

### Ionic Liquid as Solvent for Synthesis Nanoparticles

by

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Dissertation submitted in partial fulfilment of the requirements for the Bachelor of Engineering (Hons) (Chemical Engineering)

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# **CERTIFICATION OF APPROVAL**

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A project dissertation submitted to the Chemical Engineering Programme Universiti Teknologi PETRONAS in partial fulfilment of the requirement for the BACHELOR OF ENGINEERING (Hons) (CHEMICAL ENGINEERING)

Approved by,

(Dr. Moulay Rachid Babaa)

#### UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

July 2009

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# **CERTIFICATION OF ORIGINALITY**

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

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WAN MUHAMAD FAIZ WAN ABD AZIZ

# ABSTRACT

Nanoparticles are defined as the particles with the diameter size below than 100nm. Nanoparticles are useful in a lot of field for example semiconductor, electronic and catalyst. In this report there are 4 chapters which are introduction, literature review, methodology and discussion.

In the introduction, there are sub topics which are background, problem statement and objective of the project. Introduction will briefly describe on the project. The author will briefly explain on the two main parts in this project which are nanoparticles and Ionic Liquids. After, proceed with the problem statement and the purpose of this project.

Literature review is where the theory and finding will be discussed based on the reading material such as book and journal. In this project there are some of concepts need to be understand before proceeding to the next stage. Under this topic, there are nanoparticles, ionic liquids and sample characterization test topics. Theses are the main point in this project. After that in the methodology, they are the flow of the experiments. After that the outcome of the project will be discussed in the discussion part. Last part is conclusion, the project will be concluded.

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#### 1. INTRODUCTION

#### **1.1. BACKGROUND**

The title of this project is "Synthesis of Nanoparticles by Using Ionic Liquid as Solvents". From the title it self, there are two main components which are Ionic Liquid (IL) and Nanoparticle. Nanoparticle means the particles with diameters only a few nanometers, while IL is salt that contain anion and cation that in the liquids form in room temperature.

Nanoparticles have been used for a very long time, probably the earliest use being in the glazes for early dynasty Chinese porcelain. A roman cup, called the Lycurgus cup, used nanosized gold clusters to create different colors depending on whether it was illuminated from the front or the back. Nanoparticles are currently made out of a very wide variety of materials. According to the most accepted definitions, the size of nanopartiles must be less than 100 nanometers. The transition from bulk-particles to nanoparticles can lead to number of changes in physical. The main factor in this area is the increase in the ratio of surface area to volume. The increase in the surface area to volume ratio, which is gradual progression as the particle gets smaller. High surface area is a factor in the performance of catalysis and structure (Holister, Weener, & Vas, 2003). Nanoparticles are useful for application such as catalysts, optics separation, sensors and electronics.

IL is also known as room-temperature ionic liquids (RTIL). They have attracted interest in recent years due to their favorable chemical and physical properties. ILs are being thermally stable solvents with negligible vapor pressure at room temperature. ILs have been shown to have applicability in a wide range of areas including separation processes, catalysis and electrochemistry. ILs are not new, some of them have been known for many years. For instance ammonium nitrate [EtNH3[NO3] was first described in 1914 (Welton, 1999). Among imidazolium salts, one of the first 1,3-dialkylimidazolium room RTILs obtained through the mixing of 1-ethyl-3-methylimidzolium chloride with aluminum trichloride was reported more

than a half of century ago. However, these organo-aluminate ILs are unstable to air and water and they are not inert towards various organic compounds, which have most probably limited their range of applications (Dupont, 2004).

#### **1.2. PROBLEM STATEMENT**

There are a lot of techniques and method to synthesis nanoparticles such as solgel processing, microemulsions, hydrothermal, microwave and sonochemical synthesis. The common problem in synthesis nanoparticles are because of solvents that being use in the synthesis are toxic, volatile and low thermal stability. Besides that, the most important thing in synthesis of nanoparticles is to have controlled particles sizes and morphology. To overcome solvents problem, IL is the best alternative solvents and becoming an important research area in chemistry as possible replacements for conventional solvents, mainly due to their green properties. The use of the ionic liquids to synthesize nanoparticles can allow overcoming the use of high temperature and large quantities of toxic and volatile organic solvents and hence offers an alternative route to nanostructure formation with controlled particles sizes and morphology.

