CHAPTER 3 METHODOLOGY

This chapter discusses the methodology to achieve the goal of this research.

3.1 Methodology

There are five steps to complete this research as shown in Figure 3.1. It begins with studying and gathering the overview of furfural, urea and biodegradable polymer with other instruments used. Then, the complex of furfural-urea is synthesized. Next, the synthesized complex is characterized by NMR, TGA/DSC before combining with a biodegradable polymer. The mixture of complex and polymer is then entered through the extrusion process. Finally, the results are characterized and analyzed. The details of individual step are described below.

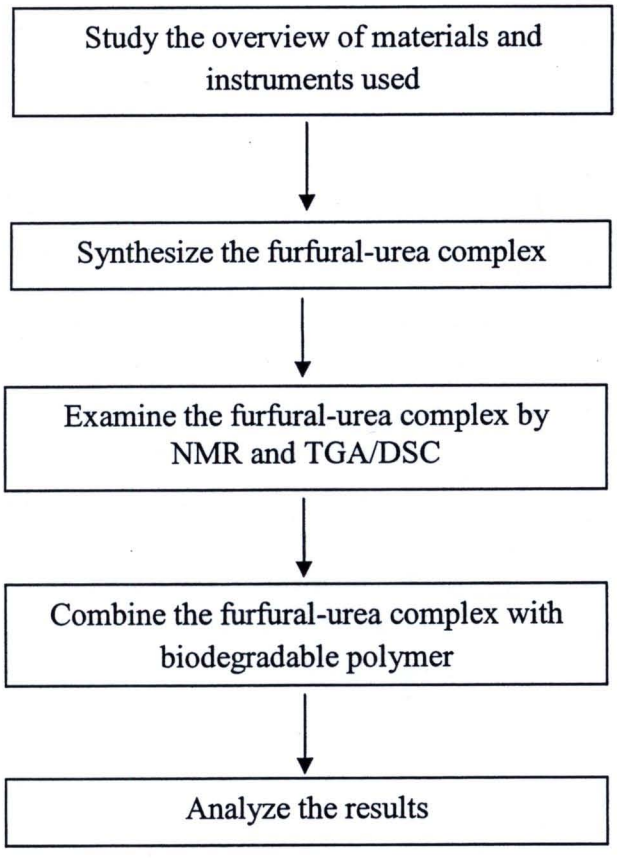

Figure 3.1 Methodology

3.1.1 Study the overview of materials and equipments used

Firstly, the natural structures and functions of both furfural and urea need to be understood. The synthesized procedure of furfural-urea complex is studied. Furthermore, the biodegradable polymer and the extrusion process are also studied.

3.1.2 Synthesize the furfural-urea complex

This step describes a material preparation in which furfural-urea complex is synthesized. The most likely structure formed is 3:2 (urea-furfural) according to the main reaction as shown in Figure 3.2.

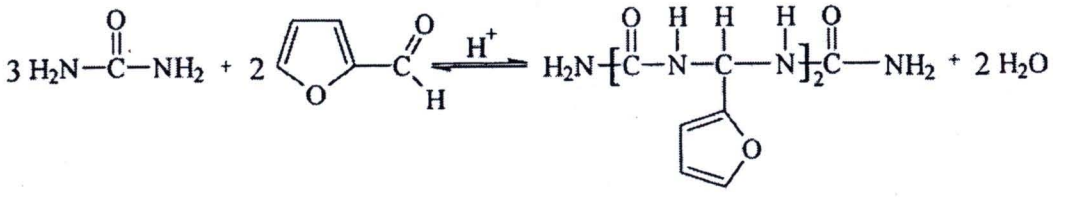

Figure 3.2 The furfural-urea complex's reaction

The synthesis is carried out through aqueous phase since the previous study was revealed by Martinez et al [4] that the reaction in homogeneous phase was faster than that in heterogeneous phase. Two to 1 ratio of urea to furfural is chosen since this proportion gives the highest yield comparing with other ratios as represented by Martinez et al [4]. To synthesize the furfural-urea complex, the following procedure is conducted. In the first step, urea is dissolved in water and furfural is added after no urea particle is observed in the solution. Normally, at least 5 minutes of stirring is required to dissolve urea completely in water. When the solution is completely mixed, HCl is incorporated into the sample before the sample is filtrated after a period of time. The filtration is carried out under the vacuum filtration after the mixture is homogeneous. Fifty ml of water is then added to eliminate the remaining reactants. Finally, the sample is dried until no water was observed. The drying time is typically 2 days. The schematic diagram of the synthesis procedure is shown in Figure 3.3

