

DEGRADATION OF PHENOL BY ULTRASONIC IRRADIATION IN  
ADDITON OF SALT

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## ABSTRACT

Phenol is a potential hazard to human health and the environment. Sonication for phenol degradation has proved to be an attractive process over the years but percentage of phenol degradation under sonication have always been quite low and the use of catalyst also is not cost effective method. The degradation of phenol in aqueous solution was investigated by use of a 60 kHz ultrasonic reactor and all solutions volume was 100mL. Experiments were performed at initial phenol concentrations varying from 20 to 50 mg/L and different temperature in the range of 40-90°C. The pH of solution ranging from 1-4 was also adjusted by using hydrochloric acid. The effects of parameters such as initial phenol concentration, pH value, and temperature on the degradation have been studied. This research also investigated the use of simple additives such as salt on phenol degradation with an aim of cost effective method. The amount of salt used in all parameter effects was 100mg. There was comparison of phenol degradation with salt and without salt by all study effects and the phenol degradation also have been studied. The higher percentage of phenol degradation was at 20mg/L of initial concentration where the percentage of degradation was 10.2% phenol degraded without salt and 14.4% phenol degraded with salt and at pH 1 which is 28.3% phenol degraded for no salt added and 31.4% for salt added. The optimum temperature was at 70°C with 17.3% of degradation without salt and 21.3% of degradation with salt. The percentage of phenol degradation was increasing with increasing the amount of salt added. The higher percentage of degradation was 47.2% when 500mg salt was added. The results of the study showed that the ultrasonic phenol degradation increased with decreasing the pH values and initial phenol concentration and also increasing the temperature of reaction In the presence of additives, the phenol is converted into

several intermediates, which are degraded faster than phenol itself so it leads to the increasing of percentage of phenol degradation.

## ABSTRAK

Phenol adalah berpotensi membahayakan kesehatan manusia dan persekitaran. Kaedah sonikasi untuk mendegradasi phenol telah terbukti merupakan proses yang menarik sejak beberapa tahun tetapi peratusan degradasi phenol melalui sonikasi agak rendah dan penggunaan pemangkin juga bukan merupakan kaedah yang berkesan untuk mengurangkan kos. Degradasi phenol dalam larutan air diselidiki dengan menggunakan reaktor ultrasonik 60 kHz dengan menggunakan larutan sebanyak 100 ml. Percubaan dilakukan pada berbagai kepekatan awal phenol iaitu 20-50 mg / L dan suhu yang berbeza dalam julat 40-90°C. pH larutan antara 1-4 juga disesuaikan dengan menggunakan asid hidroklorik. Pengaruh parameter seperti kepekatan awal phenol, nilai pH, dan suhu terhadap degradasi telah dikaji. Kajian ini juga meneliti penggunaan aditif yang sederhana seperti garam pada degradasi phenol dengan tujuan mengurangkan kos secara berkesan.. Jumlah garam yang digunakan dalam semua kesan parameter adalah 100mg. Terdapat perbandingan degradasi phenol dengan garam dan tanpa garam oleh semua kesan kajian dan degradasi phenol juga telah dikaji. Peratusan yang lebih tinggi dari degradasi phenol berada di 20mg/L kepekatan awal di mana peratusan 10.2% phenol terdegradasi tanpa garam dan 14.4% phenol terdegrasi dengan garam dan pada pH 1, 28.3% phenol terdegrasi tanpa garam dan 31.4% phenol terdegrasi ditambah garam. Suhu optimum pada 70°C dengan 17.3% phenol terdegradasi tanpa garam dan 21.3% phenol terdegradasi dengan garam. Peratusan degradasi phenol meningkat dengan meningkatnya jumlah garam ditambah. Peratusan yang lebih tinggi dari degradasi 47.2% ketika garam 500mg ditambah. Keputusan kajian menunjukkan bahawa degradasi phenol ultrasonik meningkat dengan menurunnya nilai pH dan kepekatan awal fenol dan juga meningkatkan suhu reaksi Dengan adanya aditif, fenol tersebut diubah menjadi beberapa

perantara, yang terdegradasi lebih cepat dari phenol itu sendiri sehingga boleh menyebabkan berlakunya peningkatan peratusan degradasi phenol.

