

**ADSORPTION OF CHLORAMPHENICOL BY  
ACTIVATED CARBON DERIVED FROM PALM  
KERNEL SHELL VIA MICROWAVE  
IRRADIATION**

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**by**

**FARIHAH LUQMAN**

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## LIST OF SYMBOLS

	<b>Symbol</b>	<b>Unit</b>
$M_1$	CAP Stock solution concentration	mg/L
$V_1$	CAP Stock Solution Volume	mL
$M_2$	Desired CAP Adsorbate Solution Concentration	mg/L
$V_2$	Desired CAP Adsorbate Solution Volume	mL
$A$	Function of the adsorbate concentration	-
$\epsilon$	Extinction coefficient of the product	mL/cm.g
$c$	Concentration of final adsorbate	g/mL
$d$	Path Length	cm
$A$	radiation power	W
$B$	radiation time	min
$Y$	Outcome parameter	-
$\beta_0$	Overall mean response	-
$\beta_1$	Regression model coefficient 1	-
$\beta_2$	Regression model coefficient 2	-
$X_1$	Independent parameter 1	-
$X_2$	Independent parameter 2	-
$\epsilon$	Error	-
$N$	Total number of experiments (run)	-
$Y_i$	Value of observation at $i^{\text{th}}$ experiment	-
$\bar{Y}$	Average value of observation	-
$P$	Number of regressors in the model	-

$C_o$	Initial concentration of CAP adsorbate solution	mg/L
$C_e$	Final concentration of CAP adsorbate solution	mg/L
$W_o$	Dry weight of PKS presursor	g
$W_c$	Dry weight of PKS-AC	g
$q_e$	The uptake of CAP adsorbate at equilibrium	mg/g
$V$	CAP solution volume	mL
$M$	Amount of PKS-AC	g
$R^2$	correlation coefficients	-
$q_t$	The uptake of CAP adsorbate at particular time	mg/g
$C_t$	Concentration of CAP adsorbate solution at particular time	mg/L
$q_{t.exp}$	The experimental uptake of CAP adsorbate at equilibrium	mg/g
$q_{t.cal}$	The calculated uptake of CAP adsorbate at equilibrium	mg/g
$n$	Number of data points	-
$K_L$	Langmuir's constants	L/mg
$Q_m$	maximum adsorption capacity	mg/g
$T$	Temperature in Kelvin	K
$K_F$	Freundlich constants of adsorption strength	(mg/g)(L/mg) <sup>1/n</sup>
$1/n_F$	Freundlich constants of heterogeneous surface	-
$n_F$	Freundlich constants of adsorption intensity	-
$A_T$	Tempkin isotherm equilibrium binding constant	L/mg
$B_T$	Tempkin model constant	L/mg
$E_{DR}$	mean free energy	J/mol
$k_1$	Pseudo-first-order rate constant	min <sup>-1</sup>
$k_2$	Pseudo-second-order rate constant	g/mg/min

$\Delta q_t$	The normalized standard deviation	%
$\alpha E$	Initial adsorption rate	mg/g/min
$\beta E$	Desorption constant	g/mg
$K_{p1}$	Intraparticle diffusion rate constant for 1 <sup>st</sup> region	mg/g h <sup>1/2</sup>
$K_{p2}$	Intraparticle diffusion rate constant for 2 <sup>nd</sup> region	mg/g h <sup>1/2</sup>
$K_{p3}$	Intraparticle diffusion rate constant for 3 <sup>rd</sup> region	mg/g h <sup>1/2</sup>
$C_2$	Constant related to boundary layer thickness for 1 <sup>st</sup> region	-
$C_2$	Constant related to boundary layer thickness for 2 <sup>nd</sup> region	-
$C_3$	Constant related to boundary layer thickness for 3 <sup>rd</sup> region	-
$\Delta G^\circ$	Gibbs free energy	kJ/mol
$\Delta H^\circ$	enthalpy change	kJ/mol
$\Delta S^\circ$	entropy change	kJ/mol.K
$E_a$	Arrhenius activation energy	kJ/mol
$Y_{CAP}$	CAP removal	%
$Y_{PKS-AC}$	AC's yield	%

## LIST OF ABBREVIATION

CAP	Chloramphenicol
PKS	Palm Kernel Shell
PKS-AC	Palm Kernel Shell Activated Carbons
RSM	Response Surface Methodology
IARC	International Agency For Research On Cancer
UN	United Nations
FAO	Food And Agriculture Organization
COD	Chemical Oxygen Demand
FFB	Fresh Fruit Bunch
EFB	Empty Fruit Bunch
KOH	Potassium Hydroxide
HCl	Hydrochloric Acid
NaOH	Sodium Hydroxide
CO <sub>2</sub>	Carbon Dioxide Gas
BET	Brunauer-Emmett-Teller
PSD	Pore Size Distribution
TPV	Total Pore Volume
APD	Average Pore Diameter
UV	Ultra Violet
SEM	Scanning Electron Microscope
STA	Simultaneous Thermal Analyzer
K Br	Potassium Bromide
FTIR	Fourier Transform Infrared

CCD	Central Composite Designs
CCC	Central Composite Circumscribed
CCF	Central Composite Face-Centered
CCI	Central Composite Inscribed
ANOVA	Analysis Of Variance
SSR	Sum Squares Based On Regression
SSE	Sum Squares Based On Residual
SST	Total Of Sum Squares
M	Molarity
AP	Adequate Precision
Max	Maximum
Min	Minimum
EDA	Electron-Donor-Acceptor

**PENJERAPAN KLORAMFENIKOL MENGGUNAKAN KARBON TERAKTIF  
DARIPADA TEMPURUNG BUAH KELAPA SAWIT MELALUI PEMANASAN  
GELOMBANG MIKRO**

