waste will dispose after send to Kualiti Alam Sdn Bhd. The main aim of this project is to add value of this carbonaceous waste by utilizing its carbon properties possesses by the material.

While Li-ion batteries temporarily dampened enthusiasm for capacitor-based storage, demand for power-hungry applications has sparked a resurgence of interest in supercapacitors. The overall market is expected to expand from $208 million last year, to an $877 million market in 2014, according to a new report from Lux Research[1]. Thus, further study was conducted to use carbonaceous waste as supercapacitor. By doing this, the company may get profit and cover all maintenance cost in plant.

In this project, the experimental was design to treat the waste for activation carbon purpose. Then, it will be characterized before assemble as supercapacitor. Characterization is important to monitor the structure of carbonaceous waste. The study to treat this carbonaceous material has been carried out. Some of research had been done and literature was collected in order to study the factor that may affect capacitance of carbon. The experiment is divided in 3 part, which is the first part is to do the treatment of carbonaceous waste; the second part is to Characterize the treated carbon and the last part is to use the treated carbon as supercapacitor for performance evaluation. Electrochemical performance of capacitor from commercial carbon and from carbonaceous waste is also compared and evaluated.

Further studies could be carried out to optimize the charge-discharge capacity and the efficiency of capacitor using this material. As conclusion, this study has proved that this specific carbonaceous waste is able to act as electrode in electrochemical capacitor with discharge capacity 2.185 mA.h/g and 85% efficiency

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LIST OF ABBREVIATIONS

XRD	X-Ray Diffraction
SEM	Scanning Electron Microscope
$LiClO_4$	Lithium Perchlorate
A	Ampere
V	Voltage
HTT	Heating Temperature Treatment
EDLC	Electrochemical Double Layer Capacitor

CHAPTER 1: INTRODUCTION

1.1 BACKGROUND OF STUDY

Electrochemical capacitors are currently called by a number of names - supercapacitor, ultracapacitor, or electrochemical double-layer capacitor (EDLC). Although three terms are used interchangeably, and all of these refer to a capacitor that stores electrical energy at the interface that lies between a solid electrode and an electrolyte. [2]

Supercapacitors also possess a number of desirable qualities that make them an attractive energy storage option. The mechanisms by which supercapacitor store and release charges are completely reversible, and hence they can withstand a large number of charge/discharge cycles without any visible degradation[3]. They can store or release energy very quickly, and can operate over a wide range of temperatures.

Supercapacitors are intermediate systems between dielectric capacitors and batteries. While 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. These particular properties make them suitable for numerous applications such as power electronics, spatial, military field; they can also be used in hybrid electric vehicle (HEV) in order to help the stop and go function, to provide peak power for improved acceleration, for energy recovery.[4][5]

Realizing on this important device, further study will be conducted to build the ultracapacitor by using the carbonaceous waste from ethylene production plant instead of disposing it. From the previous experiment, the characteristic and properties of carbonaceous waste had been identified. The performance of carbonaceous waste as supercapacitor also had been evaluated. Since it was proved that the carbonaceous waste possess capacitance properties, further study will be conducted in order to increase the performance of the material as supercapacitor.

In this study, the waste was used as precursor to prepare the activated carbon. The preparation process consisted of $ZnCl_2$. After, preparation process complete, the activated carbon will be used as electrode to build supercapacitor.

1.2 PROBLEM STATEMENT

In the Ethylene production Plant, one of the scheduled wastes possesses the carbonaceous properties. The waste may be used as electrode to reduce maintenance cost for the company and might give extra profit to the company. Thus, in the previous experiment, the waste was tested to see the ability of the waste to store charge. The experiment shows that this waste is able to hold charge and deliver discharge where the discharge capacity by carbonaceous waste capacitor is 1.161 mA.h/g.

However, it is noticed that non-treated carbonaceous waste gave lower specific discharge capacity compared to the commercial carbon which is 1.947 mA.h/g. This is due to the particle size and pore size of carbonaceous waste is distributing not uniformly. As a result the surface area of the waste lower compared to activated carbon.

Low surface area will give low charge capacity. In order to increase the capacitance of the waste, the surface area of the waste should be rose up. The method to increase the surface area is by applying treatment on the carbonaceous waste.

Hence, further study will be conducted on the possible way to treat carbonaceous waste in order to increase the surface area of the material. Then fabrication product will be experimented on the potential of the carbonaceous waste as a supercapacitor after applied some treatment. This product will be evaluated and compared with the commercial activated carbon at the end of the experiment.

1.3 OBJECTIVES

From the problem statement above, the study will be carried out and the experiment was design in order to achieve the objectives below:

1. To investigate chemical activation process effect on carbonaceous waste.
2. To investigate the physical properties of the treated carbonaceous waste.
3. To evaluate the supercapacitor performance of treated carbonaceous waste as supercapacitor.

1.4 SCOPE OF STUDY

The scope of study in this project is:

1. The source of carbonaceous waste will be taken from Ethylene Malaysia Sdn. Bhd (EMSB); an ethylene production plant located in Kerteh, Terengganu.
2. Carbon used in this study will be treated using $ZnCl_2$ and Heating Temperature Treatment.
3. Electrochemical capacitor using carbonaceous waste is in electrolyte of 1 M Lithium Perchlorate in Acetonitrile.

CHAPTER 2: LITERATURE REVIEW

2.1 ACTIVATED CARBON

Carbon is an indispensable element in industry. By far, the greatest single use of carbon is in the form of coke for the iron and steel industry. The major portion of this coke is used in the reduction of iron ore in blast furnaces. As in the rubber industry; the major applications for carbon blacks are in the printing ink, paint, paper and plastic industries. Minor amounts are used in the manufacture of dry cells and carbon brushes, and as insulation.

The largest single application for gas phase activated carbons is in the recovery of volatile organic solvents from air or vapor mixtures. Another large application is in the purification and separation of natural and industrial gases. Main applications for pyrographite and the fiber forms of manufactured graphite are found as components for rockets, missile and other aerospace vehicles.

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})[6]. 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.

Factors which may influence the properties of an activated carbon depend on [7]:

a. the chemical activation (parent feedstock)
b. the heating rate
c. the flow rate of the containing gas usually nitrogen
d. the final Heat Treatment Temperature of carbonization
e. the temperature of activation
f. the activating gas
g. the duration of activation
h. flow rate of the activating gas

2.2 CARBONACEOUS WASTE

The carbonaceous residue or petroleum coke is one of the by-product from Ethane cracking process. Petroleum coke is defined as a black solid residue, obtained mainly by cracking and carbonizing of petroleum derived feedstock, vacuum bottoms, tar and pitches in processes such as delayed coking or fluid coking. It consists mainly of carbon (90 to 95 percent) and has low ash content. It is used as a feedstock in coke ovens for the steel industry, for heating purposes, for electrode manufacture and for production of chemicals. The two most important qualities are "green coke" and "calcinated coke". This category also includes "catalyst coke" deposited on the catalyst during refining processes: this coke is not recoverable and is usually burned as refinery fuel.

2.3 SUPERCAPACITORS

Capacitance is defined as C =q /AV, where q is the charge stored on the application of a voltage AV between two conductors. The capacitance of a flat-plate capacitor is C = **EA/d,** where A is the area of two plates separated a distance d and **E** is the permittivity of the dielectric medium between them [8]. An electrochemical capacitor utilizes a large electrode surface area A and a small distance d across the surface between the electronic charge stored in the electrode and the compensating ionic charge in the electrolyte. The distance d is smallest for a faradaic capacitor where the stored electrons are localized on reduced surface cations and the compensating ionic charge is chemisorbed on the opposite side of the surface oxide ion separating the surface cation and the electrolyte. Where the surface area of the electrode and the distance d between the reduced electrode cation and the chemisorbed electrolyte cation remain unchanged by the applied voltage, the capacity

$$C \equiv \frac{\mathrm{q}}{\Delta V} = \frac{dq/dt}{d\,(\Delta V)/dt} = \frac{I}{v}$$

is constant and, for a constant sweep rate v, a cyclic voltammogram (CV) would have a constant faradaic current I as illustrated in **Figure 1**. On reversal of the current at the end potentials, the current would rise to a value mirrored on the I = 0 axis, and the rate of rise would depend on the CV sweep rate v relative to the rate of discharge or charge of the capacitor. A non-ideal capacitor would have a CV in which I varies with the voltage; a reversible, but displaced, maximum in I(V) would signal either an intercalation reaction or a second surface reaction[8].

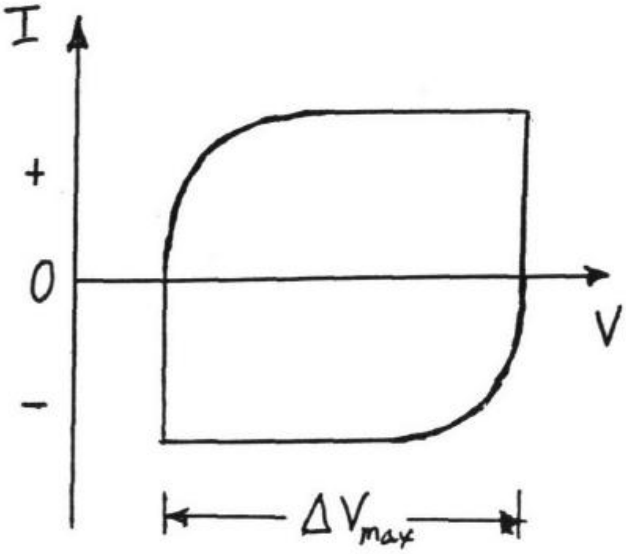


Figure 1: Ideal CV for Supercapacitor

2.4 EXPERIMENTAL

Activated carbon is a black solid substance resembling granular or powdered charcoal. It is a processed carbon material with a highly developed porous structure and a large internal specific surface area. The preparation of activated carbon with different pore sizes can be achieved by using physical or chemical activation process. In both methods, the development of porosity is different in term of practical procedures and mechanism. In physical activation the generation of porosity took place via selective elimination of the more reactive carbon of the structure and further gasification led to the production of the activated carbon with the sought pore structure.

In chemical activation process the precursor is mixed with a chemical such as $ZnCl_2$ or H_3PO_4, carbonized and washed to produce the activated carbon. Following the thermal decomposition of the precursor, the chemical reacts with the product causing reduction in the evolution of volatile matter and inhibition of the particle shrinkage. Once the chemical is removed by exhaustive washing, a large amount of porosity is formed.[9]

It was reported that the $ZnCl_2$ solution can give significant effect on carbon properties in term of surface areas. The surface areas of the resulting activated carbon prepared from gelam wood bark impregnated with various concentration of ZnCl2 solution is given in **Figure 2**.

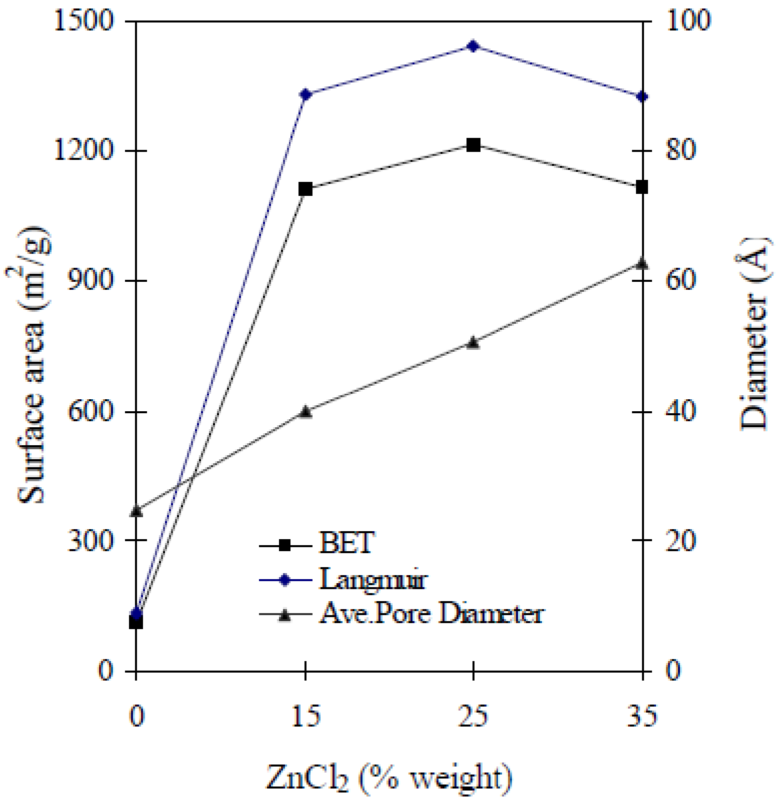


Figure 2: The effect of the concentration of ZnCl2 impregnation solution on the surface area and average pore diameter of activated carbon. [10]

Temperature has been reported to play an important role in producing optimum surface area of activated carbon. For example referencing to experimental work has shown that by heating a material at 500-700ºC for 3 hours would lead to the formation of activated carbon with large surface area[11]

Particle size of carbon can contribute to the capacitance of the material. The major advantage of nanoparticle assembly over conventional materials is the high surface area to volume ratio. The existence of such size effects offers a new pathway to regulate reactivity, either chemically or electrochemically or both by controlling the particle size. Electrochemistry plays a key role in nanoparticle science as it paves a way for coupling particle activity to external circuitry.

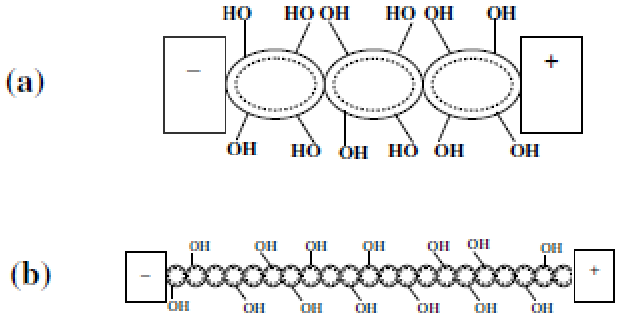


Figure 3: Effect of particle size on the mechanism of intergrain electron transport for (a) microsized, (b) nanosized particles.

Figure 3 shows the effect of particle size on the mechanism of intergrain electron transport for microsized and nanosized particles [12]. In the microsized particles, the depth of space charge layer is insignificant compared to the grain size and the electrical conduction on the application of an external bias is largely governed by the grain boundaries. On the other hand, in nanosized particles, the space charge layer is in par with grain size, so that the application of external bias of same magnitude would have a profound effect on the intergrain conduction, leading to an increase in electronic conduction and thereby, the capacitance. The nanoparticulate surface not only enhances current due to the high surface but also the scale of surface roughness removes the requirement for a solution phase species to mediate electron transfer to the active redox site of the electrode material. Under these conditions, an increase in the electronic and ionic conductance should be inversely proportional to the particle size; i.e. smaller the particle size, higher the conductance and hence the capacitance.

From the literature review, the capacitance of the material is depend on the physical properties. The surface cleanness, surface area and size particle can influence the ability of carbon to store charge. It also can be conclude that by applying physical and chemical activation can give significant effect to the physical properties of carbon.

So, the experiment will focus on the study of chemical activation and physical activation treatment towards carbonaceous waste. The chemical chosen is $ZnCl_2$ and Heating Temperature Treatment is using 500 ^{o}C. The activated carbon was characterized by its surface topography and particle size using SEM and XRD. Then comparison between treated and non-treated carbon is conducted.

CHAPTER 3: METHODOLOGY

3.1 RESEARCH METHODOLOGY

All knowledge in the journal, article and info that shared in the internet will be collected. The data will be analyzed and select appropriate info that can be used to design the experimental work. Study Material Safety Data Sheet (MSDS) to identify the hazard in the experiment and biological hazard from the chemical substance used before start the experiment. The experiment is design as below:

Part 1: Treatment Process

Objective 1 can be achieved by mix one of chemical activation process. The precursor is mixed with a $ZnCl_2$ and washed to produce the activated carbon. 4 samples were taken in various concentration of $ZnCl_2$. Then the mixture will heat up to remove all undesired and impurities component in the mixture.

Part 2: Characterization Process

Physical properties can be evaluated using two characterization methods; X-Ray Diffraction (XRD) and also Scanning Electron Microscope (SEM). Results of these characterization processes will be further discuss in **Chapter 4: Results and Discussion** section.

Part 3: Evaluation Supercapacitor Performance

In this part of study is to evaluate the electrochemical capacitor performance of carbonaceous waste. Under certain condition, the capacitor performance will be evaluated, in terms of its specific discharge capacity, charge-discharge capacity and also its efficiency. Comparison of electrochemical of electrochemical capacitor fabricated using the carbonaceous waste and capacitor using commercial carbon will be carried out to evaluate the capacitor's economic feasibility.

3.2 EXPERIMENTAL PROCEDURE

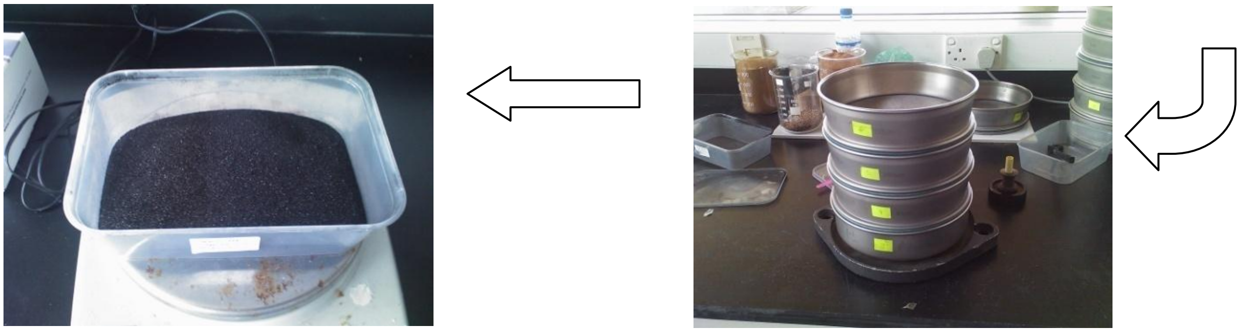
3.2.1 Part I: Preparation of chemical

High surface area of carbonaceous waste is favorable in the capacitor electrode fabrication. Thus, the carbonaceous waste is preferably in powder form. Since carbonaceous waste has hard physical structure, the press grinder is used to pound it until it became into powder form. After the grinding process, the carbonaceous waste is sieved to separates any big particles or any contaminant that might exist with the sample various size of sieve is used; the finest powder is taken to be used throughout the study. The planning of the experiment procedure is followed as below:

i. Chemical use: Carbonaceous waste
ii. Equipment use: Mortar pestle, mechanical sieve
iii. Objective : **To prepare sample for experimental used**
iv. Procedure:
    1. Clean & dry carbonaceous waste
    2. Crush & Sieve the carbonaceous waste (fine powder 65 µm)
    3. Sample ready to use for the next process

a. **Carbonaceous Waste**

b. **Crushed**



d. <u>Carbonaceous in Powder form</u>

c. <u>Sieve</u>

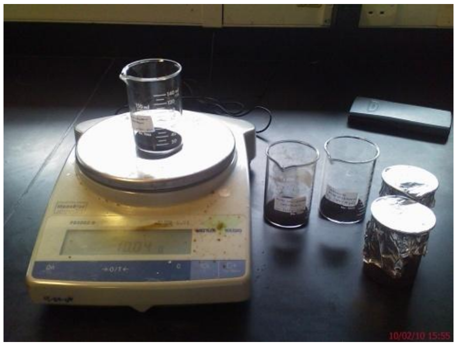
Figure 4: Chronology of Sample Preparation

3.2.2 Part II: Chemical Activation Process

The solution of $ZnCl_2$ is a good chemical activation to activate carbon. From the literature review was reported that it can increase surface area of carbon dramatically upon the introduction of $ZnCl_2$. The impregnation process was performed at 70°C to vaporize excess water and well mix the mixture. The impregnated sample was dried in an oven at 120°C overnight to remove the excess $ZnCl_2$.

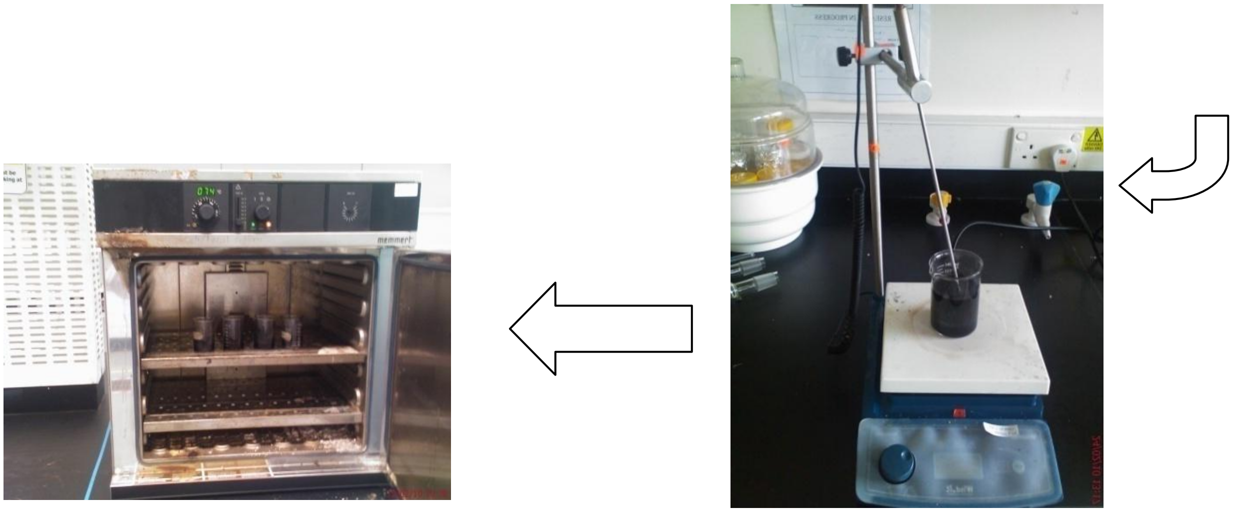
The planning of the experiment procedure is followed as below:

i. Chemical use: Sample (from Part I), $ZnCl_2$.
ii. Equipment use: Oven, Water bath & hot plate.
iii. Objective: **1. To investigate chemical activation process effect on carbonaceous waste.**
2. To activate the carbon inside carbonaceous waste
3. To increase surface area of carbonaceous waste
iv. Procedure:
   1. Take 10 grams of sample to the biker
   2. Weight $ZnCl_2$ by different mass to make various concentration of solution.
   3. Mix the sample with the solution (15-20 w/w %)(**Table 1**) and put the mixture into oil bath.
   4. Set the temperature of oil bath at 70^0C overnight.
   5. Dry the impregnated sample in oven at 120^0C overnight.



Weight the carbonaceous waste

Heat at 70^0C



Dry the sample at 120^{0}C **Heating and stir constantly at 70^{0}C**

Figure 5: Chronology of Chemical Activation Process

Table 1 shows the parameter that will be used in carbonaceous waste treatment. Sample A is non-treated carbon and Sample E will be not introduced by $ZnCl_2$ but with high temperature. A series of 10 gram of the sample B, C and D was mixed with $ZnCl_2$ solution of various concentrations.

Table 1: The various of concentration of $ZnCl_2$ were used

Treatment Sample	Chemical activation	Temperature(°C)	Concentration (w/w %)
A	-	120	0
B	$ZnCl_2$	120	15
C	$ZnCl_2$	120	25
D	$ZnCl_2$	120	35
E	-	500	0

3.2.3 Part III: Characterization Process

<u>Characterization Process Using XRD</u>

Chemical used : Treated carbonaceous waste.

Equipment used: X-ray Diffraction

Objective :

1. **To identify type of particle in sample**
2. **To measure the particle size of the sample.**
3. **To compare crystallization between treated and non-treated carbonaceous waste**

Procedure:

1. Take 2 grams of sample
2. Load sample onto the tray in front of the XRD. Sample side is facing up.
3. Run the sample and print results using software.
4. Identify the most intense peak from the results.

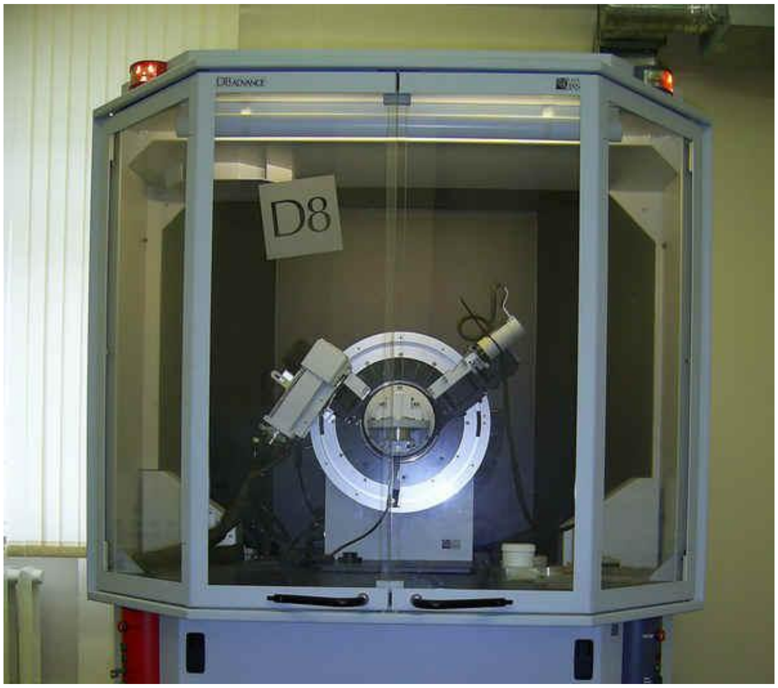


Figure 6: Scanning Electron Microscope

X-Ray Diffraction (XRD)

X-ray diffraction is a tool for the investigation of the structure of matter. X -rays are scattered by interaction with the electrons of the atoms in the material being investigated. The technique began when von Laue discovered that crystals diffract x-rays in 1912. Since then it has been applied to chemical analysis, stress and strain measurement, the study of phase equilibrium, measurement of particle size, as well as crystal structure.