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**LIST OF SYMBOLS/ABBREVIATIONS**

A	- Absorbance
C <sub>6</sub> H <sub>5</sub> OH	- Phenol
g	- Gram
HCL	- Hydrochloric Acid
kHz	- Kilo Hertz
L	- Litre
mg	- Miligram
mL	- Mililitre
min	- Minute
NaCl	- Sodium Chloride
W	- Watt
%	- Percentage
°C	- Degree Celcius

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## CHAPTER ONE

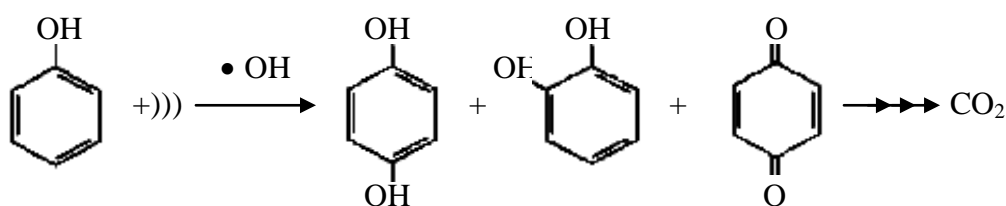
### INTRODUCTION

#### 1.1 Background of Study

Wastewater from the industries has to be treated because it causes toxicity and foul odor to the water that can be a potential hazard to human health and the environment. Phenol is the one of the common and important hazardous chemical compounds in wastewater. Its high stability and solubility in water is the main reasons why the degradation of this compound to acceptable levels is a relatively difficult process. Phenol can be found in aqueous effluents from various industries such as the manufacturing of resins and plastics, petroleum refining, steel production, coal gasification and conversion, surface runoff from coal mines, byproducts of agricultural chemicals, dyestuff, textiles, tanning, fiberboard production and paint stripping operations, pulp and paper, pesticides, medications, pharmaceuticals and even from food processing industries (Lesko, 2004; Entezari and Petrier, 2004; Entezari and Petrier, 2005; Mahamuni and Pandit, 2005; Lathasreea *et al.*, 2004; Beltran *et al.*, 2005). Phenol is one of the most abundant pollutants in industrial wastewater (Alnaizy and Akgerman, 2000; Maleki *et al.*, 2005). Even though the concentration of phenol in wastewater is low, it is considerable as toxicity. It has to be handled with great care because it causes immediate white blistering to the skin. The major hazard of phenol is its ability to penetrate the skin rapidly, particularly when liquid, causing severe injury which can be fatal. Phenol also has a strong corrosive effect on body tissue causing severe chemical burns. This compound has attracted public attention because of its exposure that can result acute and chronic effects to human health (Entezari *et al.*, 2003). Due to its toxicity, hazardous character and increasing social concern on

environment, the discharge of such compounds in the environment has become more restrict. Therefore, there is more demand for improved methods of treatment (Entezari and Petrier, 2004; Beltran et al., 2005).

Ultrasonic irradiation is a novel advanced oxidation process that has emerged as an answer to the growing need for lower levels of contaminants in wastewater (Wu et al., 2001; Nikolopoulos et al., 2005). Nowadays, the application of ultrasonic (US) in wastewater treatment has attracted great interest. It has been proposed as one of the alternative techniques for degradation of hazardous organic compounds. Ultrasonic technology as an innovative technology may be used for water and wastewater treatment for pollution removal. The basis for the present-day generation of ultrasound was established as far back as 1880 with the discovery of the piezoelectric effect by the Curies (Gelate P and Honett M, 2000). Cavitations phenomenon was first identified and reported in 1895 (Thornycroft and Sydney B, 1895). Ultrasonic irradiation results in the formation and collapse of micro scale bubbles and generating local high temperature. The bubbles are thought to work as the reaction field and the local high temperature caused by the collapsed bubbles forces the decomposed water to generate hydroxyl radicals, which promotes the degradation reaction of the organic compounds. Phenol and its chloro/nitro derivatives are largely soluble in water so that the main reaction site for their destruction during ultrasonic irradiation is the bulk liquid, where the attack of hydroxyl radicals on the ring carbons results in various oxidation intermediates. After sufficiently long contact times mineralization occurs. The sonochemical degradation of phenol and the major intermediates as detected by HPLC analysis is as shown in Figure 1.1 (Berlan, Tarbelsi and H Delmas, 1994);