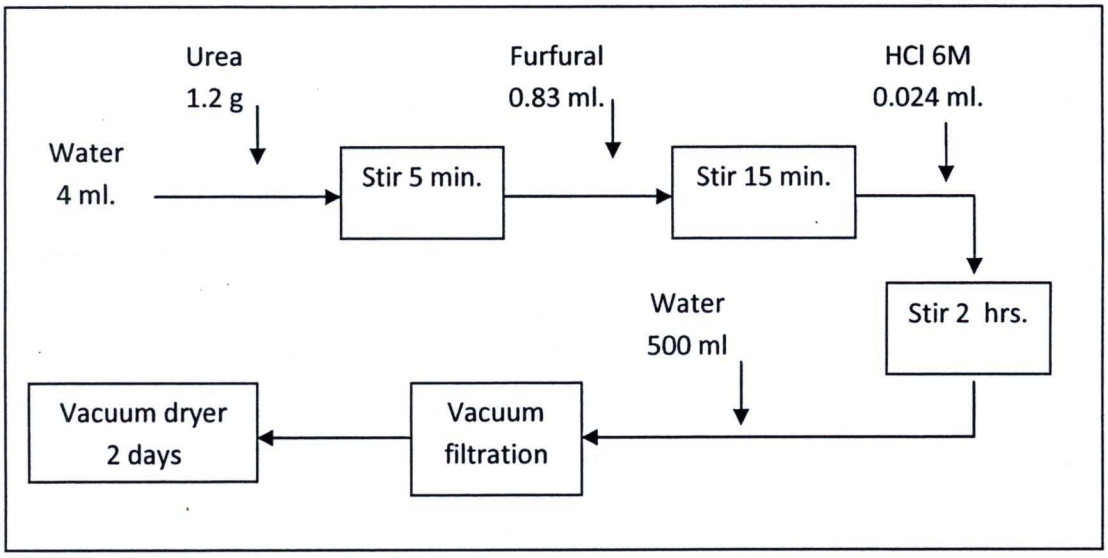

Figure 3.3 The schematic diagram of synthesis procedure

The amount of each material in a typical synthesis is shown in Table 3.1

Table 3.1 Material proportion for furfural-urea synthesis

	Proportion
Urea	1.2 g.
Furfural	0.83 ml.
Water	4 ml.
HCl (6M)	0.024 ml.

There are many significant factors that affect the qualities of the synthesized products. Firstly, the drying process is considered. Oven and vacuum dryer are two drying methods used to dry the sample after the samples is filtrated. Moreover, the reaction time is one factor that could end up with different compositions of products. For this synthesis, reaction time can be divided into 2 parts which are mixing time of urea and furfural and the other one is reaction time after adding HCl acid. Mixing times of urea and furfural to be considered are at 15 minutes and 2 hours, while time after adding HCl acid is varied from 1 hour to 1 day and 4 days. The amount of HCl acid, as it acts like a catalyst is also varied. Three and 5 times of typical acid amount are considered. Finally, the amount of water used to wash the filtrate is considered because the reactants can be dissolved in water more easily than the products. In this project, the use of 500 ml of water is compared with the typical value at 50 ml of water added.

3.1.3 Examine the furfural-urea complex by NMR and TGA/DSC

In the third step, the furfural-urea synthesized complex is characterized by using several techniques. Proton NMR is used to determine the structure of the material. To prepare the sample for proton NMR spectrometer, 3 mg of sample is dissolved in 1 ml of dimethysulfoxide (DMSO) which is selected as the NMR solvent. This solvent is deuterated solvent. The reason why deuterated solvent is used is to erase the magnetic alignment by adding 1 neutron as isotope. This added neutron does not affect the NMR magnetic by aligning opposite direction of proton. If there is only 1 proton align in the field, it should affect the signal by aligning with the NMR magnetic. In this study, proton NMR spectrometer with 300 MHz is used. Furthermore, TGA technique is a simple analytical technique that measures the weight loss or weight gain of a material as a function of temperature or time, while DSC measures the heat flow to or from a sample as a function of temperature or time.TGA/DSC of the complex is operated from room temperature to 200°C with 1°C/minute of heating rate.

3.1.4 Combine the furfural-urea complex with biodegradable polymer

After the furfural-urea complex is characterized by several techniques mentioned above, it is combined with a biodegradable polymer called poly (butylenes succinate-co-butylene adipate) (PBSA). The mixture of furfural-urea complex and PBSA is extruded via twin screw extruder to form a thin film. The sample is extruded at about 140°C with 50 screw rotational speeds. The reason why this temperature is chosen is that the operating temperature should not be significantly greater than its melting temperature so that the material will decompose. At this point 5% and 10% of furfural-urea complex imbedded with PBSA biodegradable polymer is conducted. The furfural-urea complex imbedded with PBSA polymer is mixed for 10 minutes since the longer the material stays in the extruder, the increase the risk of the material decomposition.

Moreover, solvent casting is one method that is used to study the mixing of PBSA and furfural-urea complex. Applicable solvent used in solvent casting is investigated. To form a film, the solvent is removed from the product by evaporation to form a film. The solvents used to test the solubility of furfural-urea complex and PBSA polymer are chloroform, tetrahydrofuran, ethanol and water. To produce a thin film of furfural-urea imbedded with PBSA polymer, 10% of furfural-urea complex is mixed with the PBSA polymer before adding into a solvent with high solubility. The solution is then stirred for 30 minutes to crush the complex particles by using mechanical agitator. After most particles are crushed, the sample is poured into a flat plate. The blower is used to dry the sample until the sample becomes a thin film.

The product films produced from both extrusion and solvent casting are characterized by FT-IR/ATR to determine how much furfural-urea complex imbedded in the new product.

In addition, the rate of the furfural-urea additive in the polymer film released from water is also determined by immersing the polymer film with furfural-urea complex in the water and measures the released amount of the materials. The amount of furfural-urea complex come out from the solution is then recorded by UV- visible spectrometer at different period of time. In addition, the standard curve of absorbance and the wavelength are also calibrated to determine the concentration of the released furfural and urea. Both UV and visible regions are considered to determine the absorbance range whether it is furfural or urea.

3.1.5 Analyze the results

In the last step, results are analyzed by dividing into 2 sections. The first section mentions the synthesis result while polymer film imbedded with furfural-urea complex is analyzed in the other section.