

**Effect of Ultrasound and Particle Size on Solubility of Crystal**

by

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15609

Dissertation submitted in partial fulfilment of

the requirements for the

Bachelor of Engineering (Hons)

(Chemical Engineering)

SEPTEMBER 2015

Universiti Teknologi PETRONAS

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CERTIFICATION OF APPROVAL

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Approved by,

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(Dr Md Abdus Salam)

UNIVERSITI TEKNOLOGI PETRONAS

BANDAR SERI ISKANDAR, PERAK

September 2015

## CERTIFICATION OF ORIGINALITY

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

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MUHAMMAD NIZAMUDDIN NU'AIM BIN MOHD SHAARANI

## **ABSTRACT**

Solubility of crystal plays an important role in both biological and chemical field. For this project, we will focus on two factors that affects the solubility of crystal which is effect of ultrasound and effect of particle size. In this research project, the solute, benzoic acid (AR Grade) which also categorised as sparingly soluble acid that is not easy to dissolve in water. It is a colourless crystalline solid and a simple aromatic carboxylic acid. Salts of benzoic acid are used as food preservatives and benzoic acid also is an important ancestor for the industrial synthesis of many other organic substances. This acid shows low aqueous solubility and dissolution rate and this has become the main concern in the industry that require dissolution of this chemical. In this project, the solvent that will be used to dissolve the benzoic acid is the distilled water. The experiment was conducted by applying the ultrasound during the dissolution of benzoic acid in distilled water in a different range of time and according to different particle size groups. Result shows the relation between different particle size groups and different range of time during sonication on the solubility of the crystal. The purpose of conducting this research project is because of the lack of literature data on solubility of benzoic acid which is also the acid that is categorised as sparingly soluble acid. The experimental outcome shows the crystal with smaller particle size have higher solubility compared to crystal with larger particle size and the solution with longer time of sonication also have higher solubility compared to solution that being sonication with a shorter time.

## **ACKNOWLEDGEMENT**

First and foremost, I would like to express my gratitude to God for His kind blessings by giving me strong will and determination to complete this Final Year Project (FYP) course. After all the difficulties and challenges for the past few months, this course was completed.

I would like to take this golden opportunity to thank all people who played their role for the successful completion of the Final Year Project with the topic “Effect of Ultrasound and Particle Size on Solubility of Crystal”. I would like to thank my FYP I and FYP II supervisors, Dr Taslima Khanam and Dr Md Abdus Salam for their expert guidance, continuous support and encouragement throughout the course of this research project. They have been a very supportive supervisors and willing to share their knowledge, in order to ensure that I could learn and understand every single thing regarding this project. My gratitude is also extended to FYP I and FYP II coordinators, Dr Sintayehu and Dr Nurul Ekmi for their effort in assisting us in all the possible way.

Furthermore, I express my deepest gratitude to Universiti Teknologi PETRONAS (UTP) for the platform of my course. I am also grateful to Chemical Engineering Department of UTP for the support that I received when undertaking the course work.

Finally, much love and thanks to my family and friends who never failed in helping me through thick and thin of time. It is the unfailing support from them that has enabled me to complete this FYP course. Not to forget to those who are directly or indirectly involved in the completion of this project.

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# CHAPTER 1

## INTRODUCTION

### 1.1 Background of Study

Crystallization categorized as the oldest unit operation in the chemical engineering. As for example the process of making Sodium Chloride, it has been manufactured since the beginning of civilization. Nowadays, there are few sectors of the chemical industry that do not, at some stage, utilize crystallization as a method of production, purification or recovery of solid material. Crystallization is one of the best and cheapest methods available for the production of pure solids from impure solutions. Besides that, the industrial applications of crystallization are not just narrowed to the production of pure solid substances. As for example, in the petroleum industry, in which distillation has become the major processing operation, also focus to low-temperature crystallization as a method for the separation of 'difficult' liquid hydrocarbon mixtures [1].

Crystallization from solutions is the important operation in the production of pharmaceutical solid particles such as drugs and it benefits in determining the purity (chemical and structure) and the physical properties for some materials [2]. The relation of solubility with the crystallization is that the solubility characteristics of a solute in a given solvent have a considerable influence on the choice of a method of crystallization [1].

There are many factors that affects the solubility of a crystal in solvent such as effect of ultrasound, temperature, particle size, ionic strength and impurities. There are also difference between solubility of solids. There are solid that easy to

soluble and not easy to soluble in solvent. All solids that dissociate into ions show some limit to their solubilities, but those whose saturated solutions exceed about 0.01 mol/L cannot be treated by simple equilibrium constants due to ion-pair formation that greatly complicates their behaviour and those salts fall into the “sparingly soluble” category. The important to separate the sparingly soluble solid is that the formation of such a product can effectively remove the corresponding ions from the solution, thus driving the reaction to the right. The solid dissolution in a solvent are of particular interest in several industrial. It involves in many fields such as chemical engineering, pharmaceutical mineral processing [2], petroleum engineering and nuclear engineering [3]. Therefore many studies have focused on a range of methods to enhance the dissolution of drugs as for example, the use of ultrasonic to enhance the attainment rate [4] , the effect of particle size on the dissolution [5] , the effect of temperature and the effect of mixing speed [3] .

In this research project, we are going to focus on two factors of solubility which are effect of ultrasound and particle size on the solubility of sparingly soluble crystal. This is because to show that the influence of ultrasound has a great potential in intensifying rates and altering pathways of chemical reactions [3]. Furthermore, the effect of particle size is also one of the important factors for the dissolution rate, for example in developing drug products. By decreasing the particle size, will increase the surface area and consequence will increase the dissolution rate.

## **1.2 Problem Statement**

In the effect of particle size on solubility of sparingly soluble solid, there are still has limitation. The milling method is a common technique to increase the dissolution rate by increasing the surface area but this method still has limitations and disadvantages due to the inefficient preparation process. A high energy input may disturb the crystal lattice causing physical and chemical stability issues. Defective and disordered regions in crystal are thermodynamically unstable and affect the surface energy [5].

In the effect of ultrasound on solubility of sparingly soluble acid, as reported in the literature, they were using different power level of ultrasonic at a fixed time and the results of the rate of dissolution are not so smooth.

### 1.3 Objectives and Scope of Study

The main objective of this research is to study the effect of ultrasound and particle size on the solubility of crystal. To achieve the main objective, the sub-objectives of this project are as the following:

- (a) To investigate the effect of ultrasonic on the solubility of sparingly soluble solid in a different range of time.
- (b) To investigate the particle size on the rate of dissolution of sparingly soluble solid in a different range of time.
- (c) To monitor the fluctuation of temperature during the increment of the time during sonication.

The scope of study for this project is we will use the sparingly soluble solid which is benzoic acid (AR grade) as the solute. The solvent will be distilled water. Carboxylic acids such as benzoic acid are relatively weak acids, and thus exist mostly in the acidic form when added to pure water. Since the melting point of benzoic acid also is reasonably low ( $122.41^{\circ}\text{C}$ ), this indicate that it must be a molecular crystal rather than an ionic crystal. If it were an ionic compound, the melting point would be very high (greater than  $700^{\circ}\text{C}$ ). Therefore instead of using easy-soluble acid, we use sparingly soluble acid.