**Figure 1.1** Structure of Degradation of Phenol

Phenol has been listed as the priority pollutant in the list of EPA (USA). Most of the countries specify the maximum allowable concentration of phenol in the effluent streams to be less than 1 mg/L (Mahamuni and Pandit, 2005). But degradation of phenol to such low levels has not been possible by conventional biological treatment processes. Many process such as membrane separation, wet air oxidation, radiolysis, Fenton oxidation, photocatalytic oxidation, electrochemical oxidation, ozonation, peroxidation and sonication had been used in the past for the degradation of phenol to the required low levels (Entazari *et al.*, 2003; Gogate *et al.*, 2004). These processes had been used alone or in combination with other processes to achieve the aim. But all these processes have their inherent limitations such as low rates of degradation, or lower mineralization or high costs of operation or severe operating conditions. Sonication for phenol degradation has proved to be an attractive process in terms of its normal operating conditions and lower costs. But the rates of phenol degradation under sonication have been very low. There had been some efforts to increase the rates of phenol degradation using hybrid techniques (Nafrechoux *et al.*, 2000; Esplugas *et al.*, 2002). Enhancement of ultrasonic degradation rates of phenol using simple additives is a simple but effective method for increasing the rates of degradation. It have shown and proved that the rates of ultrasonic degradation of phenol can be increased substantially using simple sodium chloride addition to the aqueous phenol solution (J.D Seymore and R.B Gupta, 1997).

## **1.2 Problem Statement**

Even in low concentration, phenol is considered as toxicity and improper handling of these compounds and/or inadequate discharge of their wastes result in long-term deterioration of the water environment and imposes considerable risk on all life forms because of their suspected carcinogenic properties. When hazardous compound spreading to environmental, more energy is needed to treat it resulting to high of energy consumption and it leads to the high of cost operation. When treating the hazardous compound from discharge of waste water, energy that will be

used is low and cost of operation will be low better than when it spread to the river which can be pollutants.

There are several problems from previous research that attributes to low percentage of phenol degradation. By doing this research, there are some improvements to achieve the high percentage of phenol degradation with the parameter such as initial concentration, pH value, temperature and amount of salt added. Furthermore, previous researches are used catalyst as an additive and this is not cost effective method because catalyst is expensive. In this research, cost will be least expensive by using salt as an additive in increasing the percentage of phenol degradation.

### **1.3 Objectives**

Based on the background of this study, the objective is listed as following:

To obtain the optimum condition in phenol degradation using ultrasonic irradiation with cost effective method in term of addition of salt.

### **1.4 Scope of Study**

Based on the objectives of this study, the scopes of study are highlighted as follows:

- i To study the parameter that affects the percentage of phenol degradation using ultrasonic irradiation such as initial concentration, pH value, temperature and amount of salt added.
- ii To compare the percentage of phenol degradation on ultrasound with salt and without salt

## **1.5 Contribution**

- i Prevent the high of phenol concentration release which can causes toxicity and foul odor that can be potential hazard to human health and environmental.
- ii Reduce the energy consumption by treating from the discharge of wastewater rather than it release in environmental.
- iii Improving current research by increasing the percentage of phenol degradation with optimum condition.
- iv Lowering the cost by using salt as an influence on phenol degradation (cost effective method).



## CHAPTER TWO

### LITERATURE RIVIEW

#### 2.1 Introduction of Ultrasonic Irradiation

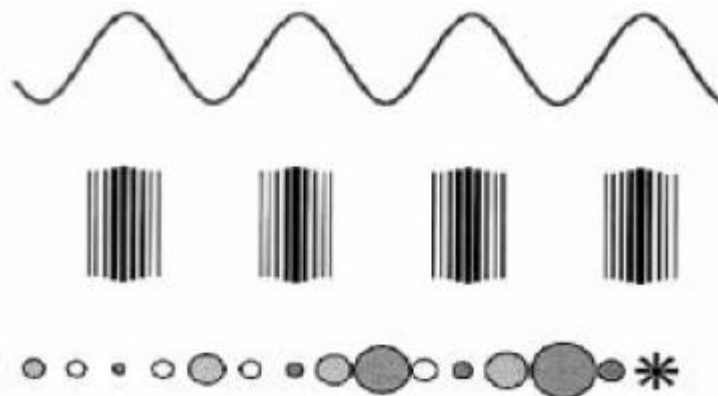
Ultrasound irradiation is a novel advanced oxidation process that has emerged as an answer to the growing need for lower levels of contaminants in wastewater (Wu *et al.*, 2001; Nikolopoulos *et al.*, 2005). Destruction of microorganisms by ultrasonic has been of considerable interest since 1920's when studies of Harvey and Loomis were published. They showed that heating injure the bacteria, but ultrasonic appeared to have a greater effect (Harvey EN and Loomis L, 1998). Since 1945, an increasing understanding of the phenomenon of cavitation has developed coupled with significant developments in electronic circuitry and transducers (i.e. devices which convert electrical to mechanical signals and vice versa). As a result of this there has been a rapid expansion in the application of power ultrasound to chemical processes, a subject that has become known as "Sonochemistry" (Gelate P and Honett M, 2000; Suslick, 1994).