#### **1.3. OBJECTIVES**

- . To develop synthetic methods for the control of diameter, shape and size distribution of nanoparticles by using ionic liquids as the solvent
- To study the relationship between the size and morphology of nanopartiles with the structure of the ILs

#### 2. LITERATURE REVIEW

#### **2.1. NANOPARTICLES**

Nanoparticles are important state of condensed matter. Nanoparticles may be considered as intermediate formations, which are limited by individual atoms on the one hand, and the solid phase on the other. Such particles exhibit the size dependence and a wide spectrum of properties. Nanoparticles can be defined as particles measuring from 1 - 100 nm. nanoparticles are larger than individual atoms and molecules buat are smaller than bulk solid. Hence they obey neither absolute quantum chemistry nor laws of classical physics and have properties that differ markedly from those expected. There are two major phenomenons that are responsible for these differences:

- High dispersity of nanocrytalline system.
- As the size of a crystal is reduced, the number of atoms at the surface of the crystal compared to the number of atoms in the crystal itself, increases. Properties which are usually determined by the molecular structure of the bulk lattice now become increasingly dominated by the defect structure of the surface.
- Size quantization.
- This phenomenon occurs only in metal s and semiconductors. It is because the size of a nanoparticle is comparable to the de Broglie wavelength of its charge carriers. Due to the spatial confinement of the charge carriers. The edge of the valance and conduction bands split into discrete, quantized, electronic levels.

#### 2.1.1. SONOCHEMICAL

The sonochemical synthesis of nanoparticles, such as gold and platinum have shown that ultrasound irradiation of aqueous solutions containing noble metal salts produce the corresponding colloids of the noble metal. The ultrasound induced reduction is a consequence of acoustic cavitation in solution. The high temperature and pressures that occur within a collapsing bubble due to acoustic cavitation cause homlytic dissociation of water and other species to produce reducing radicals (Crum, Mason, Reisse, & Suslick, 1999). In the presence of a colloid stabilizer, ultrasound reduction can produce nanometer sized particles of metal colloids such as platinum, palladium, silver and gold. The size of the metal particles is determined by the rate of reduction. The faster rates giving rise to smaller particles (Okitsu, Ashokkumar, & Grieser, 2005). This reduction rate is then determined by some factors (Fujimoto, Terauchi, Umehara, Kojima, & Henderson, 2001):

- The frequency and power of the ultrasonic field.
- The presence of additional reducing radicals obtained from additives.
- The nature of the atmospheric gas used in the acoustic cavitation.

Synthesis of platinum nanoparticles by using ultrasound has been well established compared to ruthenium because of the difficulties associated with ruthenium reduction as compared to the other metals. The ultrasound-assisted reduction of Ru(III) using polyethylene glycol in a zeolite matrix (Li, et al., 2004). However, the ultrasound does not play a role in the reduction of the Ru(III) and simply enables the ruthenium nanoparticles to penetrate the interstitial pores of the zeolite host (He, Vinodgopal, Ashokkumar, & Grieser, 2006).

#### 2.1.2. MICROWAVE

Some of the chemical reducing reactions can be carried out at room temperature (Mallin & Murphy, 2002) (He, et al., 2001). But most of them need elevated temperatures for higher reaction rate. The energy used to heat up the media can be conventional thermal heating (Sun & Xia, 2002) (Sun, Mayers, Herricks, & Xia, 2003), laser irradiation (Chen & Yeh, 2001) (Treguer, et al., 1998), ultrasonic (Carotenuto, Pepe, & Nicolais, 2000), fixed frequency microwave radiation (Yamamoto, et al., 2004) (Komarneni, Li, Newalker, Katsuki, & Bhalla, 2002) and UV irradiation (Itakura, Torigoe, & Esumi, 1995). Microwave radiation is known to have a faster heating rate than the conventional heating through conduction and convection. Some researches have reported the use of a fixed frequency microwave

(2.45 GHz) to synthesis platinum and silver nanoparticles (Komarneni, Li, Newalkar, Katsuki, & Bhalla, 2002). The microwave radiation heats up a material through its dielectric loss, which converts the radiation energy into thermal energy.

