DESIGN OF EXPERIMENT APPROACH IN MUNICIPAL WASTEWATER TREATMENT THROUGH ADSORPTION OF ACTIVATED CARBON

NUR SALWA BINTI ABDUL RASID

FACULTY OF ENGINEERING
UNIVERSITY OF MALAYA
KUALA LUMPUR

2018

DESIGN OF EXPERIMENT APPROACH IN MUNICIPAL WASTEWATER TREATMENT THROUGH ADSORPTION OF ACTIVATED CARBON

NUR SALWA BINTI ABDUL RASID

RESEARCH REPORT SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF MASTER OF ENGINEERING (MECHANICAL)

FACULTY OF ENGINEERING
UNIVERSITY OF MALAYA
KUALA LUMPUR

2018

UNIVERSITY OF MALAYA

ORIGINAL LITERARY WORK DECLARATION

Name of Candidate: Nur Salwa binti Abdul Rasid

Matric No: KGY150016

Name of Degree: Master of Engineering (Mechanical)

Title of Project Paper/Research Report/Dissertation/Thesis ("this Work"): Design of Experiment Approach in Municipal Wastewater Treatment through Adsorption of Activated Carbon.

Field of Study: Material Processing

I do solemnly and sincerely declare that:

(1) I am the sole author/writer of this Work;
(2) This Work is original;
(3) Any use of any work in which copyright exists was done by way of fair dealing and for permitted purposes and any excerpt or extract from, or reference to or reproduction of any copyright work has been disclosed expressly and sufficiently and the title of the Work and its authorship have been acknowledged in this Work;
(4) I do not have any actual knowledge nor do I ought reasonably to know that the making of this work constitutes an infringement of any copyright work;
(5) I hereby assign all and every rights in the copyright to this Work to the University ofMalaya ("UM"), who henceforth shall be owner of the copyright in this Work and that any reproduction or use in any form or by any means whatsoever is prohibited without the written consent of UM having been first had and obtained;
(6) I am fully aware that if in the course of making this Work I have infringed any copyright whether intentionally or otherwise, I may be subject to legal action or any other action as may be determined by UM.

Candidate's Signature Date: 25 January 2018

Subscribed and solemnly declared before,

Witness's Signature Date:

Name:

Designation:

ABSTRACT

This research presents an approach of application Design of Experiment (DOE) used with municipal wastewater together with activated carbon for the adsorption process. DOE is systematic approaches to engineering problem-solving by determine the relationship between factors/parameters affecting a process and the output of that process. In other words, it is used to find cause-and-effect relationships. By using the DOE, knowledge and estimate the best operating conditions of a system, process or product can be obtained. Through this research, two types of activated carbon which one the Granular Activated Carbon (GAC) and Powdered Activated Carbon (PAC) have been used as the main adsorption materials, to find the most suitable combination of factors that be that influences for neutralized the pH level in municipal wastewater. This DOE approach helps to eliminate non-value added factors while improving the time performance during the adsorption process. In order to initiate a successful process, results obtained through the experiment have been analyzed using Minitab 18 software. Neutralized pH water from the wastewater can be achieved by a combination of parameters/factors 250ml municipal wastewater adsorption with 1.25 gram of GAC within the 3 minutes and 28 seconds of duration period.

Keywords: Activated Carbon, Granular Activated Carbon (GAC), Powdered Activated Carbon, Design of Experiment.

ABSTRAK

Kajian penyelidikan ini membentangkan pendekatan aplikasi reka bentuk percubaan (DOE) yang digunakan dengan air kumbahan perbandaran bersama dengan karbon yang diaktifkan untuk proses penjerapan. DOE adalah pendekatan sistematik kepada penyelesaian masalah kejuruteraan dengan menentukan hubungan antara faktor / parameter yang mempengaruhi proses dan output. Dalam erti kata lain, ia digunakan untuk mencari hubungan sebab dan kesan. Dengan menggunakan DOE, pengetahuan dan dalam menganggarkan keadaan terbaik operasi sistem, proses atau produk boleh diperolehi. Melalui penyelidikan ini, dua jenis karbon yang diaktifkan iaitu Karbon Ketul (GAC) dan Karbon Serbuk (PAC) telah digunakan sebagai bahan utama, di mana ia membantu untuk mencari kombinasi factor yang dapat digunakan untuk meneutralkan tahap pH dalam air sisa perbandaran. Pendekatan DOE ini membantu untuk menghapuskan faktor-faktor tambahan yang tidak diperlukan sambil meningkatkan prestasi masa semasa proses penjerapan. Untuk mendapatkan proses yang berjaya, hasil yang diperoleh melalui eksperimen yang dianalisis dengan menggunakan perisian Minitab 18. Air pH yang neautral dari air sisa dapat dicapai dengan gabungan parameter / faktor 250ml penjerapan air sisa perbandaran dengan 1.25 gram GAC dalam tempoh masa 3 minit dan 28 saat.

ACKNOWLEDGEMENTS

Alhamdullilah, finally I had finished my research project completely. I am very thanks full to ALLAH S.W.T. because without HIS will, I would never be through this successfully. I also like to express my heartfelt gratitude and an infinite appreciation for the parties and individuals involved directly and indirectly in this research project. Here, I would like to dedicate thanks to those who had guidance and support from the beginning until the end of my research project. Thank you infinitely to my Supervisor, Dr.Farazila Yusof because she was monitor, guide, and train me well and gave me encouragement and motivational talk and lessons. Finally, I am highly thankful to my parents as they contribute their energy, strong moral support, and money for me during the research project. Not forgetting to Coordinator Final Year Project (FYP) thank you for organizing schedules, provide the information on FYP, discussion for the progress of the projects, intensive classes to the students.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF SYMBOLS AND ABBREVIATIONS

ANOVA	:	Analysis of Variance
DOE	:	Design of Experiment
EAC	:	Extracted Activated Carbon
FGLM	:	Fit General Linear Model
GAC	:	Granular Activated Carbon
GLM	:	General Linear Model
H_o	:	Null Hypothesis
H_1	:	Alternative Hypothesis
LSL		Lower Specification Limit
PAC	:	Powdered Activated Carbon
pH	:	Potential of Hydrogen
P-Value	:	Probability Value
SMART	:	Specific, Measurable Attainable, Reliable, Tangible
USL	:	Upper Specification Limit
WHO	:	World Health Organization

LIST OF APPENDICES

CHAPTER 1: INTRODUCTION

This chapter contains an overview about the issues of the research, problem statement of current situations, aims of the objectives and also the rationale and significance of the study.

1.1 Background of the Study

Water is essential for sustaining life on Earth. Almost 71% of the Earth's surface is covered by it, while the remaining 29% consists of continents and islands (National Oceanic and Atmospheric Administration). Hence, three-quarters of Earth is made up of water and the demand for water increasing due to the growth in the population. The main threat to this water demand increased because the shortage of continuous supply to provide clean water (Thines et al., 2016).

Based on the study carried out by World Health Organization (WHO) about approximately 1.1 billion people have no direct access to clean water supply. Generally, water is polluted due to lack of sanitation facility. So to meet the higher demand of clean water the adsorption of coal is used for water treatment in this study.

1.2 Problem Statement

Gaining access to safe drinking water is a serious issue to people of all ages worldwide. As our population and industry grew to their present size, which is indirectly contribute to the pollutant load of municipal wastewater. An approach of Design of Experiment (DOE) is conduct to identify significant parameter used for the best economical ways to produce more desirable quality water treatment.

1.3 Purpose of the Study

The purpose of this research project is to restructure the current system of water treatment used in municipal waste water by DOE approach. Thus, determination of most efficient and economical methods of determining the effect of a set of independent variables on a response variable will be conduct.

1.4 Project Objectives

There are several objectives of this research project which is:

1. To identify the best combination of factors influence for neutralized the pH level in municipal wastewater.
2. To determine the time taken required for neutralize the pH level in municipal wastewater.

1.5 Significance of the Study

The results of this research will be used as guide to providing more efficient ways for producing water treatment. In addition elimination of non-value added activities also can be focused.

1.6 Scope and Limitations

The research focused on activities of municipal wastewater treatment through DOE approach, where the process involved a lot of adsorption experiment which required the materials such as PAC and GAC, then the experiment is randomly run based on the combination of factors that influenced the output of the process/system.

There is one limitation/barriers in this research project which is the water condition of wastewater in differences location has different pH values.

CHAPTER 2: LITERATURE REVIEW

This chapter will discussed about the existing study of the title selected. There is a vast amount of research literature journals that have been collected from the secondary data.

2.1 Municipal Wastewater

The wastewater is a combination of domestic (or sanitary) wastewater, industrial wastewater, stormwater runoff, and from sewer inflow or infiltration. Domestic wastewater refers to typical household wastes discharged from residences and from commercial and institutional facilities as well as any other wastes that people may accidentally or intentionally pour down the drain (Metcalf et al., 2013).

2.2 Characteristics of Municipal Wastewater

Physically, domestic wastewater is usually characterized by a grey colour, musty odour and has a solids content of about 0.1%. The solid material is a mixture of food particles, toilet paper, grease, oil, soap, salts, metals, detergents, sand and about 30% the solids can be suspended and 70% dissolved. Chemically, wastewater is composed of 70 % organic and 30 % inorganic compounds as well as various gases. Organic compounds consist primarily of 25% carbohydrates, 65% proteins and 10% fats. Inorganic components may consist of heavy metals, nitrogen, phosphorus, pH, sulphur, chlorides, alkalinity, toxic compounds, etc. Gases commonly dissolved in wastewater are hydrogen sulphide, methane, ammonia, oxygen, carbon dioxide and nitrogen. (Liu and Liptak, 1997).

2.3 Adsorption Mechanism

The adsorption process occurs when gas or liquid solutes gets attracted and accumulated on the absorbent, which is usually solid molecule. Adsorption can be classified into two categories; physical adsorption or van der Waals adsorption and chemical adsorption or also known as activated adsorption (Eckhard Worch, 2012). Physical adsorption (Figure 2.1) occurs when the concerned molecules adsorbate and the adsorbent are attracted together while in the chemical adsorption (Figure 2.2) occurs when the concerned molecules are attracted to the surface of the adsorbent by a strong chemical bond. The performance of an adsorption process depends on the removal percentage of the contaminants from the absorbent being used (Liu and Liptak, 1997).