**ABSTRAK**

Kajian ini adalah untuk menghasilkan karbon teraktif daripada isirong sawit (PKS) untuk menyingkirkan kloramfenikol (CAP) daripada larutan akuas. Karbon teraktif isirong sawit (PKS-AC) ini dihasilkan melalui kaedah pengaktifan fizik yang melibatkan penggasan karbon dioksida (CO<sub>2</sub>) dan proses pemanasan menggunakan gelombang mikro. Kesan faktor-faktor penyediaan seperti kuasa radiasi dan masa radiasi ke atas kecekapan penyingkiran kloramfenikol dan hasilan karbon teraktif telah dioptimakan melalui metodologi permukaan sambutan (RSM). Keadaan penyediaan optima untuk karbon teraktif isirong sawit (PKS-AC) yang telah ditemui adalah 364 W untuk kuasa radiasi dan 2 min masa radiasi. Keadaan penyediaan optima ini menyumbang kepada sederhana tinggi kecekapan penyingkiran kloramfenikol (85.93%) dan hasilan karbon teraktif (37.02%). Melalui analisis hampiran, jumlah tetap karbon untuk isirong sawit mentah adalah 40.23% dan meningkat hebat kepada 71.86% (optima isirong sawit) selepas proses pengaktifan. Kajian keseimbangan melibatkan kepekatan awal kloramfenikol, masa hubungan, suhu larutan dan pH larutan berdasarkan proses penjerapan kloramfenikol di atas optima isirong sawit telah dijalankan. Penjerapan kloramfenikol adalah sesuai dengan garis sesuhu Langmuir yang mempunyai 22.83, 23.70 dan 24.88mg/g maksima penjerapan kapasiti pada 30°C, 45°C dan 60°C. Kajian kinetik mendedahkan penjerapan kloramfenikol ini sesuai dengan model kinetik pseudo tertib kedua manakala kajian termodinamik mengesahkan proses ini adalah endotermik secara semulajadi. Kajian mekanisma mendapati proses penjerapan dikawal oleh mekanisma resapan filem.

## **ADSORPTION OF CHLORAMPHENICOL BY ACTIVATED CARBON DERIVED FROM PALM KERNEL SHELL VIA MICROWAVE IRRADIATED**

### **ABSTRACT**

This study aims to synthesis activated carbon (AC) derived from palm kernel shell (PKS) to adsorb chloramphenicol (CAP) from aqueous solution. This palm kernel shell activated carbon (PKS-AC) was produced via physical activation method that involves carbon dioxide (CO<sub>2</sub>) gasification and heating process via microwave. The effects of preparation conditions of microwave radiation power and radiation time on CAP removal efficiency and AC's yield were optimized via response surface methodology (RSM). Optimum preparation conditions for PKS-AC were identified, including radiation power of 364 W and 2 min of radiation time. These optimum conditions contributed to relatively high CAP removal of 85.93% and AC's yield of 37.02%. Through elemental analysis, the total fixed carbon for raw PKS was found to be 40.23% and greatly increased to 71.86% (optimized PKS-AC) after activation process. The equilibrium studies which involved initial CAP concentration, contact time, the temperature of solution and pH solutions based on adsorption of CAP on optimized PKS-AC were performed. The adsorption of CAP onto optimized PKS-AC followed Langmuir isotherm where the maximum adsorption capacities were 22.83, 23.70 and 24.88mg/g for 30°C, 45°C and 60°C respectively. Kinetic studies revealed that adsorption of CAP onto PKS-AC followed pseudo-second order kinetic model while thermodynamic studies confirmed that the adsorption system was endothermic in nature. Mechanism studies described that the CAP adsorption process was govern by film diffusion mechanism model.

# CHAPTER 1

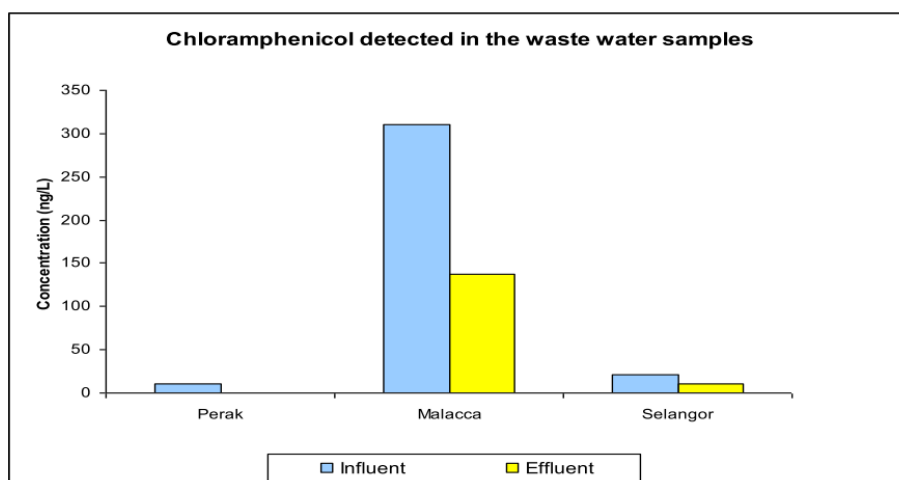
## INTRODUCTION

### 1.1 Chloramphenicol

Chloramphenicol (CAP) is a broad-spectrum antibiotic that is applied in the treatment of bacterial infections and systemic infections. The scientific names for CAP are D-threo-2,2-dichloro-N-[3-hydroxy- $\alpha$ -(hydroxymethyl)-p-nitrophenethyl]-acetamide and D(-) threo-2-dichloroacetamido-1-p-nitrophenyl-1,3-propanediol (Brock, 1961).

Previously, CAP was evaluated by International Agency for Research on Cancer (IARC) in 1990, United States Food and Drug Administration in 1985 and European Committee for Veterinary Medicinal Products in 1994 which it has been suspected carcinogen that could lead to genetic damages and possibly originate cancer (Wongtavatchai J. et. al., 2004). Besides, countries including Canada, Japan and China have banned CAP in aquaculture, food animal production and pet animal medicine as the UN Food and Agriculture Organization (FAO) urged to stop the utilization of CAP. There are several side effects related to the usage of CAP which could risk aplastic anemia, diarrhea, nausea, vomiting and inflammation of the enterocolitis and small intestine. The contaminants of pharmaceuticals including CAP were reported found in treated wastewater, untreated wastewater and drinking water (Lach & Ociepa-Kubicka, 2017). Although CAP is banned as mentioned earlier, the illegal utilization of CAP in aquaculture and wastewater discharge from antibiotic production sites still pollutes the water body. Lach & Ociepa-Kubicka (2017) described that both hospital and pharmaceutical manufacturing plants discharged wastewater with highest concentration of pharmaceutical contaminants. Figure 1.1 shows the detection of CAP in wastewater samples collected from swine facilities in particular states in Malaysia.