## **CHAPTER 2**

### **LITERATURE REVIEW AND THEORY**

Solubility is the property of a solid, liquid or gaseous chemical substance called solute to disintegrate in solid, liquid, or gaseous solvent to produce a homogenous solution of the solute in the solvent. The extent of solubility of a substance in a specific solvent is dignified as the saturation concentration where adding more solute will not make its concentration in the solution increasing [6]. Solubility take place under dynamic equilibrium meaning that the solubility outcomes from the simultaneous and opposing processes of dissolution and phase joining as for example the precipitation of solid. When two processes occur at a constant rate, the solubility equilibrium occurs and under certain conditions equilibrium solubility may be exceeded to give a solution which called as supersaturated solution [7] .

Nowadays, the solid dissolution is important in several industrial situations such as mineral processing, chemical engineering, petroleum engineering, pharmaceutical, food industry and many more [3]. The dissolution rate also can be the rate-limiting step for poorly water-soluble drugs such as griseofulvin, amiodarone, fenofibrate and amlodipine [5]. Therefore in the dissolution rate, there are two studies, mostly being examined is the effect of ultrasound and particle size on the solubility of sparingly soluble solid [3, 8-10].

Solubility is not to be confused with the capability to dissolve a substance, as these processes may occur not only because of dissolution but also because of chemical reaction. Besides, solubility does not depend on particle size or other kinetic aspects but if given enough time, even large particles will dissolve [11].

## 2.1 Effect of Ultrasound

Ultrasound is composed of sound waves with frequency away from the limit of human hearing. By tuning frequency, ultrasound can be used in many industrial applications including food. Its techniques are relatively cheap, simple and energy saving, and hence became an incipient technology for probing and modifying food products [12]. Ultrasound is known to enhance solid-liquid reactions and it is an increasingly used tool to enhance the chemical process rates [10]. It has been demonstrated to successfully increase mass transfer rates, improve yields, initiate reactions and even change reaction pathways. Ultrasound successfully used in several applications including extraction, impregnation, crystallization, emulsification cleaning and disinfections [4]. Ultrasound may influence crystallization through the mechanisms of cavitation as it appears to particularly effective as a means of encouraging nucleation, and there is evidence of improvements in reproducibility acquired through such sononucleation [13].

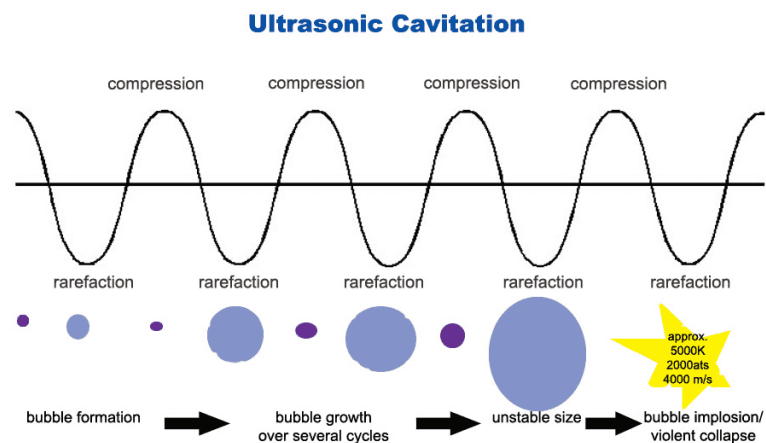


FIGURE 2.1 Ultrasonic Cavitation

In previous studies shown that ultrasound good in intensifying the rates and can alter the pathways of chemical reaction. Besides that, ultrasound had enhanced the saturation limit by inducing super saturation in the system [3]. There were also previous investigation of the dissolution phosphate rock in nitric acid, in the absence and presence of ultrasound. The results shown that the ultrasound enhances the reaction rates but it does not influence the activation energy [10]. Some previous studies choose acid concentration, temperature, and ultrasound power level as parameters to investigate their effects on the reaction kinetics [3, 9, 10].

Most of previous studies focus on intensifying rates and altering chemical reaction pathways through ultrasonic, but relative less focus has been given to its physical effects. Besides that, the problem that usually occurred during ultrasonic studies is the temperature control could not be maintained constant. Some of the researchers also suggested to perform the saturation limit experiments with more accurate temperature control [3].

The use of ultrasound will increases the driving force for mass transfer, consecutively increasing the intrinsic mass transfer and the interfacial area [14]. Besides, the use of ultrasound will drive the therapeutic action of microbubbles which have the cavitation microstreaming plays a role with it [15]. Therefore when a solid-liquid systems where the solids are discrete as particles is exposed to ultrasound, the solid particles get fragmented by the microjets that are created by the acoustic field and this fact could be demoralized to enhance mass transfer rates since the interfacial area available for the transport process increases in the presence of ultrasound [3].

## 2.2 Effect of Particle Size

The relationship between particle size and solubility, originally derived for vapour pressure in liquid-vapour systems by Thomson in 1871, utilized later by Gibbs, and applied to solid-liquid systems by Ostwald (1900) and Freundlich (1926) may be expressed in the form

$$\ln \left[ \frac{c(r)}{c^*} \right] = \frac{2M\gamma}{vRT\rho r} \quad (1)$$

where  $c(r)$  is the solubility of particles of size (radius)  $r$ ,  $c^*$  is the normal equilibrium solubility of the substance,  $R$  is the gas constant,  $T$  is absolute temperature,  $\rho$  is the density of the solid,  $M$  is the molar mass of the solid in solution and  $\gamma$  is the interfacial tension of the solid in contact with the solution [1].

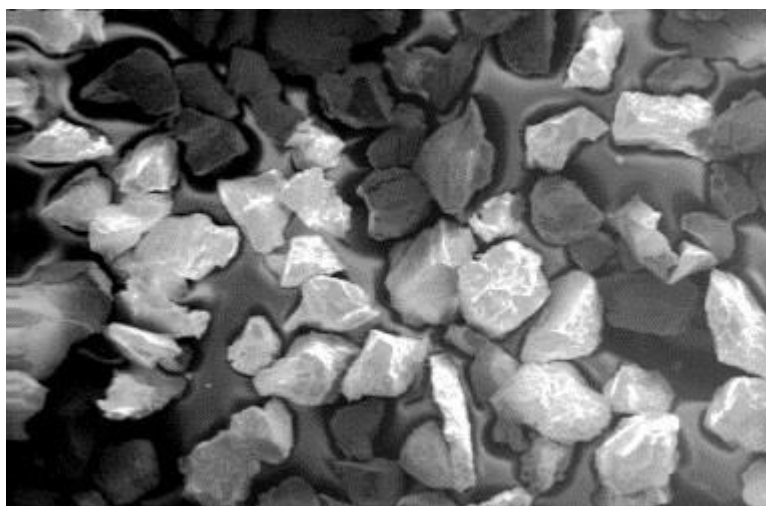


FIGURE 2.2 Example of Crystalline Particle Size

The solubility of drug is frequently related to drug particle size as a particle size becomes smaller, the surface area to volume ratio increases. The larger surface area permits better interaction with the solvent and this will increase the solubility [6]. The decreasing of particle size will increase the specific surface area, resulting in an increase in dissolution rate. Many studies have focused on a range of methods to enhance the dissolution rates and one of them is physicochemical analysis which helped explain the effect of particle size on the dissolution rates [5]. The mean dissolution time showed a good correlation with the mean particle size of each



fraction. Knowledge of these relationships can be critical because it can be eventually used for drug release control.