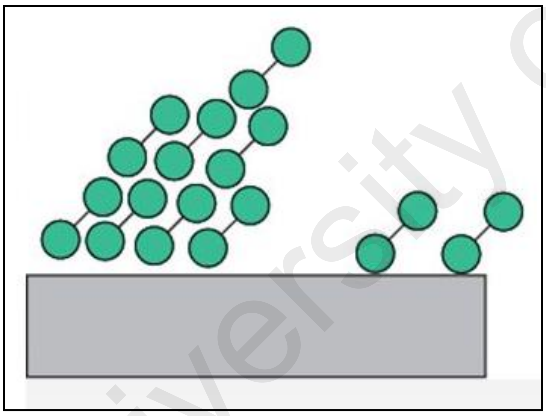


Figure 2.1: Physical Adsorption

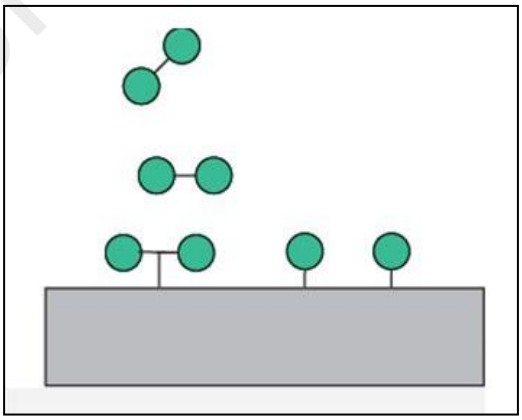


Figure 2.2: Chemical Adsorption

2.4 Activated Carbon

Adsorption of activated carbon is one of the most impressive water treatment methods that used to remove organic and inorganic contamination from any water (Björklund et al., 2015; Karelid et al., 2017), but currently has limited application for other types of contaminated water, including stormwater, which often contaminated with metals, organic pollutants and nutrients (Björklund et al., 2009, 2011; Bressy et al., 2012; Zgheib et al., 2011).

Activated carbon is also called as activated charcoal or activated coal (Tsai et al., 1997) and sometimes called as solid sponge (Zuo et al., 2012) is most powerful adsorbent which is produced from various raw materials from animal, mineral and vegetables (Khah et al., 2009; Hannafi et al., 2008; Khalkhali et al., 2005) and therefore, it presents different properties (Bonomo, 2008).The activated carbon cannot be characterized by any distinctive chemical formula (Cuhadaroglu and Uygun, 2008).

2.4.1 Characteristics of Activated Carbon

Activated carbons are applied in several forms which are granular activated carbon (GAC) with particle sizes in the range of 0.5 to 4 mm, powdered activated carbon (PAC) with particle sizes < 40 μm, extracted activated carbon (EAC) (Onyeji et al., 2011), pellet activated carbon (Wan Nik, 2006) and others. Activated carbons can be produced from different carbon-containing raw materials and by different activation processes. The most common raw materials are wood, wood charcoal, peat, lignite and lignite coke, hard coal and coke, bituminous coal, petrol coke as well as residual materials, such as coconut shells, sawdust, or plastic residuals (Eckhard Worch, 2012).

2.4.2 Types of Activated Carbon

There are two types sources of activated carbon production which is Natural Materials and agricultural by products (Lee et al., 2002). The raw materials such as coal, peat, wood, and lignite (Altenor et al., 2009) are very expensive and non-renewable (Ahmedna et al., 2000; Tan et al., 2008) but raw materials have properties of low degradation by aging, high carbon content and easy to be activated (Dias et al., 2007). Recently natural materials available in large quantities from agricultural operations have been evaluated as very inexpensive, low cost adsorbents (Sugumaran et al., 2012; Buasri et al., 2012) , effective and also environmental friendly (Khattri and Singh, 2009).

2.4.2.1 Agricultural Wastes by Products

Since sewage sludge is a carbon-rich which produced by wastewater treatment, renewable and vast amounts of resource such as almond shell, hazelnut shell, walnut shell, apricot stone (Aygun et al., 2003), cashew nut shell (Kumar et al., 2011),corncob waste (Juang et al., 2002), garlic peel (Liu et al., 2013), grape fruit peel (Torab et al., 2013),mixture almond shells (Ardejani et al., 2008) which can be obtained at low-cost adsorbents (Figure 2.3) that used to remove a wide range of contaminants from air and water. Sludge has been shown to produce high-quality carbons for adsorption of impurities in water, including metals, phenols and dyes (Smith et al., 2009; Xu et al., 2015).

2.4.2.2 Industrial Wastes by Products

As potential adsorbents for the removal of contaminants from wastewater Industrial activities generate the huge amount of solid waste materials as by-products. Therefore, the possibility of reuse in adsorption processes represents an interesting solution mainly because these industrial waste material are available almost free of cost and causes major disposal problem. In recent years, a number of industrial wastes have been investigated with or without treatment as adsorbents for the removal of pollutants from wastewaters (Bhatnagar and Sillanpaa, 2010).

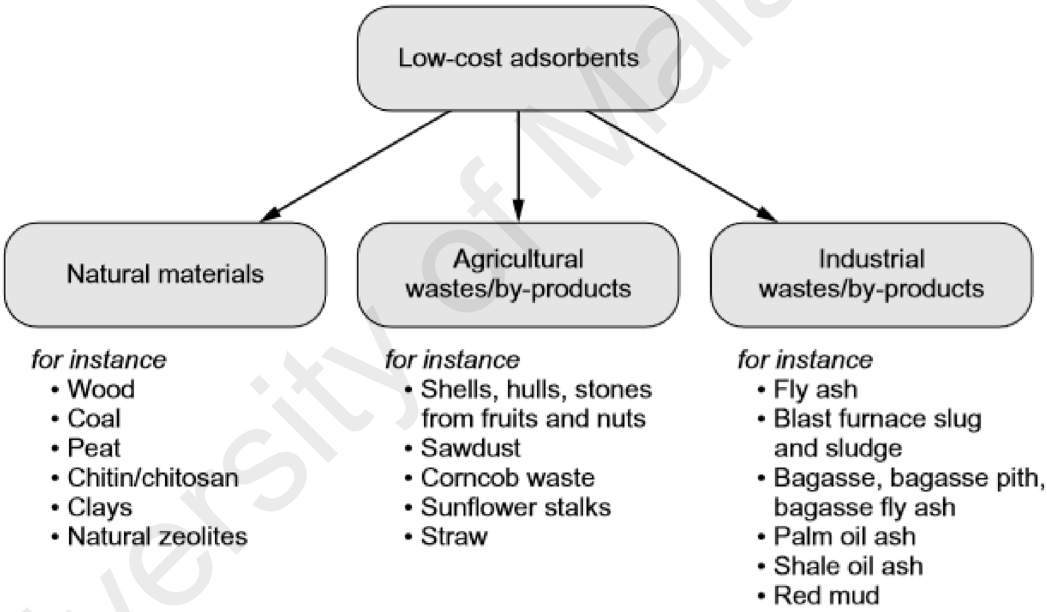


Figure 2.3: Selected Low-Cost Adsorbents

2.4.3 Applications of Activated Carbon

There are many applications o activated carbon such as water treatment (Sirichote et al., 2002) chemical and petroleum industries (Tawalbeh et al., 2005), batteries, fuel cells (Shah et al., 2006) nuclear power stations (Zabaniotou et al., 2003), electrodes for electric double layer capacitors (Kubota et al, 2009), and others. Activated carbon also can be used for decolourization, deodorization and taste removal purposes in food industries (Sahu et al., 2010). For best application of activated carbon its physical properties need to be determined. Different applications of activated carbon produced from agricultural waste materials (Table 2.1) shown below.

Table 2.1: Different Applications of Activated Carbon

Starting Material	Applications
Coconut shell	1.Reduction of hexamine cobalt 2.Adsorption of axalic acid and maleic acid
Hazelnut shell and apricot stone	Adsorption of copper
Peanut shell	Adsorption of Plumbum
Nutshell	Methylene blue adsorption
Oil palm shell	Methane adsorption
Kenaf fiber	Plumbum removal from waste water

2.5 Potential of Hydrogen (pH) Relationship with Water

The pH is importance in determining the corrosivity of water (Figure 2.5), the lower the pH then potential level of corrosion is higher which is considered acidic (Nordberg et al., 1985; Murrel, 1987; Stone et al., 1987). Generally raw sewage pH level is range between values of 5.5 to 8.0.

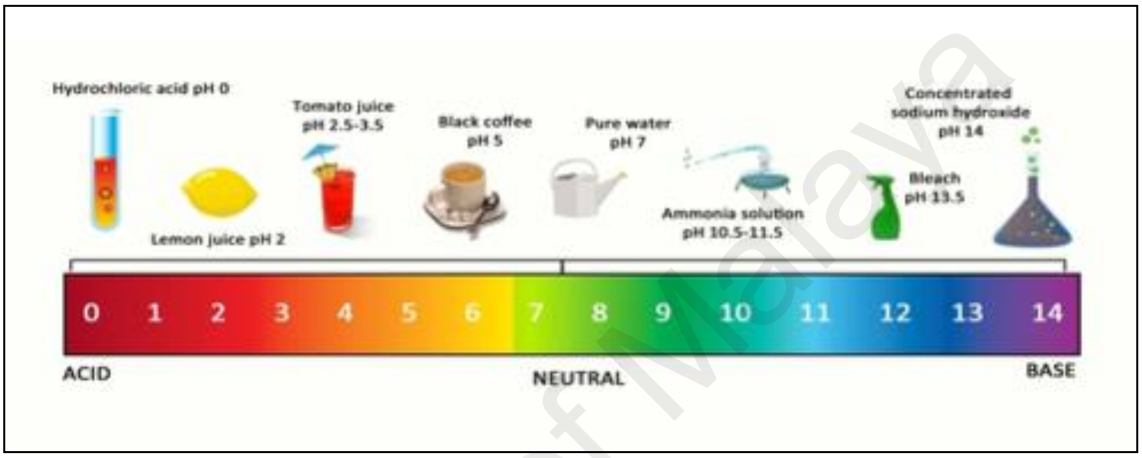


Figure 2.4: pH Scale

CHAPTER 3: METHODOLOGY

This chapter will discuss the main purpose of the research. To achieve this research on title selected several method has been applied in order to obtain as much as possible the information and data. The researchers will assess and evaluate the topic based on introduction DOE, fundamental of DOE, research instruments used, and research procedure.

3.1 Introduction of DOE

Design of experiment is the one method that introduced by Sir R.A. Fisher in early 1920, England. His primary goal was to determine the optimum water, rain, sunshine, fertilizer and soil conditions needed to produce the best crop through DOE, Fisher was able to lay out all combinations of the factors included in experimental study. The conditions were created using matrix, which allowed each factor an equal number of test conditions. He is devised the first method that made it possible to analyze the effect of more than one factor at a time.

3.2 Fundamental of DOE

Design of experiment consists series of test which the input variables are changed defer to a given rule in order to identify reasons for changes in the output response (Montgomery, 2013). Usually, noise data are involved which the results obtained can be significantly affected by noise. There are three basic principles of statistical methods in experimental design which is:

Replication	:	Repetition of experiment to obtained more precise and accurate results (mean value).
Randomization	:	Experiment runs with the random order.
Blocking	:	Isolating a known systematic bias effect and prevent it from obscuring the main effects.