Characterization Process Using SEM

Chemical used: Treated Carbonaceous waste.
Equipment used: Scanning Electronic Microscope
Objective : **1. To study the structure of treated and non-treated sample**
2. To study the effect of chemical treatment and heating treatment on the sample

Procedure:

1. Clean the sample and take 2 grams of sample.
2. Place the sample in vacuum condition in 0.5 hours.
3. Stack the sample onto the brass mount- for non conductive sample only
4. Run the sample

For this study, the characterization using XRD was carried out under these conditions:

Scan Speed : 2º/min
2θ range : 10º -80º

Figure 7: Place the sample in vacuum condition

Figure 8: Run the sample in SEM

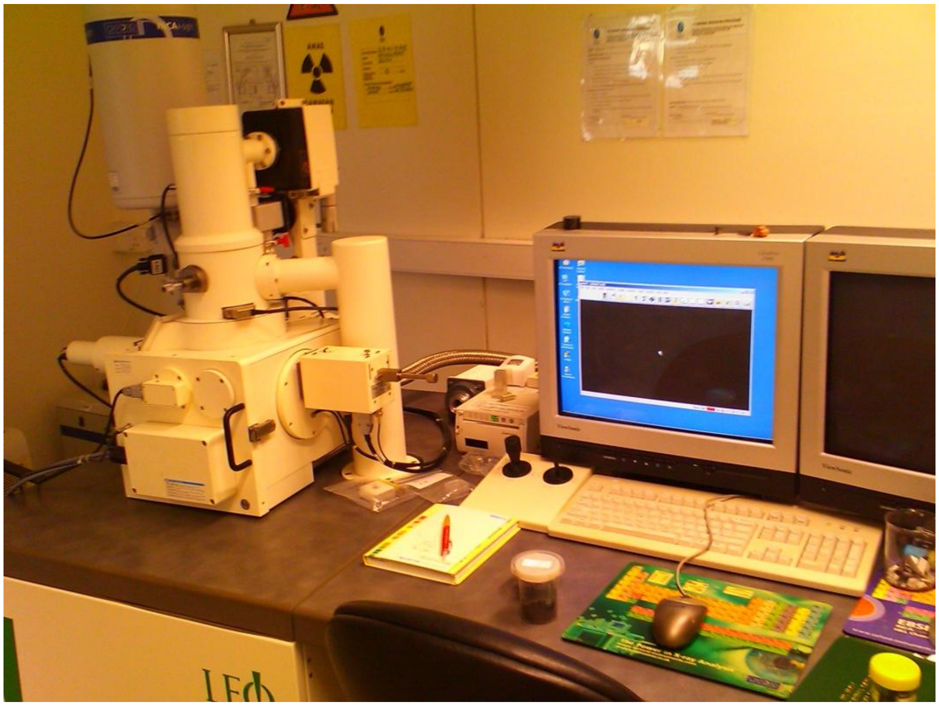


Figure 9: The view of the sample structure show on the monitor

Scanning Electronic Magnifying(SEM)

SEM can characterize nanomaterials and nanostructure. The SEM creates images by using electrons instead of light waves. In SEM, source of electrons is focused into a beam with a very fine spot size of ~5 nm and having energy range from few hundred eV to 50k eV. Images are produced by collecting the emitted electrons on a cathode ray tube.

For this study, SEM will be carried out in different magnitude of magnification. SEM results from:

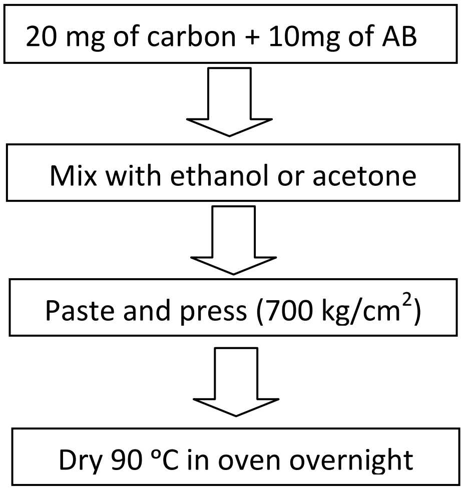
1. 200x magnification
2. 1000x magnification
3. 5000x magnification
4. 10 000x magnification

3.2.4 Part IV: Supercapacitor Performance Evaluation

i. Chemical use: Sample (from Part III), Acetone, Ethanol,
ii. Equipment use: Test Cell, Teflon
iii. Objective : **To evaluate the supercapacitor performance of treated carbonaceous waste.**
iv. Procedure:

1. Electrolyte preparation:
   i. 0.254g of lithium perchloride salt is added in the 50ml biker.
   ii. Acetonitrile is poured into biker until volume of solution is 25ml
   iii. The mixture is stirred to make a well-mix solution.

2. Cathode and Anode preparation:



3. Capacitor configuration:

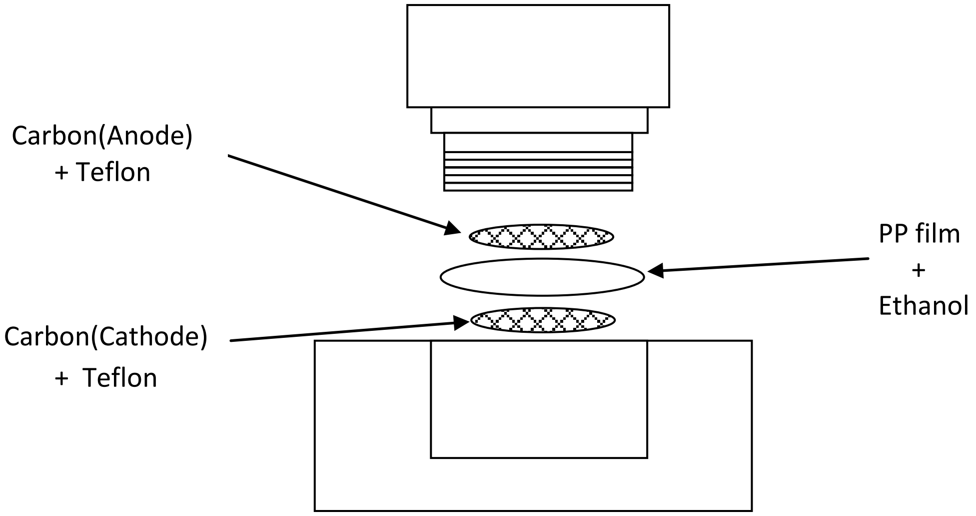


Figure 10: Battery Test Cell is used to evaluate the performance of supercapacitor

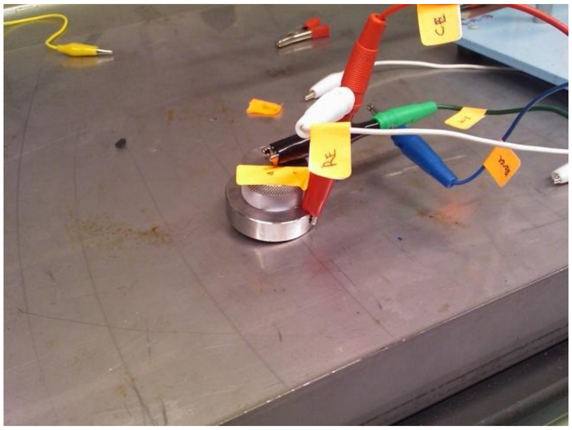


Figure 11: Connection configuration between Test Cell and supercapacitor

In this experiment, the current that will use to evaluate the performance of supercapacitor are 0.005 A, 0.01 A and 0.05 A. The potential different is set at 0~2 V and run in 10 cycle.

3.3 MILESTONE FOR THE FINAL YEAR PROJECT

No.	Detail/ Week	1	2	3	4	5	6	7	8	9		10	11	12	13	14	18	19	14
	Date	25-Jan	01-Feb	08-Feb	15-Feb	22-Feb	28-Feb	01-Mac	08-Mac	15-Mac	22-Mac	29-Mac	05-Apr	12-Apr	19-Apr	26-Apr	24-Mei	04-Jun	11-Jun
1	Preparation of Sample	■	■	■	■														
	1. Grind Carbonnaceous Waste																		
	2. Sieve with desired size																		
2	Chemical Activation Treatment					■	■	■	■										
3	Submission of Progress Report1						■												
4	Sample characterization					■	■	■	■		■	■	■	■					
	1. SEM analysis																		
	2. XRD analysis																		
5	Submission of Progress Report 2												■						
6	Pre-EDX (Poster Presentation)													■					
7	Project work continues										■	■	■	■	■				
	Evaluation electrode capacitor performance																		
8	Submission of Final Report															■			
9	Documentation	■	■	■	■	■	■	■	■		■	■	■	■	■	■	■	■	■
10	Final Oral Presentation																■	■	
																		■	
11	Submission of hardbound																		■

CHAPTER 4: RESULT & DISCUSSION

4.1 CHARACTERIZATION OF CARBONACEOUS WASTE

4.1.1 X-Ray Diffraction (XRD)

Table 2:The highest intensity of Carbon A

2-Theta	Intensity, cps
25.35	178.98
25.4	180.98
25.45	196.97
25.5	190.98
25.55	184.98
25.6	215.97
25.65	186.98
25.7	181.98
25.75	186.98
25.8	186.98
25.85	151.98

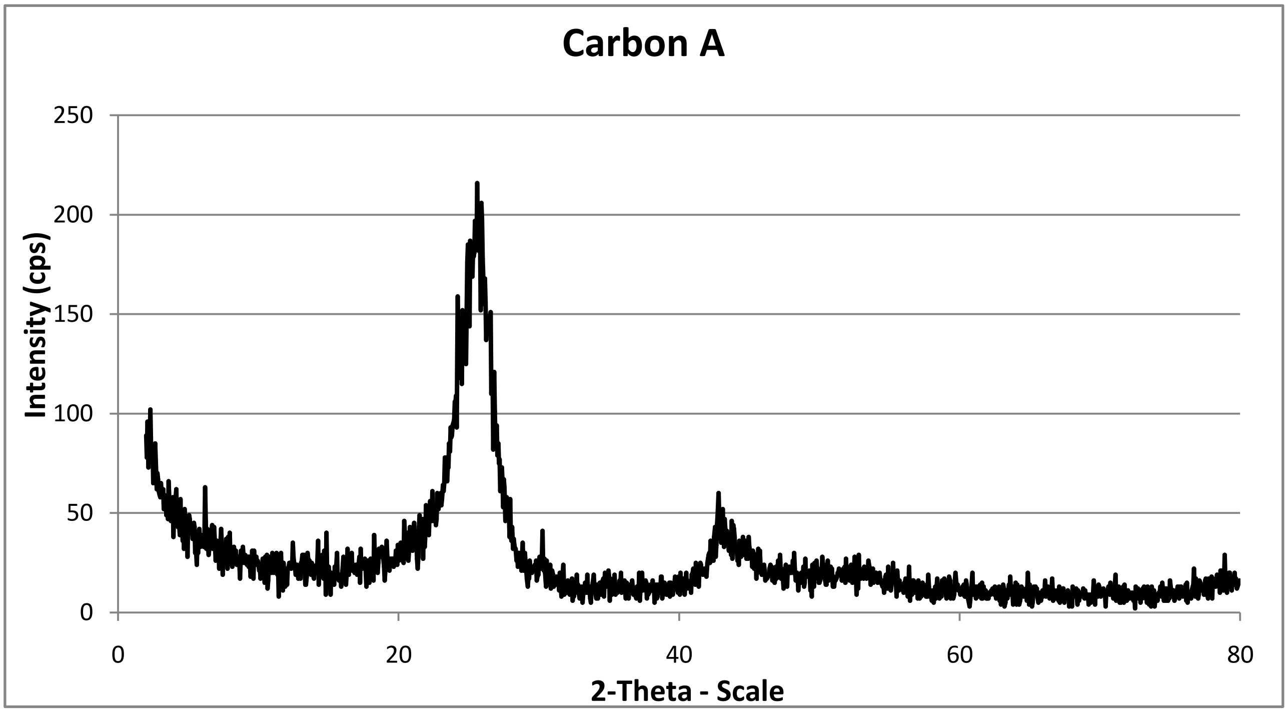


Figure 12: XRD patterns of Non-Treated Carbonaceous Waste

Table 3: The highest intensity of Carbon B

2-Theta	Intensity, cps
25.35	41.995
25.4	43.994
25.45	49.993
25.5	46.994
25.55	50.993
25.6	52.993
25.65	39.995
25.7	46.994
25.75	38.995
25.8	32.996
25.85	30.996

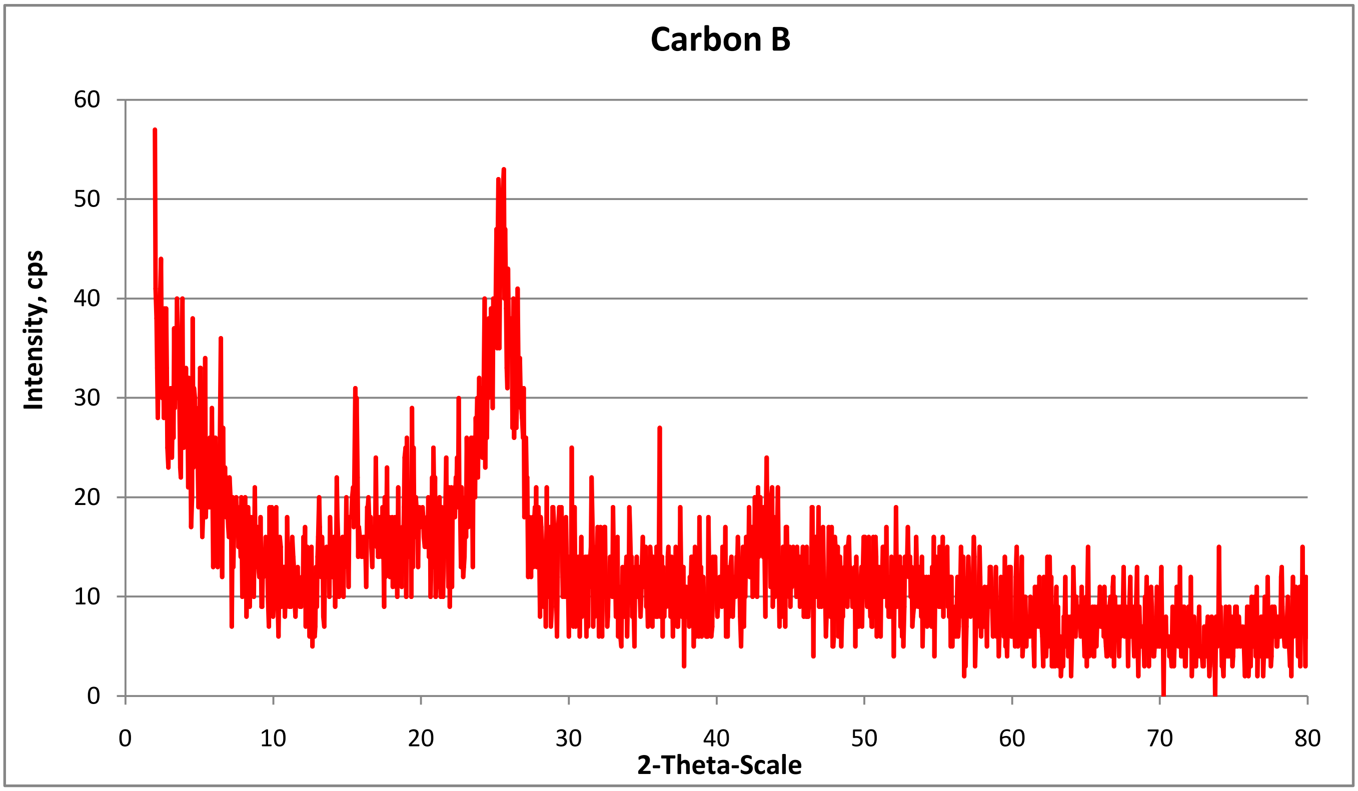


Figure 13: XRD patterns of treated Carbonaceous Waste with 15% $ZnCl_2$

Table 4: The highest intensity of Carbon C

2-Theta	Intensity, cps
25.4	34.995
25.45	30.996
25.5	30.996
25.55	34.995
25.6	24.997
25.65	38.995
25.7	37.995
25.75	29.996
25.8	25.997
25.85	34.995
25.9	30.996

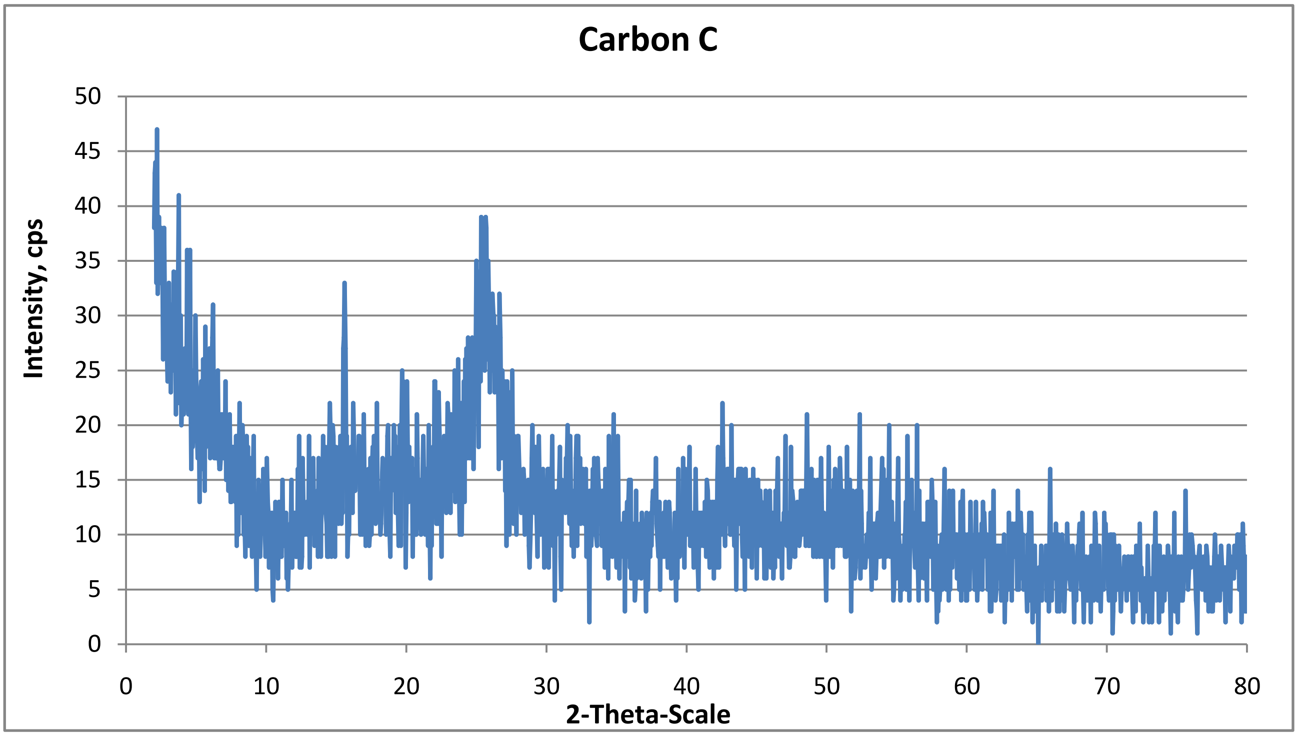


Figure 14: XRD patterns of treated Carbonaceous Waste with 25% $ZnCl_2$

Table 5: The highest intensity of Carbon D

2-Theta	Intensity, cps
25.2	16.998
25.25	17.998
25.3	22.997
25.35	17.998
25.4	15.998
25.45	28.996
25.5	20.997
25.55	18.998
25.6	14.998
25.65	11.998
25.7	22.997

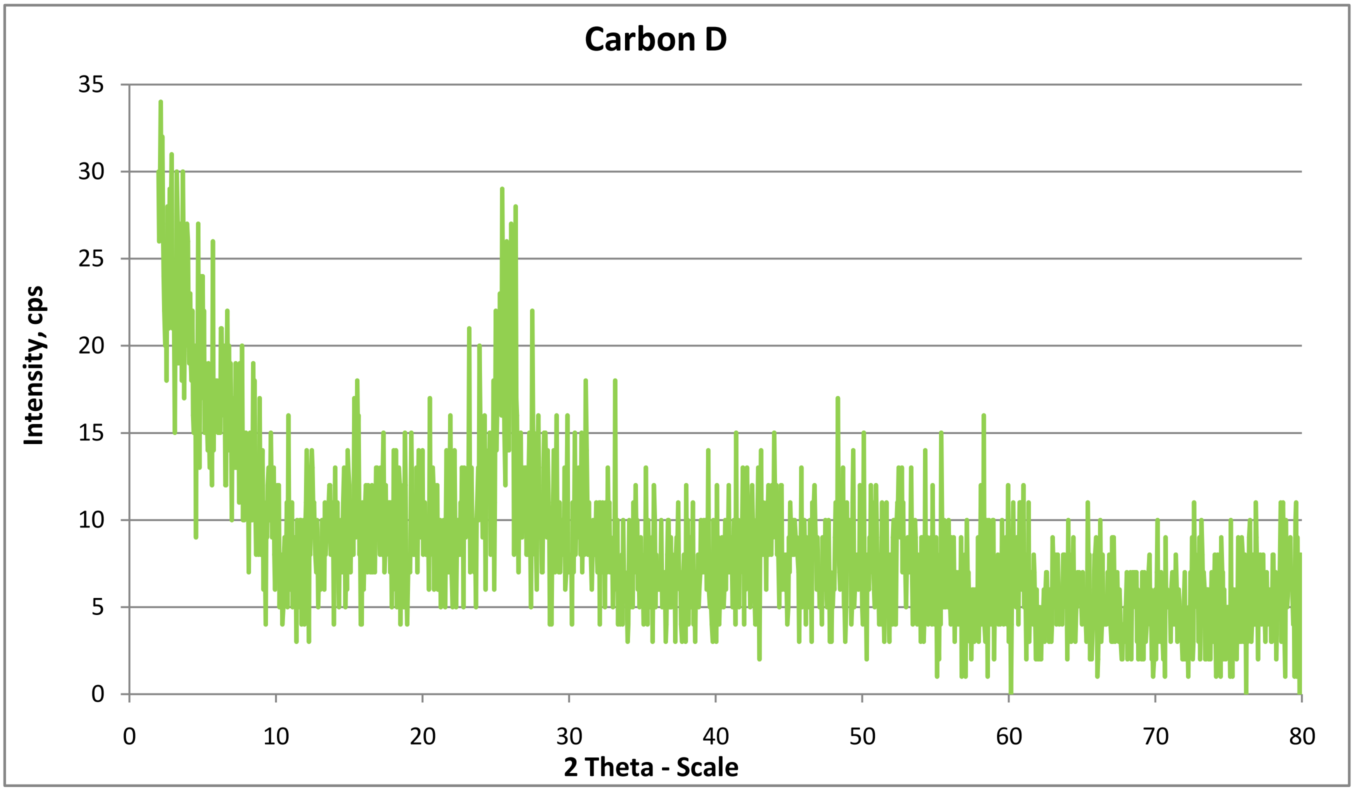


Figure 15: XRD patterns of treated Carbonaceous Waste with 35% $ZnCl_2$

Table 6: The highest intensity of Carbon E

2-Theta	Intensity, cps
25.3	159.98
25.35	129.98
25.4	162.98
25.45	138.98
25.5	164.98
25.55	171.98
25.6	158.98
25.65	161.98
25.7	164.98
25.75	135.98
25.8	149.98

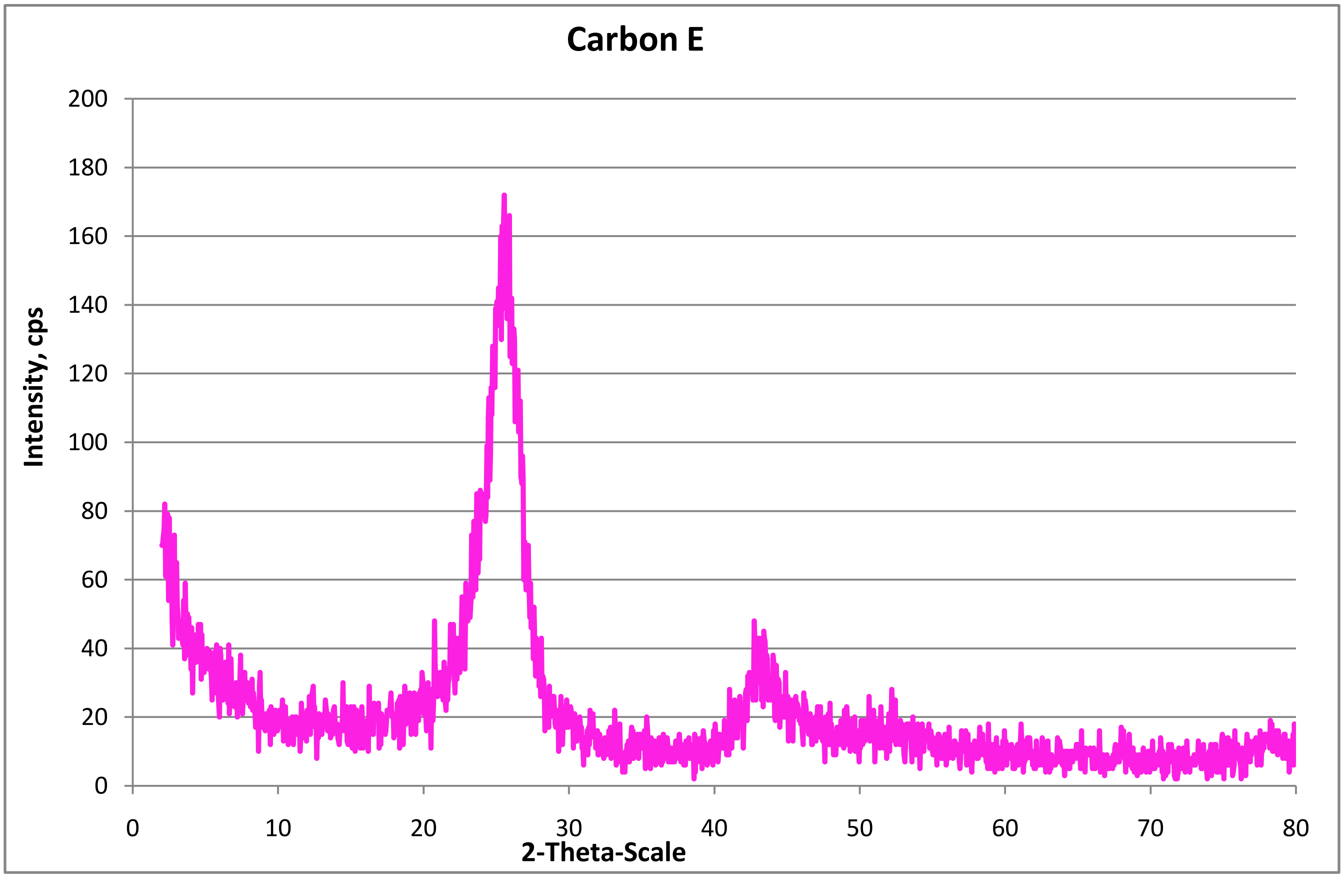


Figure 16: XRD patterns of treated Carbonaceous Waste with Heating at 500^{o}C

X-ray diffraction is a commonly employed technique to elucidate the structural features of carbonaceous materials including the average estimation of crystallite size and degree of ordering.