To reduce the particle size, the milling method is commonly used but still it has limitations and disadvantages due to the inefficient preparation process. A high energy input may disrupt the crystal lattice causing physical and chemical stability issues. Some researcher suggested to scientist that they should consider the effects of the particle size and size distribution on the dissolution rate when developing poorly soluble drugs.

### 2.3 Solute: Benzoic Acid (AR Grade)

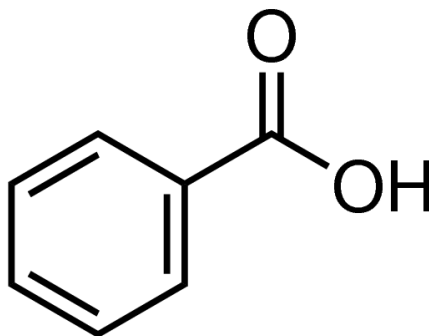


FIGURE 2.3 Skeletal Formula of Benzoic Acid

Benzoic acid is a colourless crystalline solid and a simple aromatic carboxylic acid. Salts of benzoic acid are used as food preservatives and benzoic acid also is an important ancestor for the industrial synthesis of many other organic substances.

Carboxylic acids such as benzoic acid are relatively weak acids, and thus exist mostly in the acidic form when added to pure water. Benzoic acid has low melting point and this indicate it is a molecular. It also being categorised as one of the salts that are not easy to dissolve in water.

## 2.4 Theory of Dissolution Rate

Generally, the dissolution rate can be explained by the well-known modified Noyes-Whitney equation [5]:

$$\frac{dC}{dt} = \frac{DS}{Vh}(C_s - C_t) \quad (2)$$

where  $dC/dt$  is the dissolution rate,  $D$  is the diffusion coefficient of the solute in solution,  $S$  is the interfacial surface area of the exposed solid,  $V$  is the volume of the solution,  $h$  is the thickness of the diffusion boundary layer,  $C_s$  is the concentration of saturated solution of the solute at the surface of the solid, and  $C_t$  is the concentration of the solute in the bulk medium at time  $t$ .

If the solute concentration does not exceed 10% of the amount required for equilibrium solubility ( $C_s$ ), it is called the 'sink condition' [5]. It is also well known that the rate of dissolution of a solute in a solvent depends on the interfacial area and the intrinsic mass transfer coefficient [14].

## CHAPTER 3

### METHODOLOGY AND PROJECT WORK

#### 3.1 Project Flow Chart

Below is the project flow chart for this study project that have been followed in order to achieve the objective of this study.

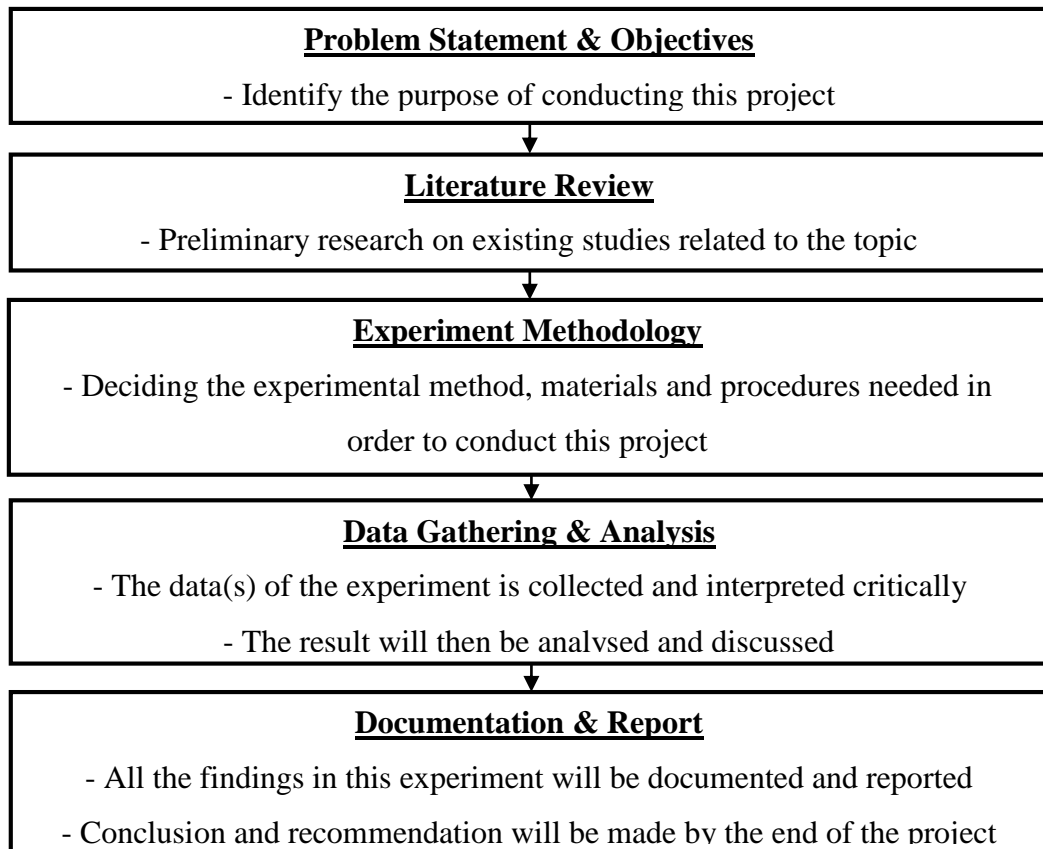


FIGURE 3.1 Project Flow Chart

### 3.2 Gantt Chart and Key Milestone

TABLE 3.1 Gantt Chart & Key Milestone (FYP I)

NO	DETAILS	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Title														
2	Preliminary Research Work & Literature Review														
3	Submission of extended proposal (1 <sup>st</sup> draft)														
4	Submission of extended proposal (Final draft)														
5	Preparation for Proposal Defence														
6	Proposal Defence														
7	Detailed Literature Review														
8	Preparation of Interim Report														
9	Submission of the draft of Interim Report														
10	Submission of Interim Report (FINAL)														



Milestones



Process

TABLE 3.2 Gantt Chart and Key Milestone (FYP II)

NO	DETAILS	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Project Work Continues															
2	Submission of Progress Report															
3	Project Work Continues															
4	Pre-SEDEX															
5	Submission of the Draft Final Report															
6	Submission of Dissertation (soft bound)															
7	Submission of Technical Paper															
8	Viva															
9	Submission of Project Dissertation (Hard Bound)															



Milestones



Process

### 3.3 Experiment Methodology

#### 3.3.1 Materials and Apparatus

- (a) Benzoic Acid (AR Grade)
- (b) Distilled water
- (c) Aluminium foil
- (d) Hot plate with magnetic stirrer
- (e) Mortar & Pestle
- (f) Sieve Shaker with sieve Tray (50  $\mu\text{m}$ , 100  $\mu\text{m}$ , 212  $\mu\text{m}$ )
- (g) Micropipette
- (h) Portable Turbidity Meter
- (i) Set of Filter Tunnel
- (j) Thermometer
- (k) Ultrasonic water bath (Bandelin Sonorex Digitec – 35kHz, 480 Watt)
- (l) UV/VIS Spectrometer (Lambda 25)