**Figure 1.1** Chloramphenicol detection in the waste water samples collected from the swine facilities in Perak, Malacca and Selangor (Marni & Faridah, 2010)

Adsorption technology has improved the ability to detect contaminants at very low concentrations in wastewater and drinking water. Thus, the application of activated carbon (AC) in the adsorption process has become the interest due to the concern related to human health. One of the benefits of AC in the process is the spent AC can be recovered back to reduce the associated costs. The recovery process can be done by desorbing the accumulated adsorbates with the objective returning back to its original structure. Recovery of AC also can lead to safe disposal management as a requirement of environmental regulations. There are two types of recovery which depend on the activation process of physical or chemical. For the adsorbent that undergoes the physical activation, recovery process can be conducted by heating and pressure reduction process. For the adsorbent that undergoes the chemical activation, recovery process can be conducted by breaking the covalent bonds from the supplied energy. Ani et al. (2020) described the properties of base material and characteristics of adsorbates also affecting the rate of recovery.

Exploration on the heating operation has contributed to the development of microwave equipment. There are various types of microwave including microwave irradiated which has been applied in the industry of food processing, rubber treatment and wood drying (Yacob et al., 2013). The principle of heating process in microwave irradiation is based on direct

interaction of char particles with electromagnetic energy. According to the study of Karim (2018), interior heat of microwave irradiation is produced under high frequency of voltage which leads to the dipole rotation and ionic conduction of the char particles. The orientation of particles changes due to the inducement of supplied voltage which leads to the agitation. The microwave irradiation has contributed to the improvement of heat transfer and uniform distribution of heat transfer (Karim, 2018). Besides, Yacob et al. (2013) described that the usage of microwave irradiation could lead to lower energy consumption due to the reduction in treatment time.

## **1.2 Water pollution**

The advancement of the fourth industrial has largely contributed to environmental pollution, especially water pollution. The water pollution directly affects the health of human and living creatures through food poisoning. Water contamination is a greater hazard for human beings as the contamination could exist in different forms of bacteria, heavy metal, viruses and organic components. Some of the water contaminants are non-biodegradable which are carcinogenic and could accumulate in the human body continuously. The pollution of water brings risk of diseases and deaths to the lives of millions of people. Designing the new technology for water purification with zero complexity and cost effectiveness still remains a challenge. An economical AC derived from palm kernel shell (PKS) via microwave irradiated for chloramphenicol (CAP) removal is proposed in this study.

### **1.3 Problem statement**

CAP might be consequential in serious adverse health effects in human body which is strictly banned in the European Union (European Commission 1994). A reliable treatment is required to maintain the environment's water from contaminants of CAP residues to avoid threatening public health. Adsorption process is the best technique to treat CAP compared to other techniques. Adsorption process has advantages over the other techniques because of its high efficiency in removing the contaminants even though at lower contaminant concentrations. Moreover, the adsorption is also known for its high selectivity, regenerability and cost-efficiency. Despite that, the adsorption process is limited by high cost of precursor implemented for production of AC. AC has been widely derived from fossil fuel resources including lignite coal, bituminous coal and peat. This type of AC has been long known for its disadvantage rather than advantage which is from non-renewable resources that could lead to climate change and pollution. Commonly, the activation step for activated carbon production is conducted by using conventional heating. However, the conventional heating has limitations including energy intensive and high operation cost. This study uses microwave irradiation technique. The benefit of microwave irradiation is able to offer selective heating mechanism and reduce complexity in the heating operation. A tool known as Response Surface Methodology (RSM) is utilized to discover the optimum conditions of adsorption process. This RSM is employed because traditional approach of changing one factor at a time while maintaining others constant is time consuming. This study is also aiming to encourage people to strive for green technology in a delicate balance addressed by standards of reducing environmental impact while prolonging the profitability.

#### **1.4 Research objectives**

There are several objectives to be achieved in this study including:

- i. To prepare and characterize physio-chemical properties of activated carbon-based palm kernel shells (PKS-AC) through irradiation for removal of CAP from aqueous solution.
- ii. To identify the optimum activation conditions for PKS-AC by using Response Surface Methodology (RSM).
- iii. To evaluate the effect of initial CAP concentration, contact time, solution temperature and solution pH towards the adsorption process.
- iv. To investigate the behaviour of CAP adsorption onto AC-PKS by studying adsorption isotherm, kinetic and thermodynamic.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Adsorption process**

Contaminants of CAP can be removed from wastewater by various conventional methods through physical, biological and chemical processes. Li et al. (2018) stated that biodegradation of traditional biological treatment is not productive enough to remove CAP due to its particular characteristics. There are several alternative treatments that can replace biological treatment which are photocatalytic degradation, ozonation and radiation-induced treatment. Li et al. (2018) raised concerns towards the mentioned alternative methods which have lower productivity, higher operation cost and photocatalytic degradation treatment that could lead to carcinogenic effects for humans.

According to that, Atikah, Taufik, Din, Khan, Yahaya, Karim, et al. (2020) stated that adsorption is one of the efficient processes which is already being applied in the wastewater treatment industry to remove contaminants from the treated water. Besides that, Ani et al. (2020) described that adsorption process is the most productive due to the ability of adsorbing contaminants at low concentration compared to ultrafiltration, ion exchange, coagulation and electrochemical adsorption treatment. The adsorption process, also known as one of the treatment processes that require lower utilization of land area which Ani et al. (2020) mentioned, only takes over  $\frac{1}{2}$  -  $\frac{1}{4}$  of biological land area. The factors that affect adsorption process are rate of mass transfer and equilibrium of solid and liquid.

#### **2.2 Type of porous materials in adsorption process**

Commonly, porous materials are being used in the adsorption process. There are several examples of porous materials which are carbon nanotubes, bamboo charcoal, mesoporous silica, earth materials and AC. However, carbon nanotubes and mesoporous silica showed risk

of toxicity, higher material cost and difficult fabrication process (Li et al., 2018). On the other hand, bamboo charcoal and Earth materials have lower material cost but lower efficiency in adsorbing contaminants (Li et al., 2018). Apart from that, Mohd Din et al. (2015) reported that AC achieves maximum adsorption capacity of CAP for about 209.7 mg/g.