2 method treatments were chosen in this project which is Heat Treatment and Chemical activation treatment. Sample A is non-treated Carbon whereas sample E is heating treatment sample. Sample B, C and D is the treatment using chemical activation which is $ZnCl_2$.

Figure 12, 13, 14, 15, and **16** shows a superimposed powder XRD pattern of the samples A, B, C, D and E respectively. For Sample A **(Figure 12)**, the peaks at 25.6º (215.97) and 42.8º (59.99) indicate that non-activated carbon consists of small domain of ordered graphene sheets, while all other peaks indicate graphitic planes. **Figure 16** shows the carbonaceous waste after treated with 500ºC. The graphene peak shown is 25.55º (171.98) and 42.75º (47.99) which is smaller than sample A.

This peak change significantly as the concentration of $ZnCl_2$ impregnating solution increased. Shown in **Figure 13, 14**, and **15** are the sample B, C and D with highest peak at 25.6º(52.99), 25.65º (38.995) and 25.45º (28.996) respectively. As the concentration of $ZnCl_2$ increased from 15% to 35% the graphene stacking peak is decrease drastically.

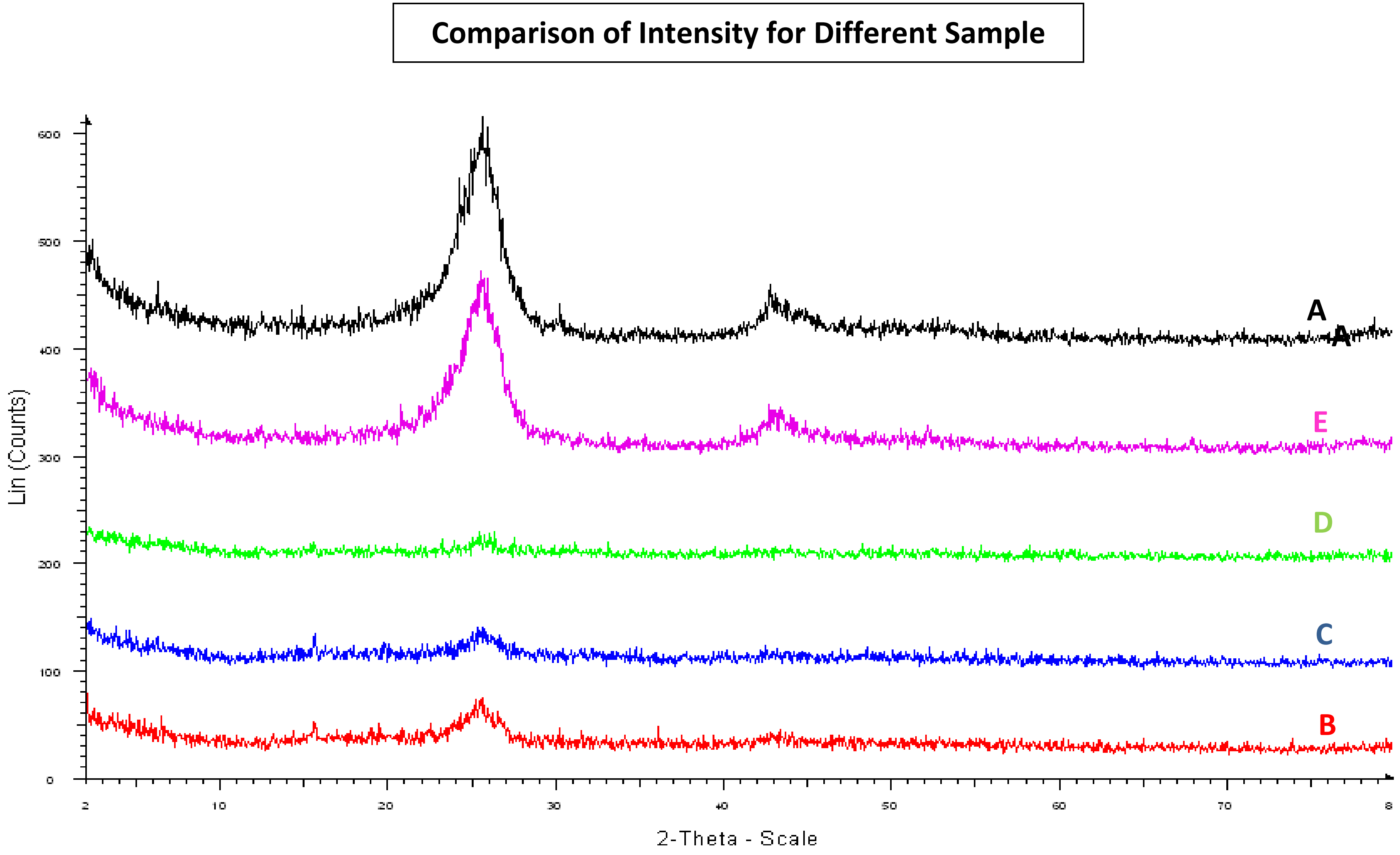


Figure 17: Superimposed XRD patterns for all samples

The entire sample was combined into **Figure 17** to see the comparison between them. According to literature review, high crystalline graphite has intensity of 700, while the sample has intensity lower than 300. The resultant intensity is in the range of high crystallized graphite and amorphous carbon. It can be concluded that Carbon A & E are low-crystalline graphite whereas carbon B & C is low-amorphous carbon. Carbon D shows no peak. Besides that, Carbon D is lost its crystalline structure.

Figure 17 showed that the crystalline structure decreased as the concentration of $ZnCl_2$ impregnating solution increased. $ZnCl_2$ introduced to the sample eliminate the crystalline structure and produced low amorphous surface and non-crystalline structure.

The XRD results also give the value of d-spacing and FWHM for this carbonaceous waste is as follows:

Table 7: The value of D-spacing & FWHM

Sample Name	d (Obs. Max)	FWHM	Raw Area	Net Area
	Angstrom	2-Theta °	Cps x 2-Theta °	Cps x 2-Theta °
Sample A	3.47689	2.613	834.8	571.5
Sample B	3.48868	1.713	182.3	73.57
Sample C	3.50599	1.648	170.5	54.2
Sample D	3.49624	1.418	74.59	17.88
Sample E	3.47385	2.317	627.4	402.8

Comparison of the *XRD's* indicates that the FWHM of the graphene stacking are affected significantly by the process temperature and $ZnCl_2$ solution, with the higher process temperatures and concentration of $ZnCl_2$ causing declined regularity of the graphene peaks. The full-width at half maximum (FWHM) for the graphene stacking peak, found at 1.4 - 3.0 Å in carbonaceous waste, measures the regularity of the stacking of the graphene layers in carbon. The interplaner distances d-spacing, for sample A, B, C, D and E are 3.48Å, 3.49Å, 3.51Å, 3.49Å and 3.47Å respectively which are comparable with the typical d value of activated carbon

(3.45Å). It is noticed that when the concentration of $ZnCl_2$ increase the interplaner distances also increase.

The means crystallite size D was obtained from the Scherrer formula [11]:

$$D = \frac{(k\lambda)}{(\beta \cos\theta)}$$

where k = 0.9, λ = 1.541Å, θ is the diffraction peak angle, and β denotes the full width at half maximum(FWHM) of the corresponding diffraction peak.

One example calculation for sample A:

$$\mathrm{D} = \frac{(k\lambda)}{\beta \cos\theta}$$

$$= \frac{(0.9)(0.154n)}{0.0456\ \cos 12.8}$$

$$= 3.194\ \text{nm}$$

The value of particle size was calculated and showed in the table below:

Table 8: Result of particle size

Sample Name	d - spacing	FWHM	Particle size, D
	Angstrom	2-Theta °	nm
Sample A	3.47689	2.613	3.194
Sample B	3.48868	1.713	4.753
Sample C	3.50599	1.648	5.065
Sample D	3.49624	1.418	5.885
Sample E	3.47385	2.317	3.602

The result is illustrated in the graphs form like shown in the graph below:

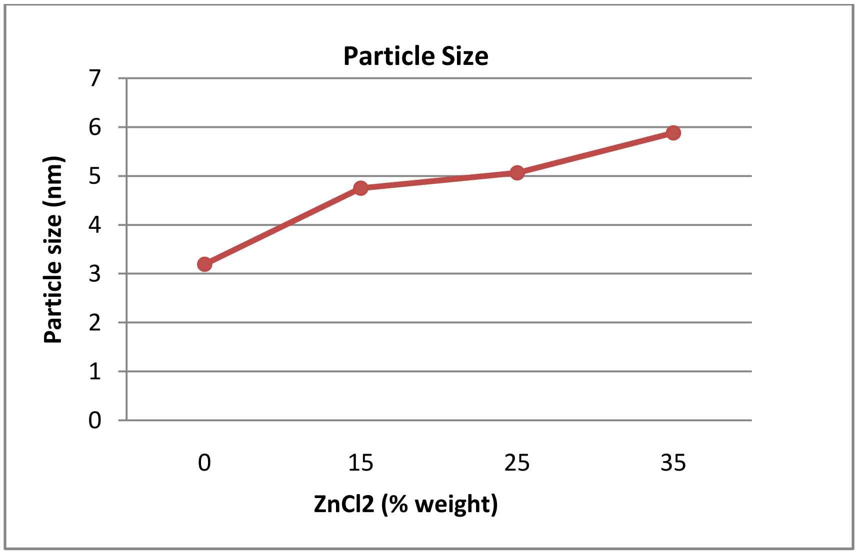


Figure 18: The effect of the concentration of ZnCl2 impregnation solution on particle size

The particle size of the resulting activated carbon prepared from carbonaceous waste impregnated with various concentration of $ZnCl_2$ solution is given in **Figure 18**. The particle size of the physically non-treated carbon (0% w/w $ZnCl_2$) is much lower compared to that of chemically treated-carbon. It increased drastically upon the introduction of $ZnCl_2$ and reached a maximum value of 5.9nm at 35% w/w $ZnCl_2$.

Similar phenomenon was observed for heating temperature treatment (shown in **Table 9**) although the particle size slightly increased. The particle size increased with increasing the temperature at 500 ºC.

Table 9: Effect of Temperature on Carbonaceous Waste

Sample Name	Temperature	Particle size, D
	°C	nm
Carbon A Carbon E	120 500	3.194 3.602

4.1.2 Scanning Electron Microscopic (SEM) Result

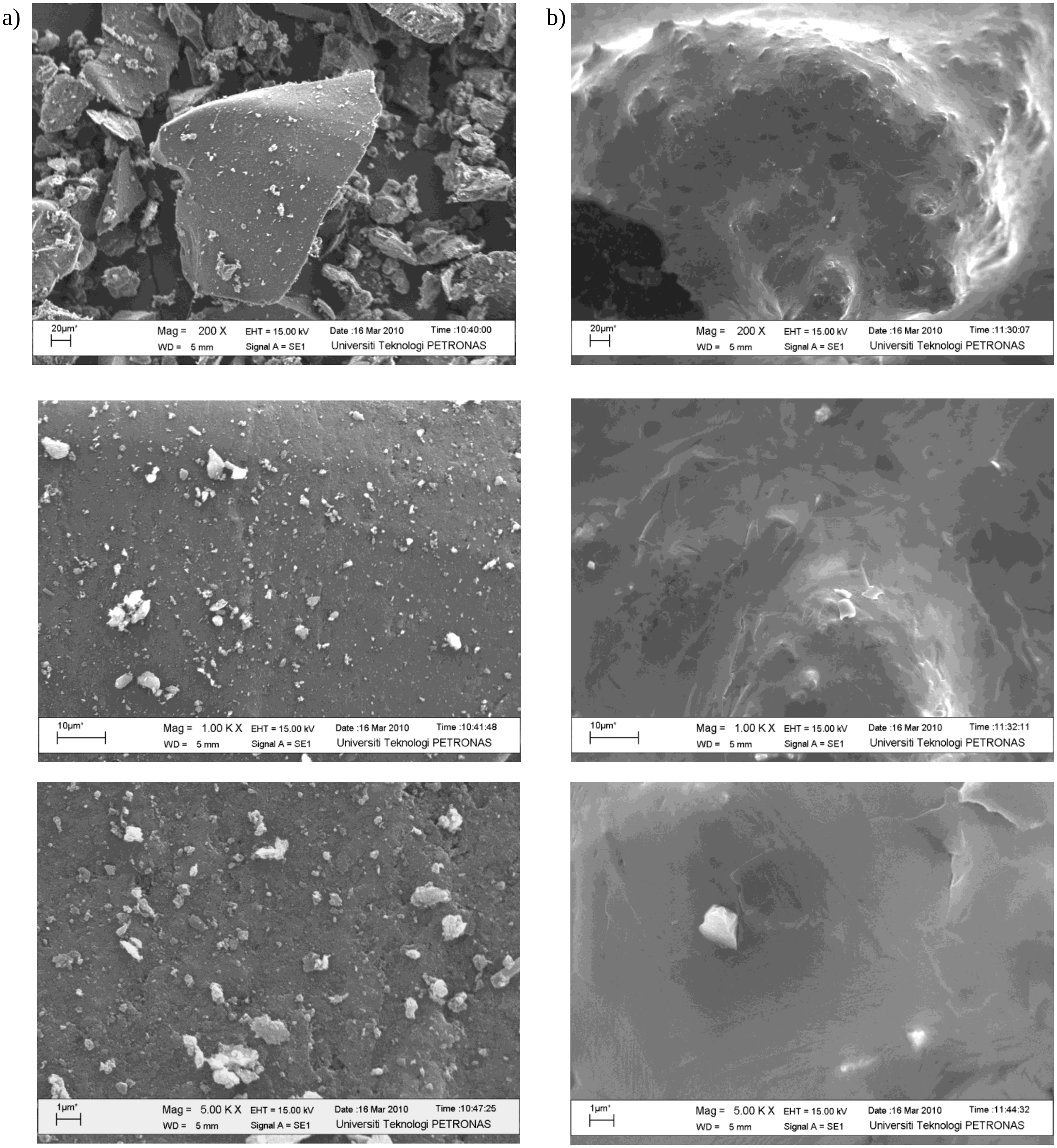


Figure 19:Scanning electron micrographs of activated carbon impregnated with (a) 0% (b) 35%

Figure 19 shows the result of SEM for treated and non-treated carbon. **Figure 19-(a)** is the result for Sample A and **Figure 19-(b)** is the result sample for Sample D. From the results it shows the differences of carbon particle distribution with various sizes in the sample. The grain size has a wide range of 195.4nm - 1.4 µm. The particle sizes of non-treated carbon are not uniform while the treated carbon posses a clean surface with uniform particle sizes. It can be seen clearly in **Figure 20** where the visual is zooming at 10 K magnification. Non-treated sample has a lot of non-uniform size of particle distribute on surface (**Figure 20.a**). A difference surface is possessed by Treated carbon where the cleanness surface and little non-uniform particles on the surface.

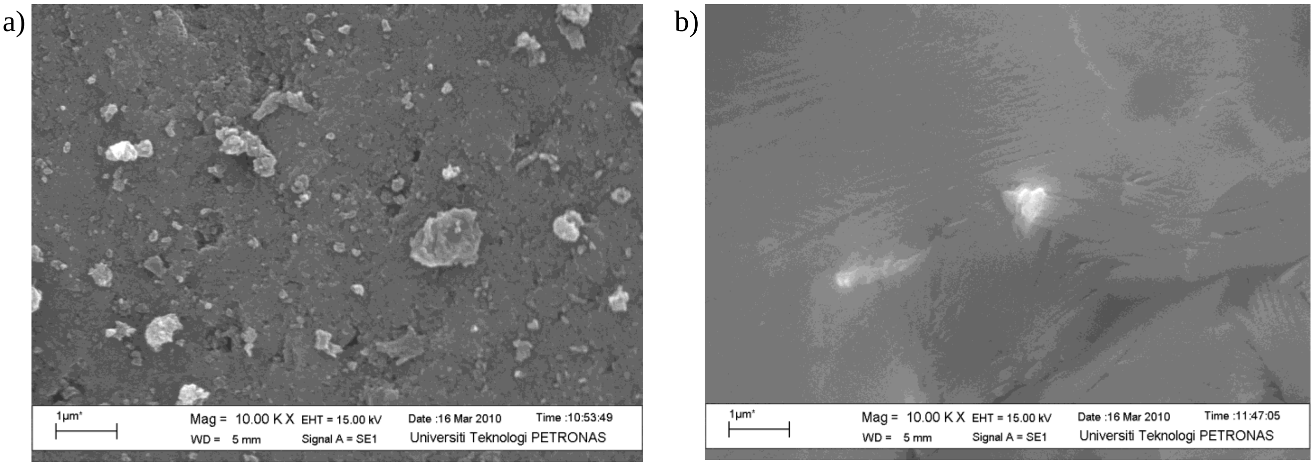


Figure 20: Comparison the clean surface of carbonaceous waste (a) Non-Treated (b) Treated

The particle size estimated by SEM micrographs as between 312.6nm -937.8nm for non-treated carbon **(Figure 21)** whereas the treated carbon posses the improvement size with range 312.6nm - 1.407 µm **(Figure 22)**. The sample that was treated by HTT also showed the increasing in size, which gives 195.4nm – 390.8nm in range size **(Figure 23)**. However, the sizes are still smaller compared to sample that was treated by chemical activation.

SEM micrographs estimate the area of non-treated carbon 76 756nm^2. After the treatment by 35% present of ZnCl2 concentration, the value of the area is about the same with uniform particle size. The sample taking from HTT gives the smallest value of area which is 119 931nm^2.

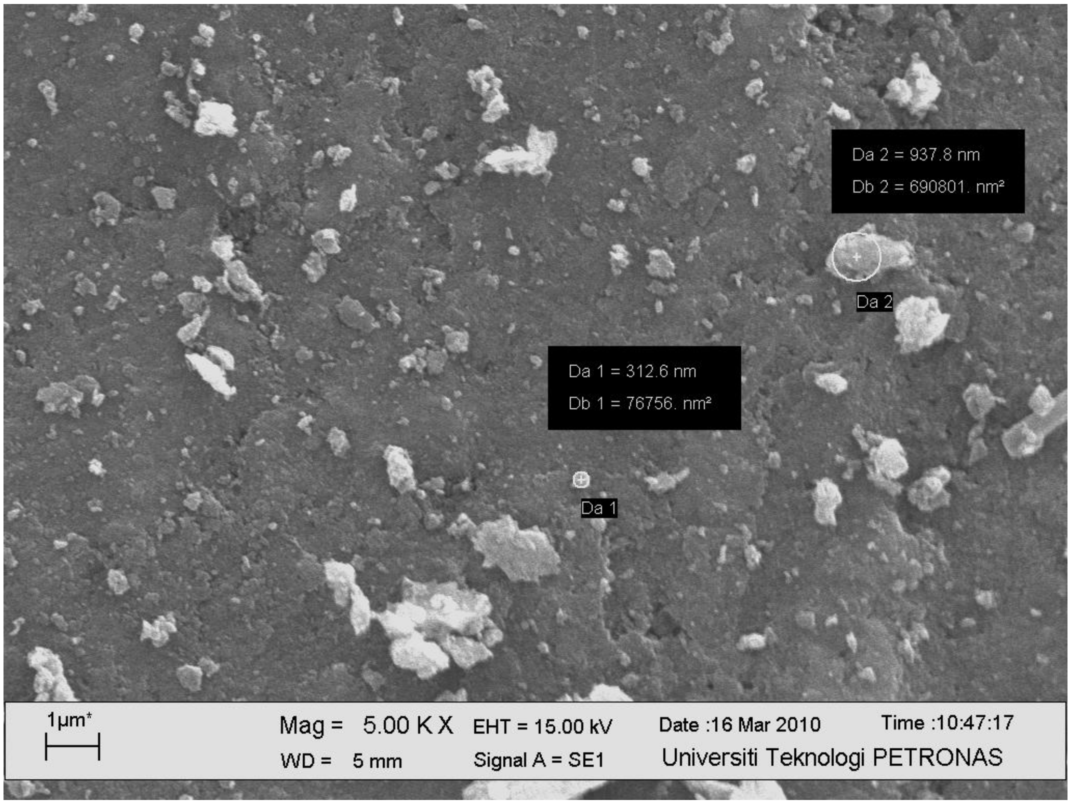


Figure 21: Size estimated using SEM for Sample A

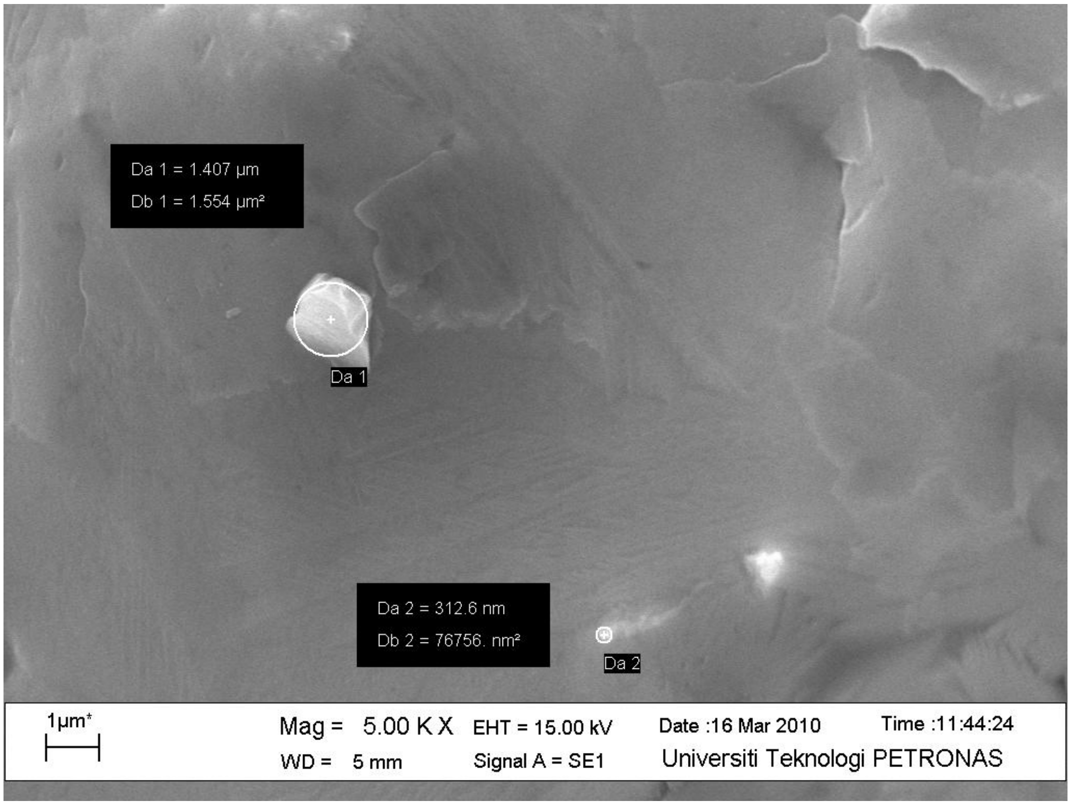


Figure 22: Saiz estimated using SEM for Sample B

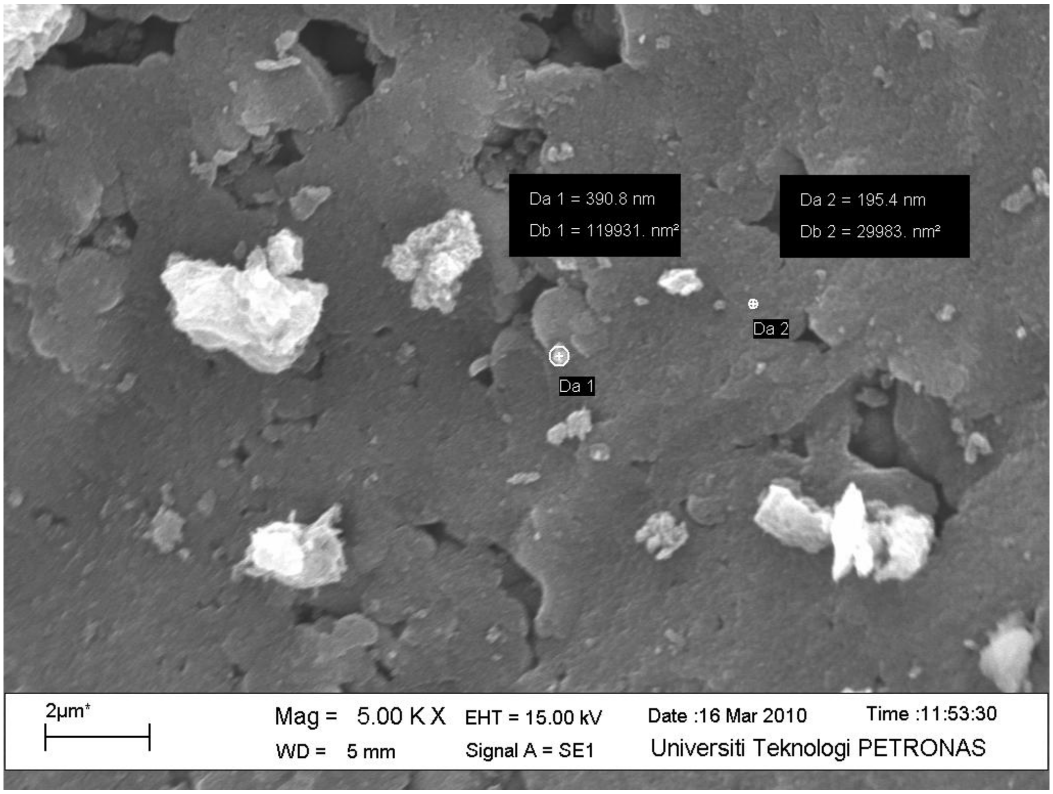


Figure 23: Size estimated using SEM for Sample E (HTT)

Figure 22 represents the morphology of the resulting activated carbon prepared via chemical activation methods. In the present of $ZnCl_2$ solution, the amount of $ZnCl_2$ can be distributed uniformly with high dispersion throughout the interior of the particle. Hence, after washing, produced a uniformly microporous carbon. The pore volume increased with a homogeneous pore size distribution. **Figure 23** represents the morphology of the resulting activated carbon prepared via physical activation method. At high temperature, a larger swelling of the particle was produced but it is not give the uniform size distribution.