To perform the DOE, the problem must be necessary identified and choose the variables which also known as factors or parameters. The fundamental of DOE consists of three terminologies (Figure 3.1) which each of them has their own function.

Input	:	Problem that needed to start the system and sustain its function.
System	:	This is the heart of the process that models your application. It consists of mechanism, parts, machines or assemblies required to make the process operation.
Factors	:	The factors which also called as controllable variables or independent variable that are requires by the system to achieve the output/objective.
Output	:	The things or objectives that want to achieved.

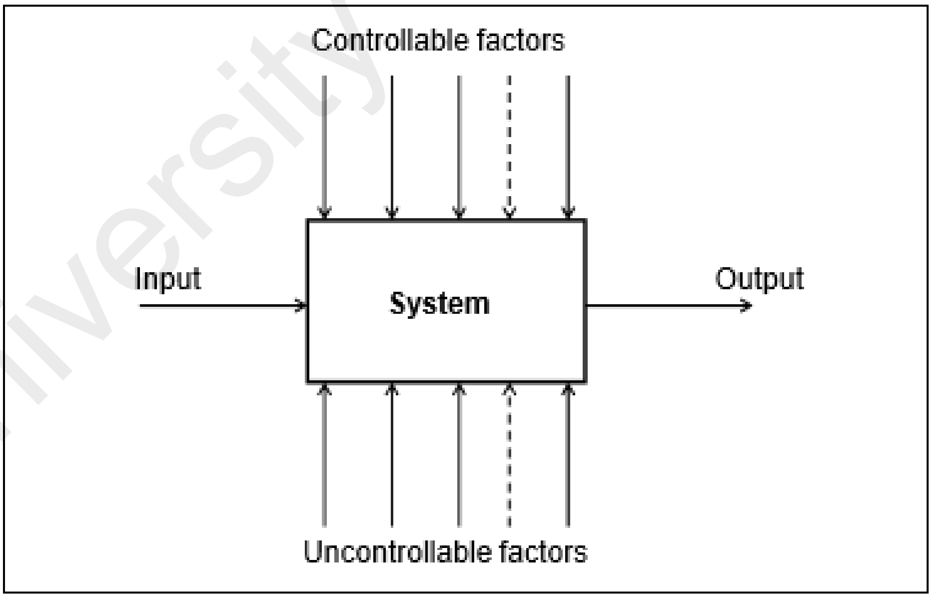


Figure 3.1: Fundamental of DOE

3.3 Research Instruments Used

The quantitative data is obtained through the conducted experiments. This project required statistical methods such as Minitab software (Figure 3.2), which it helps to analyze the data and interpret the graphical methods.

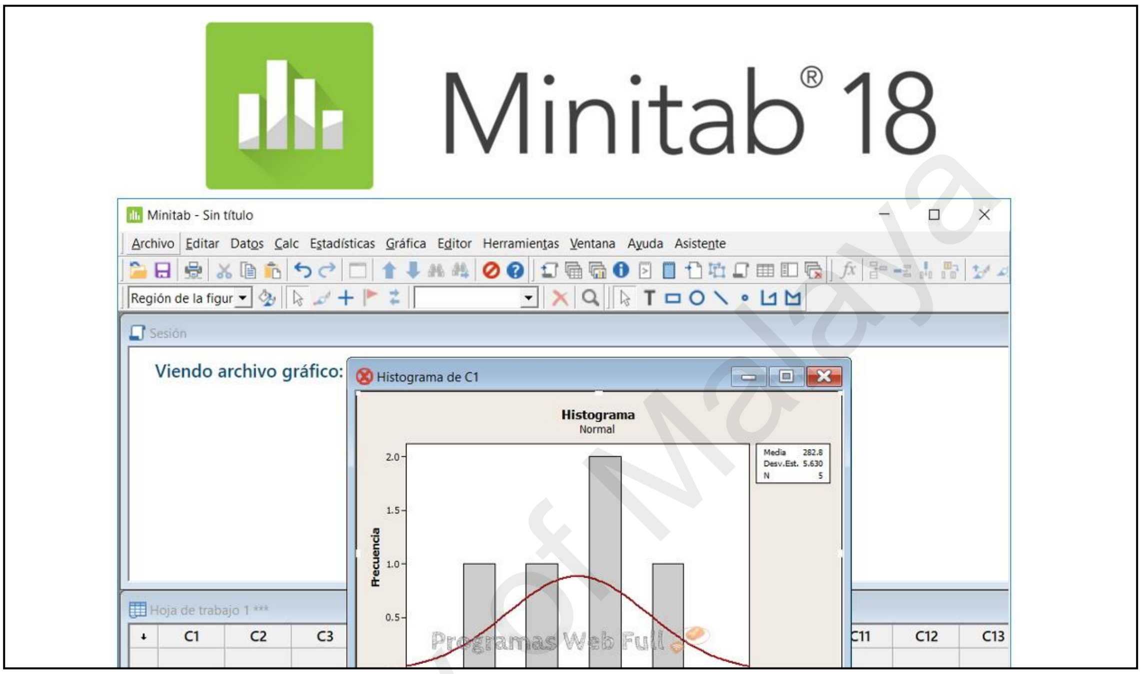


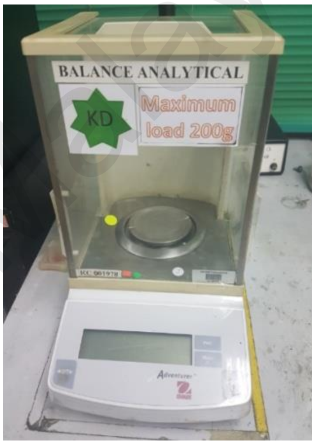
Figure 3.2: Minitab Software

3.4 Instrumentation and Material

The research project required the equipment and materials for performing the experiment which shown below (Table 3.1).

Table 3.1: Equipments and Materials

No	Equipments/Materials	
1	Activated Carbon	
2	Beaker and Conical Flask (250ml)	
3	Magnetic Stirrer (34532)	
4	pH Indicator Strips (1-14)	

5	pH Meter	
6	Magnetic Stir Bar	
7	Analytical Balance	
8	Wastewater	

3.5 Procedure for Performing the Experiment

An experiment is conducted in the Surface Laboratory which the order is randomly run according the provided worksheet (Figure 4.5).The process flow for the experiment shown below:

1. Weight the required amounts of activated carbon.
2. Prepare the beaker.
3. Measuring the level of municipal wastewater.
4. Insert the activated carbon and magnetic stirrer inside the beaker.
5. Mixing the liquid solutions by using the magnetic stirrer with the setting speed.
6. Record the time taken required for neutralized the pH level.
7. Repeat until the results (Figure 4.6) completely obtained.

3.6 Research Procedure

The research divided into two phases in implementing the project which shown below in activity process (Figure 3.3).

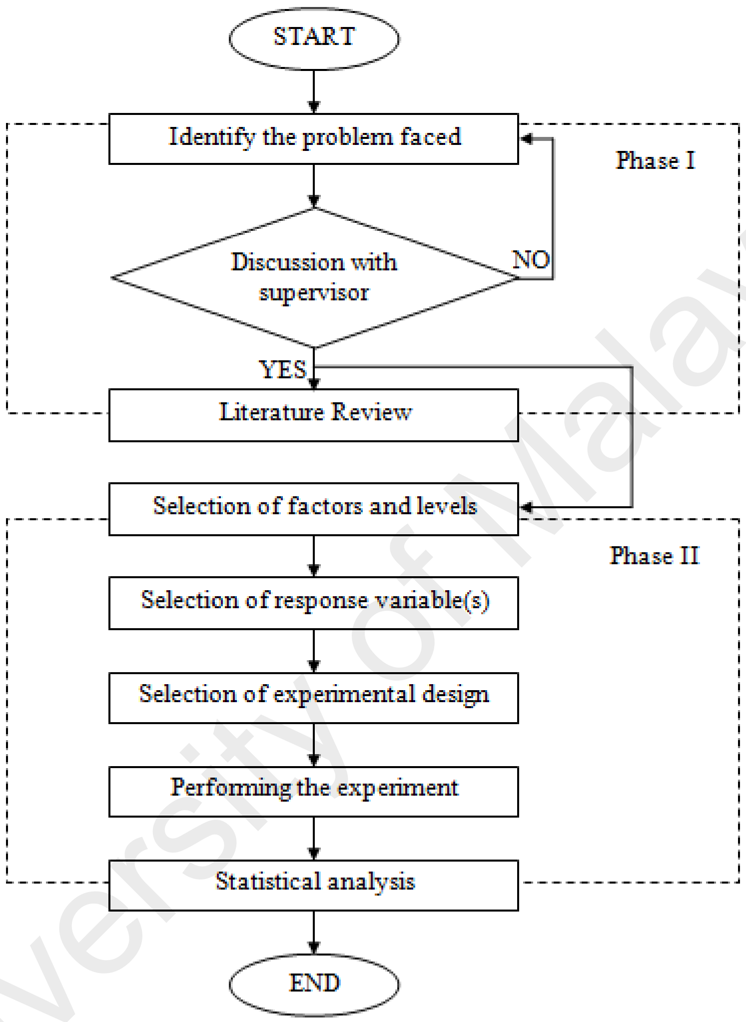


Figure 3.3: Flow Chart of Research Methodology

*(a) **Phase I***

The first phase is literature review on an application the activated carbon used in municipal wastewater which been collected from the secondary data.

(b) ***Phase II***

The second stage is more to experimental design and statistical techniques which the researcher need to run an experiment. The quantitative data obtained through the conducted experiments used together with Minitab software, the statistical tools that help the researcher to analyze the data and interpret the graphical methods in the best ways.

CHAPTER 4: FINDINGS AND DISCUSSION

This chapter will discuss about the findings obtained from the research. The researcher will interpret and explain the findings by comparing the earliest categories types of cluster methods into the both qualitative and quantitative ways, so that it is easier to understand by the researcher.

4.1 Selection of Factors and Levels

The researcher need to brainstorming the factors that related in the experiment based on past research studies. If more than 5-7 factors, screening process such as SMART concept been used to reduce the factor list, where combination of practical experiences and theoretical understanding is required to do this. Some constant factors such as raw material types need to be hold. It is usually best to keep the low number (two levels) of factor level.

SMART concept means goal setting at any level. Each of the alphabets has their own meanings which are:

Specific	:	What the goals want to accomplish?
Measurable	:	What metrics has been used to gather a valid data for further action?
Attainable	:	How the problem can be solved and accomplished?
Reliable	:	What results can realistically be achieved?
Time-based	:	Enough time to achieve the goal?