#### 3.3.2 Experimental Procedures



FIGURE 3.2 UV/Vis Spectrometer



FIGURE 3.3 Ultrasonic Water Bath Indicator

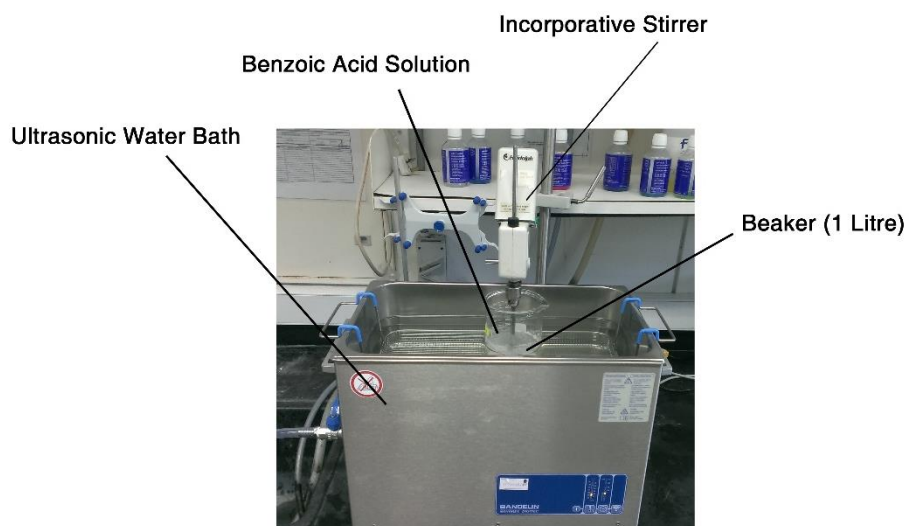


FIGURE 3.4 Experimental Setup

Figure 3.4 illustrates the experimental setup. The Benzoic acid solution in a beaker is immersed in an Ultrasonic water bath. An incorporative stirrer is placed in the benzoic acid solution to maintain the stirring speed. The thermometer will be immersed in the benzoic acid solution to monitor the temperature of the solution.



### A) Calibration

1. Prepare 5 sample of benzoic acid as per below in Table 3.3 (measured using analytical balance) :

TABLE 3.3 Standard Solution Preparation

Standard Solution	MASS (g)
Std1	2
Std2	4
Std3	6
Std4	8
Std5	10

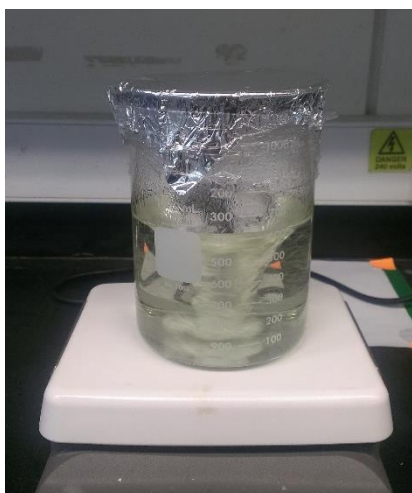


FIGURE 3.5 Standard Solution Setup

2. Each sample dissolve it in 0.5L of distilled water in a beaker (1 Litre) until dissolve completely. Use a hot plate with a magnetic stirrer as an apparatus to accomplish the task as in Figure 3.5. The dissolution checked by using a turbidity meter.
3. The temperature of the solution is maintained at 30°C and for 3 hours to ensure complete dissolution of benzoic acid. Cover the beaker with aluminium foil to avoid evaporation in 3 hours.

4. Each standard solution is transferred into the cuvette and its absorbance is measured using the Perkin Elmer UV/VIS Spectrometer (Lambda 25).
5. The calibration curve of absorbance versus concentration is plotted.

## B) Solubility Analysis

1. After calibration, a new set of sample will be setup.
2. Prepare 10g/L of benzoic acid solution for a different set of time with different set of particle size (*Size 1: 50 $\mu$ m, Size 2: 100 $\mu$ m, Size 3: 212 $\mu$ m*) by dissolving 5 g of benzoic acid in 500mL distilled water as categorised below in Table 3.4:

TABLE 3.4 Sample Solution Preparation

SAMPLE		TIME (min)
A	A1 ( <i>Size 1</i> )	0
	A2 ( <i>Size 2</i> )	
	A3 ( <i>Size 3</i> )	
B	B1 ( <i>Size 1</i> )	5
	B2 ( <i>Size 2</i> )	
	B3 ( <i>Size 3</i> )	
C	C1 ( <i>Size 1</i> )	10
	C2 ( <i>Size 2</i> )	
	C3 ( <i>Size 3</i> )	
D	D1 ( <i>Size 1</i> )	15
	D2 ( <i>Size 2</i> )	
	D3 ( <i>Size 3</i> )	
E	E1 ( <i>Size 1</i> )	30
	E2 ( <i>Size 2</i> )	
	E3 ( <i>Size 3</i> )	

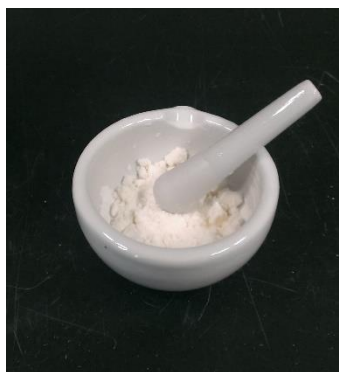


FIGURE 3.6 Milling Process



FIGURE 3.7 Sieving Process

3. Mills the particle of benzoic acid by using a mortar and pestle as in Figure 3.6, and classified into three different particle size groups by passing a through a sieve corresponding to the particle sizes of 50  $\mu\text{m}$ , 100  $\mu\text{m}$  and 212  $\mu\text{m}$ , respectively as in Figure 3.7.
4. Put sample B, into a beaker (1 Litre) containing 0.5 litre of distilled water. Immerse the beaker in the Ultrasonic water bath.
5. The temperature of ultrasonic water bath will be maintained 30°C throughout the experiment.
6. Start the experiment by turning on the frequency of ultrasonic and stir the solution by using an incorporative stirrer.
7. Stop the experiment after 5 minutes.

8. 10 ml of the solution is extracted using a micropipette and filtered to remove any crystals.
9. 2 ml of the filtered sample was extracted and transferred into the cuvette. The absorbance reading is obtained using the Perkin Elmer UV/VIS Spectrometer (Lambda 25) and recorded.
10. The concentration of sample B is obtained using the absorbance-concentration calibration curve of benzoic acid.
11. Repeat the experiment for sample B, C, D and E with their corresponding time and particle group size.
12. The concentrations of each sample versus the corresponding time and corresponding size of particles is plotted.