These result indicates that degree of grain size tend to increase with higher calcinations temperature and the present of chemical activation. The carbonaceous particle size estimated by SEM micrographs as between 195.4nm - 1.4 μm and it is different in size by Scherrer's equation since Scherrer's equation is calculated from XRD patterns while SEM micrographs are calculated from selected area in sample.

4.2 ELECTROCHEMICAL CAPACITOR PERFORMANCE

Supercapacitor performance is the critical part in determining the ability of material to work as capacitor. So, the capacitor performance will be evaluated in terms of:

a. Charge-discharge time
b. Specific Discharge capacity
c. Capacitor Efficiency
d. Comparison between commercial carbon capacitor with carbonaceous waste capacitor.

4.2.1 Charge-Discharge Time

4.2.1.1 Voltage Profile for I = 5 mA

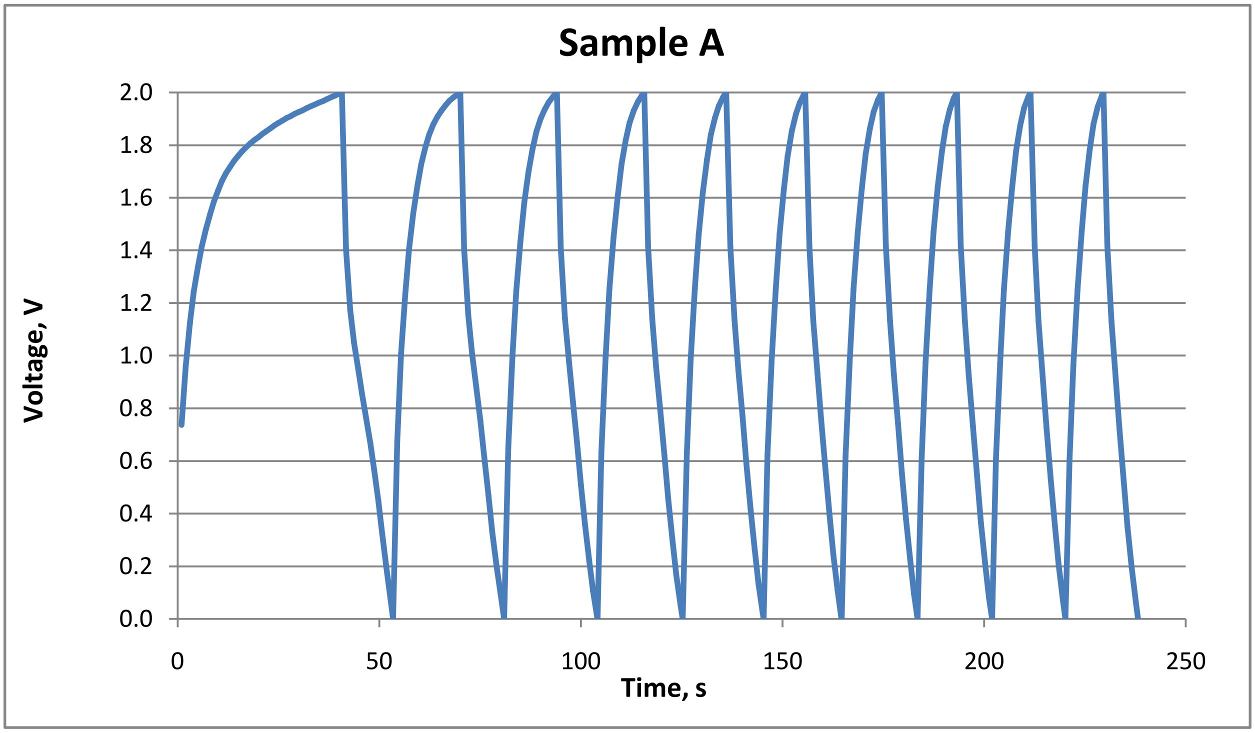


Figure 24: Voltage profile for Sample A at I= 5mA

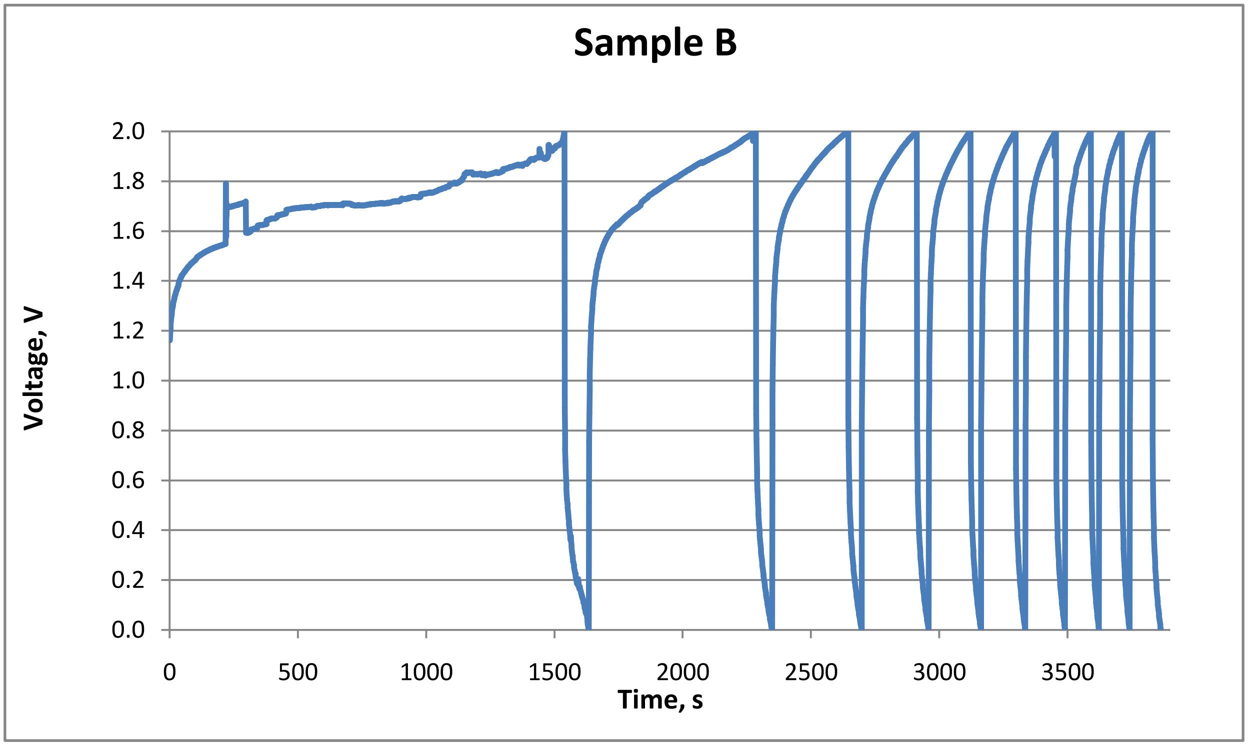


Figure 25: Voltage profile for Sample B at I= 5mA

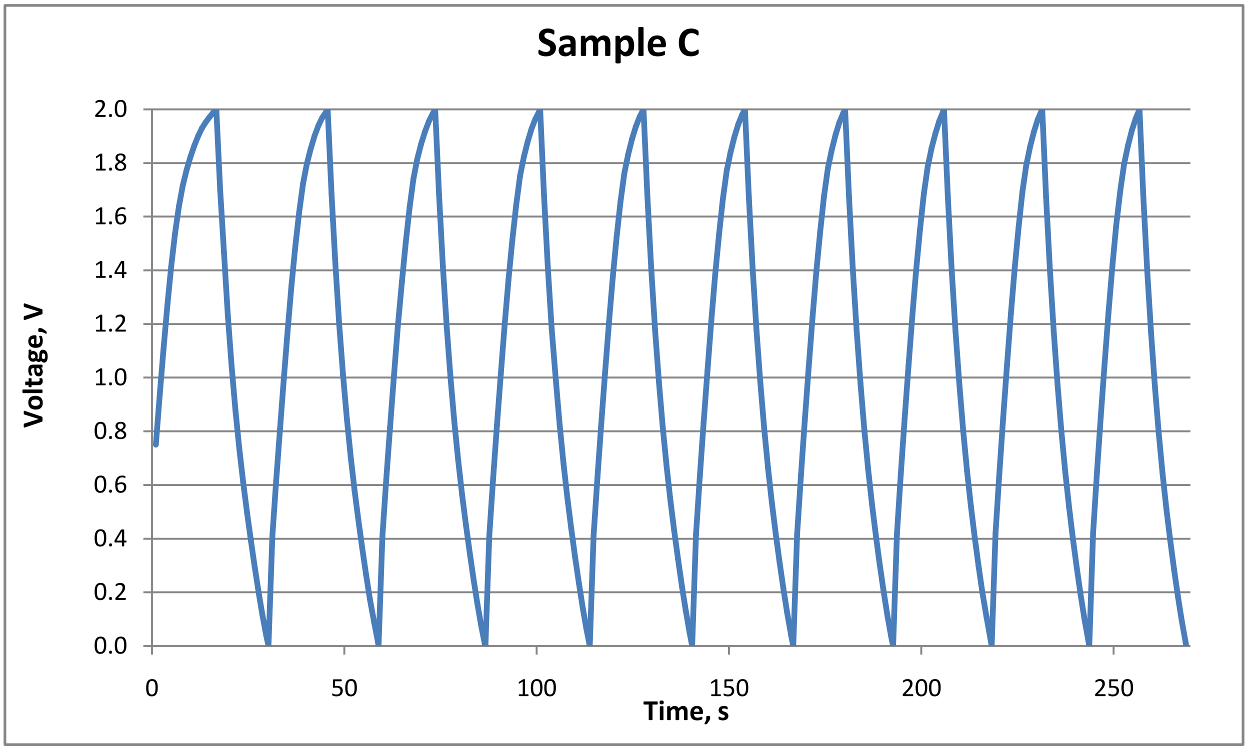


Figure 26: Voltage profile for Sample C at I= 5mA

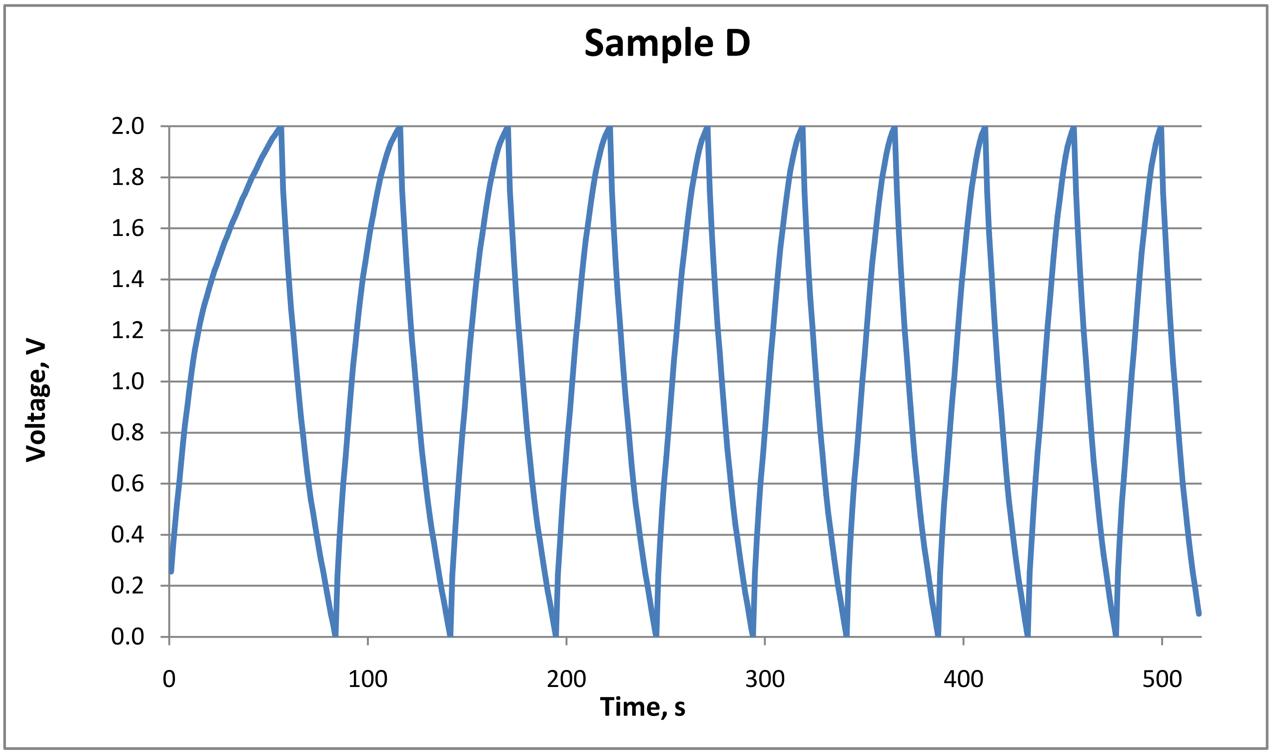


Figure 27: Voltage profile for Sample D at I= 5mA

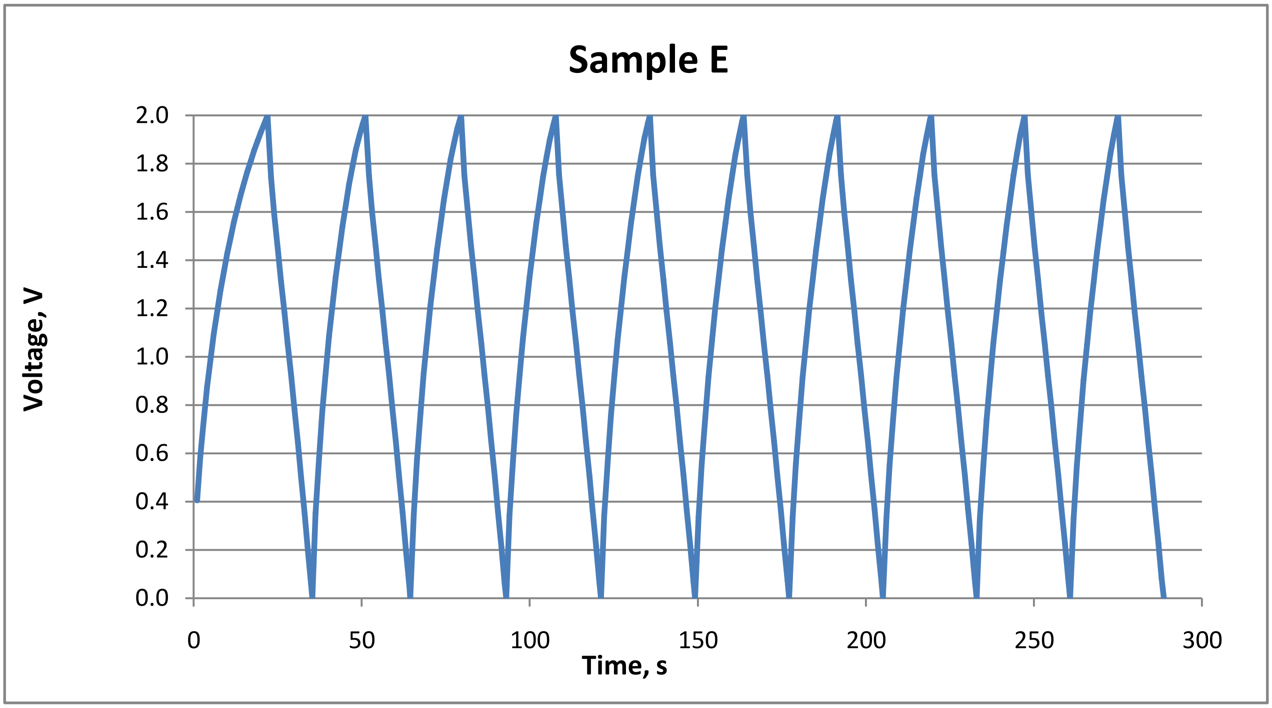


Figure 28: Voltage profile for Sample E at I= 5mA

4.2.1.2 Voltage Profile for I = 10 mA

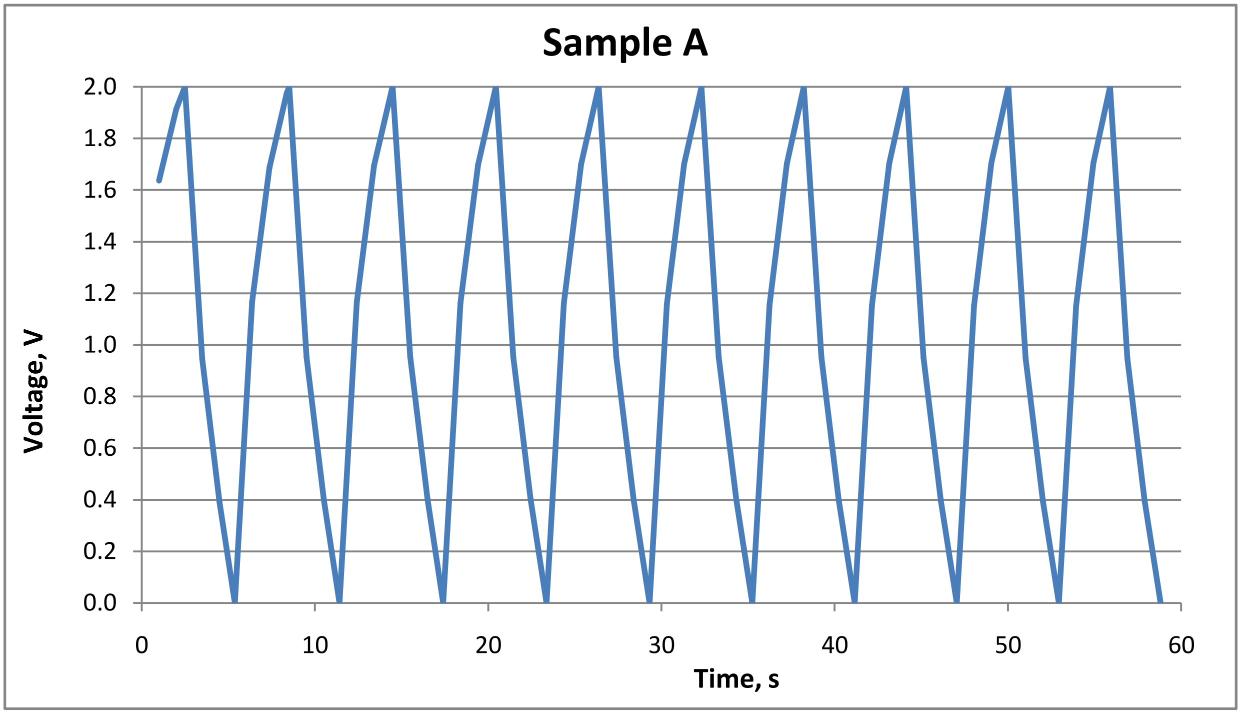


Figure 29: Voltage profile for Sample A at I= 10mA

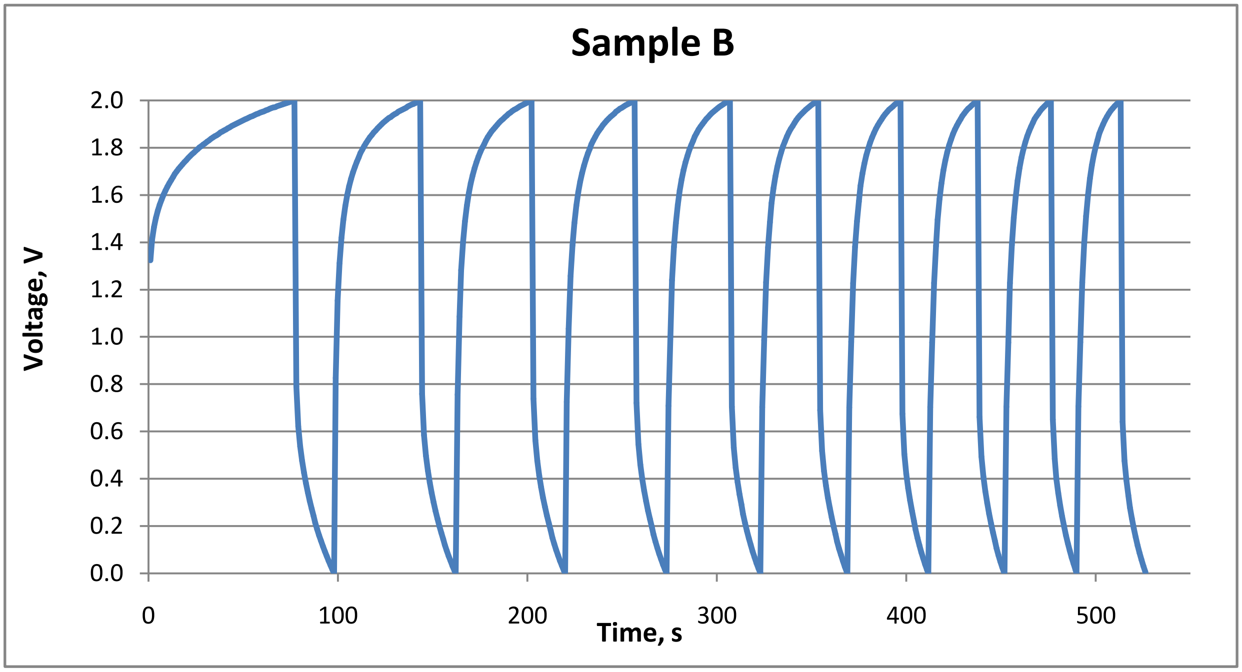


Figure 30: Voltage profile for Sample B at I= 10mA

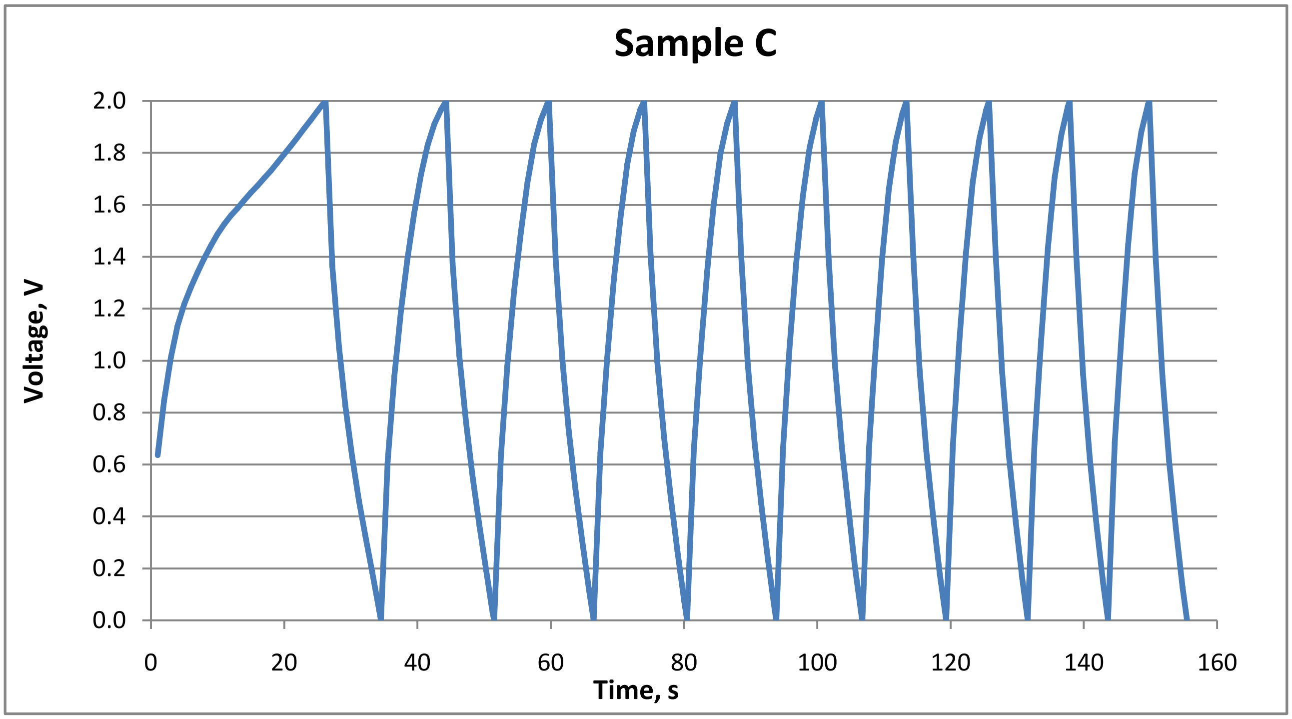


Figure 31: Voltage profile for Sample C at I= 10mA

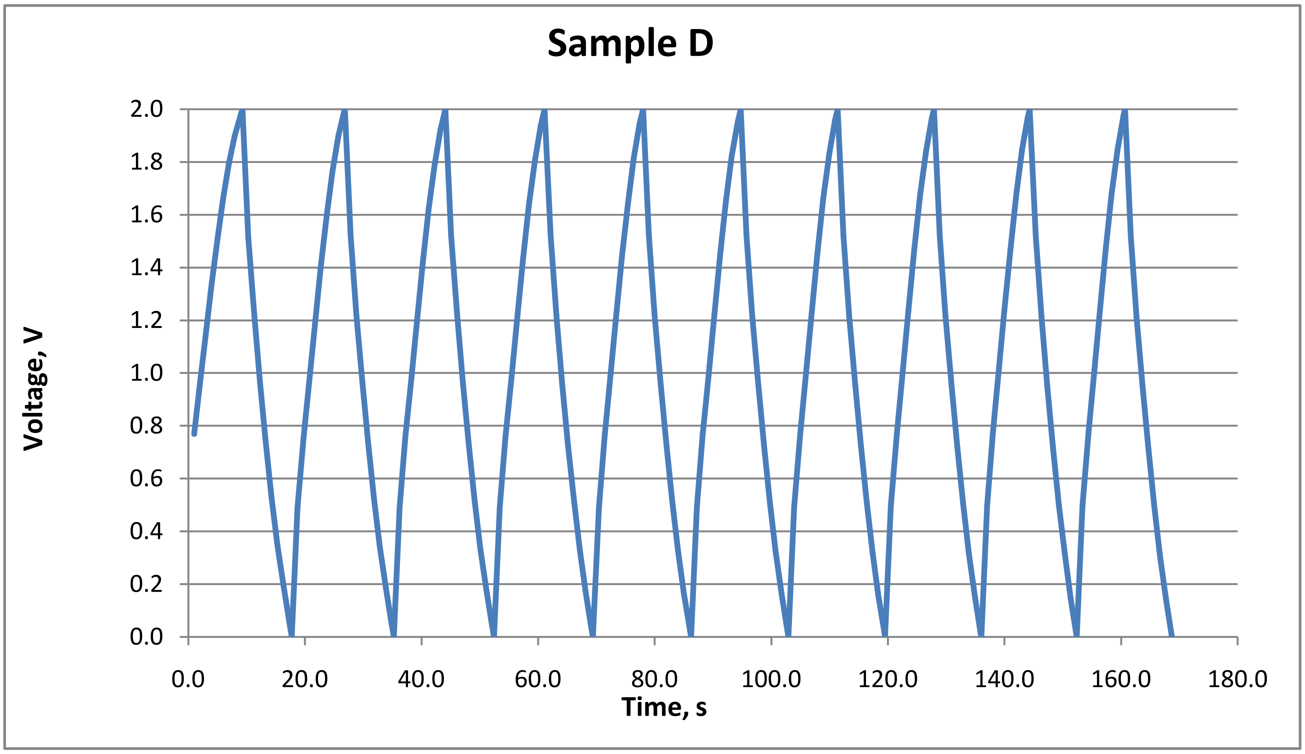


Figure 32: Voltage profile for Sample D at I= 10mA

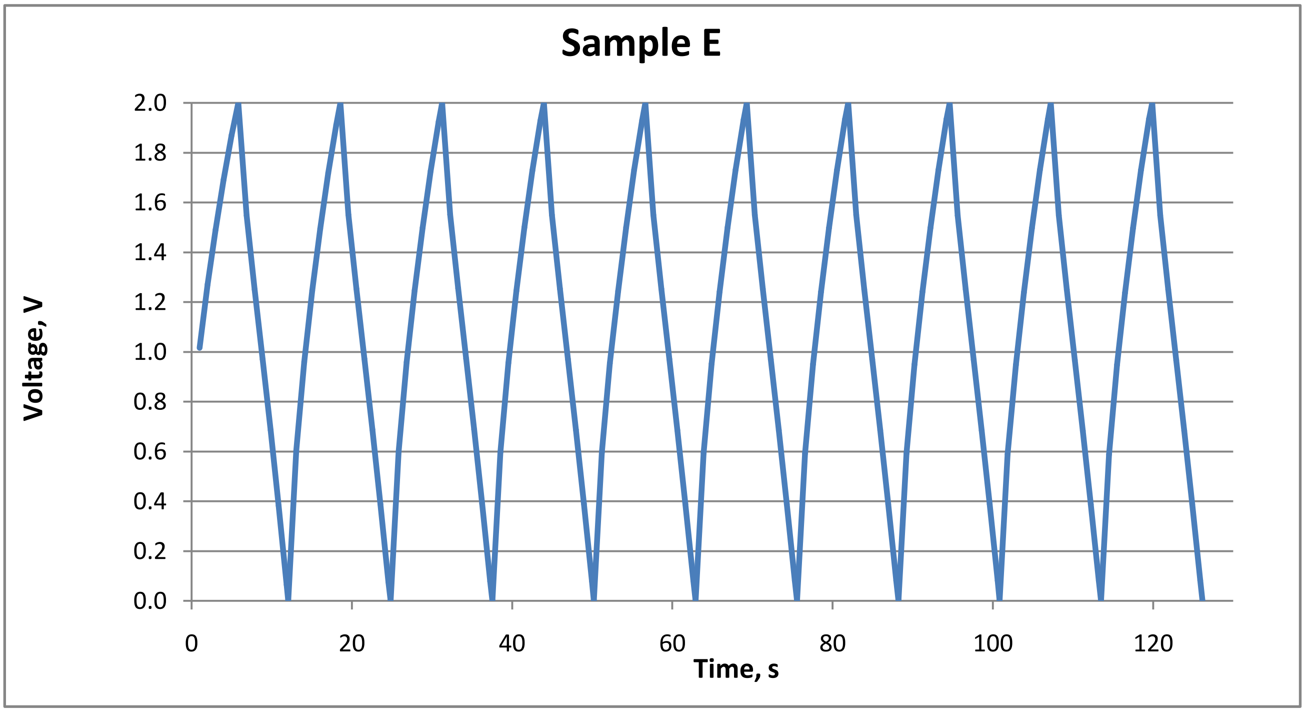


Figure 33: Voltage profile for Sample E at I= 10mA