### **2.3 Activated carbon**

AC is a type of carbonaceous material which have characteristics such large surface area, suitable pore structure, higher physiochemical stability, regenerable, large surface reactivity, mostly from sp<sup>2</sup> hybridized carbon and have a good mechanical strength (Ani et al., 2020) (Atikah, Azmier, Khan, Yahaya, Taufik, Din, et al., 2020). Waste from the agricultural sector usually is being incinerated which leads to air pollution. Transforming the generated waste from agriculture and industry into AC could innovate new disposal methods which are able to reduce environmental impact. With the objective to reduce the cost of preparations, researchers have moved to use the less valuable biomass. Ani et al. (2020) described that by-products of agriculture such as almond shell, coconut shell, hazelnut shell and apricots seeds can be derived to produce AC. Besides that, Li et al. (2018) stated that aquatic plants such as *Typha orientalis* also can be produced to form AC.

### **2.4 Activated carbon derived from palm kernel shell in Malaysia**

In 2015, Malaysia was the second largest producer of palm oil behind Indonesia, responsible for 32% (19.9 million tonnes) for global palm oil production (GreenPalm, 2016). A fresh fruit bunch (FFB) consists of 27% palm oil, 23% empty fruit bunch (EFB), 14-15% fibres, 6-7% kernel and 6-7% shell for a basic weight (Rugayah et al., 2014). Accordingly, Abnisa et al. (2011) reported that palm shell waste including empty fruit bunches and shells were generated more than 2.4 million tonnes for each year in Malaysia. For sustainable purposes, PKS can be recycled and reused back as a boiler fuel (heating value of 17 MJ kg<sup>-1</sup>)

and derived to produce AC as mentioned by Rugayah et al. (2014). The research also described a PKS having low content of ash, high content of carbon and high volatile content which is suitable to produce AC.

## **2.5 Activation process**

Activation of precursors can be done in two ways which is physical or chemical activation processes (Ani et al., 2020). Physical activation requires the carbonization process of raw material at 500-600°C and activation of char at 800-1100°C with the continuous supply of oxidizing agents (carbon dioxide, steam and air). Chemical activation requires carbonization and activation of precursor to be performed at the same time which the raw material must be immersed with specified chemicals first.

Impregnation of AC with specific additives before activation process is encouraged to enhance the adsorption performance and improve the selectivity of AC. In the research of Hidayu and Muda (2017), the impregnation of PKS-AC with cerium oxide, barium oxide and titanium oxide were conducted. It was found that PKS-AC impregnated with cerium oxide achieved the highest adsorption capacity compared to barium oxide and titanium oxide. According to Oginni et al. (2019), potassium hydroxide (KOH) is the most productive among chemical activation agents due to the observation that it can enhance the formation of alcohol (-OH) functional groups on the surface of AC. Besides, the contribution of potassium alone could improve the formation of intercalation compounds with carbon. Furthermore, the utilization of KOH as an additive in chemical activation leads to the enhancement of pore formation due to the carbon dioxide emission from carbon gasification (Oginni et al., 2019).

## CHAPTER 3 MATERIALS AND METHODS

Chapter three explored the methodology in conducting this experiment including materials and chemicals used, equipment involved, preparation of PKS-AC, experimental design and theory of adsorption studies.

### 3.1 Materials

The materials involved in this research study can be classified into three types which are precursors, chemicals and gases.

#### 3.1.1 Precursors

The precursor used in this study is the palm kernel shell (PKS). A fresh fruit bunch (FFB) was collected at a small oil palm plantation owned by Encik Fauzi bin Ahmad located in Kuala Ketil, Kedah, Malaysia. The structure of an oil palm fruit consists of exocarp, mesocarp, endocarp and kernel. Some oil palm fruit was removed from the bunch and it is manually processed to acquire only the palm kernel shell. The structure of an oil palm fruit consists of exocarp, mesocarp, endocarp and the kernel itself.

#### 3.1.2 Chemicals

The chemicals utilized in this experiment are tabulated in Table 3.1 while the properties of adsorbent CAP are listed in Table 3.2.

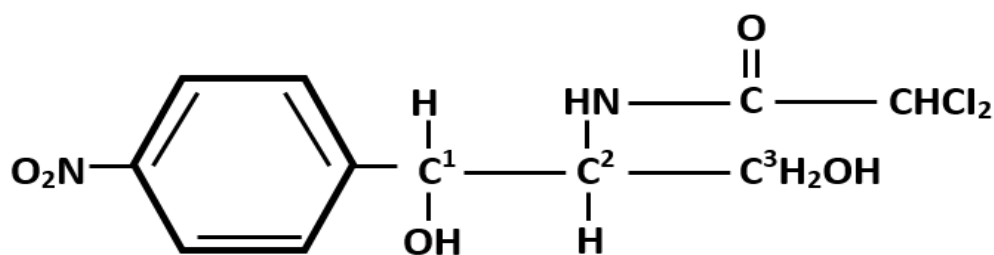
**Table 3.1** Chemicals for PKS-AC preparation

Chemical	Purpose of use
Hydrochloric Acid (HCl)	pH Adjustment
Sodium Hydroxide (NaOH)	pH Adjustment
Chloramphenicol (CAP)	Adsorbate



**Table 3.2** CAP properties

Properties	Description
Scientific Name	2,2-dichloro-N-[(1R,2R)-1,3-dihydroxy-1-(4-nitrophenyl)propan-2-yl]acetamide
Common Name	Chloromycetin; Chlornitromycin; Levomycetin
Molecular Dimension	278 nm
Molecular Formula	C <sub>11</sub> H <sub>12</sub> Cl <sub>2</sub> N <sub>2</sub> O <sub>5</sub>
Molecular Weight	323.132 g/mol
Melting Point	150.5-151.5 °C
Chemical Structure	