In the 1960's, research concentrated on understanding the mechanisms of ultrasonic interaction with microbial cells. Cavitation phenomenon and associated shear disruption, localized heating and free radical formation were found to be contributory causes (Everett WC 1978). By 1975 it was shown that brief exposure to ultrasonic lead to thinning of cell walls which was attributed to release cytoplasm membrane from the cell wall. Fecal coliforms inactivation most likely results from a combination of physical and chemical mechanisms which occur during acoustic cavitation, so it is expected that higher intensities will enhance inactivation rates.

The correlation of chemical reaction rates and ultrasonic intensity has been reported previously. However, for most processes, increase in process rate not continues with higher sound intensities (Neppiras EA, 1980; Scherba G and Weigel, 1991). Since 1990, several studies have focused on the use of ultrasound to remove organic xenobiotics from water (Entezari MH, Petrier C and Devidal P, 2003).

## 2.2 Sound theory

Most modern ultrasonic devices rely on transducers which are composed of piezoelectric materials. Such materials respond to the application of an electrical potential across opposite faces with a small change in dimensions. This is the inverse of the piezoelectric effect. If the potential is alternated at high frequencies, the crystal converts electrical energy to mechanical vibration (sound) energy. At sufficiently high alternating potential, high frequency sound (ultrasound) will be generated. When more powerful ultrasound at a lower frequency is applied to a system, it is possible to produce chemical changes as a result of acoustically generated cavitation (Gelate P and Honett M, 2000; Suslick, 1994). Frequencies above 18 kHz are usually considered to be ultrasonic. The frequencies used for ultrasonic cleaning, range 20 kHz to over 100 kHz. The most commonly used frequencies for industrial cleaning are those between 20 and 50 kHz (Suslick, 1999; Entezari, 2003; Zheng W, 2004). Ultrasound has wavelengths between successive compression waves measuring roughly 10 to  $10^{-3}$  cm. These are not comparable to molecular dimensions as shown in Figure 2.2. Because of this mismatch, the chemical effects of ultrasound cannot result from a direct interaction of sound with molecular species (Suslick, 1999, Gong C and Hart DB, 1998).



**Figure 2.2:** Compression and expansion cycle of ultrasound (Suslick, 1999; Entezari, 2003; Zheng W, 2004)

### 2.3 Bubble cavitation

Ultrasound reactor technology (USRT) in a liquid leads to the acoustic cavitation phenomenon such as formation, growth, and collapse of bubbles (cavitation), accompanied by generation of local high temperature, pressure, and reactive radical species ( $^{\circ}\text{OH}$ ,  $^{\circ}\text{OOH}$ ) via thermal dissociation of water and oxygen. These radicals penetrate into water and oxidize dissolved organic compounds. Hydrogen peroxide ( $\text{H}_2\text{O}_2$ ) is formed as a consequence of  $^{\circ}\text{OH}$  and  $^{\circ}\text{OOH}$  radical recombination in the outside of the cavitation bubble (Jiang Wai *et al*, 2002; Visscher AD and Langenhove 1998). Concentration of  $^{\circ}\text{OH}$  at a bubble interface can be as high as  $4 \times 10^{-3}$  M, which is 108-109 times higher than that in the other advanced oxidation processes. Pyrolysis of pollutants could lead to radical formation and starting chain reactions.