4.2.1.1 Voltage Profile for I = 50 mA

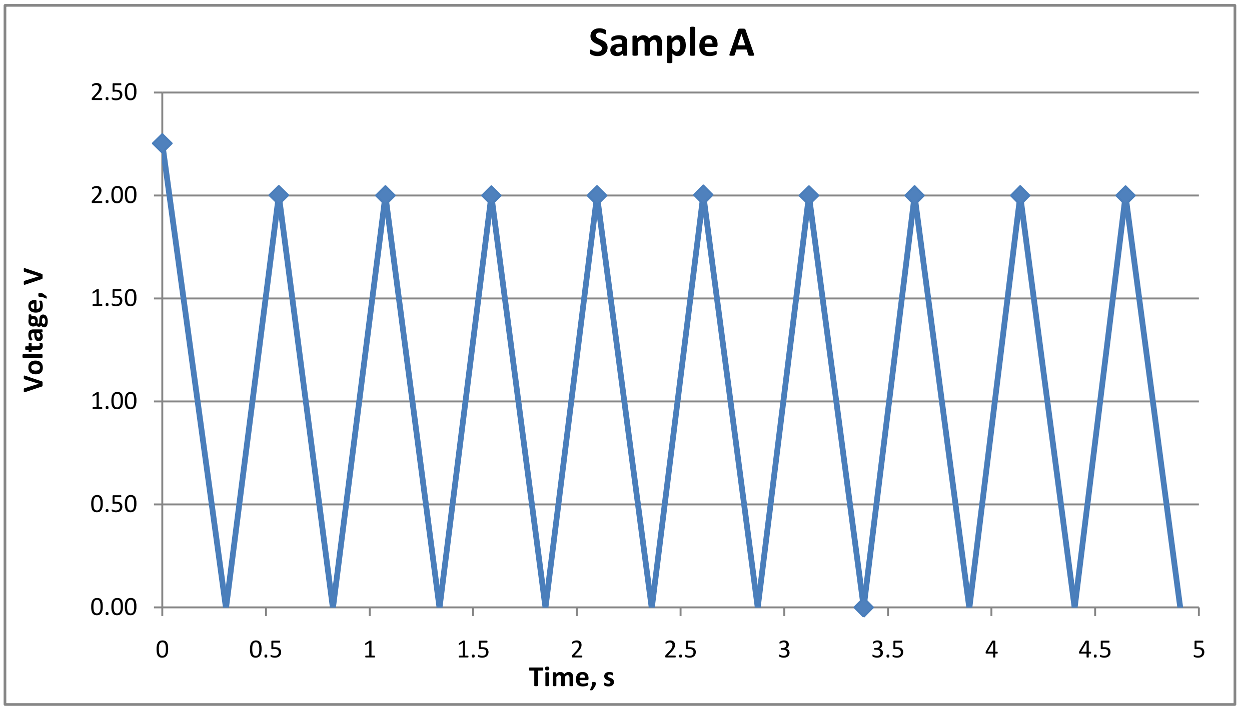


Figure 34: Voltage profile for Sample A at I= 50mA

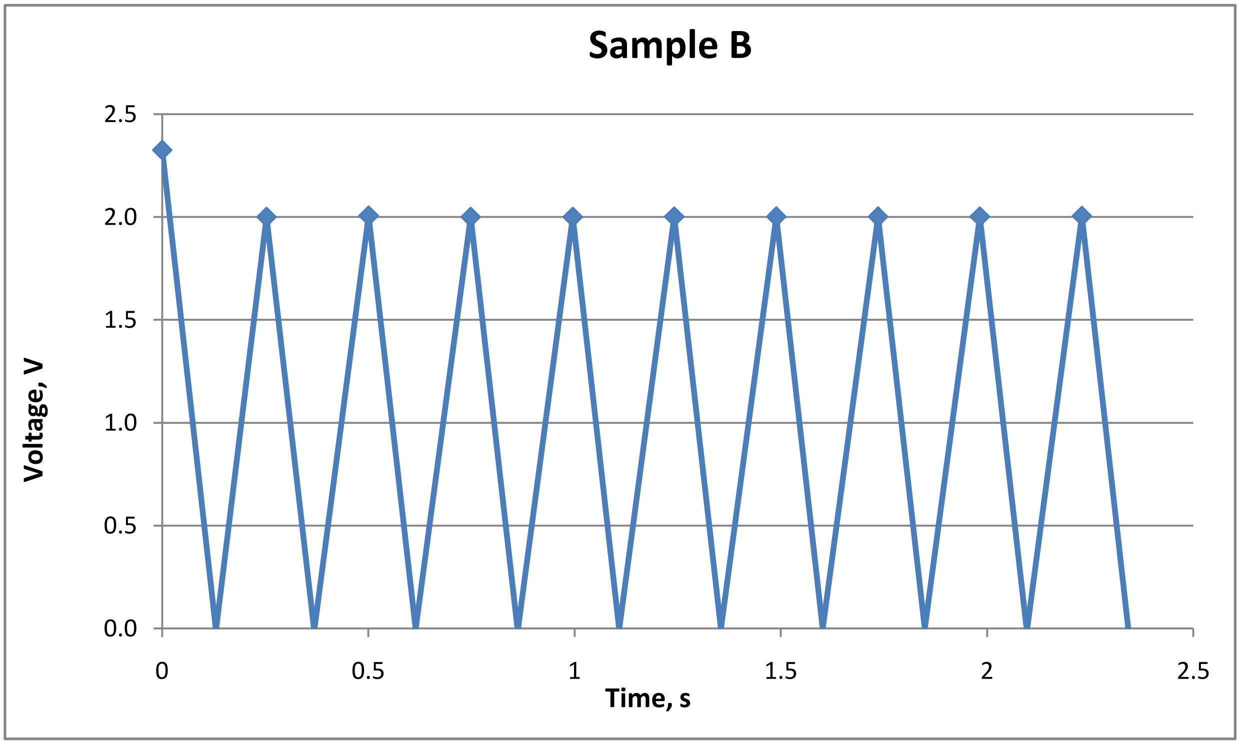


Figure 35: Voltage profile for Sample B at I= 50mA

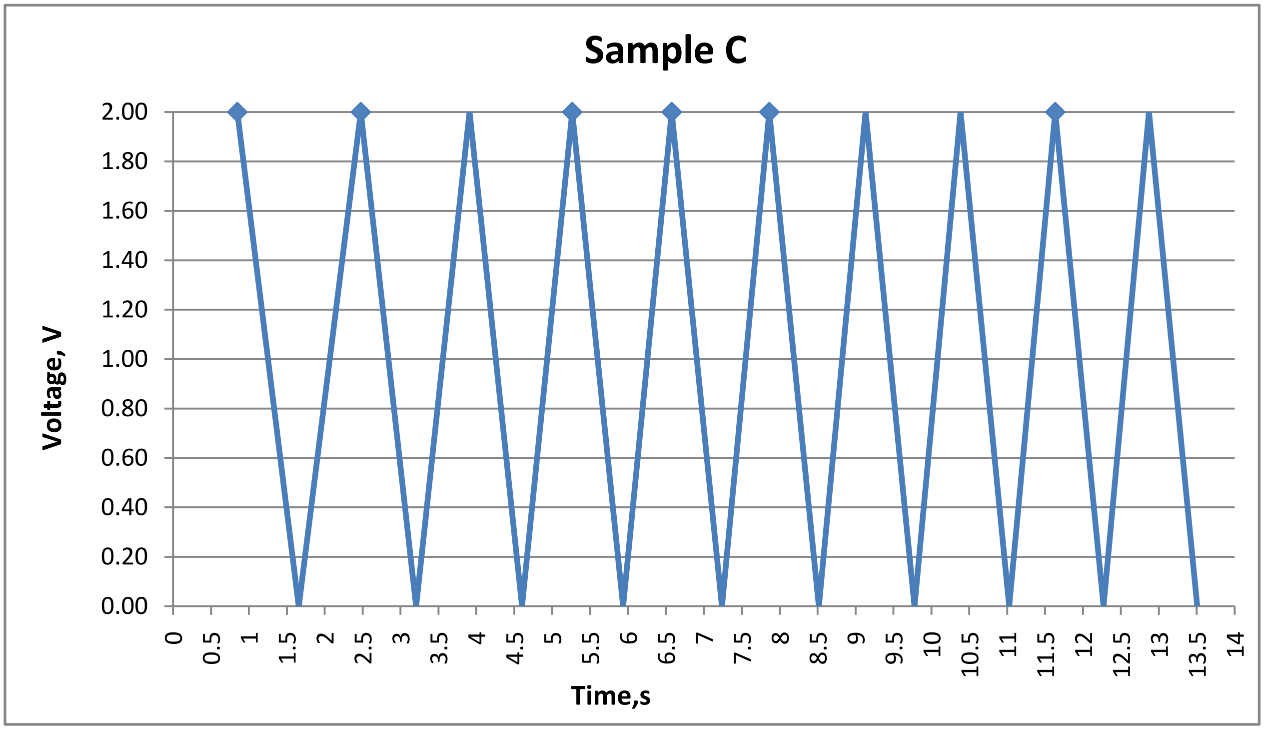


Figure 36: Voltage profile for Sample C at I= 50mA

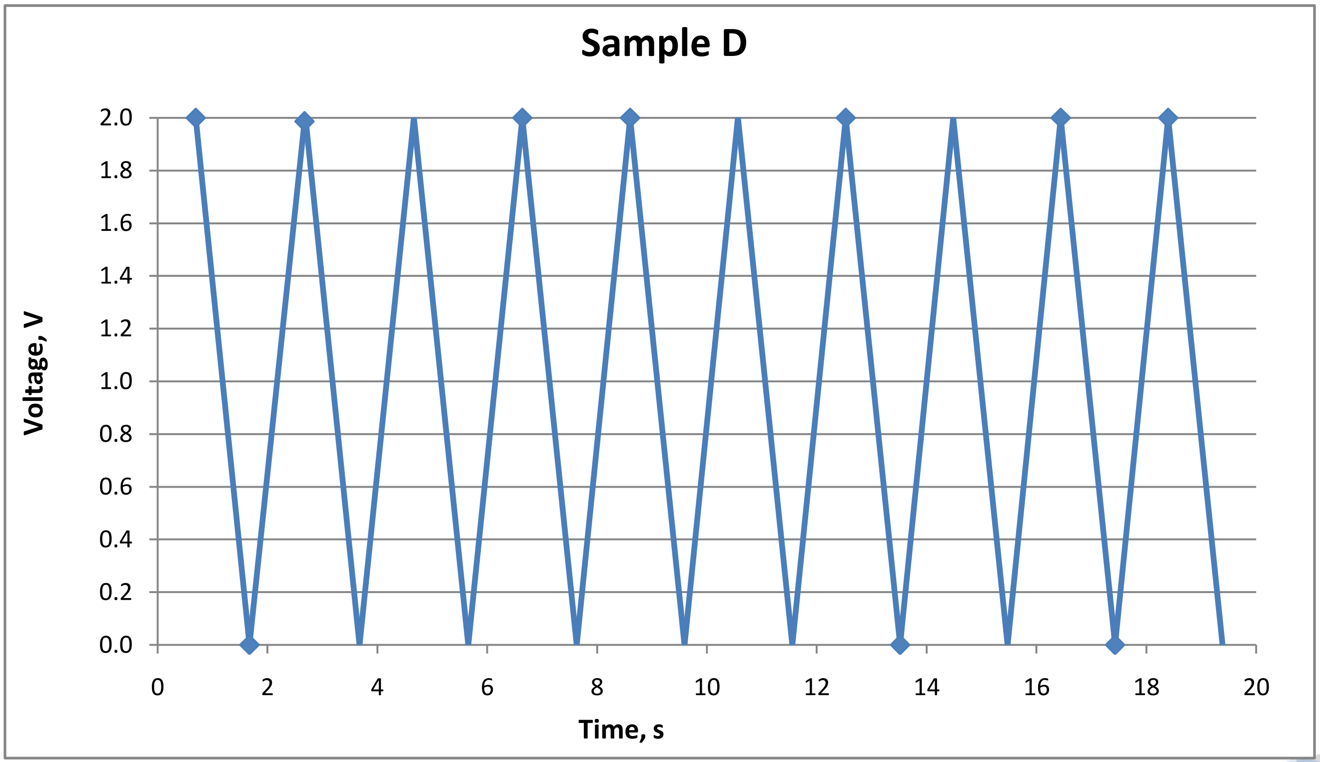


Figure 37: Voltage profile for Sample D at I= 50mA

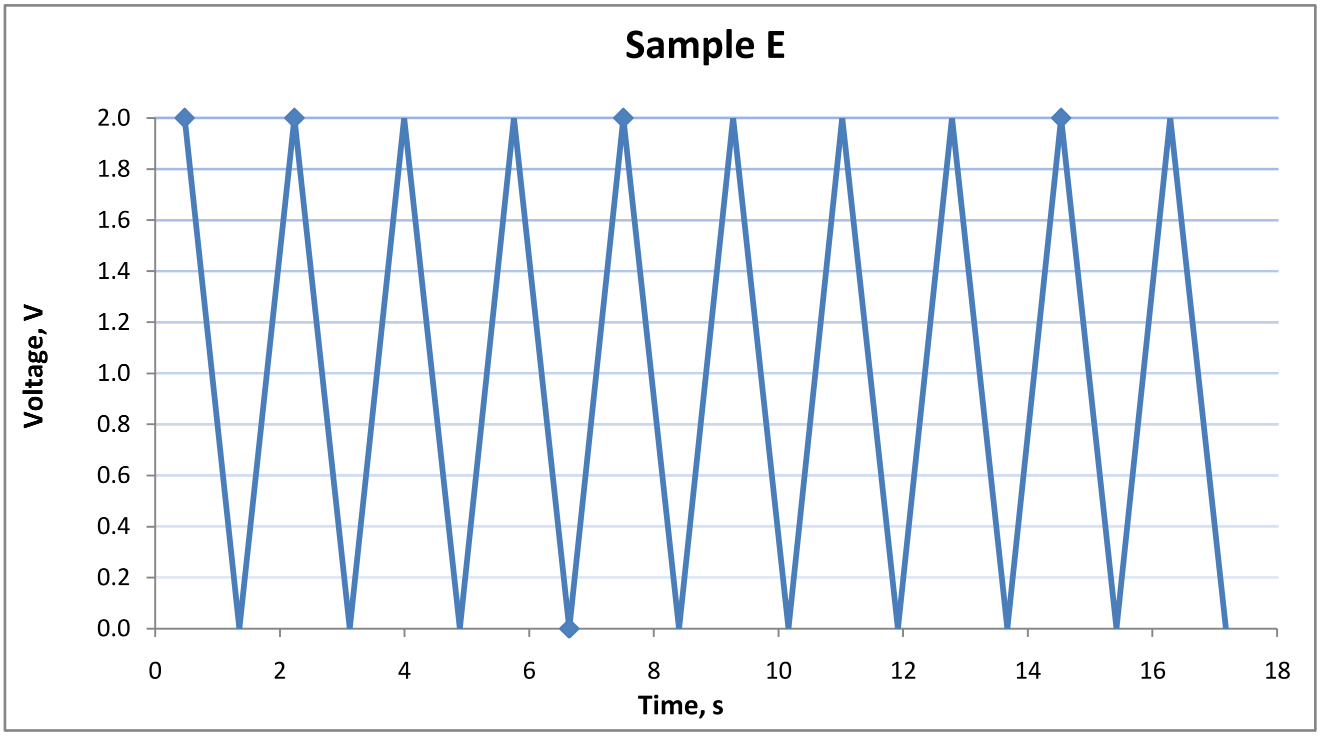


Figure 38: Voltage profile for Sample E at I= 50mA

The graph voltage vs. time is drawn in **Figure 24 -38** to see the time taken for the sample to store charge and discharge ion. From the figure, it is noticed that when the impregnation concentration of ZnCl2 increase, the time taken to finish 10 cycle become shorter. Again the treatment using impregnation of ZnCl2 gives significant effect on time taken to complete 10 cycles than Heating Temperature Treatment.

When a super capacitor is charged, electronic charge accumulates on the electrodes (conductive carbon) and ions (from the electrolyte) of opposite charge approach the electronic charge. This phenomenon is coined "the double layer phenomenon". Between charging and discharging, ions and electrons shift locations. In the charged state a high concentration of ions will be located along the electronically charged carbon surface (electrodes). As the electrons flow through an external discharge circuit, slower moving ions will shift away from the double layer. During Electric Double Layer Capacitor (EDLC) cycling electrons and ions constantly move in the capacitor, yet no chemical reaction occurs. Therefore electrochemical capacitors can undergo millions of charge and discharge cycles.

4.2.2 Specific Discharge Capacity

Discharge capacity for current, I = 5 mA.