Table 4.1: Selection of Factors

No	Possible Factor	S	M	A	R	T	Result
1	Condition of water	/	/	/	/	/	Accepted
2	Types of glassware	/	/	/	/	/	Accepted
3	Level of water	/	/	/	/	/	Accepted
4	Quantity of carbon used	/	/	/	/	/	Accepted
5	Stirrer speed (rpm)	/	/	/	/	/	Accepted
6	Types of carbon	/	/	/	/	/	Accepted
7	Unpredicted surrounding	/					Rejected

There are only six factors that influence the output (Table 4.1) which is condition of water, types of glassware, level of water, quantity of carbon used, stirrer speed, and types of carbon.

4.2 Selection of Response Variables

Select an appropriate response is really important for the experiment, which it can provides useful information about the process under study and often reflects the product quality. The response should cover the input-output relationship for the process. The variables consist of two factors which each factor involved have direct influence on the output.

Controllable Factors (Table 4.2)	:	It is the main factor in the system requires achieving intended output/objectives.
Uncontrollable Factor (Table 4.3)	:	This factor also known as noise factors. These variables may influence on the output but are either unidentifiable, difficult to control or not economically to control.

Table 4.2: Controllable Factors

No.	Controllable Factors	Why	How
1	Types of glassware	To determine which are best in controlling the water?	Beaker and conical flask has been used.
2	Level of water	To determine either it influences the results.	Standard level is used. (250ml and 125ml)
3	Quantity of carbon used	Remove contamination from water.	1.25g and 2.5g
4	Stirrer speed (rpm)	To know how efficient the equipment mixing the solutions.	Different speed of stirrer is been used
5	Types of carbon	To know which the most effective.	Comparison between PAC and GAC.

Table 4.3: Uncontrollable Factor

No.	Uncontrollable Factors	Why
1	Condition of water	Difference wastewater has difference pH level depending on the location of the water taken.

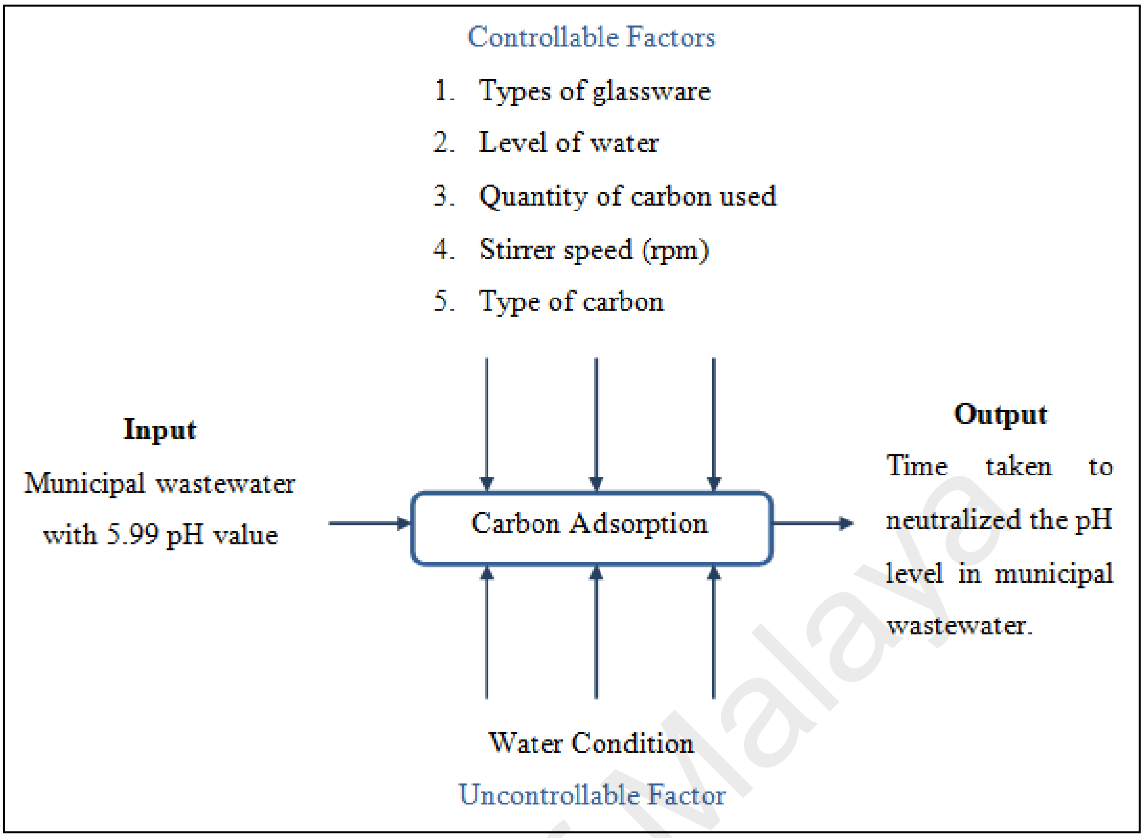


Figure 4.1: DOE Diagram

All the parameters/factors that influenced the process and the output have been interpreted in DOE diagram shows above (Figure 4.1). Next, the selection of factors and levels (Table 4.4) is required for performing the experiment.

Table 4.4: Selection of Factors and Level

		Level	
No.	**Factors**	**High**	**Low**
1	Types of glassware	Beaker	Conical Flask
2	Level of water	250ml	125ml
3	Quantity of carbon used	2.5g	1.25g
4	Stirrer speed (rpm)	560 rpm	1120 rpm
5	Types of carbon	Granular	Powdered

4.3 Experimental Design for Half Factorial

The choice of design involves consideration of sample size, number of replicates; selection of a suitable run order for the experimental trials, and whether or not blocking or other randomization restrictions are involved.

Stat → DOE → Factorial → Create Factorial Design

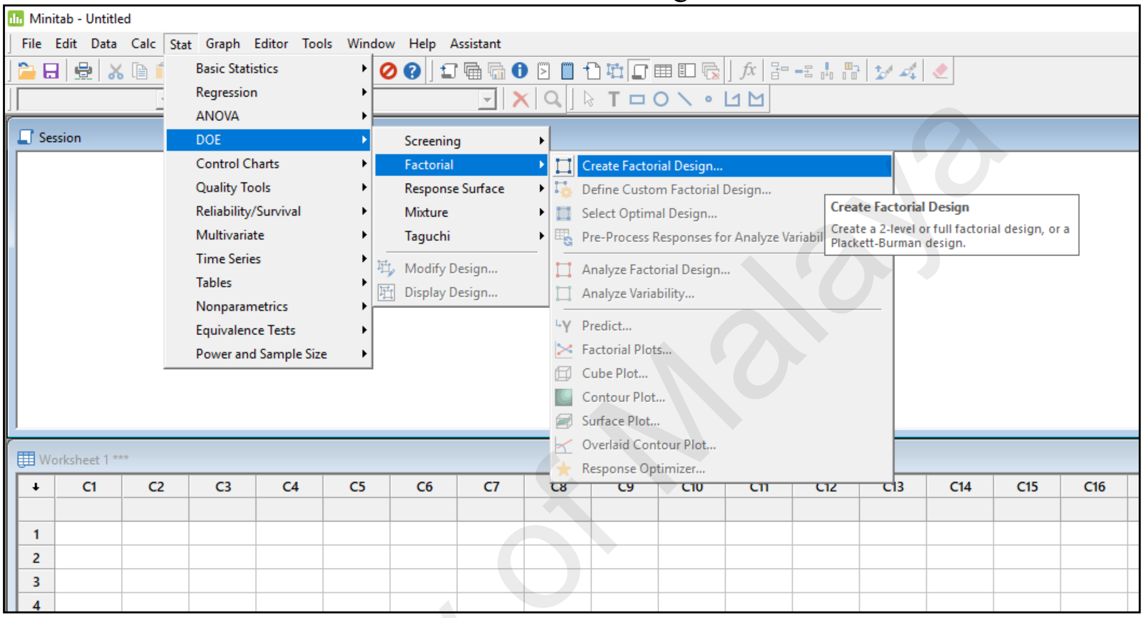
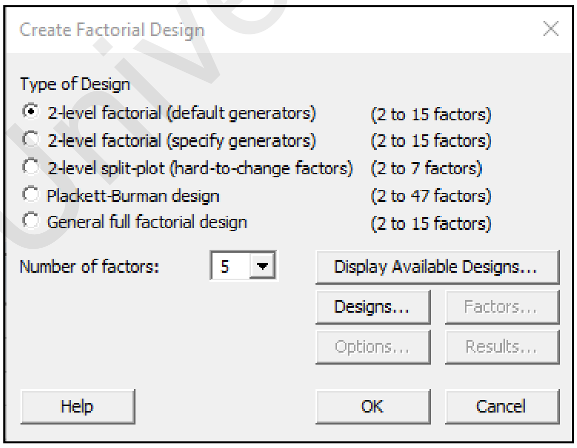


Figure 4.2: Screenshot of the Minitab Software for Half Factorial Design

Select number of factors → Click Designs → Select Designs: ¼ fraction with III resolution → OK



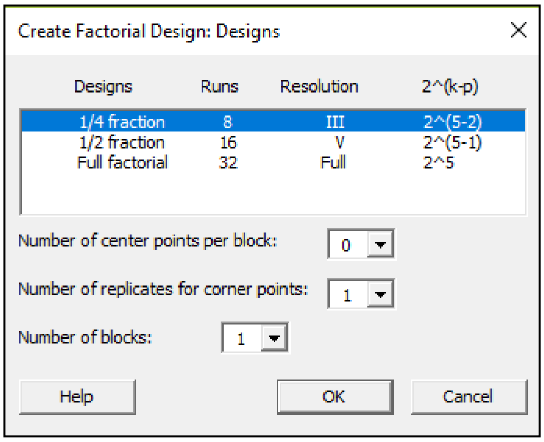


Figure 4.3: Create Factorial Design **Figure 4.4: Designs**

Design summary for the experiment that generate by the software.