The basis for ultrasound irradiation applications is that acoustic cavitation can create a number of mechanical, acoustical, chemical and biological changes in a liquid (Laborde, 1998; Louterbone W, 1997). Bubbles form, grow and subsequently collapse through compression-rarefaction cycles. Temperature in collapsing bubbles can reach to 3000- 5000°K and pressure to 500-10000 atm. Under such extreme conditions, water molecules undergo homolysis to yield hydroxyl radicals and

hydrogen atoms. Since oxidation by hydroxyl radical is an important degradation pathway, amount of the hydroxyl radicals present in the sonolysis system is directly related to the degradation efficiency (Zheng W and Maurin, 2005). There are two main mechanisms in sonolysis system for pollutant decomposition:

-Pyrolysis reactions in cavitation bubbles

-Radical reactions by radical species ( $^{\circ}\text{H}$ ,  $^{\circ}\text{OH}$ ) from water sonolysis.

These two mechanisms are as below;



In elastic media such as air and most solids, there is a continuous transition as a sound wave is transmitted. In non-elastic media such as water and most liquids, there is continuous transition as long as the amplitude or loudness of the sound is relatively low (AH Mahvi, 2009). As amplitude is increased the magnitude of the negative pressure in the areas of rarefaction eventually becomes sufficient to cause the liquid to fracture because of the negative pressure, causing a phenomenon known as cavitation. Cavitation bubbles are created at sites of rarefaction as the liquid fractures or tears because of the negative pressure of sound waves in the liquid. As the wave fronts pass, the cavitation bubbles oscillate under influence of positive pressure, eventually growing to an unstable size (AH Mahvi, 2009). Finally the violent collapse of the cavitation bubbles results in implosions, which causes radiation of shock waves from the sites of the collapse. The collapse and implosion of myriad cavitation bubbles throughout an ultrasonically activated liquid result in the effect commonly associated with ultrasound (Hua I and Hoffman, 1997; Kalumuk K, 2003). Thus, sonochemical destruction of pollutants in aqueous phase generally occurs as the results of imploding cavitation bubbles and involves several reaction pathways and zones such as pyrolysis inside the bubble and/or at the bubble-liquid interface and hydroxyl radical-mediated reactions at the bubble liquid interface and/or in the liquid bulk (Vassilakis and Pantidou, 2004).

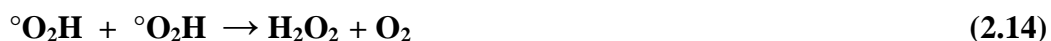
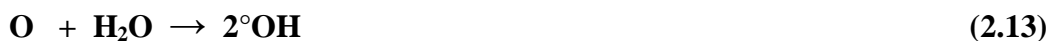
## **2.4 Advantages and Disadvantages of Ultrasound**

There are no additives introduced into the ultrasonic system and no by products generated by ultrasonic technology. Therefore, there are no anticipated environmental concerns associated with this technology (Buchholz, Tanis D and Macomber, 1998). In contrast to many other processes which are negatively affected when suspended solids of effluent increase, US efficiency may even improve by increase of turbidity or suspended solids (Manson T and Lorimer, 2002). Although the technology has been shown to be feasible on a small scale, the commercialization of sonolysis is still a challenge, due to the high energy requirement of the process (Crittenden JC *et al.*, 2004).

## **2.5 Applications of ultrasound in phenolic effluents treatment**

Phenols are widely consumed in the industry as preservatives in paint, leather and textile goods, and in the production of phenolic resins, disinfectants, medicine, caprolactam and bisphenol A. Improper handling of these compounds and/or inadequate discharge of their wastes result in long-term deterioration of the water environment and imposes considerable risk on all life forms because of their suspected carcinogenic properties (J.H Kim *et al.*, 2002). Sonochemistry is a field, which studies the enhancement of chemical reactions and mass transfer rates under various ultrasonic conditions. It is based on the fact that when a liquid is exposed to a sufficiently large acoustic field, the pressure waves lead to the formation of liquid voids or cavitation holes, which contain small quantities of dissolved gases and vapour from the surrounding medium (Suslick, 1997). As these microbubbles expand during the rarefaction cycles of the pressure waves, they ultimately become too large to sustain themselves and undergo violent implosions with the release of very extreme temperatures and pressures at “local hot spots” in the liquid (Suslick, Hemmerton and Cline, 1990). Under these conditions, gas molecules entrapped in the cavitation bubbles are thermally fragmented (by pyrolysis) to dissociate into a variety of short-lived energetic free radical species.