	Discharge capacity, mA.h				
No. Cycle	Carbon A	Carbon B	Carbon C	Carbon D	Carbon E
1	0.00425	0.0112222	0.0136111	0.01819444	0.01223611
2	0.003625	0.010125	0.0137611	0.01611111	0.01233333
3	0.003625	0.0096111	0.0137778	0.01583333	0.01234722
4	0.003625	0.0093333	0.0136528	0.01583333	0.01234722
5	0.00365278	0.0091667	0.0135556	0.01555556	0.01236111
6	0.00365278	0.0090417	0.0135278	0.01569444	0.01234722
7	0.00365278	0.0089583	0.0135139	0.01569444	0.012375
8	0.00366667	0.0088889	0.0134722	0.01569444	0.01234722
9	0.00365278	0.0088611	0.0134444	0.01583333	0.01233333
10	0.00366667	0.0088056	0.0134306	0.01569444	0.01236111

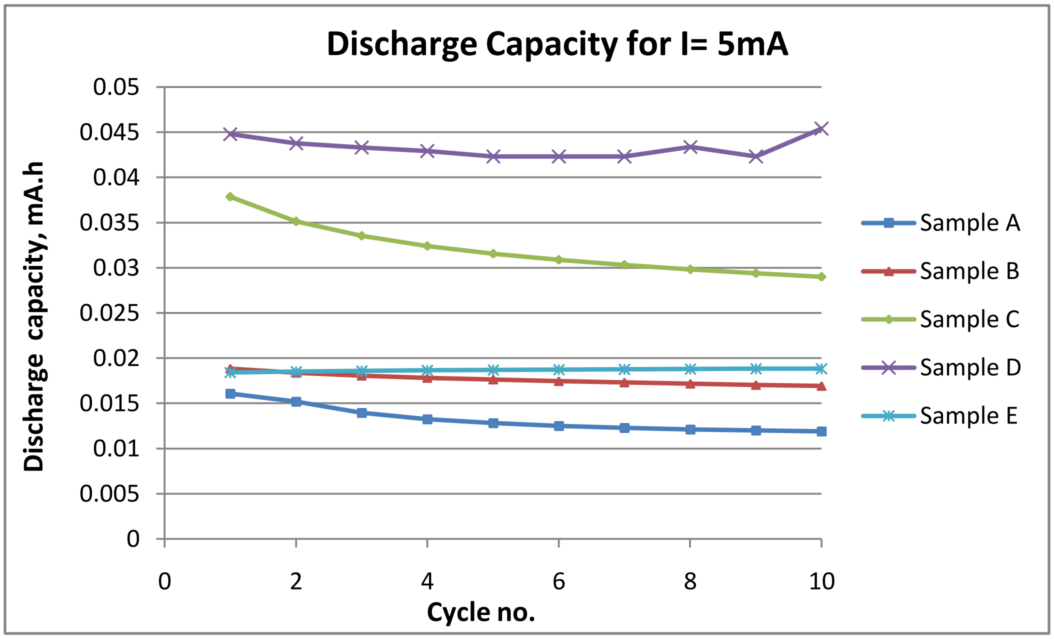


Figure 39: Discharge capacity vs. number of cycle at I=5mA

Discharge capacity for current, I = 10 mA.

	Discharge Capacity, mA.h				
Cycle no.	Sample A	Sample B	Sample C	Sample D	Sample E
1	0.0069222	0.023089	0.0235028	0.0466472	0.017225
2	0.0087111	0.020017	0.0233333	0.0414517	0.0173361
3	0.0085306	0.018722	0.0231583	0.0413681	0.0173667
4	0.0084444	0.017928	0.0230056	0.0416808	0.0173806
5	0.0083833	0.017344	0.0228583	0.0418083	0.0173917
6	0.0083444	0.016894	0.0227278	0.0406808	0.0174
7	0.0083028	0.016517	0.0226056	0.0396808	0.0174056
8	0.0082722	0.016219	0.0224944	0.0391778	0.0174083
9	0.0082472	0.01595	0.0223833	0.0378889	0.0174083
10	0.0082278	0.015614	0.0222889	0.0368083	0.0174167

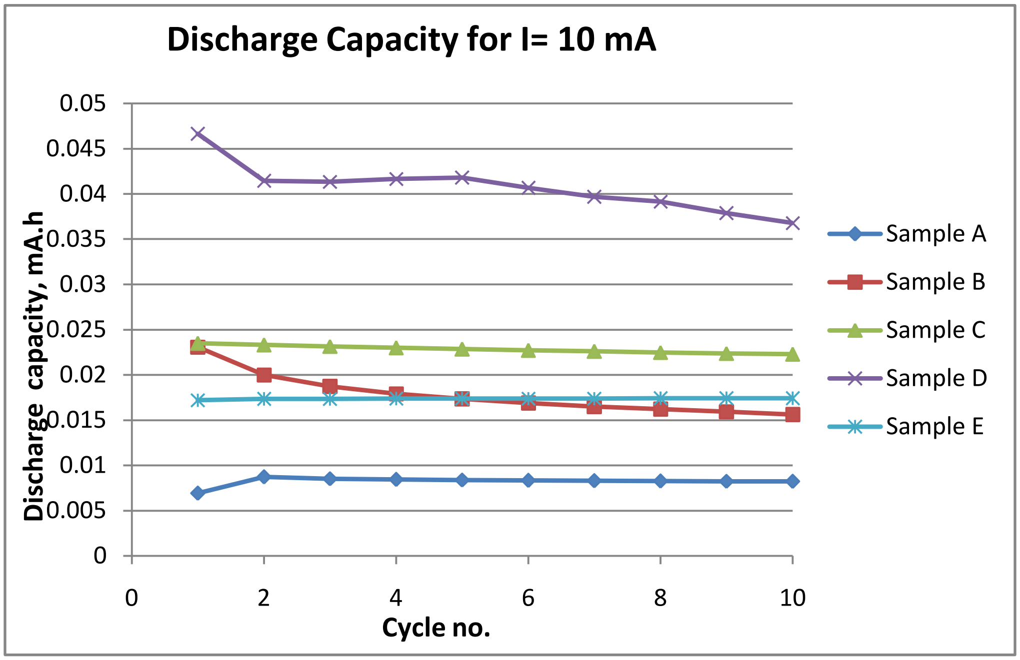


Figure 40: Discharge capacity vs. number of cycle at I=10mA

Discharge capacity for current, I = 50 mA.

	Discharge capacity, mA.h				
No. Cycle	Carbon A	Carbon B	Carbon C	Carbon D	Carbon E
1	0.00425	0.0112222	0.0136111	0.01819444	0.01223611
2	0.003625	0.010125	0.0137611	0.01611111	0.01233333
3	0.003625	0.0096111	0.0137778	0.01583333	0.01234722
4	0.003625	0.0093333	0.0136528	0.01583333	0.01234722
5	0.00365278	0.0091667	0.0135556	0.01555556	0.01236111
6	0.00365278	0.0090417	0.0135278	0.01569444	0.01234722
7	0.00365278	0.0089583	0.0135139	0.01569444	0.012375
8	0.00366667	0.0088889	0.0134722	0.01569444	0.01234722
9	0.00365278	0.0088611	0.0134444	0.01583333	0.01233333
10	0.00366667	0.0088056	0.0134306	0.01569444	0.01236111

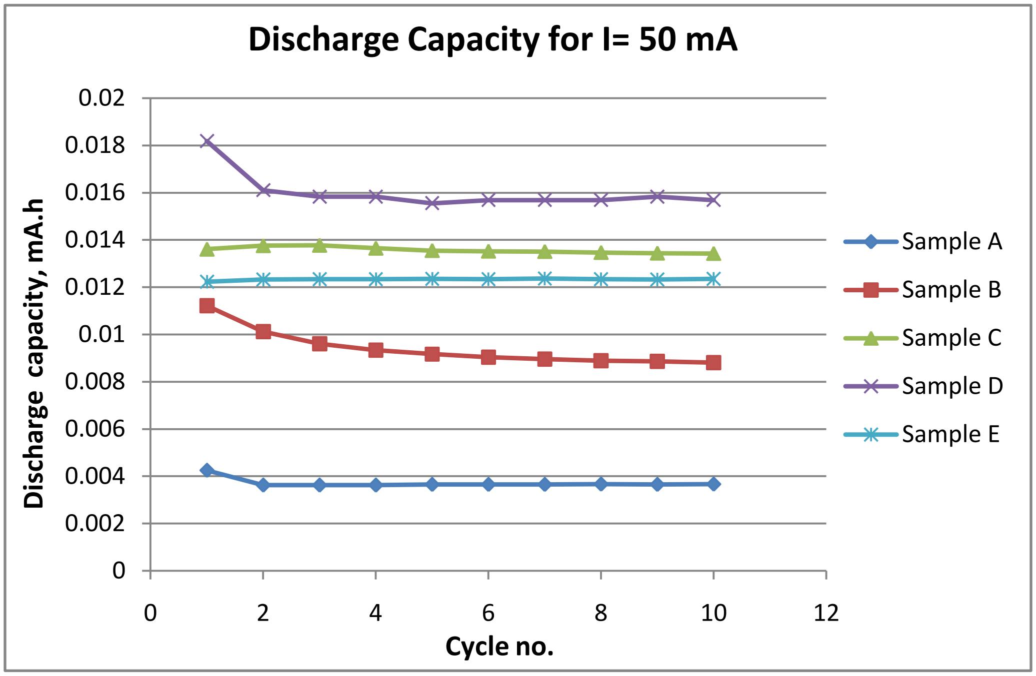


Figure 41: Discharge capacity vs. number of cycle at I=50mA

From the time obtained in voltage profile, discharge capacity of a capacitor could be determined. Discharge capacity is calculated using Coulombs' law:

$$Q = It$$

Where; Q = discharge capacity (mA.h)

I = current applied (mA)

t = discharge time (h)

This equation describes the quantity of charge pass through to the point of conductor at certain time while the current remain the same. Then, the graph discharge capacity vs. cycle no. is plotted to see the variation of discharge capacity for each cycle. All Raw data is provided in **Appendix D**.

Figure 39-41 is the comparison of discharge capacity between each sample. When current increase, it cause the discharge capacity for each sample decrease. So it shows that the structure of this carbon waste could not store and deliver the charge when higher current is induced. The best range current for the treated carbon is 5 mA-10 mA.

In **Figure 39**, with current 5 mA shows the non-treated carbon (Sample A) has the lowest value of discharge capacity. Accordingly, the obtained maximum average discharge capacity is 0.031±0.014 mA.h corresponding to activation parameter that involves $ZnCl_2$ as an activation agent. The higher concentration of $ZnCl_2$, the higher discharge capacity. So it is prove that with chemical activation treatment, it can improve the performance of carbonaceous waste.

Impregnation with $ZnCl_2$ again proved that by introducing $ZnCl_2$, it give significantly effect on carbonaceous waste discharge capacity if compared to HTT. However, at 15% concentration of $ZnCl_2$ (Sample B) the value of discharge capacity is lower than Sample E with HTT. In this case the HTT can increase discharge capacity of carbon better than 15% concentration of $ZnCl_2$ impregnation.

The value of discharge capacity can go further if the concentration of $ZnCl_2$ is increase. So, further research should be conducted in the future to see the maximum improvement can be done by the carbonaceous waste in order to apply it as supercapacitor. Since it is prove that the highest concentration of $ZnCl_2$ give highest discharge capacity, for the next discussion will consider on result with current I = 5 mA, Sample A, D & E.

4.2.3 Capacitor efficiency

Efficiency of capacitor is ability for the capacitor to deliver the charge that had been stored. Some material might capable in receiving the charge when certain amount of current induced, but may fail to deliver the charge when it is needed. So by estimating the efficiency of capacitor, we can judge how long the performance of supercapacitor can work.

Efficiency of capacitor can be calculated using this formula:

$$\zeta = \frac{Discharge\ Capacity}{Charge\ Capacity}$$

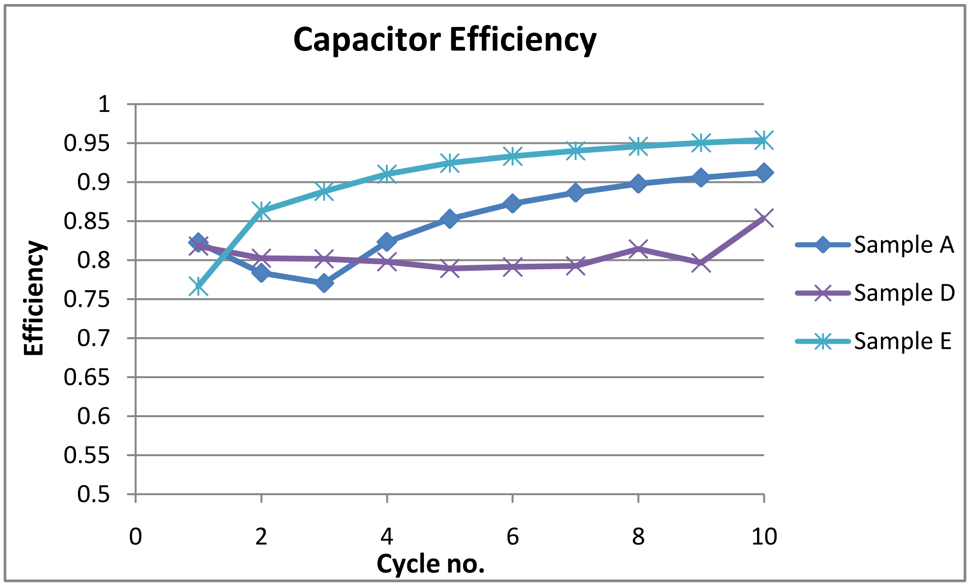


Figure 42: Capacitor efficiency evaluation

When charging and discharging a capacitor, conductor loses energy due to the already existing ESR (Equivalent Series Resistance). This resistance is always present since all of the charge has to be transported to the capacitor plates and, since the materials are not superconducting, it means that there is loss in transport that is converted into heat. We have a loss of energy, which we originally wanted to store or retrieve from the capacitor.

4.2.4 Comparison of Specific Discharge Capacity (SDC)

Capacity can also be known as charge delivered in certain period of time when there is voltage potential difference. Specific capacity is capacity of capacitor with respect to weight of active material in the electrode. To calculate specific discharge capacity (mA.h/g), the following formula is used:

$$SDC = \frac{Discharge\ capacity}{Weight\ of\ activated\ carbon}$$

Where;

Weight of activated carbon is weight of carbonaceous waste used as electrode = 20 mg.

The experiment is carried out to test commercial carbon performance with the current 5 mA and voltage limit 2V. Four cycles of charge –discharge process are evaluated. The result of the experiment is used to compare the specific discharge capacity with the Sample A, D and E. The comparison can give the first view to the sample whether the properties of the sample can compete with the commercial one. The result is shown in **Table 10** and illustrated in **Figure 42**.

Table 10: Comparison of specific discharge capacity

	Specific Discharge Capacity, mA.h/g			
Cycle no.	Commercial Carbon (mA.h/g)	Sample A	Sample D	Sample E
1	1.952	0.804236	2.240833	0.922083
2	2.006	0.760139	2.188583	0.927292
3	1.975	0.697917	2.165833	0.930833
4	1.947	0.663333	2.145833	0.933542

Commercial carbon has undergone pre-treatment processes beforehand. This pre-treatment process has change the structure of the carbon; they posses higher surface-area, smaller carbon size and more porous. So, the commercial carbon is comparable with the sample.

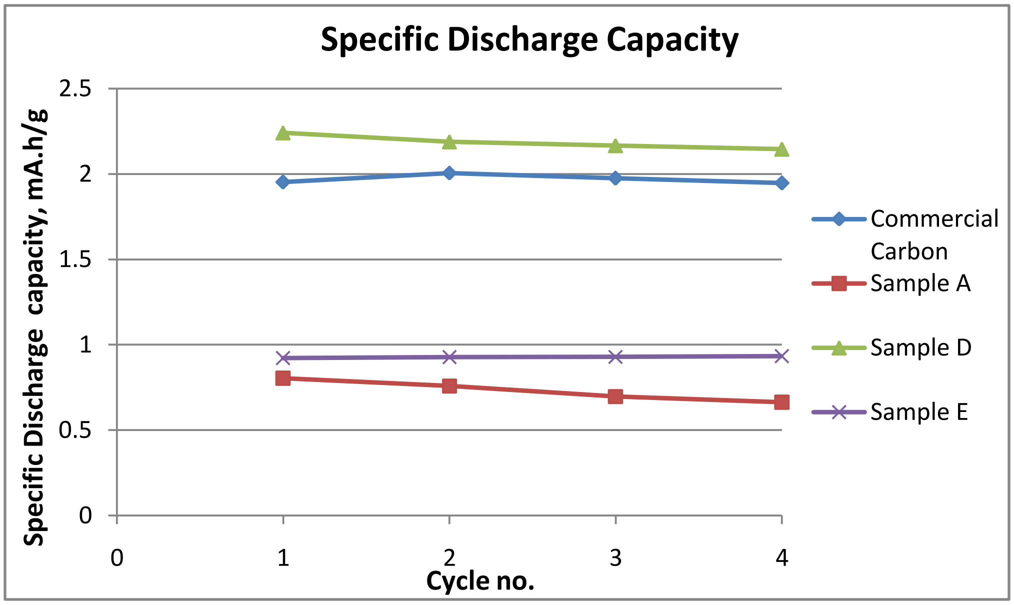


Figure 43: Comparison of Discharge Capacity between commercial carbon and Carbonaceous Waste.

Figure 43 illustrate the comparison of 4 type's carbon where Sample D & E are pretreated carbon with chemical and physical activation. Sample A is non-treated carbon. From the figure, non-treated carbon only can store small charge. After the treatment, the performance of carbonaceous waste is increase with high specific discharge capacity and able to beat commercial carbon. It can be concluded that, carbonaceous waste has high potential to work as electrode supercapacitor and this ability can be increased if the experiment continues with the higher concentration of $ZnCl2$.

CHAPTER 5: CONCLUSION & RECOMMENDATION

This project presents performances of carbonaceous waste as supercapacitor. From the XRD results, structure of carbonaceous waste used in the study can be determined. The structure lies between high crystalline graphite and amorphous carbon. It shows that carbonaceous waste used in this study is low-crystalline graphite, not petroleum coke.

The two x-ray characterization methods, wavelength dispersive scanning electron microscope and x-ray diffraction, each provide limited information about carbonaceous waste. Unfortunately, neither method provides definitive information about the structural and/or bonding roles of particles in the activated carbon.

After evaluating the electrochemical capacitor performance of the carbonaceous waste, the results shows that this specific material is able to hold charge and deliver charge; thus proves that this material is capable in acting as electrode in capacitor. The average discharge capacity of the carbonaceous waste is 2.185 mA.h/g with 85% efficiency. This value is higher than commercial carbon than used as supercapacitor. Thus, it is prove that the carbonaceous waste has high potential to become supercapacitor.

Since, this is carbonaceous waste; a lot of improvement on treatment of this waste should be conducted. There are a lot of factors that may effecting on capacitance of this carbonaceous waste such as temperature of activation, flow rate of activating gas, the duration of activation etc. the treatment should consider all in factors in order to get highest optimization of this carbonaceous waste. The improvement can be made for further study by:

a. Using both treatment physical and chemical activation.
b. Using voltmetry to investigate electrochemical redox reaction in carbonaceous waste.
c. Performing BET to provide surface area data.
d. Using various results of characterization techniques such as pore size distribution and FTIR spectroscopy.

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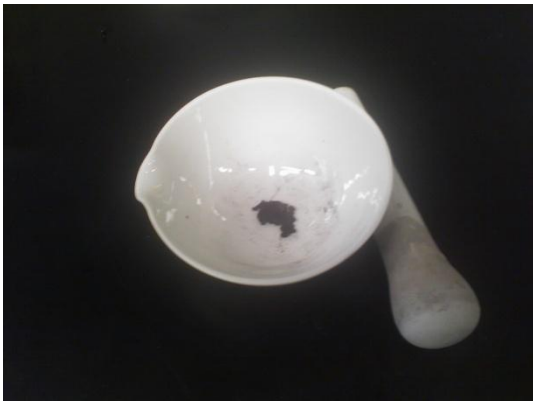
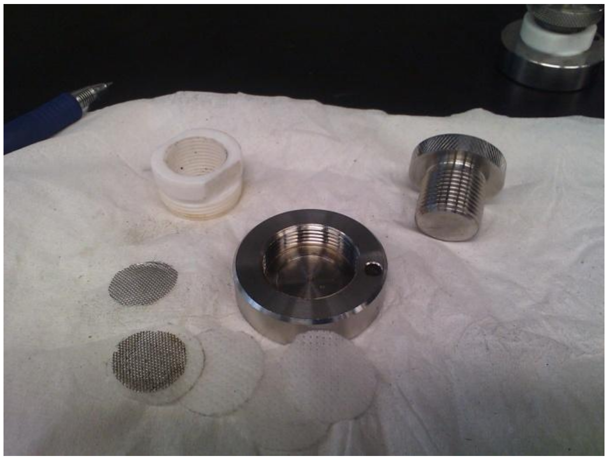
APPENDICES

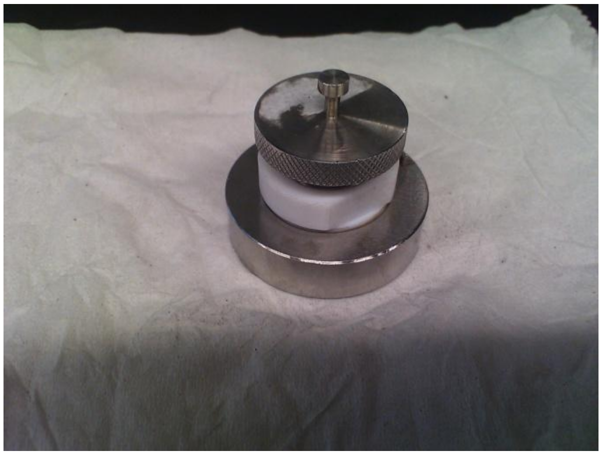
APPENDIX A: METHODOLOGY

1. Binder and carbon is well mixed:

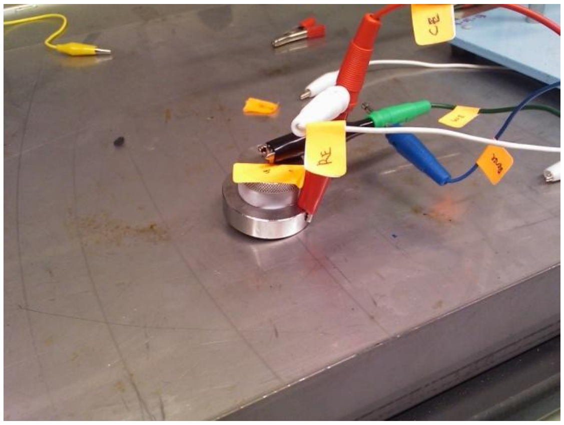


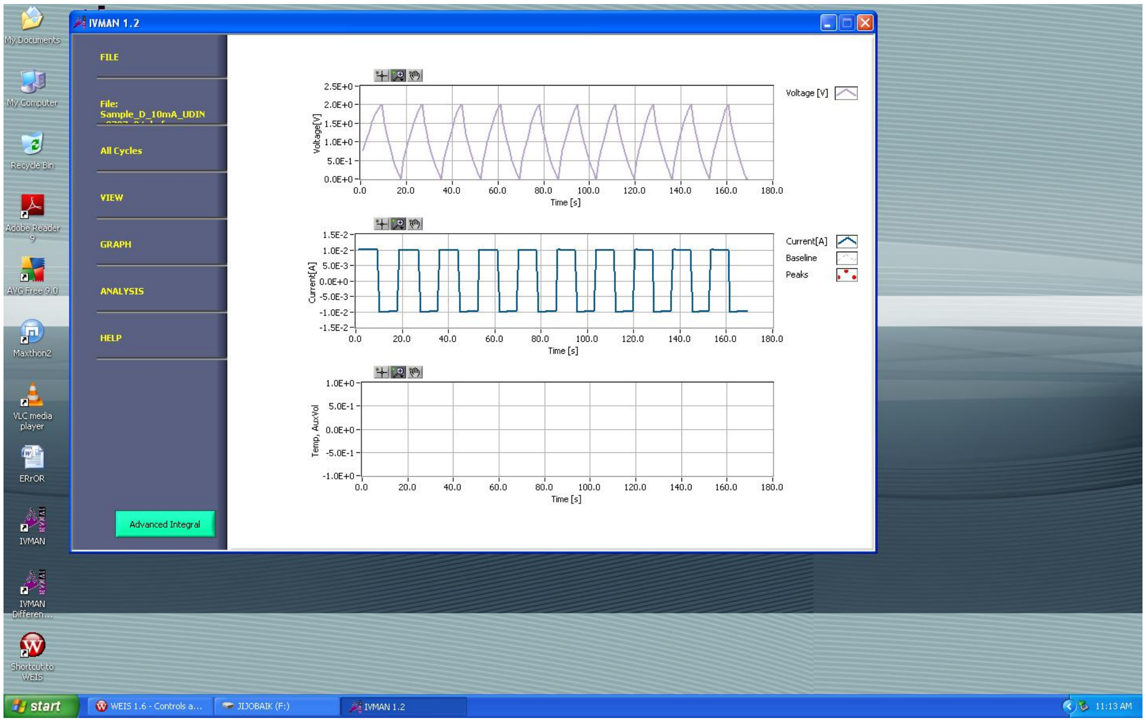
2. Test cell component, configuration with complete installation