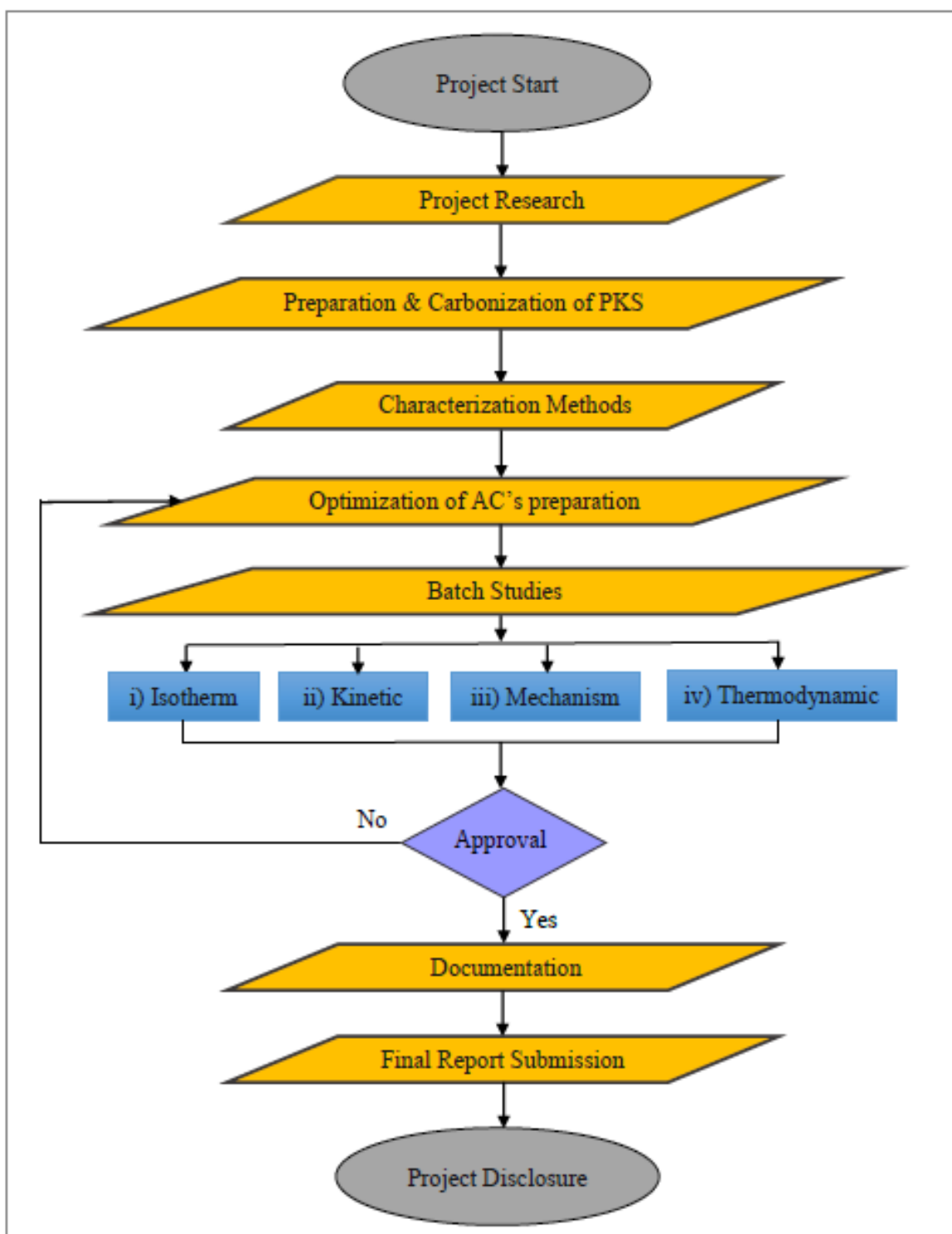
### 3.1.3 Gases

Carbon dioxide gas (CO<sub>2</sub>) having purity of 99.99% was used in this experiment. The CO<sub>2</sub> was used in the carbonization process where it is purged into the modified microwave.

## 3.2 Experimental activities

Figure 3.1 shows the flow activity of the research in this final year project. As an overall, PKS-AC will be used to study the efficiency for removal of CAP from aqueous solution. Its performance on the capacity adsorption of CAP will be studied in this project. The project research has been conducted in semester 1 while activity of PKS preparation and carbonization process is initiated in semester 2. Next, the prepared AC will be characterized in terms of measurement of BET surface area, Barrett-Joyner-Halenda pore size distribution

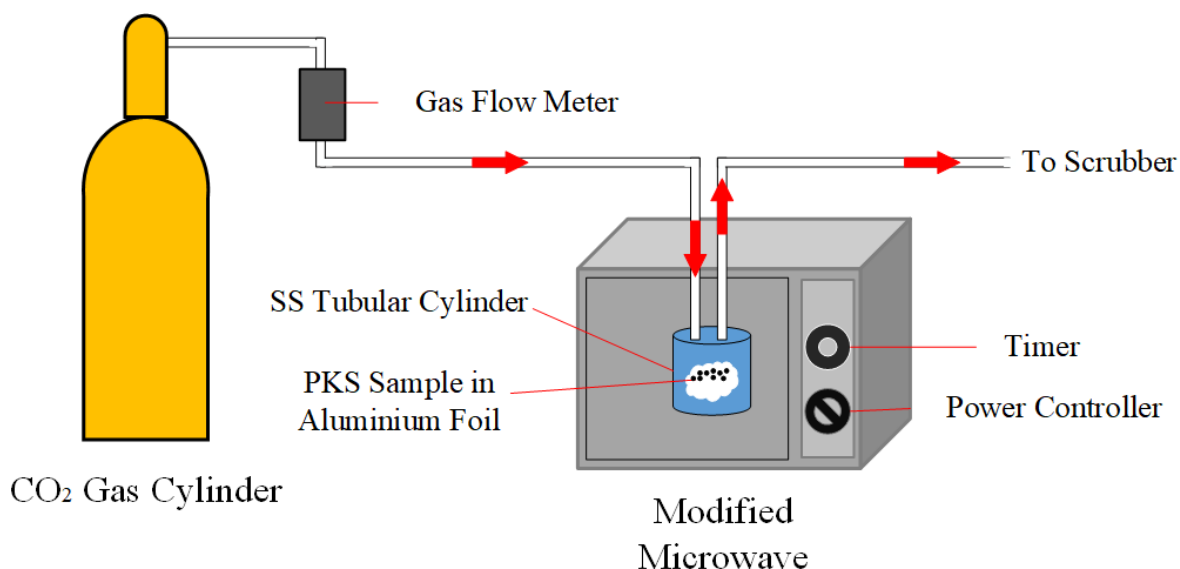
(PSD), total pore volume (TPV), average pore diameter (APD) and more. Then, the optimum preparation conditions involved parameters of radiation power and radiation time to analyse the responses of CAP removal and AC's yield by using standard response surface methodology (RSM). Next, the adsorption process for removal CAP was conducted. The batch studies involved the effect of initial CAP concentration and contact time, effect of solution temperature and effect of solution pH. Furthermore, the studies related to the isotherm, kinetic, mechanism and thermodynamic were performed to analyse the performance of optimized PKS-AC in removing CAP. The documentation process conducted after obtaining approval from the supervisor.