Session

Fractional Factorial Design

Design Summary

Factors:	5	Base Design:	5, 8	Resolution:	III
Runs:	8	Replicates:	1	Fraction:	1/4
Blocks:	1	Center pts (total):	0		

Worksheet 1 ***

↓	C1	C2	C3	C4	C5	C6	C7	C8	C9
	StdOrder	RunOrder	CenterPt	Blocks	Types of glassware	Level of water	Quantity of carbon used	Stirrer speed (rpm)	Types of carbon
1	2	1	1	1	1	-1	-1	-1	-1
2	6	2	1	1	1	-1	1	-1	1
3	1	3	1	1	-1	-1	-1	1	1
4	3	4	1	1	-1	1	-1	-1	1
5	8	5	1	1	1	1	1	1	1
6	7	6	1	1	-1	1	1	-1	-1
7	5	7	1	1	-1	-1	1	1	-1
8	4	8	1	1	1	1	-1	1	-1

Figure 4.5: ¼ Fraction Worksheet

Worksheet 1 ***

↓	C1	C2	C3	C4	C5-T	C6	C7	C8	C9-T	C10
	StdOrder	RunOrder	CenterPt	Blocks	Types of glassware	Level of water (ml)	Quantity of carbon used	Stirrer speed (rpm)	Types of carbon	Results (minutes)
1	2	1	1	1	Beaker	125	1.25	560	Powdered	2
2	6	2	1	1	Beaker	125	2.50	560	Granular	4
3	1	3	1	1	Conical flask	125	1.25	1120	Granular	3
4	3	4	1	1	Conical flask	250	1.25	560	Granular	12
5	8	5	1	1	Beaker	250	2.50	1120	Granular	10
6	7	6	1	1	Conical flask	250	2.50	560	Powdered	5
7	5	7	1	1	Conical flask	125	2.50	1120	Powdered	1
8	4	8	1	1	Beaker	250	1.25	1120	Powdered	7

Figure 4.6: Results for ¼ Fraction

The normality test is conduct to ensure either the experiment may proceed or not. The decision of the experiment is made by calculated the normality test based on the results obtained.

P-Value > 0.05	H_o	=	Data is normal
	H_1	=	Data is not normal

Stat → Basic Statistics → Normality Test → Variable: Results → OK

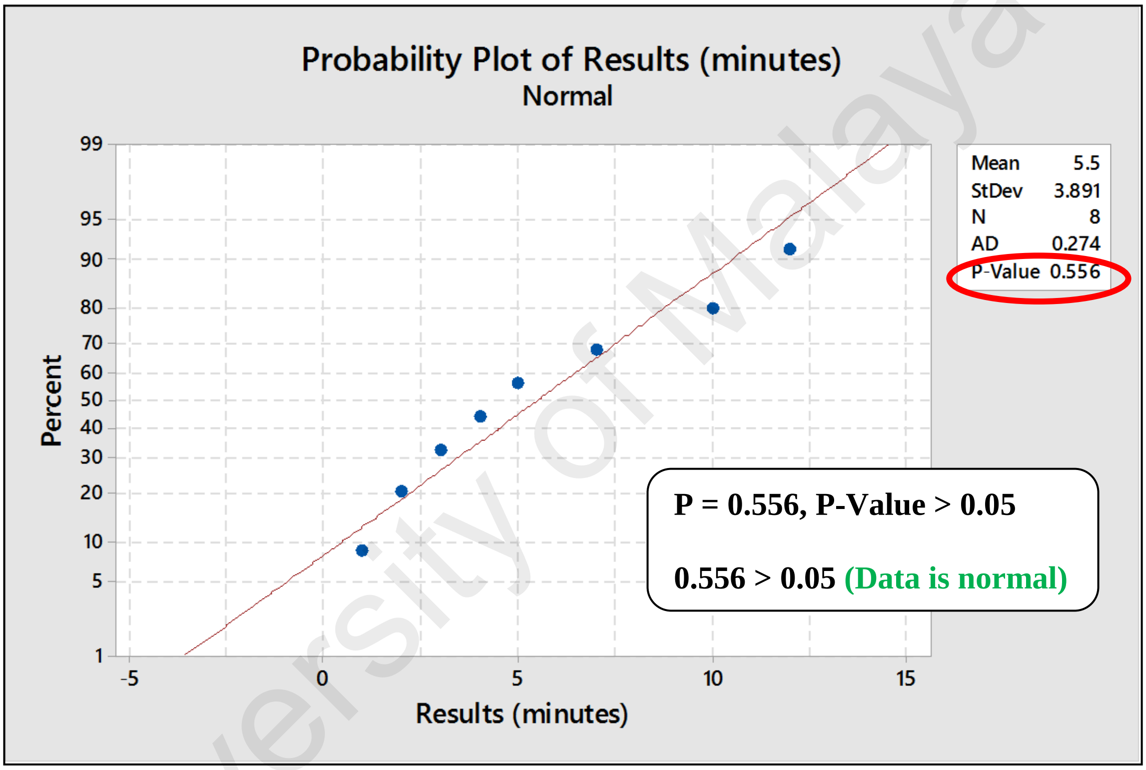


Figure 4.7: Normality Test for ¼ Design Fraction

Normality Test (Figure 4.7) shows that the P-Value is 0.556 more than 0.05, means that the Null Hypothesis is accepted and the data is normal. Next the main effect analysis has been calculated of the effect on the response solely due to one factor. Main effects are the difference between average responses at high level of the factor – average response at low level. It helps in display the variable effect.

Stat → ANOVA → Main Effects Plot → Select responses → Select factors → OK

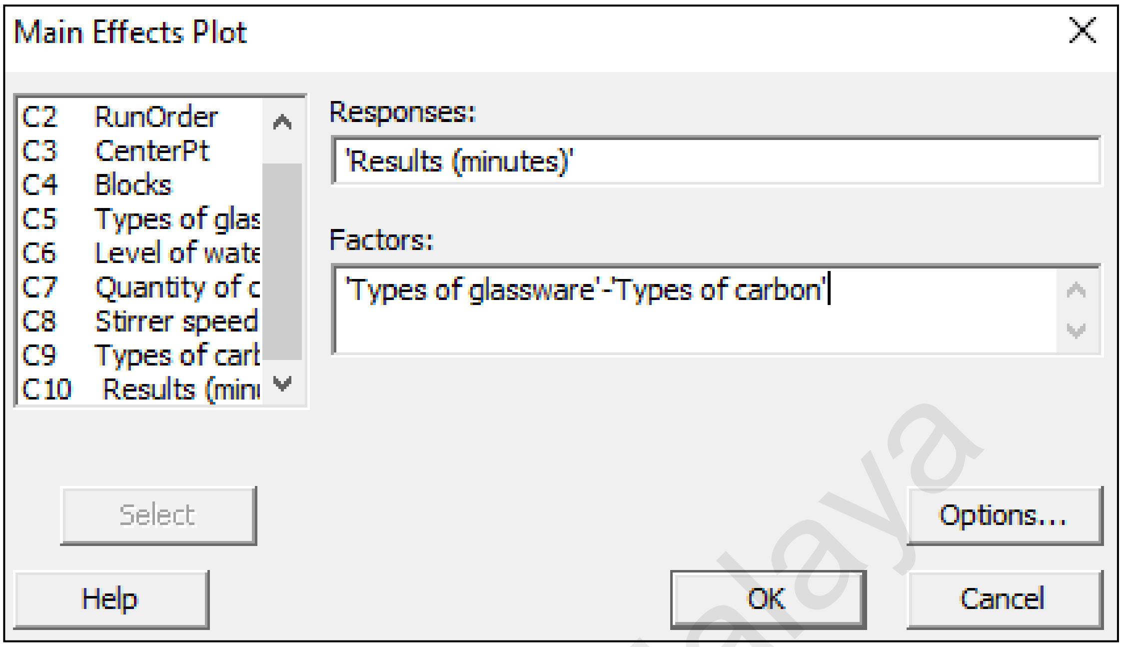


Figure 4.8: Screenshot of the Minitab Software for Main Effects Plot Selection

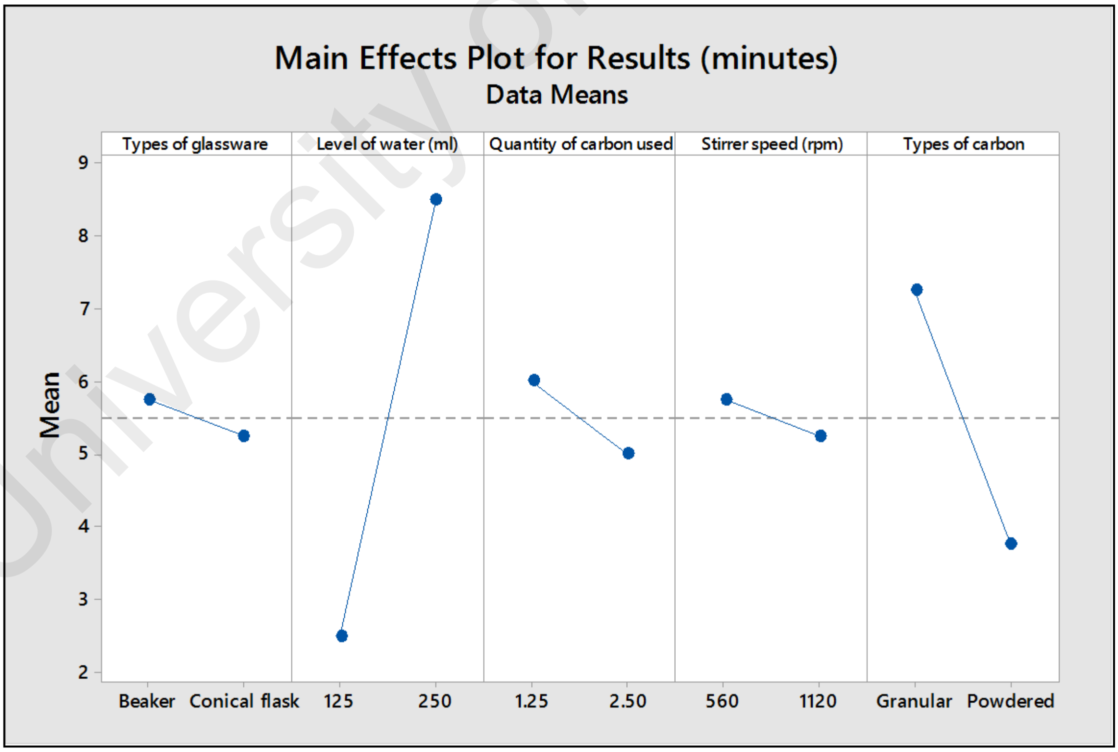


Figure 4.9: Main Effects Plot for ¼ Design Fraction

From the main effects plot (Figure 4.9), it can show which variable has the strongest effect or significant factors on the response. There are three general patterns to look for:

No Effect : Each level of the factor affects the response in the same way, and the response mean is the same across all factor levels.

Strong Effect (Positive) : Variable has the strongest effect on the response.

Strong Effect (Negative) : A negative effect means the response is reduced as the variable increases.

The analysis in the main effects plot (Figure 4.9) shows one strong positive for level of water and strong negative for the others variables. In this case, Analysis of variance is been used to select the factors according to the P-Value.