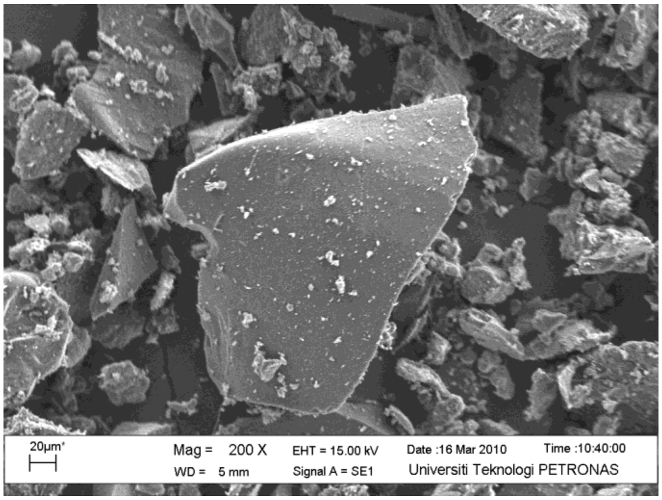
3. Evaluate the performance with WEIS

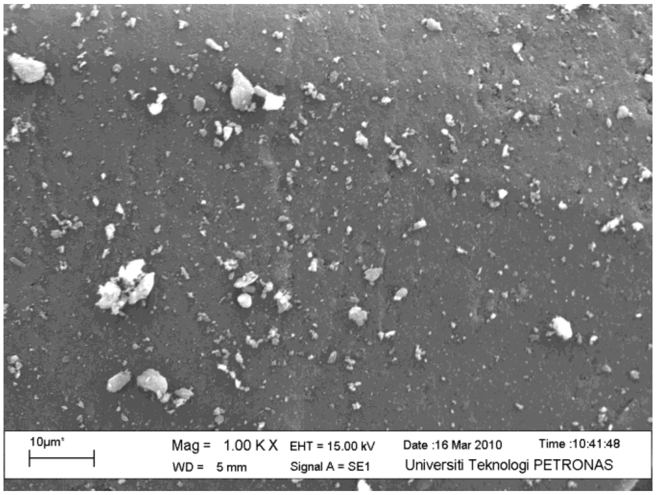


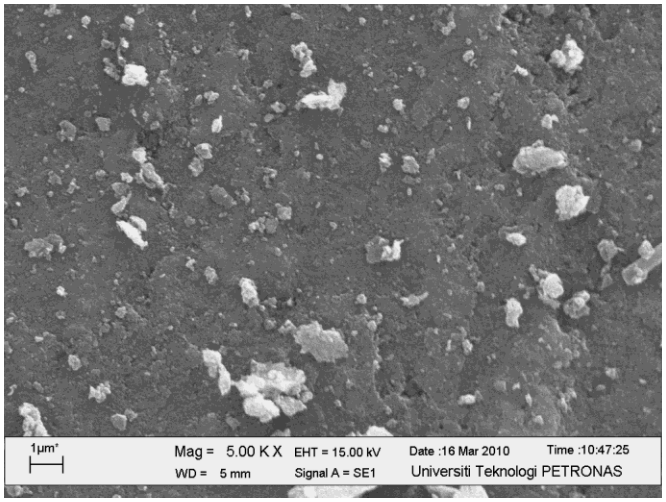


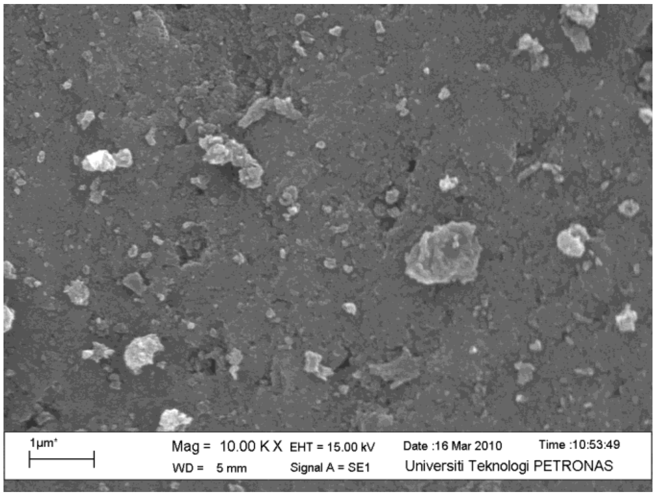
APPENDIX B: SCANNING ELECTRON MICROGRAPH

Sample A

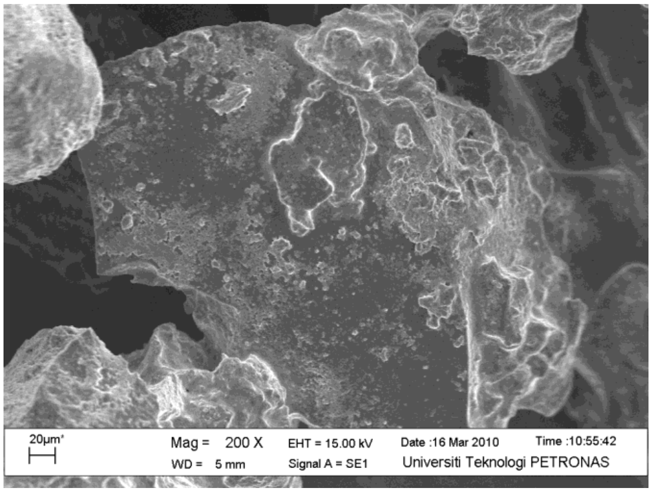








Sample B


20µm*
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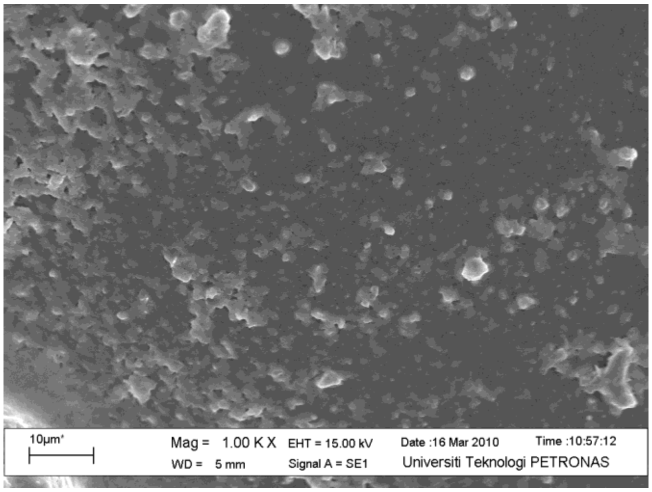
WD = 5 mm
EHT = 15.00 kV
Signal A = SE1
Date :16 Mar 2010
Time :10:55:42
Universiti Teknologi PETRONAS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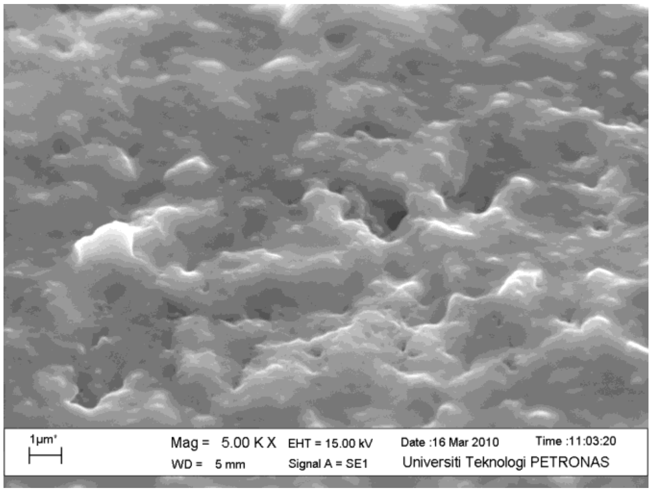
Universiti Teknologi PETRONAS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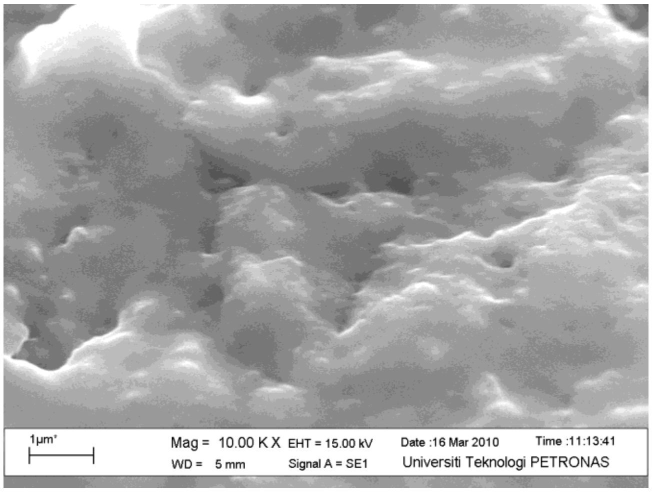
Universiti Teknologi PETRONAS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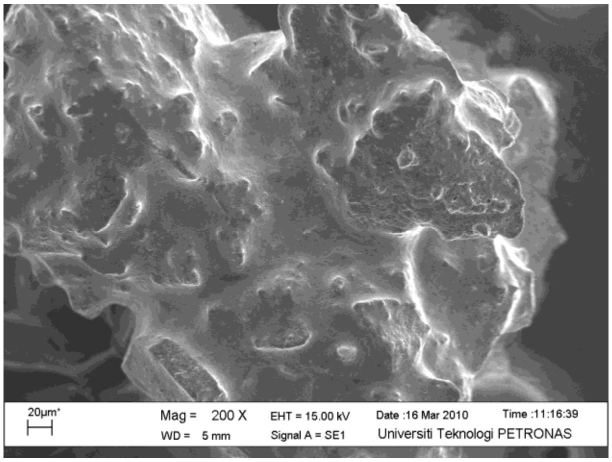
Signal A = SE1
Date :16 Mar 2010
Time :11:13:41
Universiti Teknologi PETRONAS

Sample C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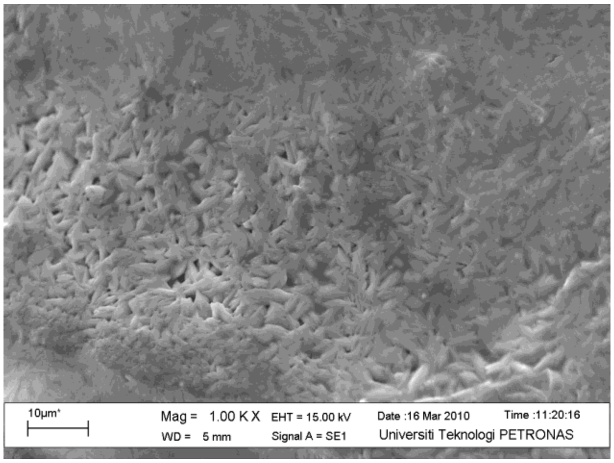
Universiti Teknologi PETRONAS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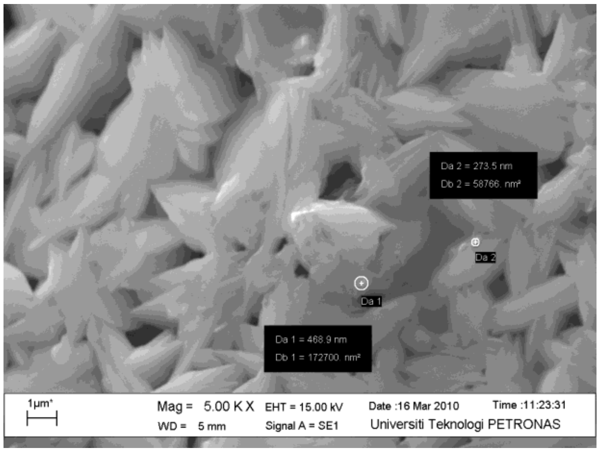
Signal A = SE1
Date :16 Mar 2010
Time :11:20:16
Universiti Teknologi PETRONAS


Da 2 = 273.5 nm
Db 2 = 58766. nm²
Da 2
Da 1
Da 1 = 468.9 nm
Db 1 = 172700. nm²
1µm*
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WD = 5 mm
EHT = 15.00 kV
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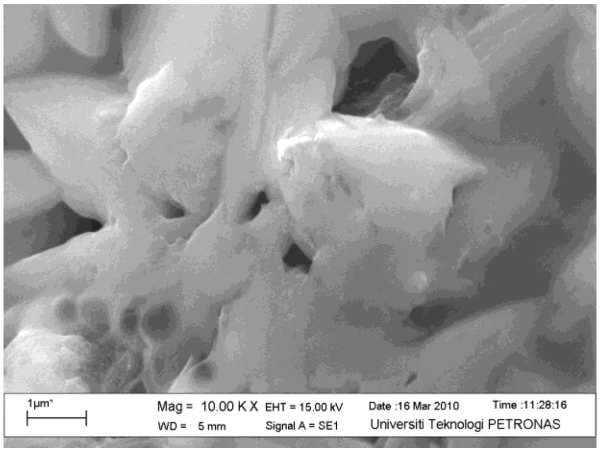
Time :11:23:31
Universiti Teknologi PETRONAS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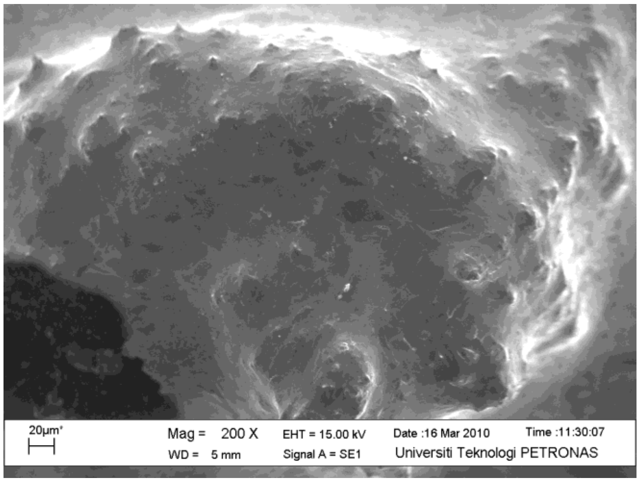
Universiti Teknologi PETRONAS

Sample D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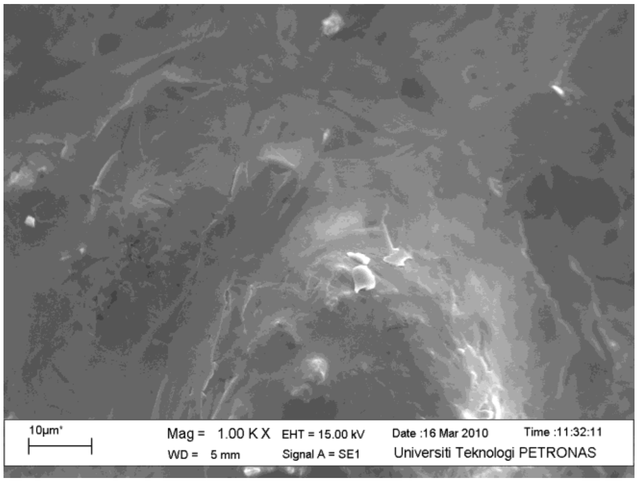
Universiti Teknologi PETRONAS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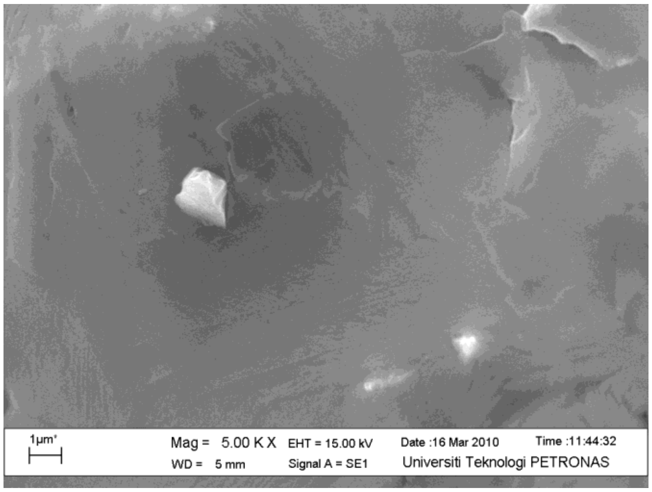
Time :11:32:11
Universiti Teknologi PETRONAS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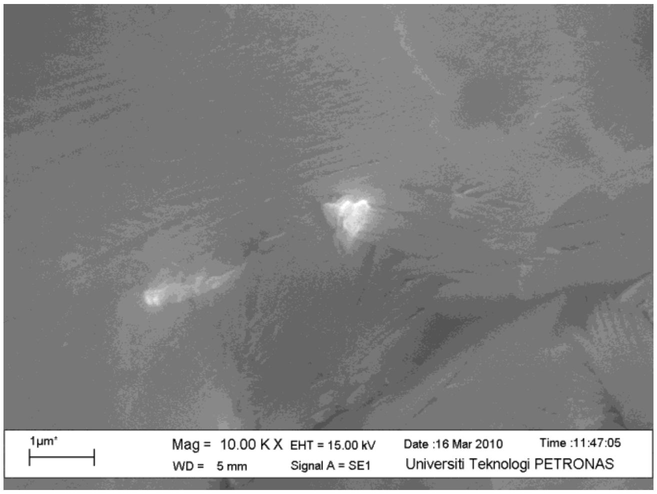
Universiti Teknologi PETRONAS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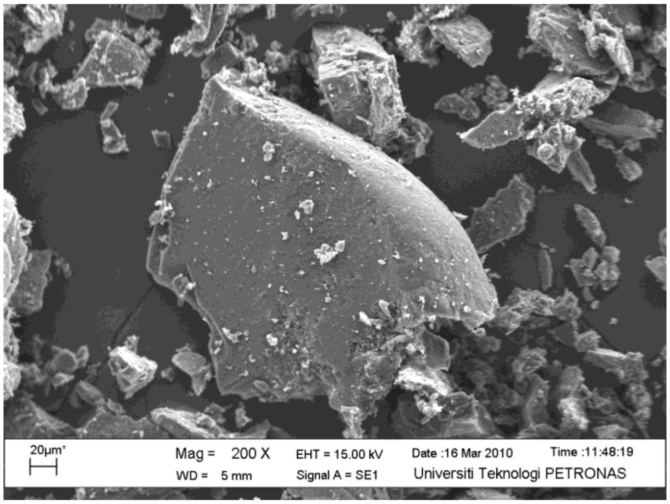
Universiti Teknologi PETRONAS