Variable frequency microwave (VFM) was developed to heat up the material quickly and uniformly. VFM has been used for thermoset polymers curing and Sn alloys soldering. These studies have shown that the VFM heating provided faster and more uniform heating profile than the conventional thermal heating methods such as convection or conduction ovens. VFM synthesis of silver nanoparticles has been shown that using fixed frequency microwave radiation, fast reaction and narrow distribution of the nanoparticles can be achieved in comparison with the conventional heating methods. Compared to the fixed frequency microwave, VFM provides more uniform heating that can lead to a more homogenous nucleation. In addition, VFM can also heat metals without an arcing problem. The fast reaction rate of VFM method provides an important advantage for the in situ formation of nanoparticles in a thermosetting polymer. In such case, uniformly distributed nanoparticles must be formed prior to curing of the thermosetting polymers since nucleation and growth of nanoparticles could be hindered by crosslinking reaction. Therefore, a fast heating rate, uniform heating profile and precise temperature control are required (Jiang, Moon, Zhang, Pothukuchi, & Wong).

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#### **2.2. IONIC LIQUIDS**

Ionic liquids are perceived as "green" since they do not evaporate under ambient conditions. Besides that there are other factors that make ionic liquid is quit an interesting solvent to research which are:

- Synthetic chemists are limited by the available molecular solvents in which they can conduct chemistry.
- Increasingly the solvents chemists like to use are banned by international protocols determined to reduce pollution, of which volatile organic compounds represent a significant part.
- The prospect of discovering new chemistry or the ability to design a solvent that facilities/allows a specific reaction to occur.

The properties of an ionic liquid are summarized in table 1 below:

A salt	Cation and or anion quite large
Freezing Point	Preferably below 100°C
Liquidus Range	Often >200°C
Thermal Stability	Usually high
Viscosity	Normally < 100 cP, workable
Dielectric Constant	Implied $\leq 30$
Polarity	Moderate
Specific Conductivity	Usually $< 10 \text{ mScm}^{-1}$ , "good"
Molar Conductivity	< 10 S cm <sup>2</sup> mol <sup>-1</sup>
Electrochemical Window	More than 2V, even 4.5V
Solvent and/or Catalyst	Excellent for many organic reactions
Vapor Pressure	Usually negligible

#### **Table 1 : Properties Of Ionic Liquid**

Particularly significant are the low vapor pressures in most instances which contrast the environmental problems of volatile organic solvents and moderate specific conductivities, usually in the same range as those of aqueous electrolytes. It is found that many such systems are excellent solvents or catalysts. Table 2 below show the comparison between ionic liquid with other liquids:

Compound	Temp (°C)	Molarity (Mol L <sup>-1</sup> )	Spec Cond (Scm <sup>-1</sup> )	Molar Cond (Scm <sup>2</sup> mol <sup>-</sup> <sup>1</sup> )	Viscosity (cP)		
Ionic Liquids							
NaCl	900	25.3	3.88	154	1.05		
LiCl	780	35	7.59	217			
NaNO <sub>3</sub>	450	21.4	0.72	34	6.9		
Na <sub>2</sub> SiO <sub>3</sub>	1750	18	4.8	270			
AlCl <sub>3</sub> (63%) – NaCl	175	5.8	0.24	41	3.85		
LiCl-KCl	450	29.7	1.57	53	2.44		
[(CH <sub>3</sub> ) <sub>3</sub> S][HBr <sub>2</sub> ]	25	7.3	0.034	4.7	20.5		
Emim Cl(60%)- AlCl <sub>3</sub>	25	5.3	0.0065	1.2	47		
Emim Al <sub>2</sub> Cl <sub>7</sub>	25	3.4	0.015	4.4	14		
Bmim CF <sub>3</sub> CO <sub>2</sub>	20	5.1	0.0032	0.6	73		
Bupy BF <sub>4</sub>	25	5.5	0.0019	0.3	103		
Emim [(CF <sub>3</sub> SO <sub>2</sub> ) <sub>2</sub> N]	mim CF <sub>3</sub> SO <sub>2</sub> ) <sub>2</sub> N] 25 3.7		0.0057	1.5	31		
Others							
H <sub>2</sub> O	25	55.3ª	4 x 10 <sup>-8</sup>	7 x 10 <sup>-7</sup>	0.895		
0.1 M aq. KCl	25	0.1 <sup>b</sup>	0.013	129	0.9		
Na	100	40.4	1.04 x	2.6 x 10 <sup>6</sup>	1.058		