P-Value < 0.05 = Factor is significant

Stat → ANOVA → GLM → FGLM → Results → Analysis of variance

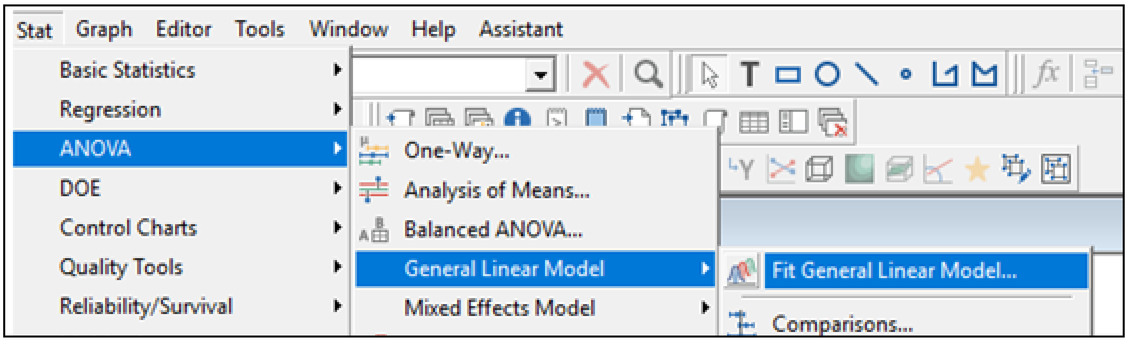


Figure 4.10: Fit General Linear Model

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Types of glassware	1	0.500	0.5000	0.15	0.733
Level of water (ml)	1	72.000	72.0000	22.15	0.042
Quantity of carbon used	1	2.000	2.0000	0.62	0.515
Stirrer speed (rpm)	1	0.500	0.5000	0.15	0.733
Types of carbon	1	24.500	24.5000	7.54	0.111
Error	2	6.500	3.2500		
Total	7	106.000			

Figure 4.11: Analysis of Variance

Figure 4.11 above shows the results which level of water are significant factor because the P-Value = 0.042 < 0.05. At least three factors are required to performing a full factorial experiment. The P-Value results above (Figure 4.11) highlight with the red circles shows two factors that near to 0.05 which is 0.515 quantity of carbon used and 0.111 types of carbon.

As the conclusion, the full factorial is conducting by three significant factors involved which is level of water, quantity of carbon used and types of carbon.

4.4 Performing Full Factorial Experiment

Stat → DOE → Factorial → Create Factorial Design → Select number of factors → Click Designs → Select Designs: Full Factorial → OK

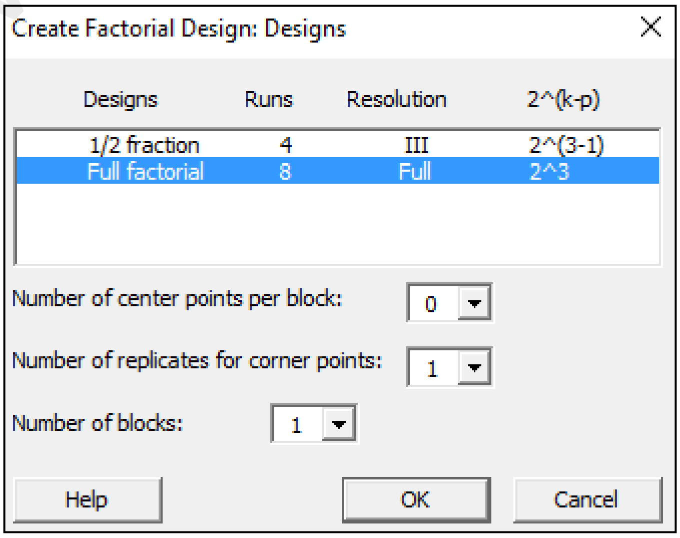


Figure 4.12: Full Factorial Designs

The experiment (Figure 4.13) is conducted by follow a Minitab worksheet, fill all the details required in the experiment. Run 8 experiment regarding to $2^n = 2^3 = 8$.

Worksheet 1 ***

↓	C1	C2	C3	C4	C5	C6-T	C7	C8
	StdOrder	RunOrder	CenterPt	Blocks	Level of water (ml)	Types of carbon	Quantity of carbon used (g)	Results (s)
1	1	1	1	1	125	Powdered	1.25	25
2	5	2	1	1	125	Powdered	2.50	30
3	6	3	1	1	250	Powdered	2.50	60
4	2	4	1	1	250	Powdered	1.25	90
5	7	5	1	1	125	Granular	2.50	140
6	4	6	1	1	250	Granular	1.25	240
7	3	7	1	1	125	Granular	1.25	180
8	8	8	1	1	250	Granular	2.50	140

Figure 4.13: Experiment Worksheet

Then, come out with normality test to confirm the data is normal. Refer to the probability plot below (Figure 4.14).

P-Value > 0.05 $\quad H_o$ = Data is normal

H_1 = Data is not normal

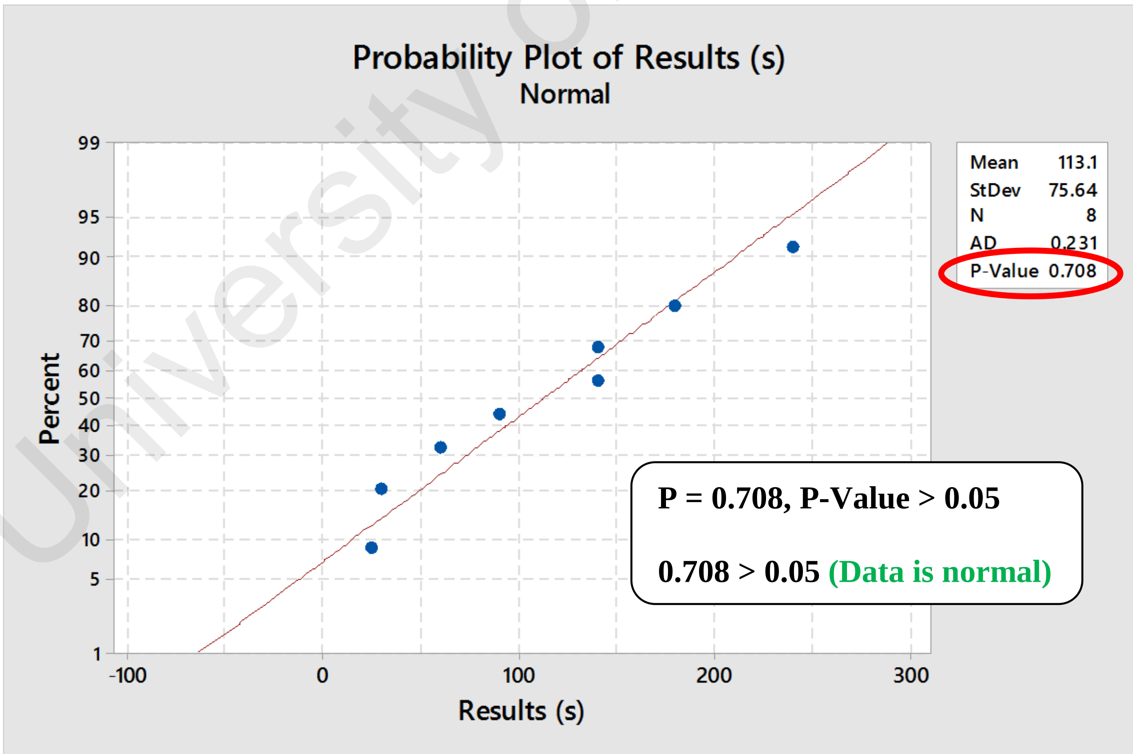


Figure 4.14: Normality Test for Full Factorial

So, Null Hypothesis is accepted because P-Value 0.708 > 0.05. The results obtained through the full factorial result shown below (Figure 4.15 and Figure 4.16).

Figure 4.15: Municipal Wastewater before Treatment

Figure 4.16: Municipal Wastewater after Treatment

4.5 Statistical Analysis

1. Construct the Hypothesis testing

H_o : μ = $\mu 0$
H_1 : μ $\neq$ $\mu 0$

Table 4.5: Hypothesis Testing

Factors	Hypothesis Testing
Level of water	H_o : $\mu = \mu 0$ H_1 : $\mu \neq \mu 0$
Quantity of carbon used	H_o : $\mu = \mu 0$ H_1 : $\mu \neq \mu 0$
Types of carbon	H_o : $\mu = \mu 0$ H_1 : $\mu \neq \mu 0$

2. P-value < 0.05= Data is normal; the factor effect is significant to the experiment. Refer to Figure 4.18 for results of P-Value.

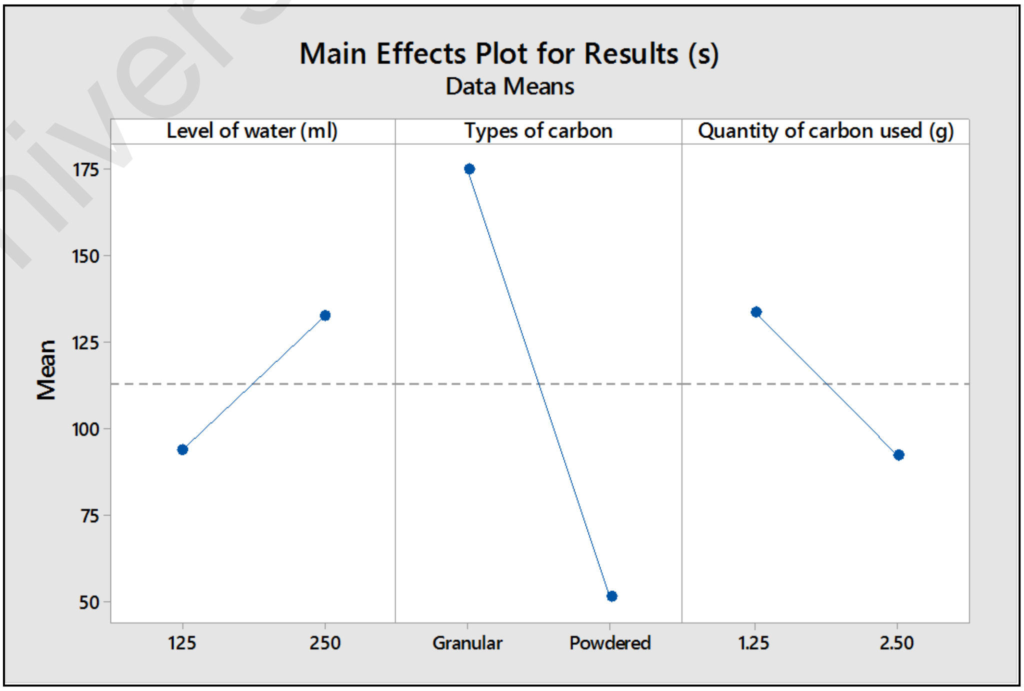


Figure 4.17: Main Effects Plot for Full Factorial

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Level of water (ml)	1	3003	3003.1	3.99	0.117
Types of carbon	1	30628	30628.1	40.67	0.003
Quantity of carbon used (g)	1	3403	3403.1	4.52	0.101
Error	4	3012	753.1		
Total	7	40047			

Figure 4.18: Analysis of Variance

Conclusion (Table 4.6) is made based on the results obtained from the Analysis of Variance (Figure 4.18).