In the case of ozone injection into solution, excess hydroxyl radicals are generated by decomposition of ozone in the aqueous (at pH > 9.5) and gas phases as shown in equation below (Serpone *et al.*, 1994; J.W Kang *et al.*, 1998; Weavers *et al.*, 1998):



## 2.7 Sonochemical degradation of phenol and its derivatives: impact of operational parameters

The study of Berlan *et al.* with 20 and 541 kHz irradiation in the presence of varying dissolved gases, catalysts and hydrostatic pressure showed that regardless of the reaction conditions 20 kHz was ineffective in phenol decomposition, while they could detect oxidation intermediates such as di-hydroxybenzene and quinone in effluents sonicated at 541 kHz (J Berlan *et al.*, 1994). They also reported that pyrolytic destruction of phenol in the gas phase is negligible; the degradation occurs mainly in the bulk solution and is accelerated with the addition of Fenton's reagent. In other study by Petrier and Francony *et al.*, where the relative efficiencies of 20, 200 and 500 kHz were compared, it was reported that the most effective frequency for destroying phenol in solution was 200 kHz, which was explained by the larger

potential at this frequency for ejection of radical species to the bulk solution (Petrier and Francony *et al.*, 1997). Moreover, comparison of 487 kHz and 20 kHz by Petrier *et al.* have shown that the rate of degradation by the former was 10 times faster owing to the much faster hydroxyl radical transfer from the gas phase to the aqueous solution at this frequency than at 20 kHz (Petrier *et al.*, 1994). They reported that the larger efficiency observed at 487 kHz arises from the fact that under these higher frequency conditions radicals are concentrated at the surface of the solution, whereas at 20 kHz they are located intensely around the tip of the transducer.

Papadaki *et al.* have found that sonication at 20 kHz in the presence of Fenton's reagent was not an efficient method of phenol degradation because both  $\text{Fe}^{+2}$  and ultrasound compete for  $\text{H}_2\text{O}_2$  (by catalytic and thermal decomposition routes, respectively), thus reducing the concentration of Fenton's reagent in solution (Papadaki *et al.*, 2004). Wu *et al.* have studied the effect of UV irradiation on sonochemical degradation of phenol, reporting that while total phenol destruction was possible the degree of mineralization was no more than 20.6% however this could be effectively improved by the addition of Fenton's reagent to the solution. Moreover, their investigation of the effects of sparging with gas showed that the effectiveness was in the order: nitrogen < air < oxygen. In a similar study by Naffrechoux *et al.*, the synergy observed in the degradation of phenol by combined UV irradiation and sonolysis at 500 kHz was explained by the concurrent action of three mechanisms on the overall process: photodecomposition; sono decomposition and oxidation by ozone, produced by photolysis of atmospheric oxygen in solution (Wu *et al.*, 2001).



## CHAPTER THREE

### METHODOLOGY

#### 3.1 Chemical and Apparatus used

During the research, few chemical and apparatus are used to complete the research process. The chemical and apparatus used are as following:

List of glassware and apparatus used:

**Table 3.1:** List of Apparatus

Apparatus	
Volumetric Flask 100mL	Measuring Cylinder 10mL, 25mL, 100mL
Beaker 100mL	Cylinder Cone 100mL
Glass rod	Cuvette
Vials 30mL	Dropper

List of chemical used:

**Table 3.2:** List of chemicals and reagents

Chemicals	Assay	Supplier	Purpose of Use
Phenol	99.0% min	Fishers Chemicals	As a raw material in this research
Sodium Chloride	–	SYESTERM	As an additives in

(NaCl)			degradation of phenol
Folin-ciocalteu's reagent	–	R & M Chemicals	To colorize phenol so that it can be detected by UV-Vis Spectrophotometer
Sodium Carbonate	–	MERCK Ltd Japan	To colorize phenol so that it can be detected by UV-Vis Spectrophotometry
Hydrochloric acid (HCl)	37% fuming	R & M Chemicals	To adjust pH of solutions
Distilled Water	–		To dissolve phenol to prepare a aqueous solutions

The following equipment used in this study:

**Table 3.3:** List of Equipments

<b>Equipment</b>	<b>Model</b>	<b>Frequency</b>	<b>Power/ Voltan</b>	<b>Purpose of Use</b>
Ultrasonic Cleaner	Daihan	60kHz	100W	To degrade phenol
UV-Vis Spectrophotometer	U1800 Hitachi	–	250V	To analyze phenol to get the absorbance
pH meter	Cyberscan500	–	–	To measure the pH of solutions

### 3.2 The Overall Methodology

The experimental procedures can be divided by into two categories:

1. Preparation of phenol calibration curve
2. Preparation of phenol degradation