**Figure 3.1** Flow diagram of research project on CAP removal

### 3.2.1 PKS-AC preparation

The PKS sample was washed and dried in an oven at 105°C for 24 hours. Physical activation of the PKS by microwave irradiation was conducted at three different powers (364 W, 490 W and 616 W) at three different radiation times (2 min, 4 min and 6 min) using the modified conventional 2.45 GHz microwave oven. The activation process was supplied with continuous flow of CO<sub>2</sub> gas at 150 cm<sup>3</sup>/min. The schematic diagram of microwave irradiation setup is presented in Figure 3.2. The equipment used consists of a rectangle microwave oven having dimension roughly 21-24 inches of width and 13 inches of height. The different power levels can be regulated by using a power controller and the exposure heated time can be controlled by using the timer of the microwave. A stainless-steel tubular cylinder is utilized for placing the aluminium foil containing PKS sample in the centre of the oven. The oven is connected directly to the CO<sub>2</sub> gas cylinder and gas flow meter was installed to measure the CO<sub>2</sub> flow rate that supplied to the oven.



**Figure 3.2** Schematic diagram of microwave irradiation setup

The procedure of the impregnation process was explained specifically for better understanding. Firstly, the dried PKS sample was placed in the small size of aluminium foil.

The aluminium foil containing the sample was allocated in a stainless-steel tubular cylinder and was loaded in the chamber of the microwave. After that, the microwave irradiation was conducted based on the set radiation power and radiation time under a constant CO<sub>2</sub> flow of 150 cm<sup>3</sup>/min. After passing the radiation time of the activation process, the PKS sample was unloaded and cooled to room temperature. Then, the activated sample was kept inside air-tight containers and labelled as “AC”. This product is ready for further studies.

### **3.2.2 Batch adsorption and analysis system**

In this adsorption system, 200 mL of 100 mg/L CAP solution was prepared from 1000 mg/L stock solution. The prepared solution was then transferred into the 250 mL conical flasks with the addition of 0.2 g of the produced AC. Next, the filled conical flasks were loaded into the isothermal water-bath shaker for a particular time. The temperature and the agitation speed (maximum 250 rpm) of water-bath shaker were regulated according to the category of experiment. Subsequently, the CAP concentration of final solution in the conical flask after adsorption process was determined by using double beam UV-Visible spectrometer (Model Shimadzu UV-1800, Japan).

### **3.2.3 Characterization system**

The characterization of PKS-AC's was performed to diagnose the surface area, pore characteristic, surface morphology, proximate analysis and surface chemistry.

#### **3.2.3(a) Surface area**

The surface area of AC was determined through an isotherm model based on the Brunauer-Emmett-Teller (BET) mathematical equation. The equipment of Micromeritics ASAP 2020 volumetric adsorption analyzer was being utilized to discover the pore volume and average pore diameter besides the surface area. This equipment operates by identifying the

number of nitrogen gas adsorbed into an internal surface at equilibrium vapour pressure (77 K) via static volumetric technique.

### **3.2.3(b) Surface morphology**

For this characterization genre, the equipment used is a scanning electron microscope (SEM) (Quanta 450 FEG, Netherland). According to Ahmad et al. (2014), the AC was required to be attached to a sample plate before being laminated with a gold-fine layer to create the reflection of electrons. Then, the AC was analysed by using an electron microscope applying the magnification concept in the range of 500-4000x.

### **3.2.3(c) Proximate analysis**

Proximate analysis of characterization schemes capable of extracting information related hydration percentage, quantity of volatile matter, number of fixed carbon and ash contents. The hydration percentage was determined by heating operation of investigated AC from normal temperature of 25°C to 110°C under nitrogen flow. Besides, the quantity of volatile matter was identified via decomposition process conducted at 850°C. Moreover, the number of fixed carbon and ash contents were measured by replacing the nitrogen flow with the oxygen flow (Ahmad et al., 2016). This approach was performed by using the equipment of Simultaneous Thermal Analyzer (STA).

### **3.2.3(d) Surface chemistry**

The characterization methods also implement the analysis on the surface chemistry of the AC. The main objective of this analysis is to understand the chemical properties in terms of the functional group that present on the surface of AC. The AC with the addition of 0.1 wt% potassium bromide (K Br) required to be in the form of fine powder without any moisture and

shaped into a thin disc (Atikah, Taufik, Din, Khan, Yahaya, Khasri, et al., 2020) . The analysis is conducted by a Fourier Transform Infrared (FTIR) spectroscope.

### **3.3 Experimental procedure**

This section emphasized the procedure of experiment that was conducted during the investigation.

#### **3.3.1 Chloramphenicol stock solution and adsorbate solution preparation**

Stock solution of CAP was prepared by diluting a concentrated CAP adsorbate with deionized water in a 1 L or 1000 mL of a volumetric flask. Firstly, a very small amount of 0.1 g CAP powder was measured by using the electronic balance weighing scale in the laboratory. This weighing process was ensured to be conducted at a condition without any disruption due to environmental factors including air pressure, vibrations and humidity. Secondly, the measured CAP powder was placed in the volumetric flask and the deionized water was added until it reached the indicated level. The filled volumetric flask was shaken in water-bath shaker for better mixing and the concentration of the produced stock solution is 100 mg/L. The various concentrations of CAP adsorbate solution ranging from 1 to 30 mg/L were prepared for the construction of CAP calibration curve and further continuous and batch studies. All this procedure was established by using the dilution formula as shown below.

$$M_1V_1 = M_2V_2 \quad \text{Equation 3.1}$$

where;

$M_1$ = CAP stock solution concentration (mg/L)

$V_1$  = CAP stock solution volume (mL)

$M_2$  = Desired CAP adsorbate solution concentration (mg/L)

$V_2$  = Desired CAP adsorbate solution volume (mL)