Table 4.6: Main Effects of the Hypothesis Testing

Factors	Hypothesis Testing	Summary
Level of water	$H_o : \mu = \mu 0$ $H_1 : \mu \neq \mu 0$	P-Value = 0.117 > 0.05 Null hypothesis is rejected
Quantity of carbon used	$H_o : \mu = \mu 0$ $H_1 : \mu \neq \mu 0$	P-Value = 0.003 < 0.05 Null hypothesis is accepted
Types of carbon	$H_o : \mu = \mu 0$ $H_1 : \mu \neq \mu 0$	P-Value = 0.101 > 0.05 Null hypothesis is rejected

3. Interaction plot

The interaction plot is used to show the relationship between one categorical factor and a continuous response depends on the value of the second categorical factor.

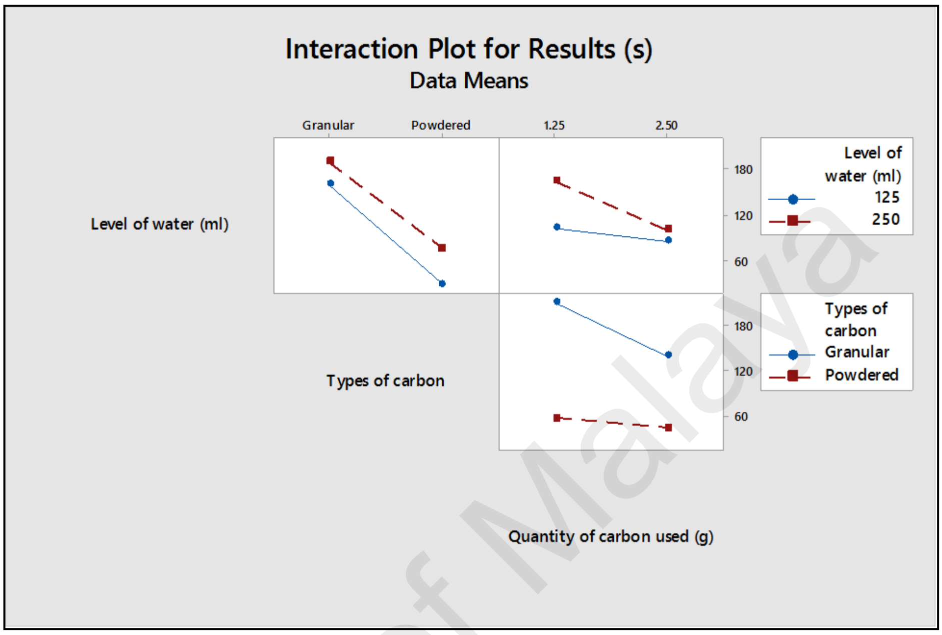


Figure 4.19: Interaction Plot

From the results obtained (Figure 4.19) shows a non- parallel interaction plot pattern.Full results shows in Table 4.7.

Table 4.7: Results of Interaction Hypothesis Testing

Factors	Hypothesis Testing			Summary
Level of water	H_o H_1	: :	Factor 1 and factor 2 is dependent Factor 1 and factor 2 is not dependent	Null hypothesis is rejected; level of water and types of carbon is not intersected.
Quantity of carbon used	H_o H_1	: :	Factor 1 and factor 2 is dependent Factor 1 and factor 2 is not dependent	Null hypothesis is rejected; quantity of carbon used and level of water is not intersected.
Types of carbon	H_o H_1	: :	Factor 1 and factor 2 is dependent Factor 1 and factor 2 is not dependent	Null hypothesis is rejected; types of carbon and quantity of carbon used is not intersected.

4. Test analysis

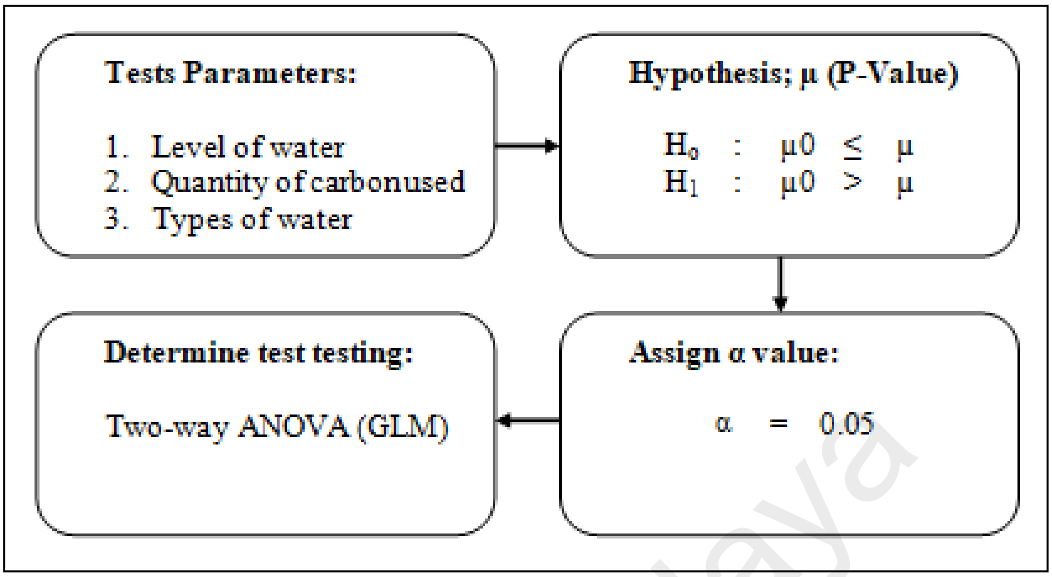


Figure 4.20: Test Analysis

GLM is use to identify if there is an interaction between the two factors with more details and specific.

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Level of water (ml)	1	3003.1	3003.1	1.92	0.238
Types of carbon	1	30628.1	30628.1	19.56	0.011
Level of water (ml)*Types of carbon	1	153.1	153.1	0.10	0.770
Error	4	6262.5	1565.6		
Total	7	40046.9			

Figure 4.21: Level of water (ml) versus Types of carbon

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Types of carbon	1	30628	30628	28.08	0.006
Quantity of carbon used (g)	1	3403	3403	3.12	0.152
Types of carbon*Quantity of carbon used (g)	1	1653	1653	1.52	0.286
Error	4	4362	1091		
Total	7	40047			

Figure 4.22: Types of carbon versus Quantity of carbon used (g)

Analysis of Variance

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Level of water (ml)	1	3003	3003	0.37	0.576
Quantity of carbon used (g)	1	3403	3403	0.42	0.553
Level of water (ml)*Quantity of carbon used (g)	1	1128	1128	0.14	0.728
Error	4	32512	8128		
Total	7	40047			

Figure 4.23: Level of water (ml) versus Quantity of carbon used (g)

From the results obtained (Figure 4.21-Figure 4.23) the conclusions is made which shown in the Table 4.8

Table 4.8: Interaction results of two factors

Factors	Hypothesis Testing	Summary
Level of water	$H_o : \mu 0 \leq \mu$ $H_1 : \mu 0 > \mu$	P-Value = 0.770 > 0.05 Null hypothesis is rejected, there is no significant between level of water and types of carbon.
Quantity of carbon used	$H_o : \mu 0 \leq \mu$ $H_1 : \mu 0 > \mu$	P-Value = 0.728 > 0.05 Null hypothesis is rejected, there is no significant between quantity of carbon used and level of water.
Types of carbon	$H_o : \mu 0 \leq \mu$ $H_1 : \mu 0 > \mu$	P-Value = 0.286 > 0.05 Null hypothesis is rejected, there is no significant between types of carbon and quantity of carbon.

5. Confirmation test

First, choose optimize setting, refer to highest reading of result highlight in red colour (Figure 4.24).

Level of water (ml)	Types of carbon	Quantity of carbon used (g)	Results (s)
125	Powdered	1.25	25
125	Powdered	2.50	30
250	Powdered	2.50	60
250	Powdered	1.25	90
125	Granular	2.50	140
250	Granular	1.25	240
125	Granular	1.25	180
250	Granular	2.50	140

Figure 4.24: Results for Confirmation Test

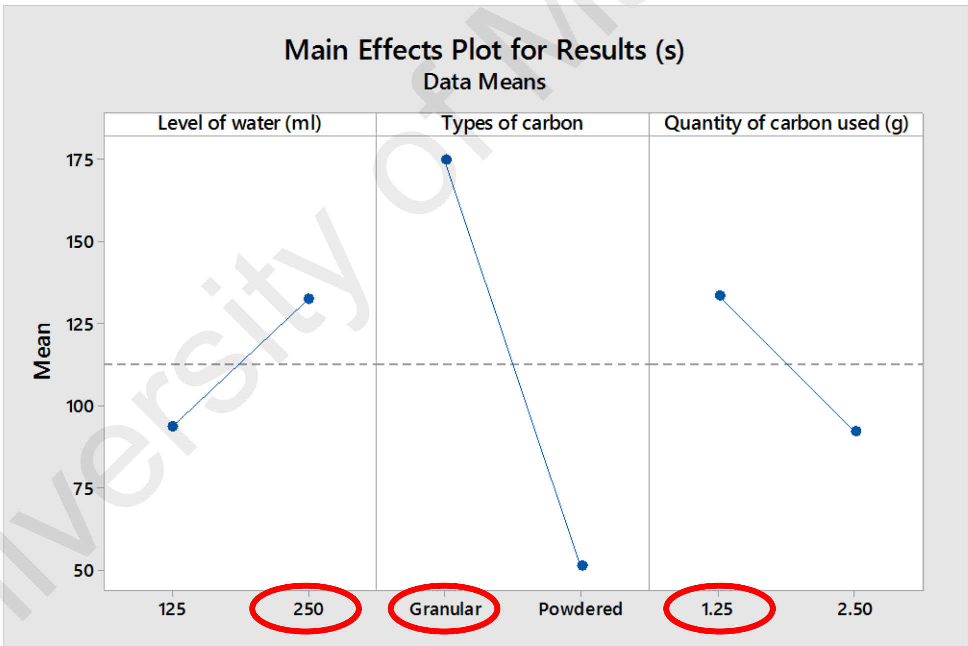


Figure 4.25: Main Effects Results for Confirmation Test

The results obtained (Figure 4.25) higlight in a red circle; 250ml level of water(municipal wastewater), Type of carbon: GAC and quantity of carbon used is 1.25gram.

Second, continue the confirmation testing by adding two more result or reading (Figure 4.24) and calculate the mean value.