### Table 2 : Comparison between Ionic Liquids with Other Liquids

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			10 <sup>5</sup>	and Second States (1997) All Second States (1997)					
H <sub>2</sub> SO <sub>4</sub>	25	0.049 <sup>b</sup>	0.0104	212	24.55				
CH <sub>3</sub> COOH	25	17.5 <sup>a</sup>	8 x 10 <sup>-9</sup>	4.6 x 10 <sup>-7</sup>	1.056				
HF	0	50.1 <sup>a</sup>	1 x 10 <sup>-6</sup>	2 x 10 <sup>-5</sup>	0.256				
a = total molarity, b = ionic molarity, emim = 1-ethyl-3-methyl-1H- imidazolium, bmim = 1-butyl-3-methyl-1H-imidazolium, bupy = 1- butylpyridinium									

One property that been emphasized recently is the molarity of the liquid, the molarity is important regarding kinetic measurements, including conductivity. Table 2 indicates a range of molarities of many liquid from 1 to 60, with water at 55, liquid alkali halides up to 35 and most organic salts less than 10. Specific conductivities span a far grater range from the metal sodium through molten inorganic salts in the Scm<sup>-1</sup> region to organic salts and aqueous solution in the mScm<sup>-1</sup> region and finally to the near non-conducting but ionizing acetic acid and water at  $\mu$ Scm<sup>-1</sup>.

Synthetics chemist have geared up virtually everything of what they do to being conducted in volatile solvents. After all, the solvent is not part of the product; just something added prior to the reaction and then later removed. While removal of the solvent by evaporation is no longer an option the reverse, distillation of the product may become possible instead, if it is sufficiently volatile. Other, well established techniques for the isolation of a product include extraction with supercritical CO2 or simple phase separation, provided that the product is immiscible in the ionic liquid.

#### 2.2.1. IONIC LIQUIDS AS SOLVENTS IN SYNTHESIS NANOPARTICLES

Nanoparticle catalysts have attracted large interest because they exhibit a high surface to bulk metal ratio, which allows more efficient use in practice. This aspect is frequently accompanied by large enhancement in the activity and selectivity where are used as catalysts (Roucoux, Schulz, & Patin, 2002). The recent achievement in this field allows control over the composition (alloy) and size (Furstner, Bonnenmann, & Richards, 1996). In spite of these advantages, nanocatalysts are

kinetically unstable with respect to agglomeration to the bulk metal and therefore require stabilization. Therefore, to be used they need stabilization by either surfaceligating anions or other ligands. In this way it is possible to realize additional control, mostly on the solubility in an appropriate solvent and their isolation (Feldheim & Foss, 2002). Combining different stabilizing effects may even improve the performance of nanoparticle catalysts (Aiken & Finke, 1999).

An important advantage of using ionic liquids in such preparations is that they facilitate inorganic synthesis from highly polar starting materials under ambient conditions and under anyhydrous or water-poor conditions. In this way, hydroxide or oxy-hydrate formation can be suppressed. The ionic liquid thought to limit diffusion of the nanoparticles and thus promote formation of small particles and limit their growth. Although imidazolium-based ionic liquids have been widely demonstrated as a good stabilizing agent for nanoparticles, in some cases it is found that agglomeration still leads to a loss of activity, indicating that ionic liquid stabilization alone does have limitations. Adding other materials to ionic liquids can combine different types of stabilizing effects, leading to more stable nanoparticles (Prvulescu & Hardacre, 2007).

The important character of ILs that makes the ILs suitable in nanoparticles can be summarized as below:

- High polarity and low suface tension
  - This combination enables fast formation of crystallites.
  - Growth inhibition of crystallites controlled by ILs.
  - Leads to very small particles with small size distributions.