## CHAPTER 4

### RESULTS AND DISCUSSION

#### 4.1 Calibration Curve of Standard Solution

TABLE 4.1 Standard Solution Results

Standard Solution	MASS (g)	CONCENTRATION (g/L)		Absorbance (A)
		Before Dilution	After dilution	
Std1	2	4	0.1	0.9895
Std2	4	8	0.2	1.5071
Std3	6	12	0.3	1.966
Std4	8	16	0.4	2.3122
Std5	10	20	0.5	2.7016

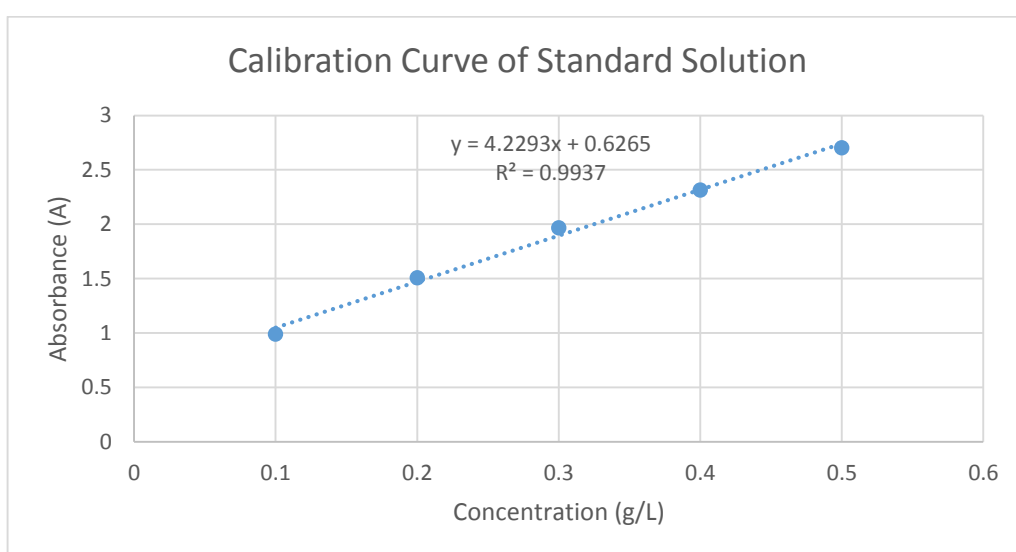


FIGURE 4.1 Calibration Curve of Standard Solution

These samples of known concentration were prepared and the absorbance of each sample was measured using a UV/VIS Spectrometer (Lambda 25). The results for their absorbance which have the highest peak of wavelength at  $\lambda_{\text{max}} = 272$  nm are recorded in Table 4.1. Then, from these results, the calibration curve of standard solution was plotted.

Based on the calibration curve of standard solution plotted as in Figure 4.1, the graph shows a linear relationship, which is typical for diluted concentrations and we get a trend line of  $y = 4.2293x + 0.6265$  with  $R^2=0.9937$ . This calibration curve of standard solution is very important as the graph was used for the determination of concentration of unknown samples.

## 4.2 Sample Solution Results (Effect of Ultrasound and Particle Size)

TABLE 4.2 Sample Solution Results

SAMPLE		MASS OF BENZOIC ACID (g)			VOLUME OF DISTILLED WATER (L)	TIME (min)	ABSORBANCE (A)	CONCENTRATION (g/L)
		Particle Size 1 (50 µm) - (A1, B1, C1, D1, E1)	Particle Size 2 (100 µm) - (A2, B2, C2, D2, E2)	Particle Size 3 (212 µm) - (A3, B3, C3, D3, E3)				
A	A1 (Size 1)	5	5	5	0.5	0	1.9513	0.3132
	A2 (Size 2)						1.7988	0.2773
	A3 (Size 3)						1.5303	0.2142
B	B1 (Size 1)	5	5	5	0.5	5	2.0845	0.3445
	B2 (Size 2)						1.9113	0.3038
	B3 (Size 3)						1.6196	0.2352
C	C1 (Size 1)	5	5	5	0.5	10	2.1060	0.3495
	C2 (Size 2)						2.0746	0.3421
	C3 (Size 3)						1.9217	0.3062
D	D1 (Size 1)	5	5	5	0.5	15	2.1853	0.3681
	D2 (Size 2)						2.1376	0.3569
	D3 (Size 3)						1.9579	0.3147
E	E1 (Size 1)	5	5	5	0.5	30	2.1874	0.3686
	E2 (Size 2)						2.1471	0.3592
	E3 (Size 3)						2.1198	0.3528



Table 4.2 shows the result of all samples that have been executed experimentally. The absorbance value we get from the UV/VIS Spectrometer and from that absorbance value we can find the concentration from the calibration curve graph.

The absorbance value of each samples was taken at highest peak of wavelength which is at  $\lambda_{\text{max}} = 272$  nm. This is relevant to literature which shows the highest peak of wavelength for benzoic acid solution is around  $\sim 270$  nm.

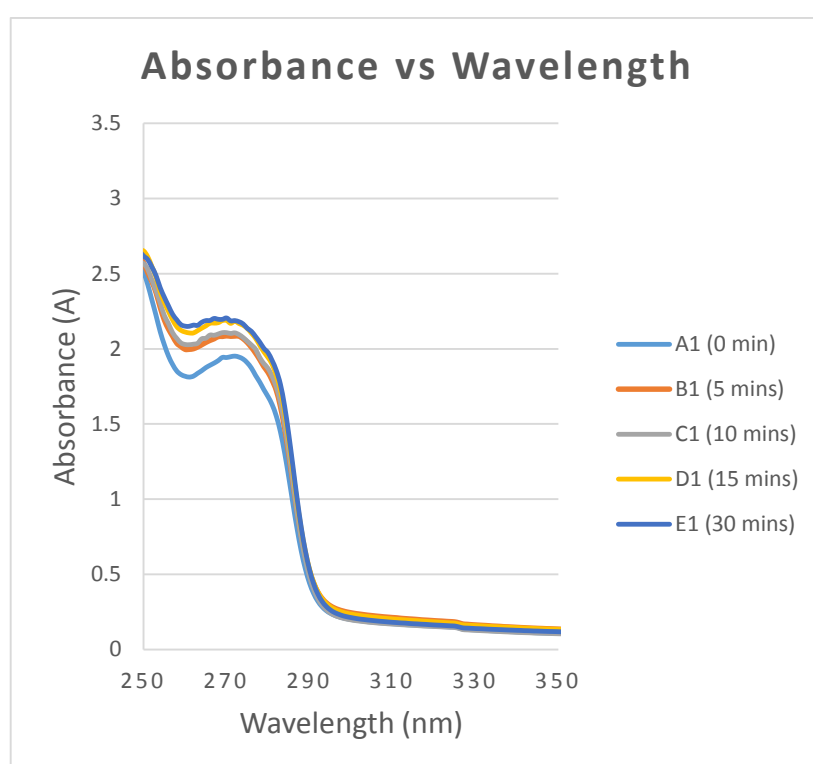


FIGURE 4.2 Graph for Absorbance vs. Wavelength for Samples Size 1 ( $50 \mu\text{m}$ )

Figure 4.2 shows the graph for absorbance versus wavelength for samples for particle size group 1 which are having  $50 \mu\text{m}$ . As had been categorised, sample A1 being sonicated at 0 minute while sample B1, C1, D1 and E1 being sonicated at 5 minutes, 10 minutes, 15 minutes, and 30 minutes respectively. These data plots generated from the software in UV/Vis Spectrometer during sample testing. As we can see, all the samples have highest peak of wavelength around  $\sim 270$  nm and from this graph also, we can get the highest peak is at  $\lambda_{\text{max}} = 272$  nm.