	R1	:	240	
	R2	:	240	→ Mean Value:
	R3	:	240	240 + 240 + 240 +180 + 140 = 208
Add two more readings	R4	:	180	
	R5	:	140	

Third, construct T-test analysis to ensure the testing is achieved targeted value.

Hypothesis	Confident Interval (CI)	α Value
H_0 : $\mu = 208$ H_1 : $\mu \neq 208$	95	$\alpha = 0.05$

One-Sample T: T-Test

Descriptive Statistics

N	Mean	StDev	SE Mean	95% CI for μ
5	208.0	46.0	20.6	(150.8, 265.2)

μ: mean of T-Test

Test

Null hypothesis	H_0 : $\mu = 208$
Alternative hypothesis	H_1 : $\mu \neq 208$

T-Value	P-Value
0.00	1.000

Next, the tolerance design is calculated to determine Upper Specification Limit (USL) and Lower Specification Limit (LSL) of range reading.

Mean = 208, Standard deviation, 46.0 and Tolerance = 6 σ where σ = 0.0516

Specification	=	mean ± tolerance
	=	208 ± tolerance
	=	208 ± 6 (0.0516)
	=	208 ± 0.3096
	=	207.7 seconds ~ 208.3 seconds
	=	3 minutes 28 seconds

Test is fail to reject H_o, so it valid to claim the specification of means of 208.

CHAPTER 5: CONCLUSION

The problem of gaining clean water can be eliminated by following the approach of DOE. Based on the results obtained from T-test analysis, at 95% Confident Interval and $\alpha = 0.05$; neutralized pH water from the wastewater can be achieved by a combination of parameters/factors 250ml municipal wastewater adsorption with 1.25 gram of Granular Activated Carbon.

The findings obtained from the calculation of tolerance design, the naturalized pH can be archived within the 3 minutes and 28 seconds of duration period.

5.1 Recommendation

This combination of factors/parameters may be used by the others people as their guideline, where this DOE concept will focus on neutralized pH from the wastewater. Since the research study about the approach of DOE in municipal wastewater through adsorption of activated carbon which is GAC and PAC, it is recommended that further studies on this research by using another adsorption materials such as reusable carbon. This is to see and to identify the changes happen in term of time taken during the current and after implementation the different adsorption materials. Further research might explore the DOE concept of combination factors/parameters to the others types of contaminated water such as stormwater, industrial wastewater and etc.

REFERENCES

Altenor, S., Carene, B., Emmanuel, E., Lambert, J., Ehrhardt, J. J., Gaspard, S. (2019). Adsorption studies of methylene blue and phenol onto vetiver roots activated carbon prepared by chemical activation. J Hazard Mater, 165:1029–39.

Ahmedna, M., Marshall, W. E., Rao, R. M. (2000). Production of granular activated carbons from select agricultural by-products and evaluation of their physical, chemical and adsorption properties. Bioresour Technol, 71:113–23.

Ardejani, F. D., Badii, K., Limaee, N. Y., Shafaei, S., & Mirhabibi, A. (2008). Adsorption of Direct Red 80 dye from aqueous solution onto almond shells: Effect of pH, initial concentration and shell type. Journal of Hazardous Materials, 151(2-3), 730-737.

Aygun, A., Yenisoy-Karakaş, S., & Duman, I. (2003). Production of granular activated carbon from fruit stones and nutshells and evaluation of their physical, chemical and adsorption properties. Microporous and Mesoporous Materials, 66(2-3), 189-195.

Bhatnagar, A., & Sillanpaa, M. (2010). Utilization of agro-industrial and municipal waste materials as potential adsorbents for water treatment—A review. Chemical Engineering Journal, 157(2-3), 277-296.

Björklund, K., Palm Cousins, A., Strömvall, A.-M., Malmqvist, P.-A. (2009). Phthalates and nonylphenols in urban runoff: occurrence, distribution and area emission factors. Sci. Total Environ. 407 (16), 4665e4672.

Björklund, K., Strömvall, A.-M., Malmqvist, P.-A. (2011). Screening of organic contaminants in urban snow. Water Sci. Technol. 64 (1), 206e213.

Björklund, K. and L. Li (2015). "Evaluation of low-cost materials for sorption of hydrophobic organic pollutants in stormwater." Journal of Environmental Management 159(Supplement C): 106-114.

Bonomo, L. (2008). Wastewater Treatment. Italy: McGraw Hill Education.

Bressy, A., Gromaire, M.C., Lorgeoux, C., Saad, M., Leroy, F., Chebbo, G. (2012). Towards the determination of an optimal scale for stormwater quality management: micropollutants in a small residential catchment. Water Res. 46 (20), 6799e6810.

Dias, J.M., Almeida, M. F., Rivera-Utrilla J., Sanchez-Polo M. (2007). Waste materials for activated carbon preparation and its use in aqueousphase treatment: a review. J Environ Manag, 85:833–46.

Hannafi, N. E., Boumakhla, M. A., Berrama, T., Bendjama, Z. (2008). Elimination of phenol by adsorption on activated carbon prepared from the peach cores: Modelling and optimisation. Desalination, 223:264–8.

Juang, R., Wu, F., & Tseng, R. (2002). Characterization and use of activated carbons prepared from bagasses for liquid-phase adsorption. Colloids and Surfaces A: Physicochemical and Engineering Aspects, 201(1-3), 191-199. doi:10.1016/s0927-7757(01)01004-4.

Karelid, V., et al. (2017). "Pilot-scale removal of pharmaceuticals in municipal wastewater: Comparison of granular and powdered activated carbon treatment at three wastewater treatment plants. Journal Environment Management, 193: 491-502.

Khah, A. M., Ansari, R. (2009). Activated charcoal: preparation, characterization and applications: a review article. International Journal of ChemTech Research, 1(4):859–64.

Khalkhali, R. A., Omidvari, R. (2005). Adsorption of mercuric ion from aqueous solutions using activated carbon. Polish Journal of Environment Studies, 14(2):185–8.

Khattri SD, Singh MK. (2009). Removal of malachite green from dye wastewater using neem sawdust by adsorption. J Hazard Mater 167:1089–1094.

Kubota, M., Hata, A., Matsuda, H. (2009). Preparation of activated carbon from phenolic resin by KOH chemical activation under microwave heating. Carbon, 47:2805–11.

Kumar, P. S., Ramalingam, S., Kirupha, S. D., Murugesan, A., Vidhyadevi, T., & Sivanesan, S. (2011). Adsorption behavior of nickel(II) onto cashew nut shell: Equilibrium, thermodynamics, kinetics, mechanism and process design. Chemical Engineering Journal, 167(1), 122-131.

Lee, Y. W., Park, J. W., Choung, J. H., Choi, D. K. (2002). Adsorption characteristics of SO2 on activated carbon prepared from coconut shell with potassium hydroxide activation. Environ Sci Technol, 36:1086–92.

Liu, D. H., & Liptk, B. G. (1997). Environmental engineers handbook. Boca Raton, FL.

Liu, W., Liu, Y., Tao, Y., Yu, Y., Jiang, H., & Lian, H. (2013). Comparative study of adsorption of Pb(II) on native garlic peel and mercerized garlic peel. Environmental Science and Pollution Research, 21(3), 2054-2063.

Metcalf, L., Eddy, H.P., & Tchobanoglous, G. (2013). Wastewater engineering treatment and resource recovery (5th ed.). New York: McGraw-Hill Education.

Murrel NE. (1987). Impact of metal solders on water quality. In: Proceedings of the annual conference of the American Water Works Association, Part 1. Denver, CO, American Water Works Association, pp. 39–43.

Nordberg GF, Goyer RA, Clarkson TW. (1985). Impact of effects of acid precipitation on toxicity of metals. Environmental Health Perspectives, 68:169–180.

Sahu, J. N., Acharya, J., Meikap, B. C. (2010). Optimization of production conditions for activated carbons from tamarind wood by zinc chloride using response surface methodology. Bioresour Technol, 101:1974–82.

Sirichote, O., Innajitara, W., Chuenchom, L., Chunchit, D., Naweekan, K. (2002). Adsorption of iron (III) ion on activated carbons obtained from bagasse, pericarp of rubber fruit and coconut shell. Songklanakarin J Sci Technol, 24 (2):235–42.

Shah, J., Jan, M R., Mabood, F., Shahid, M. (2006). Conversion of waste tyres into carbon black and their utilization as adsorbent. J Chin Chem Soc, 53:1085–9.

Smith, K.M., Fowler, G.D., Pullket, S., Graham, N.J.D. (2009). Sewage sludge-based adsorbents: a review of their production, properties and use in water treatment applications. Water Res. 43 (10), 2569e2594.

Sugumaran, P., Susan, V. P., Ravichandran, P., Seshadri, S. (2012). Production and characterization of activated carbon from banana empty fruit bunch and Delonix regia fruit pod. J Sustain Energy Environ, 3:125–32.

Tan, A. W., Ahmad, A. L., Hameed, B. H. (2008). Preparation of activated carbon from coconut husk: optimization study on removal of 2,4,6-trichlorophenol using response surface methodology. J Hazard Mater, 153:709–17.

Tawalbeh, M., Allawzi, M. A., Kandah, M. I. (2005). Production of activated carbon from Jojoba seed residue by chemical activation using a static bed reactor. J Appl Sci, 5(3):482–7.

Torab-Mostaedi, M., Asadollahzadeh, M., Hemmati, A., & Khosravi, A. (2013). Equilibrium, kinetic, and thermodynamic studies for biosorption of cadmium and nickel on grapefruit peel. Journal of the Taiwan Institute of Chemical Engineers, 44(2), 295-302.

Thines, R. K., Mubarak, N. M., Nizamuddin, S., Sahu, J. N., Abdullah, E. C., & Ganesan, P. (2016). Application potential of carbon nanomaterials in water and wastewater treatment: A review. Journal of the Taiwan Institute of Chemical Engineers.

Tsai, W., Chang, C., & Lee, S. (1997). Preparation and characterization of activated carbons from corn cob. Carbon, 35(8), 1198-1200.

Worch, E. (2012). Adsorption technology in water treatment: fundamentals, processes, and modeling. Berlin: Walter de Gruyter.

Xu, G., Yang, X., Spinosa, L. (2015). Development of sludge-based adsorbents: preparation, characterization, utilization and its feasibility assessment. J. Environmental Management. 151, 221e232.

Zgheib, S., Moilleron, R., Chebbo, G. (2012). Priority pollutants in urban stormwater: Part 1 e case of separate storm sewers. Water Res. 46 (20), 6683e6692.

Zuo, S., Xiao, Z., & Yang, J. (2012). Evolution of gaseous products from biomass pyrolysis in the presence of phosphoric acid. Journal of Analytical and Applied Pyrolysis, 95, 236-240.