#### Hydrophobicity

- Synthesis under non-aqueous conditions.
- Formation of hydroxides is suppressed
- Formation of molecular structures
- Weakly coordinating properties
  - The removal of suitable designed ILs is much easier compared to conventional solvents.

#### 2.3. SAMPLE CHARACTERIZATION TEST

#### 2.3.1. TRANSMISION ELECTRON MICROSCOPY (TEM)

In a TEM, the electrons are accelerated at high voltage (100-1000 kV) to a velocity approaching the speed of light (0.6-0.9 c). They must therefore be considered as relativistic particles. The associated wavelength is five orders of magnitude smaller than the light wavelength (0.04-0.008 Å). Nevertheless, the magnetic lens aberrations limit the convergence angle of the electron beam to  $0.5^{\circ}$  (instead of 70° for the glass lens used in optics), and reduce the TEM resolution to the Å order. This resolution enables material imaging and structure determination at the atomic level. A modified version of his technique, the energy dispersive spectrometry energy dispersive spectrometry (EDS) is nowadays usually added to the TEM.

A transmission electron microscope is constituted of two or three condenser lenses to focus the electron beam on the sample, an objective lens to form the diffraction in the back focal plane and the image of the sample in the image plane, some intermediate lenses to magnify the image or the diffraction pattern on the screen. If the sample is thin (< 200 nm) and constituted of light chemical elements, the image presents a very low contrast when it is focused. To obtain amplitude contrasted image, an objective diaphragm is inserted in the back focal plane to select the transmitted beam (and possibly few diffracted beam) the crystalline parts in Bragg orientation appear dark and the amorphous or not Bragg oriented parts appear bright. This imaging mode is called bright field mode BF. If the diffraction is constituted by many diffracting phases, each of them can be differentiated by selecting one of its diffracted beams with the objective diaphragm. To do that, the incident beam must be tilted so that the diffracted beam is put on the objective lens axis to avoid offaxis aberrations. This mode is called dark field mode DF. The BF and DF modes are used for imaging materials to nanometer scale. Figure below show the BF and DF mode.



condenser lens 1

condenser lens 2 condenser diaphragm

objective lens

objective diaphragm (back focal plane)

selected area diaphragm

intermediate lens

projective lens

final image screen

Figure 1 : TEM



# Figure 2: BF and DF Modes for Imaging

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#### 2.3.2. SCANNING ELECTRON MICROSCOPY (SEM)

The scanning electron microscope (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm). The SEM is also capable of performing analyses of selected point locations on the sample, this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions (using EDS), crystalline structure, and crystal orientations.

Accelerated electrons in an SEM carry significant amounts of kinetic energy, and this energy is dissipated as a variety of signals produced by electron-sample interactions when the incident electrons are decelerated in the solid sample. These signals include secondary electrons (that produce SEM images), backscattered electrons (BSE), diffracted backscattered electrons (EBSD that are used to determine crystal structures and orientations of minerals), photons (characteristic X-rays that are elemental analysis used for and continuum X-rays), visible light (cathodoluminescence--CL), and heat. Secondary electrons and backscattered electrons are commonly used for imaging samples. Secondary electrons are most valuable for showing morphology and topography on samples and backscattered electrons are most valuable for illustrating contrasts in composition in multiphase samples. X-ray generation is produced by inelastic collisions of the incident electrons with electrons in discrete ortitals (shells) of atoms in the sample. As the excited electrons return to lower energy states, they yield X-rays that are of a fixed wavelength. Thus, characteristic X-rays are produced for each element in a mineral that is "excited" by the electron beam. SEM analysis is considered to be "non-

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destructive". That is, x-rays generated by electron interactions do not lead to volume loss of the sample, so it is possible to analyze the same materials repeatedly.