### 3.3.2 System of sample analysis

The continuous analysis of the sample in determining the concentration of CAP was run by the equipment known as double beam UV-Visible spectrophotometer. This type of analysis only requires 2.5 mL of investigated CAP adsorbate solution that was sucked by 3 mL disposable syringe. This specific equipment is necessary to set the maximum wavelength of the study adsorbate which in this case is CAP adsorbate at 278 nm. A UV-Visible spectrophotometer determines the intensity of a light ray that passes through a CAP solution in a cuvette which then compares it to the light intensity before passing through the CAP sample. The measured light intensity is proportional to the CAP sample concentration which explains the higher concentration of solution, the greater the absorb light. This relationship is established as Lambert-Beer law which also involves other parameters as shown below (A. De Caro, 2015). The graph of absorbance against the concentration of CAP solution was plotted as CAP calibration curve and attached in the Appendix A.

$$A = \epsilon \cdot c \cdot d \quad \text{Equation 3.2}$$

where;

$A$  = Function of the adsorbate concentration (Absorbance)

$\epsilon$  = Extinction coefficient of the product (mL/cm.g)

$c$  = Concentration of final adsorbate (g/mL)

$d$  = Path length (1 cm)

### 3.3.3 Experimental design for PKS-AC preparation

The adsorption process of CAP was conducted in a water bath shaker using a 250 mL conical flask at an initial CAP concentration of 10 mg/L, PKS-AC dosage of 0.2 g (13 different samples) and shaker speed at 150 rpm which conducted at 30 °C. This experiment has two



different parameters or variables which are radiation power (A) and radiation time (B) as shown in Table 3.3 to identify the optimum activation conditions for PKS-AC according to the obtained AC's yield and CAP removal efficiency.

**Table 3.3** Experimental variables

Variable	Code	Unit
Radiation power	A	Watt (W)
Radiation time	B	minutes (min)

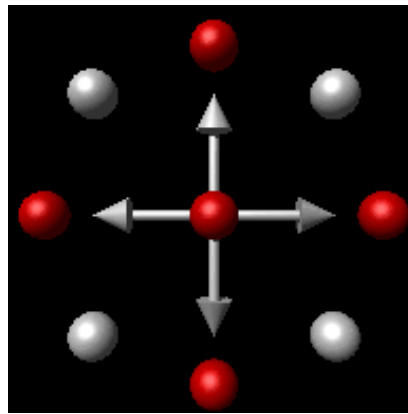
### 3.3.4 Development of regression model statistical analysis

A total of 13 runs of experiments were conducted which comprised various radiation powers (364 W, 490 W and 616 W) and radiation times (2 min, 4 min and 6 min) as listed in Table 3.4 below. This regression model statistical analysis was performed in order to discover the optimum condition for PKS-AC for better adsorption process.

**Table 3.4** Design matrix for PKS-AC preparation

Run	PKS-AC's Preparation Variables			
	Level	Radiation Power, A (watt)	Level	Radiation Time, B (min)
1	-1	364.00	0	4.00
2	+1	616.00	0	4.00
3	+1	616.00	+1	6.00
4	-1	364.00	+1	6.00
5	0	490.00	+1	6.00
6	0	490.00	0	4.00
7	0	490.00	0	4.00
8	0	490.00	0	4.00
9	0	490.00	0	4.00
10	0	490.00	-1	2.00
11	+1	616.00	-1	2.00
12	0	490.00	0	4.00
13	-1	364.00	-1	2.00

There are three design types of central composite designs (CCD) including circumscribed design (CCC), face-centered (CCF) and inscribed (CCI). The utilized design in this process was CCF which is based on the principle of  $\alpha = \pm 1$  points. The star points are positioned on the faces of the experimental domain that employ a three-level factorial design. This model applied 8 non center points and 5 center points which produced a total 13 experiments. The Figure 3.3 below shows the generated design layout of the CCD.



**Figure 3.3** Design layout of CCF

The quadratic model or known as second-order model was constructed based on the equation 3.3 below (Bhattacharya, 2021).

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{12} X_1 X_2 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \epsilon \quad \text{Equation 3.3}$$

where;

$Y$  = Outcome parameter

$\beta_0$  = Overall mean response

$\beta_1$  = Regression model coefficient 1

$\beta_2$  = Regression model coefficient 2

$X_1$  = Independent parameter 1

$X_2$  = Independent parameter 2

$\epsilon$  = Error

### 3.3.5 Exploration of variance (ANOVA)

The produced results from the analysis of both responses are interpreted in the form of analysis of variance (ANOVA). ANOVA can be defined as a statistical technique that classified the investigated variance data into various classes for further studies. This depiction method is able to highlight the relationship between the observed parameters and particular responses.

ANOVA involves several specifications including sum squares based on regression (SSR), sum squares based on residual (SSE), total of sum squares (SST) and F values. The mathematical equation of the specifications of the fitted model and non-fitted model are tabulated as Table 3.5 below. The SST specification is the total of SSR and SSE. The F value is capable of identifying the null hypothesis whether it is true or false. The true hypothesis shows the same value of mean squares of SSR and SSE. The false hypothesis indicates the SSR value must be higher than SSE. Additionally, the Prob > F value of ANOVA result should be obtained less than 0.05 to ensure a significance of a particular term model (Tounsadi et al., 2016).

**Table 3.5** Specifications of ANOVA representation

Specification	Symbol	Equation	Degree of Freedom	Mean Squares	Reference
Sum Squares based on Regression	SSR	$SSR = \sum_{i=1}^N (Y_i - \bar{Y})^2$ (Equation 3.6)	$P - 1$	$= \frac{SSR}{P - 1}$	(Chen et al., 2005)
Sum Squares based on Residual	SSE	$SSE = \sum_{i=1}^N (Y_i - \bar{Y})^2$ (Equation 3.7)	$N - P$	$= \frac{SSE}{N - P}$	(Chen et al., 2005)
Sum of Squares	SST	$SST = \sum_{i=1}^N (Y_i - \bar{Y})^2$ (Equation 3.8)	$N - 1$	-	(Analysis, 2008)
F Values	F-Value	$F_{value} = \frac{\frac{SSR}{P - 1}}{\frac{SSE}{N - P}}$ (Equation 3.9)	-	$= \frac{\frac{SSR}{P - 1}}{\frac{SSE}{N - P}}$	(Analysis, 2008)

Where  $N$  = Total number of experiments (run);  $Y_i$  = Value of observation at  $i^{\text{th}}$  experiment;  $\bar{Y}$  = Average value of observation;  $P$  = Number of regressors in the model

