3 MATERIALS AND METHODS

3.1 Overview

This chapter presents a methodology to conduct the torrefaction experiment by using certain raw materials and experiment setup. Preparation of EFB samples and procedure for torrefaction experiment is included here. Several characterization tests are done to determine the characteristic of EFB and the effectiveness of torrefaction process.

3.2 Preparation of biomass samples

The raw materials that will be used in this study is the Empty Fruit Bunches (EFB) shown in Figure 3-1. EFB is the residue generated at the thresher, where fruits are removed from fresh fruit bunches. EFB were collected from an oil palm plantation in Lepar, Pahang. About 5 kg EFB samples were collected from the site. The raw EFB is dried in the sun before proper drying process. For the drying process, a bunch of EFB samples is weighed and then placed in a tray. Then, the EFB samples were dried in an oven at the temperature of 65°C for 24 hours to provide a basis for experiments. The step was repeated until 500 gram of the EFB samples was accumulated. For this, the raw EFB samples were further ground using the mechanical grinder and sieved using sieve shaker. From the sieved samples it was determined that the largest particle size that would allow for an adequate sample amount during torrefaction experiment were samples below 1 mm size. The ground samples were removed and placed in airtight plastic bags and stored until needed.

For the initial analysis, the samples will be analysed for proximate, elemental (ultimate) analyses as well as the calorific value (CV). For further preparation that is required for use in TGA experiment, the EFB will be chopped into small pieces by using a chopper and grounded using a laboratory grade blender. The Figure 3-2 shows the flow chart of preparation of samples.
The EFB samples are collected from the oil palm plantation in Felda Lepar

Then, the EFB samples is dried in an oven with the temperature of 65°C for 24 hours

After that, the EFB samples are grinded into a mechanical grinder to get about 8 mm to 5 mm in the range of size.

EFB samples is blended inside the laboratory blender to minimise the size into 2mm and below.

Next, the samples are sieved into different range of size such as 2 mm-1 mm, 0.99 mm to 0.5 mm and 0.49 mm to 0.25 mm.

The sample is analyzed to get the proximate, ultimate and particle density for initial analysis.

The samples are cooled and stored in the airtight plastic bags until further use.

**Figure 3-1** Raw Empty Fruit Bunches (EFB)

**Figure 3-2** Flow Chart of Preparation of Samples
3.3 Torrefaction experiments

The experimental setup consists of a N$_2$ cylinder tank, CO$_2$ cylinder tank, and a vertical stainless-steel tubular reactor equipped with an electrically heated furnace and a liquid/gas collection unit. The experiment consists of two parts; the first part is inert torrefaction that only nitrogen gas flowed into the reactor. The second part is carbon dioxide and nitrogen that co-feed flowed into the reactor. The nitrogen and carbon dioxide tank was used to supply nitrogen and carbon dioxide as a carrier gas for providing an inert environment in the torrefaction experiment. The volumetric flow rate of nitrogen was controlled by an electronic mass flow controller. A schematic diagram of the experiments is shown in Figure 3-3.

A total of 3 g of EFB sample was weighed and put in a tubular reactor. The reactor was placed at the center of the furnace. When the EFB samples were in the furnace, they are heated in the furnace at a heating rate of 10 °C/min until the desired torrefaction temperature is reached at 200 - 300°C. N$_2$/CO$_2$ was used to purge the reactor depending on the medium used for torrefaction.

A constant flow of 50 mL/min of N$_2$/CO$_2$ was set using variable area mass flow controller to maintain inert/non reacting environment during the process. After flushing the reactor with torrefaction gas and different concentration of CO$_2$ the temperature is set at 240°C and 280°C. The samples were heated from room temperature to the desired temperature and kept constant within 30 minutes. After being torrefied for 30 minutes, the furnace is turn off and the reactor is left to cool to ambient temperature. Then the torrefied sample is recovered, weighed and kept in airtight plastic bags prior to further analysis. The gas product from the decomposition reaction of biomass is channelled to the bottom of reactor, passed through a condenser at room temperature. The concentration of carbon dioxide in the gas stream is also adjusted to 0%, 10%, 15% and 21vol% to investigate the effect of carbon dioxide concentration on torrefaction. The torrefaction gas is prepared by mixing the nitrogen with carbon dioxide under ambient temperature. Figure 3-4 summarizes the work flow of experiments after 30 minutes in the furnace.