The SEM is routinely used to generate high-resolution images of shapes of objects and to show spatial variations in chemical compositions (usually EDS, also BSE and CL). This instrument is also widely used to identify phases based on qualitative chemical analysis and/or crystalline structure. Precise measurement of measurement of very small features and objects down to 50 nm in size is also accomplished using the SEM. Backescattered electron images can be used for rapid discrimination of phases in multiphase samples. SEMs equipped with diffracted backscattered electron detectors can be used to examine microfabric and crystallographic orientation in many materials (Argast & Tennis, 2004) (Beane, 2004) (Moecher, 2004). Figure below show the schematic drawing of SEM:



**Figure 3: Schematic Drawing of SEM** 

#### 2.3.3. THERMOGRAVIMETRIC ANALYSIS (TGA)

TGA is a method that is based on the measurement of weight change in relation to temperature. The measurement provides basic information about the thermal stability of a chemical and its composition. TGA is one of the most commonly requested thermal techniques and it is used to characterize both inorganic and organic materials including polymers. The measurement is typically carried out on milligram size sample with a constant heating rate from 20°C to 1000°C and usually an inert environment of nitrogen or argon. This may employ a high resolution heating method which is based on adjusting the heating rate at the temperature at which the conversion of solid mass into gaseous degradation products reaches a maximum, has significantly increased sensitivity as compared to the standard methodology. In addition to gaining resolution by adjusting the heating method, the sample's surrounding atmosphere may be change to cause degradation of particular component (Philips & Blazley, 1991) (Rennecker, Zink-Sharp, Ward, & Glasser).



Figure 4: TGA Graph

Specific measurements made by TGA include :

- Moisture and Volatiles Content
- Composition of Multicomponent Systems
- Themal Stability
- Oxidative Stability

- Shelf-Life Studies Using Kinetic Analysis
- Decomposition Kinetics
- Effect of Reactive Atmospheres



Figure 5: TGA System

#### 2.3.4. X-RAY DIFFRACTION (XRD)

XRD is a very important method to characterize the structure of crystalline material. The technique can typically be used for the lattice parameters analysis of single crystals, or the phase, texture or even stress analysis of polycrystalline materials. The technique is widely used in research and development applications and its use for production or quality control issues is also growing, benefiting from developments in hardware and software for high throughput capability. Most of the application XRD requires a beam with well defined spatial and spectral characteristics. X-ray optics is a critical component for obtaining the required beam specification at the sample. Multilayer X-ray optics is now widely used in XRD due to their balanced performances in terms of divergence, spectral purity and flux.



Figure 6: Reflection of x-rays From Two Planes of Atoms in a Solid



Figure 7 : Rietveld refinement of b-TCP with 1.7% HA

Figure above shows a portion of the experimental diffraction patern of a mixture b-Tricalcium Phosphate(b-TCP) mixed with trace amounts of Hydroxyapetite (HA). Individual data points are shown as discrete dots and the computed profile is shown as the continuous line. At the top of the figure is shown the residual error between the computed and experimental curves. Finally, at the bottom of the figure are shown the stick patterns for the phases identified. Once the phases have been identified and the necessary atomic information extracted from the database, a computed profile is calculated. Then, by use of a least-squares technique, the residual error is minimized by modifying the lattice parameters, the scale factor, atomic parameters such as the thermal vibrations, atomic positions and occupancies. When the process is completed, the scale factors then provide the quantitative analysis.





In those cases when the details about the atomic and crystalline structure are unknown, then the use of calibration standards is often used instead. In this method, a number of control samples are made from the pure materials over composition range that overlaps the expected range in the target. Diffraction patterns are then prepared for each control sample, and the intensity ratio is prepared based upon two unobscured peaks from each phase. From above figure, one peak for each phase is highlighted and the ratio of the intensities is computed. This process is then repeated for the other synthesized controls sample. All of the data are used to construct the calibration curve shown as figure below. When an unknown sample is tested, the curve is used in reverse to estimate the quantitative amount through the measured intensity ratio.

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Figure 9: Calibration curve for TCP/HA mixtures

# 3. METHODOLOGY

# **3.1. RESEARCH METHODOLOGY**



Figure 10 : First Experiment (Using ILs as the solvent)

0.5M of Zinc Nitrate Hexahydrate (3.624g) and 5M of Sodium Hydroxide (6g) were prepared.

Zn(NO<sub>3</sub>)<sub>2</sub>6H<sub>2</sub>O and NaOH were mixed under manual stirring and followed by sonication for 30minutes.