### 3.3.6 Batch adsorption equilibrium studies

The adsorption of CAP was conducted at different initial CAP concentrations of 1, 5, 10, 20, 25 and 30 mg/L and at different temperatures of 30, 45 and 60°C using a water bath shaker for 24 hours. A 0.2 g of PKS-AC was added into a 200 mL CAP solution for each test. The experimental results of equilibrium study were examined through the effect of initial CAP concentration, contact time, CAP solution temperature and pH value of the solution. The concentration of each observed solution at a particular time was measured by using UV-Vis spectrophotometer. The response of CAP removal was computed by applying the measured concentrations into the following equation 3.4. Besides, the response of AC's yield was computed by equation 3.5 that involved the measurement of the dry weight of PKS-AC and precursor PKS. This measurement was determined during the activation process of PKS.

$$CAP\ Removal\ (\%) = \frac{(C_o - C_e)}{C_o} \times 100 \quad \text{Equation 3.4}$$

$$AC's\ Yield = \frac{W_c}{W_o} \times 100 \quad \text{Equation 3.5}$$

where;

$C_o$  = Initial concentration of CAP adsorbate solution (mg/L)

$C_e$  = Final concentration of CAP adsorbate solution (mg/L)

$W_o$  = Dry weight of PKS presursor (g)

$W_c$  = Dry weight of PKS-AC (g)

The uptake of CAP adsorbate on the surface of adsorbent was measured using the equation 3.6 below. Then, these values were used to further investigate via adsorption isotherms, adsorption kinetics, adsorption mechanics and thermodynamics.

$$q_e = \frac{(C_o - C_e)V}{M} \quad \text{Equation 3.6}$$

where;

$q_e$  = The uptake of CAP adsorbate at equilibrium (mg/g)

$C_o$  = Initial concentration of CAP adsorbate solution (mg/L)

$C_e$  = Final concentration of CAP adsorbate solution (mg/L)

$V$  = CAP solution volume (mL)

$M$  = Amount of PKS-AC (g)

### **3.3.6(a) Effect of initial concentration and contact time**

The following procedures were conducted to understand the effect of CAP initial concentration and effect of contact time towards the optimized PKS-AC. Firstly, a 200 mL of CAP adsorbate solutions with six different initial concentrations of 1, 5, 10, 20, 25 and 30 mg/L at original pH were prepared in the 250mL conical flasks. Secondly, the 0.2 g of PKS-AC was added into each conical flask and the flasks were enclosed with parafilm. This precaution was conducted to avoid any loss of the important samples. Thirdly, the six different conical flasks were loaded into an isothermal water shaker bath at constant temperature of 30°C and constant agitation speed of 150 rpm. This experiment that takes for about 24 hours was conducted while the absorbance value of the sample was recorded at a particular time.

### **3.3.6(b) Effect of solution temperature**

Performance of CAP adsorption onto the optimized PKS-AC at different solution temperatures were performed. Three conical flasks contained 200 mL of CAP adsorbate solution at initial concentration of 30 mg/L and at original pH were prepared. Next, a 0.2 g of PKS-AC was added into each conical flask and similarly, the flasks were enclosed with parafilm. Then, the conical flasks were loaded into the respective water shaker baths that

operate at different temperatures of 30, 45 and 60°C. The parameter of agitation speed was maintained at 150 rpm and the adsorbate solutions were withdrawn after 24 hours.

### **3.3.6(c) Effect of solution pH**

An amount of 0.2 g of optimized PKS-AC was added into conical flasks filled with 200 mL of CAP adsorbate solutions with concentration of 30 mg/L at four different pH solutions of 4, 6, 8 and 10. This experiment involved chemicals of 0.1 M HCl and 0.1 M NaOH for pH alteration. Several additional equipment is necessary during the pH alteration process including a portable pH meter for pH measurements and a magnetic stirrer for complete chemical mixing. Similarly, the agitation speed is kept constant at 150 rpm and the experiment was performed in the same water shaker bath that operates at a temperature of 30°C. The solution samples were withdrawn after 24 hours and the concentration of sample was measured by using UV-Vis spectrophotometer.

### **3.3.6(d) Adsorption of isotherm model**

The construction of adsorption isotherm involves four distinguished models which are Langmuir, Freundlich, Temkin and Dubinin-Radushkevich isotherm models. The qualification of these isotherm models with the equilibrium data were dependent on the value of correlation coefficients  $R^2$ . The  $R^2$  value of a more approaching digit of 1 is considered a more appropriate model.

### **3.3.7 Study of kinetics and diffusion mechanism model**

The obtained data in equilibrium study was used for further studies for kinetic models. The kinetic studies utilized three different kinetic models including pseudo-first order, pseudo-second order and Elovich model. The formula 3.7 below used to compute the CAP adsorption uptake onto the optimized PKS-AC at particular time,  $q_t$ . Similar to adsorption isotherm, kinetic

model also was observed in terms of correlation factor  $R^2$  with further addition of normalized standard deviation. The smaller the value of normalized standard deviation, the fitted the kinetics data. The normalized standard deviation formula can be expressed as in equation 3.8.

$$q_t = \frac{(C_o - C_t)V}{M} \quad \text{Equation 3.7}$$

Where;

$q_t$  = The uptake of CAP adsorbate at particular time (mg/g)

$C_o$  = Initial concentration of CAP adsorbate solution (mg/L)

$C_t$  = Concentration of CAP adsorbate solution at particular time (mg/L)

$V$  = CAP solution volume (mL)

$M$  = Amount of PKS-AC (g)

$$\Delta q_t(\%) = \sqrt{\frac{\sum \left[ \frac{(q_{t.exp} - q_{t.cal})}{q_{t.cal}} \right]^2}{n-1}} \quad \text{Equation 3.8}$$

Where;

$q_{t.exp}$  = The experimental uptake of CAP adsorbate at equilibrium (mg/g)

$q_{t.cal}$  = The calculated uptake of CAP adsorbate at equilibrium (mg/g)

$n$  = Number of data points

The produced results based on the kinetic study were used for further studies for mechanism models. The mechanism studies consist of intraparticle diffusion and boyd plot models.

### 3.3.8 Study of thermodynamic model

The adsorption system of adsorbate CAP and adsorbent of the prepared PKS-AC was evaluated via thermodynamic study. The graph of  $\ln q_e / C_e$  against  $q_e$  was plotted at each solution temperature of 30, 45 and 60°C. Based on the plotted graph, the values of  $K_L$  were obtained and the values were used to generate another graph known as Van't Hoff which