Sample E


20µm*
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WD = 5 mm
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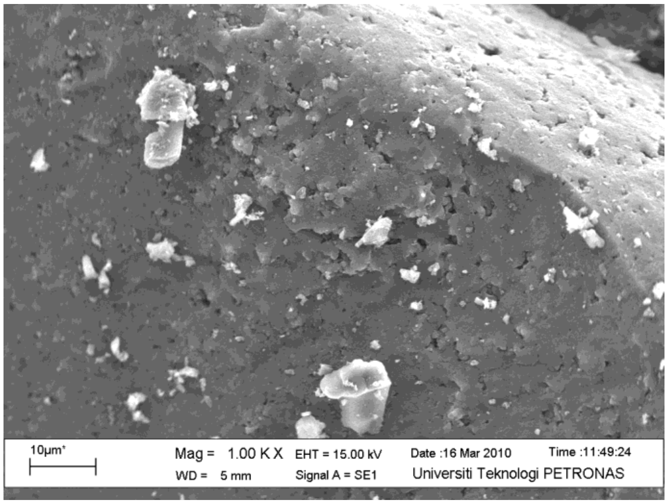
Date :16 Mar 2010
Time :11:48:19
Universiti Teknologi PETRONAS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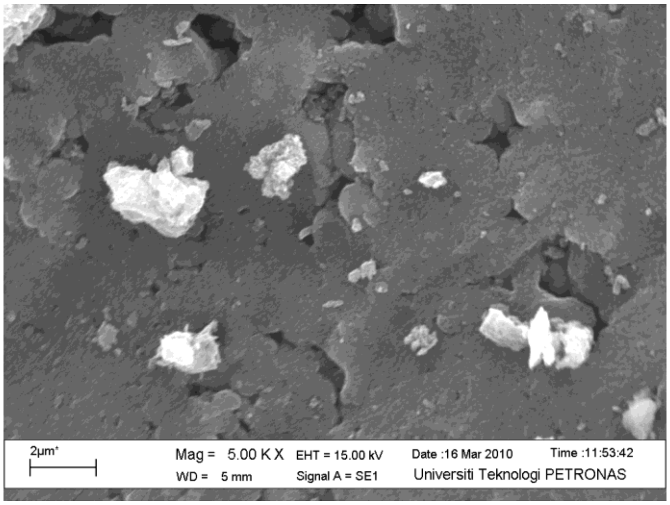
Universiti Teknologi PETRONAS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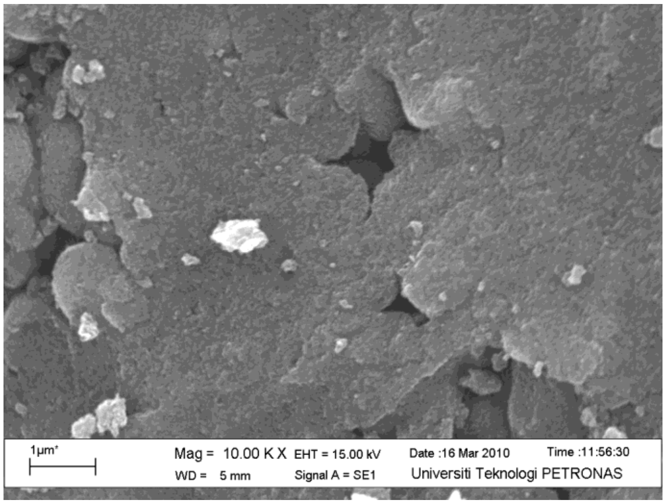
Universiti Teknologi PETRONAS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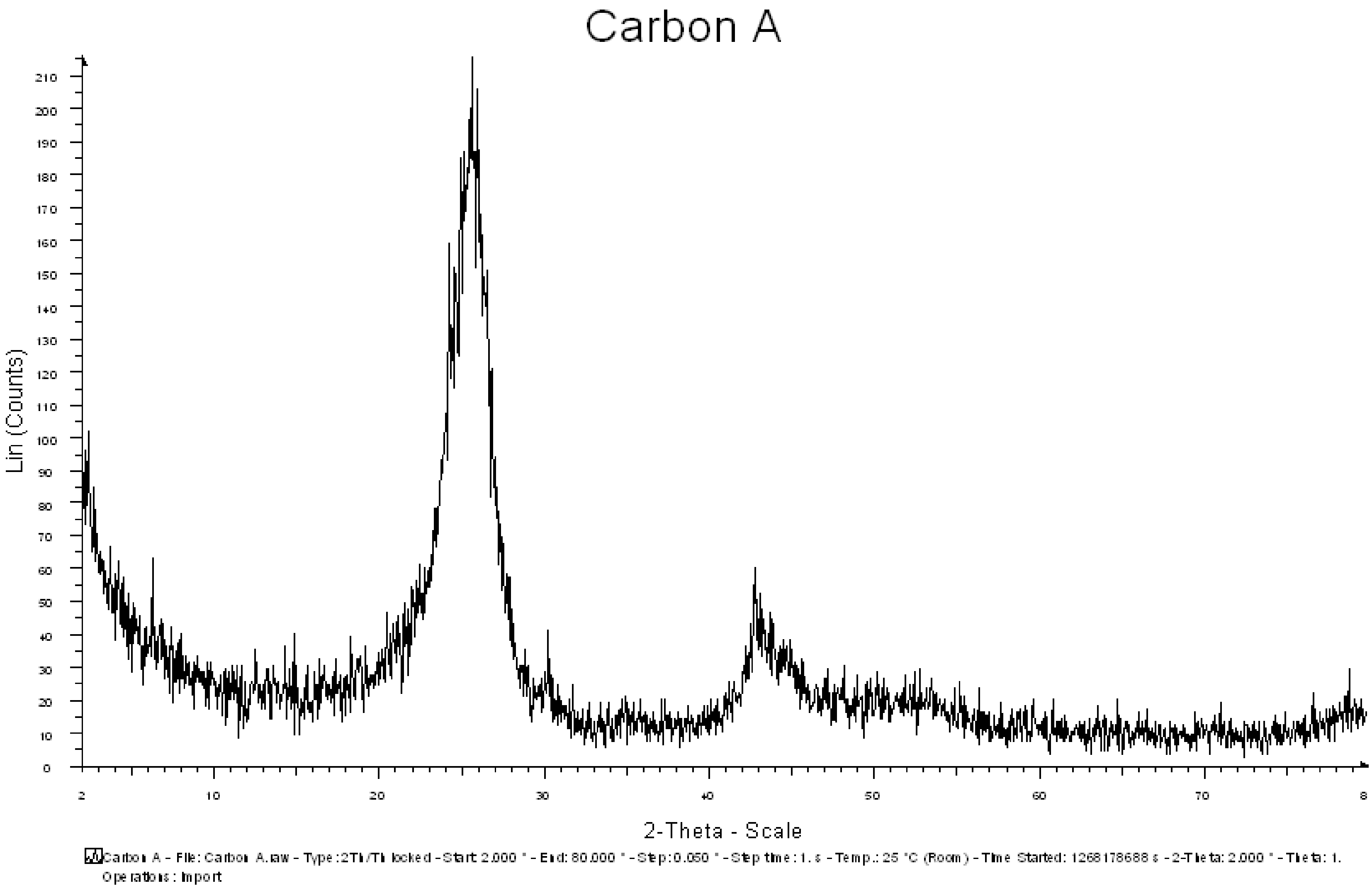
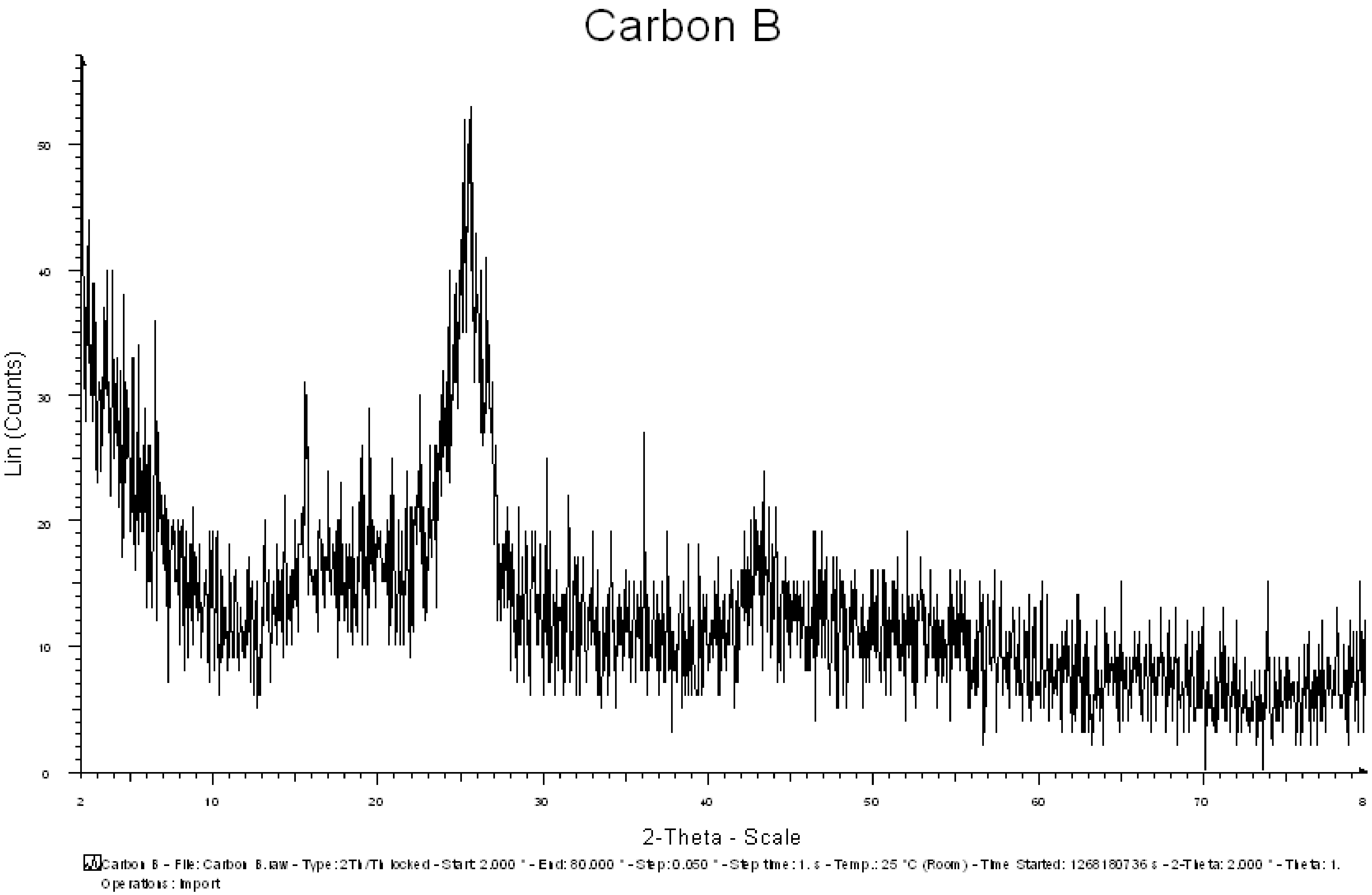
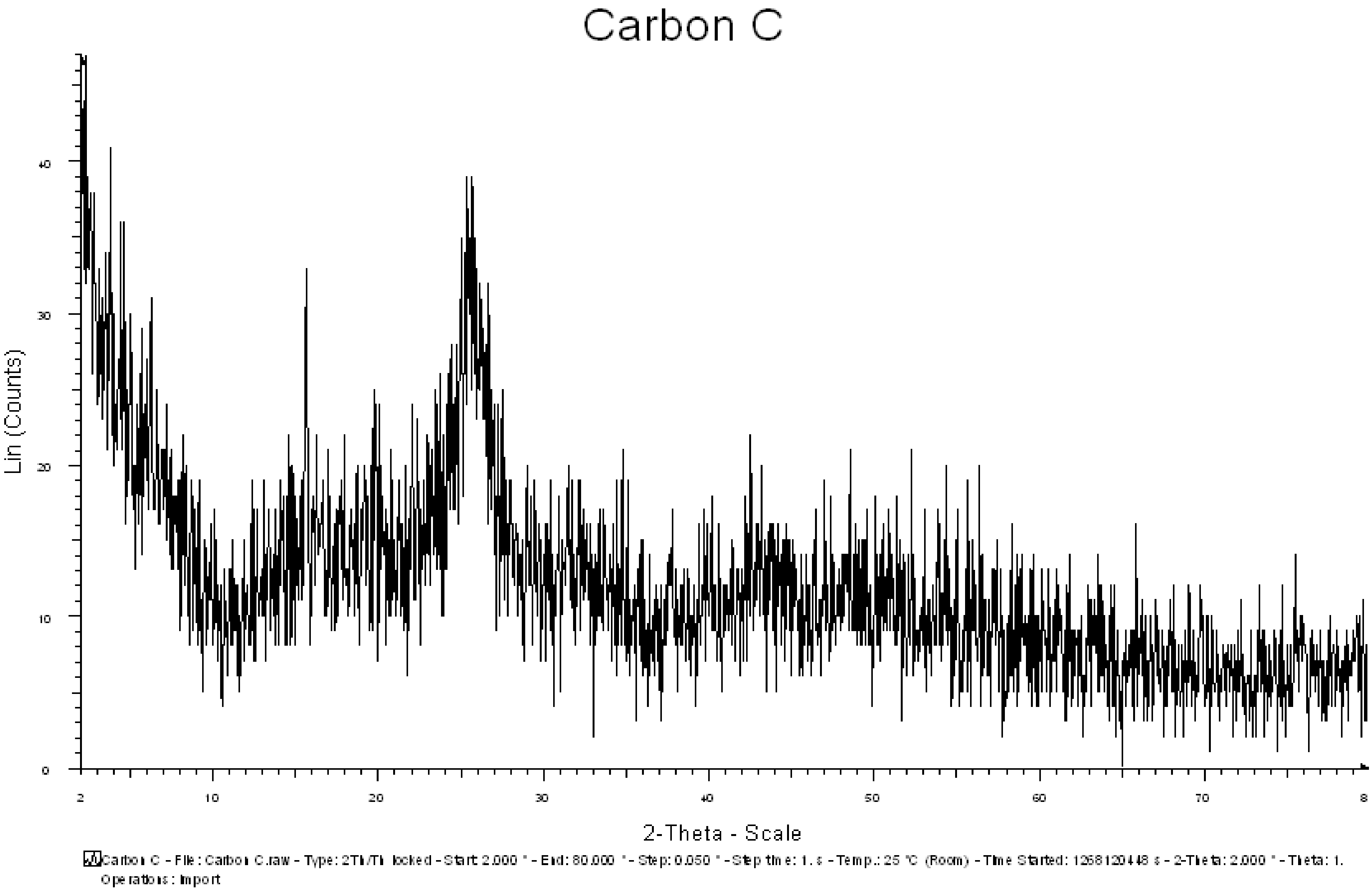
Universiti Teknologi PETRONAS

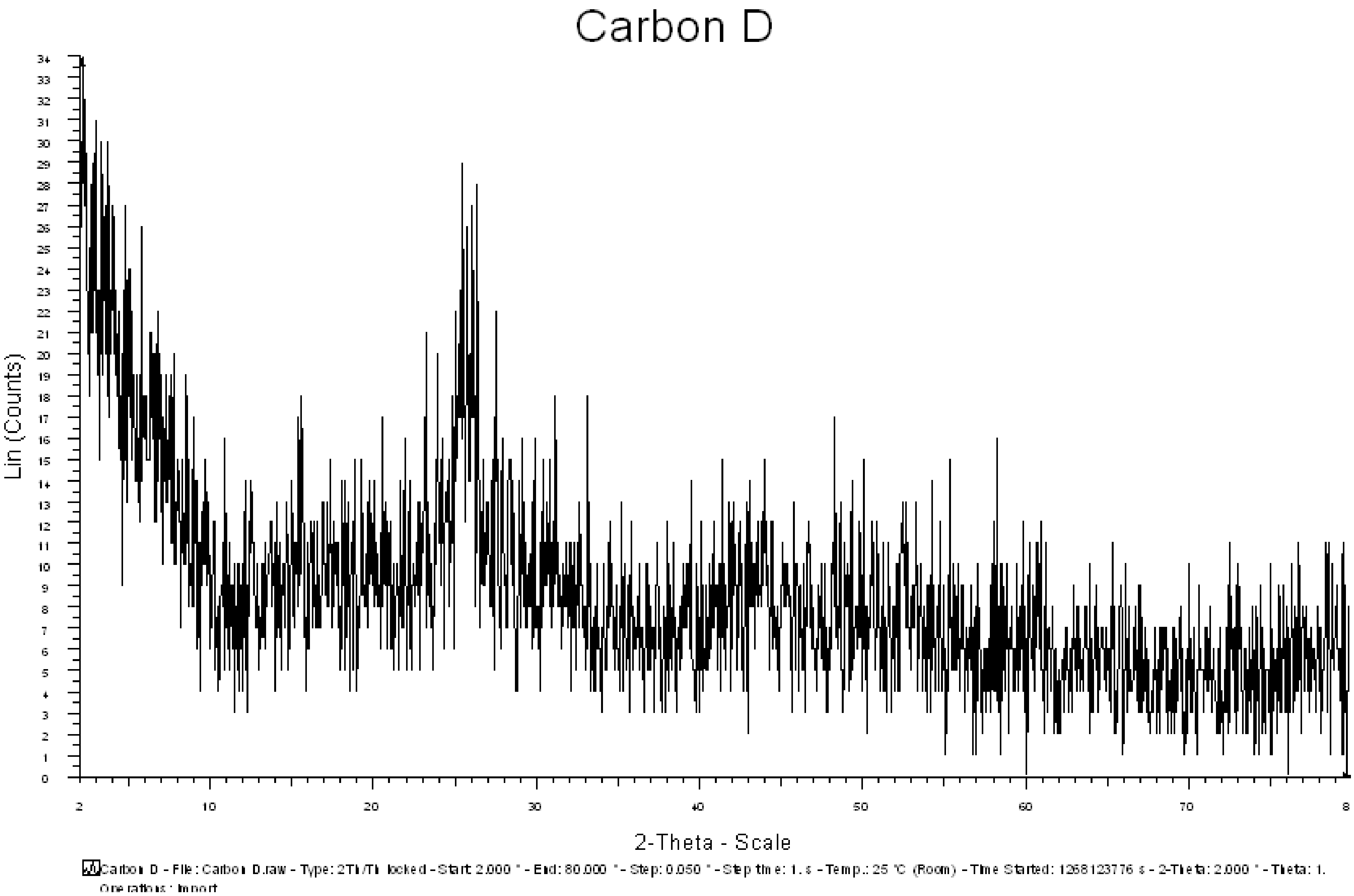
APPENDIX C: XRD – RAW DATA

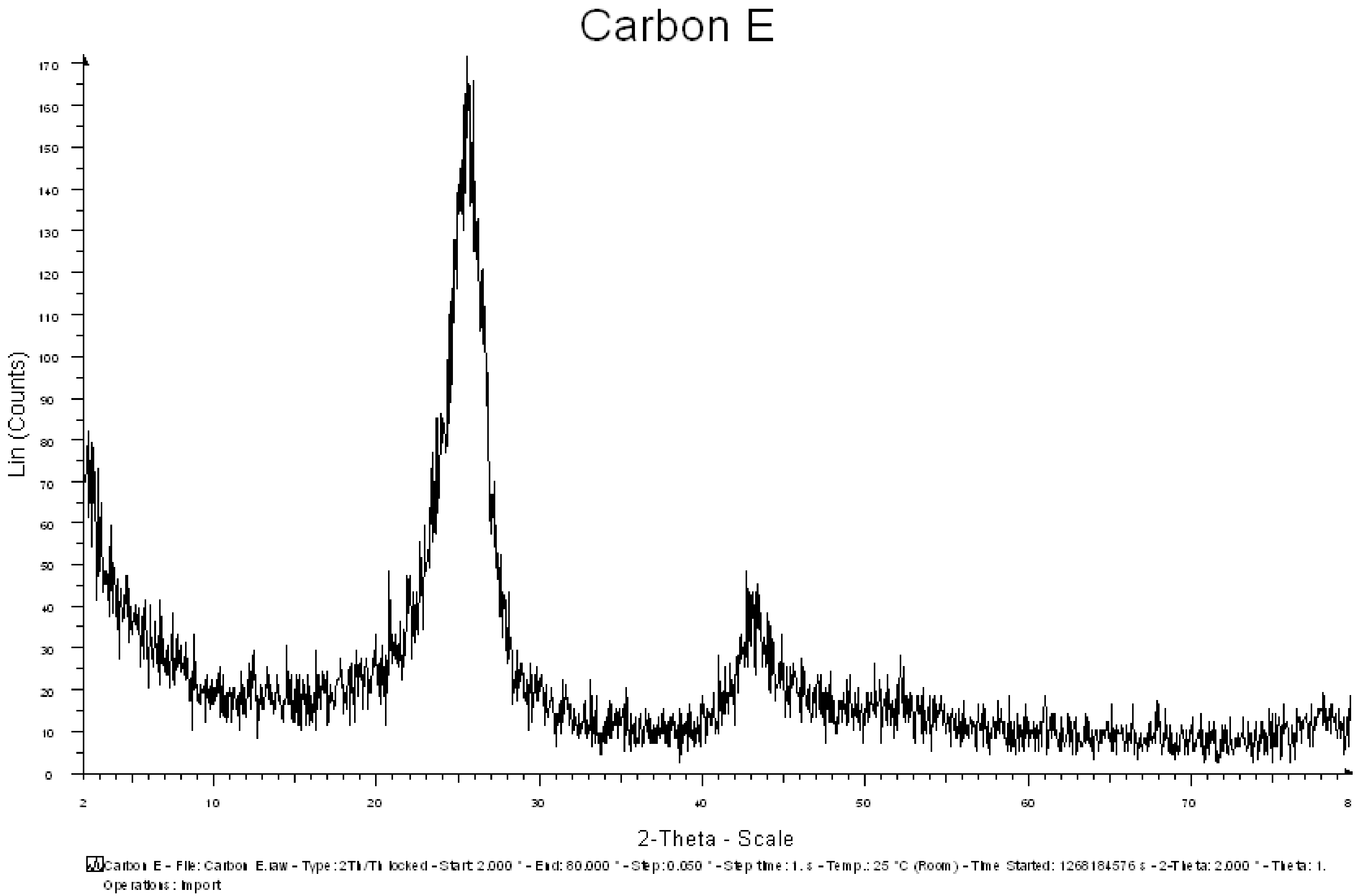

Carbon A

Carbon A - File: Carbon A.raw - Type: 2Th/Th locked - Start: 2.000 ° - End: 80.000 ° - Step: 0.050 ° - Step time: 1. s - Temp.: 25 °C (Room) - Time Started: 1268178688 s - 2-Theta: 2.000 ° - Theta: 1.
Operations: Import


Carbon B
Lin (Counts)
0
10
20
30
40
50
2
10
20
30
40
50
60
70
8
2-Theta - Scale
Carbon B - File: Carbon B.raw - Type: 2Th/Th locked - Start: 2.000 ° - End: 80.000 ° - Step: 0.050 ° - Step time: 1. s - Temp.: 25 °C (Room) - Time Started: 1268180736 s - 2-Theta: 2.000 ° - Theta: 1.
Operations: Import


Carbon C
Lin (Counts)
0
10
20
30
40
2
10
20
30
40
50
60
70
8
2-Theta - Scale
Carbon C - File: Carbon C.raw - Type: 2Th/Th locked - Start: 2.000 ° - End: 80.000 ° - Step: 0.050 ° - Step time: 1. s - Temp.: 25 °C (Room) - Time Started: 1268120448 s - 2-Theta: 2.000 ° - Theta: 1.
Operations: Import


Carbon D
Lin (Counts)
0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
28
29
30
31
32
33
34
2
10
20
30
40
50
60
70
8
2-Theta - Scale
Carbon D - File: Carbon D.raw - Type: 2Th/Th locked - Start: 2.000 ° - End: 80.000 ° - Step: 0.050 ° - Step time: 1. s - Temp.: 25 °C (Room) - Time Started: 1268123776 s - 2-Theta: 2.000 ° - Theta: 1.
Operations: Import


Carbon E
Lin (Counts)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
2
10
20
30
40
50
60
70
8
2-Theta - Scale
Carbon E - File: Carbon E.raw - Type: 2Th/Th locked - Start: 2.000 ° - End: 80.000 ° - Step: 0.050 ° - Step time: 1. s - Temp.: 25 °C (Room) - Time Started: 1268184576 s - 2-Theta: 2.000 ° - Theta: 1.
Operations: Import

APPENDIX D: CAPACITOR PERFORMANCE EVALUATION - RAW DATA

	Sample A		Sample B		Sample C		Sample D		Sample E	
Cycle no.	Charge time, h	Discharge Time, h	Charge time, h	Discharge Time, h	Charge time, h	Discharge Time, h	Charge time, h	Discharge Time, h	Charge time, h	Discharge Time, h
1	0.0039111	0.00321694	0.0046461	0.0037728	0.0105556	0.0075747	0.0109556	0.0089633	0.00481	0.0036883
2	0.00388	0.00304056	0.0042783	0.0036772	0.0090611	0.00703	0.0109061	0.0087543	0.0042969	0.0037092
3	0.0036222	0.00279167	0.004085	0.0036161	0.0080567	0.0067092	0.0108057	0.0086633	0.0041922	0.0037233
4	0.0032217	0.00265333	0.0039694	0.0035681	0.0075353	0.0064856	0.0107535	0.0085833	0.0041022	0.0037342
5	0.0030075	0.002565	0.0038756	0.0035278	0.0071944	0.0063147	0.0107194	0.0084633	0.0040475	0.0037422
6	0.0028683	0.00250306	0.0037981	0.0034953	0.0069383	0.0061761	0.0106938	0.0084633	0.0040172	0.0037494
7	0.0027733	0.00245917	0.0037408	0.0034647	0.0067433	0.0060622	0.0106743	0.0084633	0.0039956	0.0037569
8	0.0027025	0.0024275	0.0036886	0.0034383	0.0065817	0.0059653	0.0106582	0.0086803	0.0039783	0.0037636
9	0.0026536	0.00240389	0.0036431	0.0034111	0.0062242	0.0058789	0.0106224	0.0084633	0.0039656	0.0037692
10	0.0026144	0.00238556	0.0036094	0.0033892	0.0063244	0.0058006	0.0106324	0.0090819	0.0039544	0.0037725

	Specific Discharge Capacity, mA.h/g				
Cycle no.	Sample A	Sample B	Sample C	Sample D	Sample E
1	0.80423611	0.9431944	1.8936806	2.2408333	0.9220833
2	0.76013889	0.9193056	1.7575	2.1885833	0.9272917
3	0.69791667	0.9040278	1.6772917	2.1658333	0.9308333
4	0.66333333	0.8920139	1.6213889	2.1458333	0.9335417
5	0.64125	0.8819444	1.5786806	2.1158333	0.9355556
6	0.62576389	0.8738194	1.5440278	2.1158333	0.9373611
7	0.61479167	0.8661806	1.5155556	2.1158333	0.9392361
8	0.606875	0.8595833	1.4913194	2.1700694	0.9409028
9	0.60097222	0.8527778	1.4697222	2.1158333	0.9422917
10	0.59638889	0.8472917	1.4501389	2.2704861	0.943125

Current, I= 10 mA

	Sample A		Sample B		Sample C		Sample D		Sample E	
Cycle no.	Charge time, h	Discharge time, h	Charge time, h	Discharge time, h	Charge time, h	Discharge time, h	Charge time, h	Discharge time, h	Charge time, h	Discharge time, h
1	0.0006922	0.000801	0.0072836	0.0023089	0.002585	0.0023503	0.013943	0.0046647	0.0016233	0.0017225
2	0.0008711	0.000808	0.0027125	0.0020017	0.0025275	0.0023333	0.0126639	0.0041452	0.0017386	0.0017336
3	0.0008531	0.00081	0.0022836	0.0018722	0.0024564	0.0023158	0.0114278	0.0041368	0.0017936	0.0017367
4	0.0008444	0.000811	0.0020958	0.0017928	0.0024164	0.0023006	0.0102128	0.0041681	0.0017853	0.0017381
5	0.0008383	0.000812	0.0019817	0.0017344	0.0023883	0.0022858	0.0093067	0.0041808	0.0017803	0.0017392
6	0.0008344	0.000812	0.0019006	0.0016894	0.0023661	0.0022728	0.0085067	0.0040681	0.0017778	0.00174
7	0.0008303	0.000811	0.0018381	0.0016517	0.0023464	0.0022606	0.0077925	0.0039681	0.001775	0.0017406
8	0.0008272	0.000811	0.0017883	0.0016219	0.0023303	0.0022494	0.0072411	0.0039178	0.0017728	0.0017408
9	0.0008247	0.000811	0.0017469	0.001595	0.002315	0.0022383	0.0067997	0.0037889	0.0017714	0.0017408
10	0.0008228	0.00081	0.0017136	0.0015614	0.0023014	0.0022289	0.0064464	0.0036808	0.0017706	0.0017417

	Specific Discharge Capacity, mA.h/g				
Cycle no.	Sample A	Sample B	Sample C	Sample D	Sample E
1	0.3461111	1.154444	1.1751389	2.3323611	0.86125
2	0.4355556	1.000833	1.1666667	2.0725833	0.8668056
3	0.4265278	0.936111	1.1579167	2.0684042	0.8683333
4	0.4222222	0.896389	1.1502778	2.0840417	0.8690278
5	0.4191667	0.867222	1.1429167	2.0904167	0.8695833
6	0.4172222	0.844722	1.1363889	2.0340417	0.87
7	0.4151389	0.825833	1.1302778	1.9840417	0.8702778
8	0.4136111	0.810972	1.1247222	1.9588889	0.8704167
9	0.4123611	0.7975	1.1191667	1.8944444	0.8704167
10	0.4113889	0.780694	1.1144444	1.8404167	0.8708333

Current, I= 50 mA

	Sample A		Sample B		Sample C		Sample D		Sample E	
Cycle no.	Charge time, h	Discharge time, h	Charge time, h	Discharge time, h	Charge time, h	Discharge time, h	Charge time, h	Discharge time, h	Charge time, h	Discharge time, h
1	0.0000798	0.0000850	0.0002375	0.00022444	0.0002730	0.000272	0.0003289	0.0003639	0.000253	0.0002447
2	0.0000711	0.0000725	0.0002275	0.0002025	0.0002731	0.000275	0.0003389	0.0003222	0.000245	0.0002467
3	0.0000703	0.0000725	0.0001961	0.00019222	0.00027417	0.000276	0.0003667	0.0003167	0.000243	0.0002469
4	0.0000694	0.0000725	0.0001839	0.00018667	0.00027472	0.000273	0.0003694	0.0003167	0.000242	0.0002469
5	0.0000692	0.0000731	0.0001781	0.00018333	0.00027417	0.000271	0.0003722	0.0003111	0.000241	0.0002472
6	0.0000692	0.0000731	0.0001739	0.00018083	0.00027472	0.000271	0.0003722	0.0003139	0.000241	0.0002469
7	0.0000686	0.0000731	0.0001714	0.00017917	0.00027472	0.00027	0.0003722	0.0003139	0.000241	0.0002475
8	0.0000683	0.0000733	0.0001694	0.00017778	0.00027472	0.000269	0.0003722	0.0003139	0.000241	0.0002469
9	0.0000683	0.0000731	0.0001681	0.00017722	0.00027444	0.000269	0.0003722	0.0003167	0.00024	0.0002467
10	0.0000681	0.0000733	0.0001672	0.00017611	0.00027472	0.000269	0.0003694	0.0003139	0.00024	0.0002472

	Specific Discharge capacity, mA.h/g				
No. Cycle	Carbon A	Carbon B	Carbon C	Carbon D	Carbon E
1	0.2125	0.5611111	0.6805556	0.90972222	0.61180556
2	0.18125	0.50625	0.6880556	0.80555556	0.61666667
3	0.18125	0.4805556	0.6888889	0.79166667	0.61736111
4	0.18125	0.4666667	0.6826389	0.79166667	0.61736111
5	0.18263889	0.4583333	0.6777778	0.77777778	0.61805556
6	0.18263889	0.4520833	0.6763889	0.78472222	0.61736111
7	0.18263889	0.4479167	0.6756944	0.78472222	0.61875
8	0.18333333	0.4444444	0.6736111	0.78472222	0.61736111
9	0.18263889	0.4430556	0.6722222	0.79166667	0.61666667
10	0.18333333	0.4402778	0.6715278	0.78472222	0.61805556