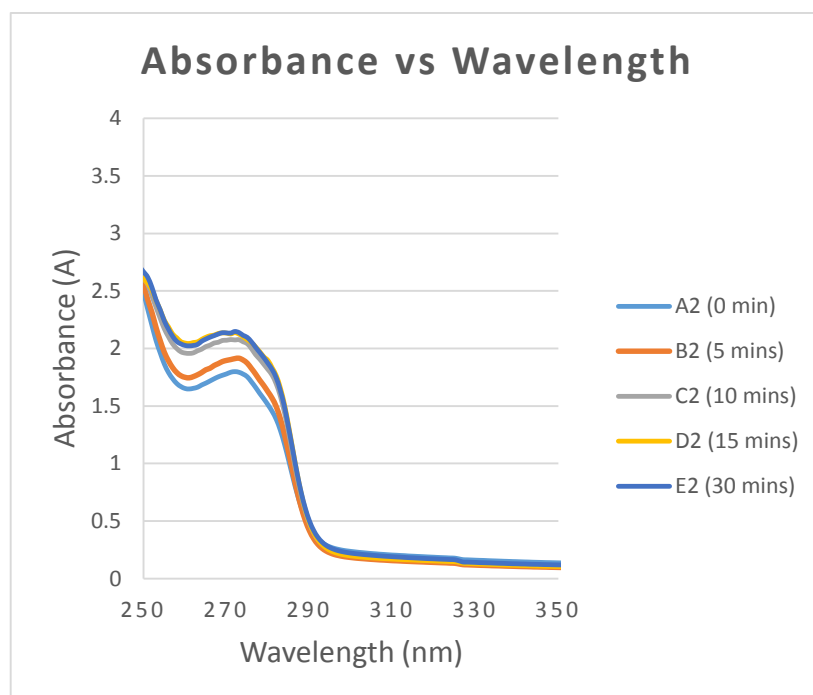


FIGURE 4.3 Graph for Absorbance vs. Wavelength for Samples Size 2 (100 μm)

Figure 4.3 shows the graph for absorbance versus wavelength for samples for particle size group 2 which are having 100 μm. As had been categorised, sample A2 being sonicated at 0 minute while sample B2, C2, D2 and E2 being sonicated at 5 minutes, 10 minutes, 15 minutes, and 30 minutes respectively. The highest peak is also at  $\lambda_{\max} = 272$  nm.

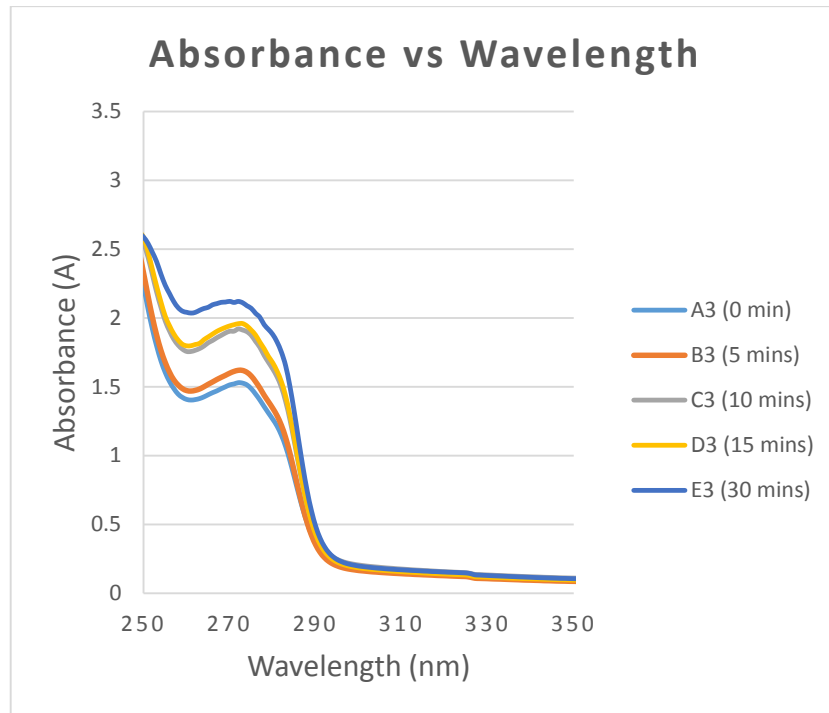


FIGURE 4.4 Graph for Absorbance vs. Wavelength for Samples Size 3 (212 μm)

Figure 4.4 shows the graph for absorbance versus wavelength for samples for particle size group 3 which are having 212 μm. As had been categorised, sample A3 being sonicated at 0 minute while sample B3, C3, D3 and E3 being sonicated at 5 minutes, 10 minutes, 15 minutes, and 30 minutes respectively. As we can see, all the samples have highest peak of wavelength around ~270 nm and as we zoom in, we can detect the highest peak is at  $\lambda_{\max} = 272$  nm. Therefore, the absorbance value for each samples we took at 272 nm of wavelength and for further analysis, the unknown concentration of samples were determined from the calibration curve. All graph in Figure 4.2, Figure 4.3 and Figure 4.4 shows that when the time of experiment with sonication increase, the absorbance value also increase, thus we get the increasing of concentration values as tabulated in Table 4.2.

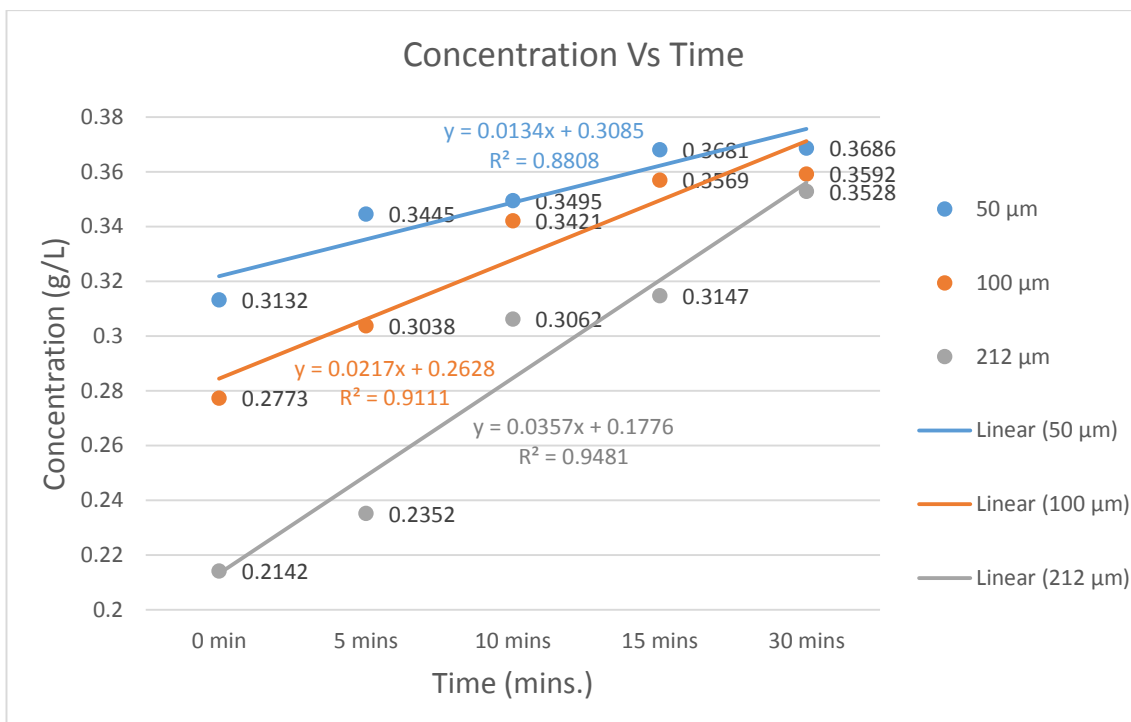


FIGURE 4.5 Concentration Against Time for Different Particle Size Groups

By using this sonicator, Bandelin Sonorex Digital with 35kHz of frequency and 480 Watt ultrasonic power, we get the result and the data was plotted as in Figure 4.5. As we can see the longer the time taken for sonication with smallest particle size will give higher concentration.