The mixture were transferred into a Teflon-lined autoclave, which was subsequently filled with 2ml of ethanol.

The autoclave was put into the oven under 200°C

Filtrate the mixture and dried under 100°C in the oven for 1 hour

Figure 11 : Second Experiment (without Using ILs as Solvent)

Both figures show the experimental procedure of synthesis nanoparticles. For the first experiment, author use IL in order to synthesis the IL while for the second experiment, the author ran the experiment without using ILs in the synthesis of nanoparticles.

# **3.2. PROJECT ACTIVITIES**

Literature Review

Experimentation

# Sample Testing

Report & Presentation

**Data Analysis** 

#### **Figure 12 : Project Activities**

Above figure show the overall of the project activities. The first activity is searching for the literature review and reading material that related to the project in order to have the better understanding and make the feasibility study of the project. After that, the author proceeds the project with the Experiment. After getting the sample from the experiment, author makes the testing for each sample. The tests needed are TGA, SEM, TEM and XRD. The author makes the analysis from the data before proceed with the report and presentation.

# **3.3. GANTT CHART**

No	Detail/Week	1	2	3	4	5	6	7	in discussion designed	8	9	10	11	12	13	14
1	Project work begin			diger.	1.19	- 1 <sup>13</sup>	an e						al said an a'			
2	Submission of Progress Report 1															
3	Project work continues	5 A		100												
4	Submission of Progress Report 2					100		land Data	Seme							
5	Seminar		=(		1.1	- 2 K			ste							
6	Project work continues			12		apres.	100	and the								
7	Poster Exhibition						and the		] Te	301	12					The states
8	Submission of Dissertation			10 - 10 - 10 - 10 - 10 - 10 - 10 - 10 -					×							
9	Oral Presentation			127							100					1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1
10	Submission of Project Dissertation															

### Figure 13 : Final Year Project Gantt Chart

Figure above shows the gantt chart of final year project. It has been suggested by the coordinator of final year project. Every student must send 2 progress report and a dissertation before the final oral presentation at week 13. The hardbound final report will be submitted after the final presentation which expected on week 14.

# 4. RESULT AND DISCUSSION

In the ILs there are three main parts or structure that will contribute on controlling the size and morphology of the nanoparticles, which are alkyl chain, cation and anion. Figure below show the structure of the ILs.



**Figure 14 : Ionic Liquid Structure** 

From the first experiment, the author can study the relation of the cation and anion in the ILs with the size and morphology of the nanoparticles. Because of some problems, the author did not manage to get the result for the experiment even though the experiments have been run twice. The author will discuss based on the expected result. The size of the nanoparticles depends on the nucleation rate. The faster nucleation rate, the smaller particle size will be. The anions in the ILs will control the nucleation rate. With different anion in the ILs will give different effect to the nucleation rate. Besides that, the anion in the highly hydrophilic could have a strong interaction with water due to formation of strong hydrogen bonds which can lead to blocking the interaction of the IL with the particles. Anion is important role in controlling the size of the nanoparticles but cation will control the morphology of the nanoparticels.



Figure 15 : Sem for Cu

In the second experiment the author produced nanoparticles of Zn and Cu. Above figure show the SEM for the Cu. The size of the nanoparticles is less than 100nm. However, there are a lot of augmentations in the product. Unfortunately because of some problem the author did not manage to get the sem for Zn.

# 5. CONCLUSION AND RECOMMENDATION

Unfortunately the author did not achieve the objective of the project. However the author can conclude from the reading and some research, the factors that will control the size are anion and cation in the ILs. IL is suitable as the solvent in the synthesis nanoparticles. With the nature of the IL, the size of the nanoparticles can be controlled.

For the future study the author recommend, to extend the research on the affect of alkyl chain in the ILs structure to the size of nanoparticles and study on the optimum condition for experiment in order to produce high quality product. Diameter of the particles will decrease with the increase of the carbon in the alkyl chain. The size of nanoparticles varies with the kind of ILs used because of the prevention of coalescence between particles by the strong adsorption of ILs species on the surface and by electrostatic repulsion between the resulting particles.

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