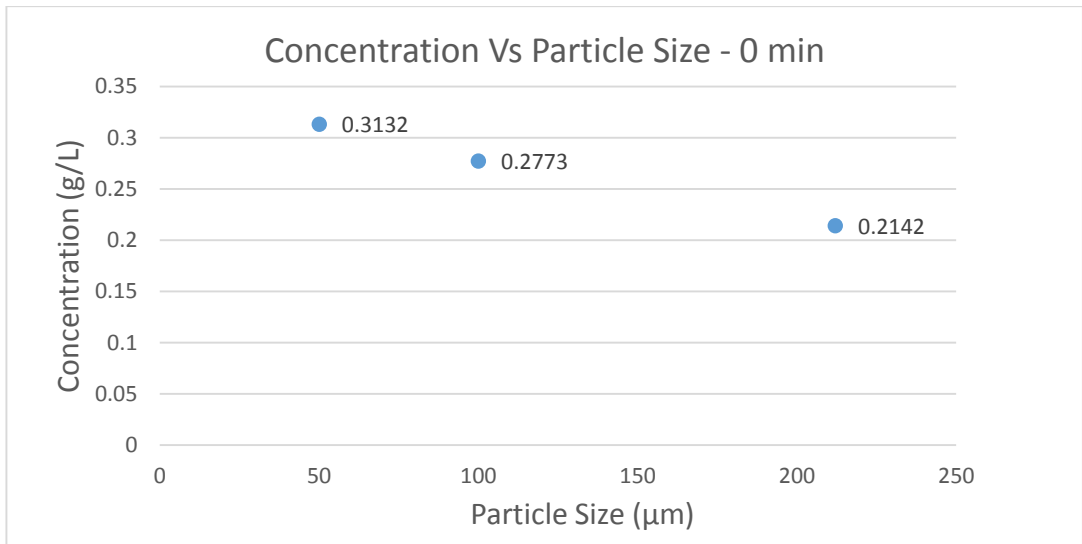


FIGURE 4.6 Concentration Against Particle Size for Time = 0 min

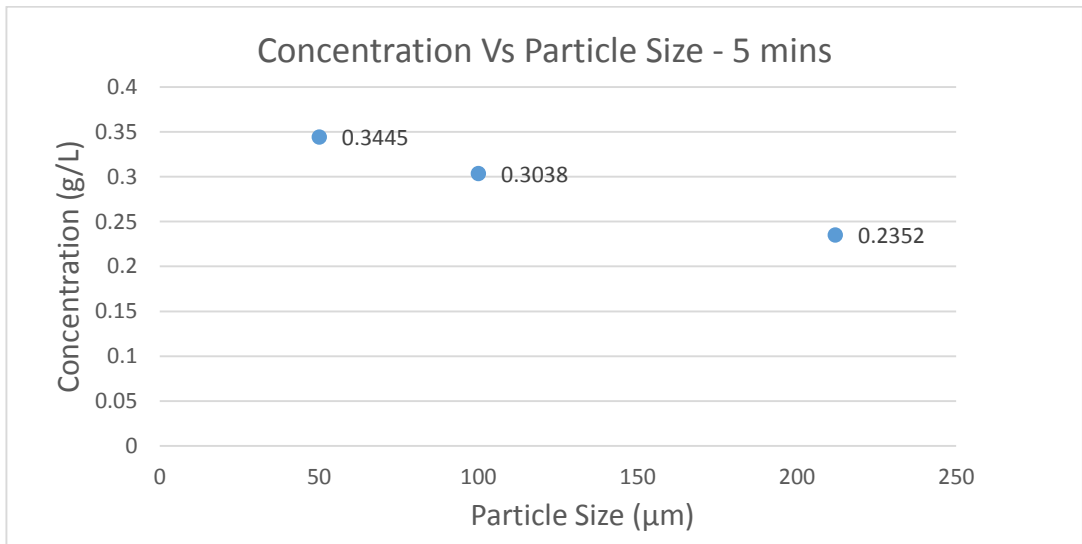


FIGURE 4.7 Concentration Against Particle Size for Time = 5 mins

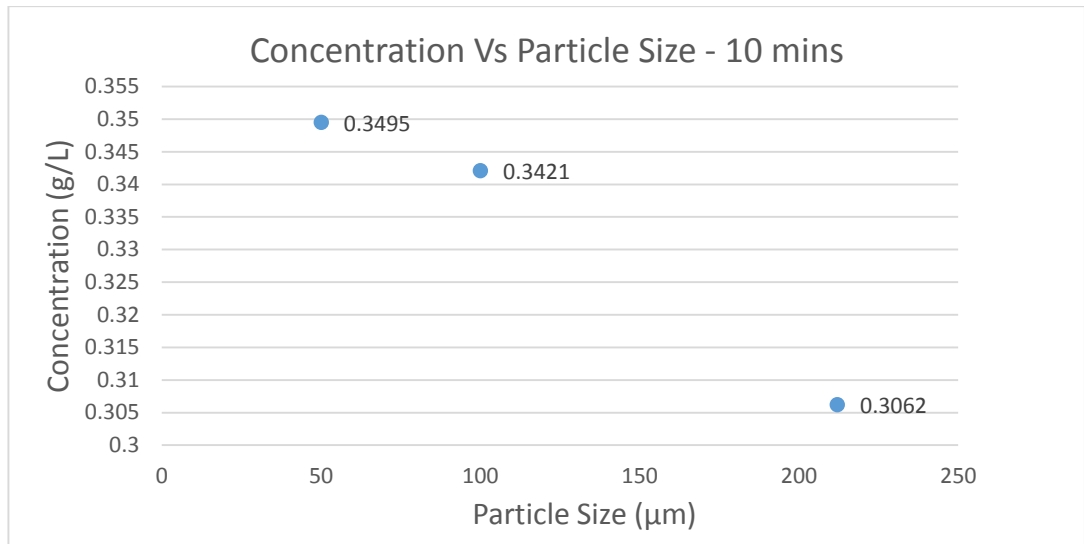


FIGURE 4.8 Concentration Against Particle Size for Time = 10 mins

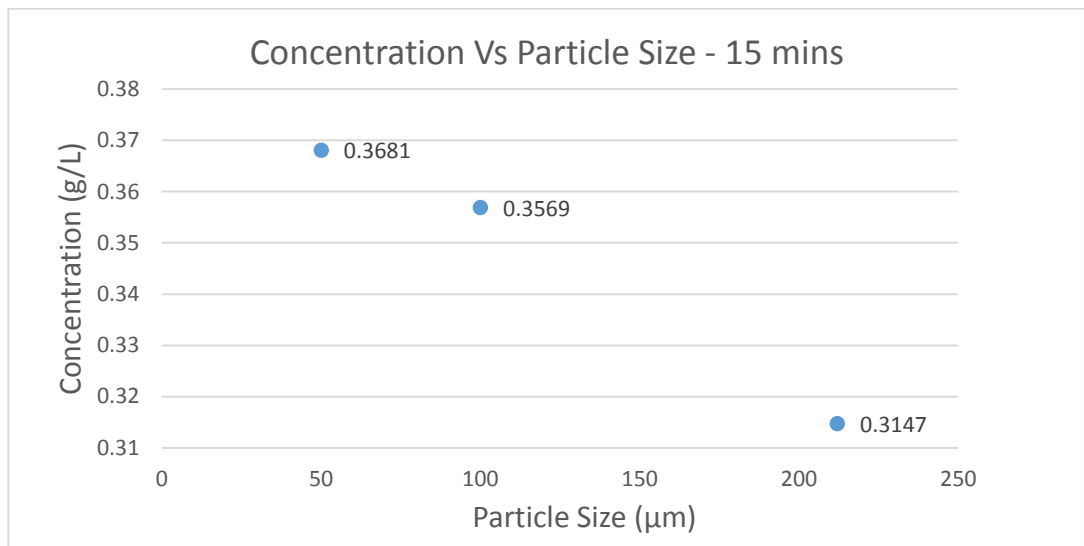


FIGURE 4.9 Concentration Against Particle Size for Time = 15 mins

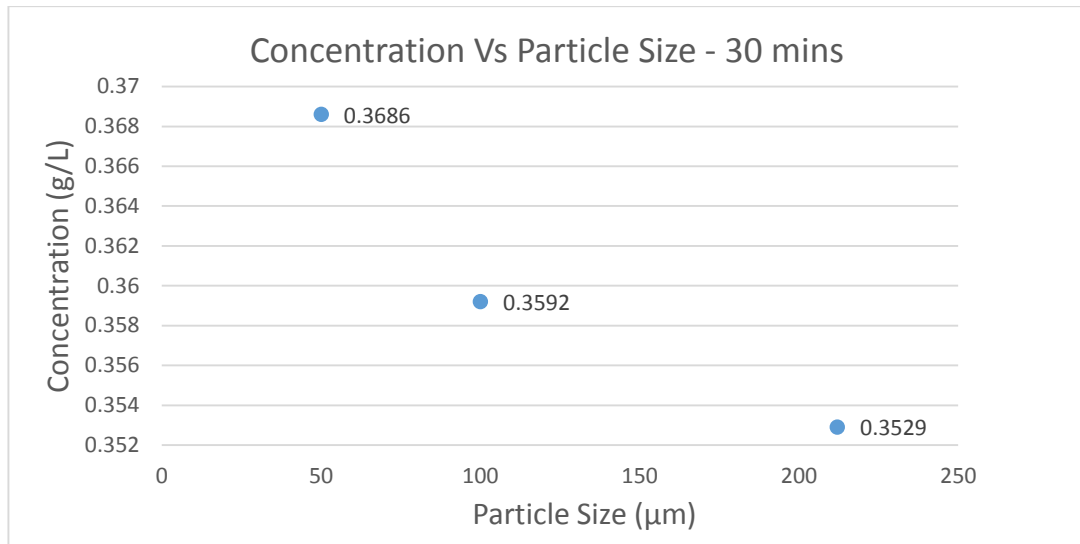


FIGURE 4.10 Concentration Against Particle Size for Time = 30 mins

Figure 4.6, Figure 4.7, Figure 4.8, Figure 4.9 and Figure 4.10 shows the graph of concentrations against different particle sizes for 0 min, 5 mins, 10 mins, 15 mins, and 30 mins respectively. From all the figure above, we can see that the decreasing of particle size will increase the concentration.

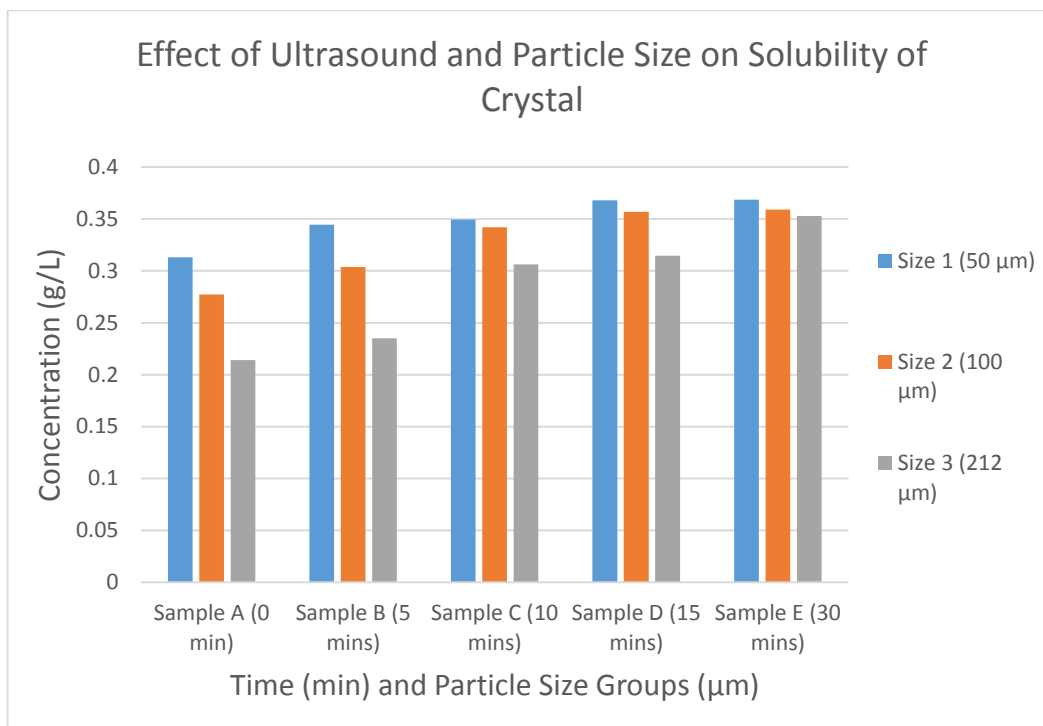


FIGURE 4.11 Effect of Ultrasound and Particle Size on Solubility of Crystal

Figure 4.11 shows the effect of ultrasound and particle size on solubility of crystal. As we can see the sample E1, with particle size group 1 (50µm) and longest time taken (30 mins) with sonication for the experiment was having highest concentration compared to the sample A3, with particle size group 3(212µm) and shortest time taken(0 min) with sonication for the experiment. Meaning that, the sample with the smallest particle size and being sonication with longest time have highest solubility. This is because, the time of dissolution will affect the solubility of benzoic acid. The longer the time for dissolution will have higher reaction between benzoic acid and distilled water. The benzoic acid will dissolve more and thus increase the concentration of benzoic acid solution.

The smaller particle size of benzoic acid will have larger surface area which means that it will react and dissolve quickly in distilled water. Therefore the concentration of benzoic acid solution will also increase.



## **CHAPTER 5**

### **CONCLUSION AND RECOMMENDATION**

As a conclusion, the objectives of this research project are met which is to study the effect of ultrasound on solubility of crystal at different set of time and different particle size groups. The results show that dissolution of benzoic acid with sonication are recommended especially in pharmaceutical, food industry and many more. Furthermore, the crystal with smaller particle size give a higher solubility compared to the crystal with bigger particle size and the longer the time taken for the dissolution, will increase the concentration of solution and solubility. It is recommended to maintain constant temperature during all the experimental procedures for solubility determination. It is essential that any thermometers, thermostat, etc. are accurately calibrated with reference to a standard thermometer.

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