CHAPTER 3

MATERIALS AND METHODS

3.1 INTRODUCTION

In this chapter, the methodology of the research as well as the materials used will be explained in detail. These include the preparation of ACs using different activation agents, step by step procedures of material characterizations using different techniques. The performance of ACs was then evaluated using electrochemical procedures. Figure 3.1 displayed the flow of the procedure taken in completing this work.

3.2 MATERIALS

The PKS were charred in a electrical furnace. Chemical used as activating agents were sodium hydroxide (NaOH), potassium hydroxide (KOH), sulfuric acid 98% (H_2SO_4) and zinc chloride ($ZnCl_2$). Hydrochloric acid (HCl) was used in post-treatment of ACs. In electrode preparation process polyvinylidene fluoride (PVDF) used as binder N-methyl-2-pyrrolidone (NMP) as solvent and Super-P (carbon black) was use as conductive carbon.. All the solutions in this work were prepared using deionized (DI) water.

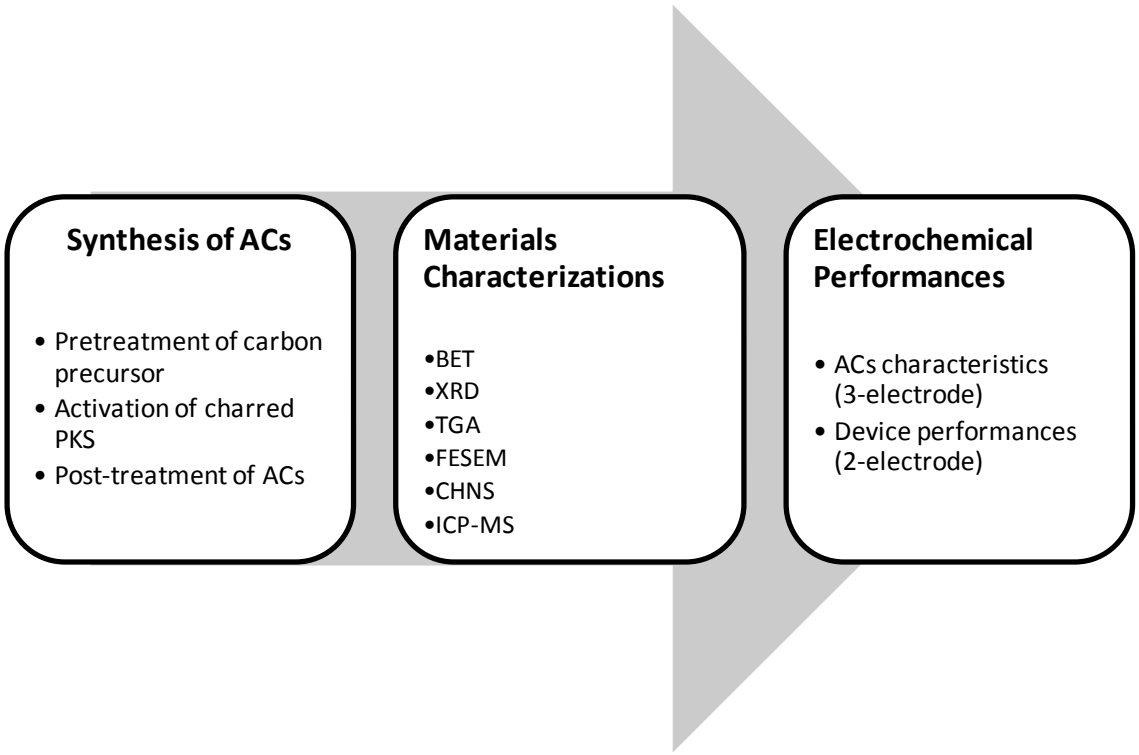

Figure 3.1: Flow chart of this work.

3.3 PREPARATION OF ACTIVATED CARBON

3.3.1 Pre-Treatment of Carbon Precursor

The PKS waste was collected from oil mill factory. The PKS was washed thoroughly with distilled water to remove soil and dirt by sonication process and then dried at 105 °C to remove moisture. PKS was charred at 500 °C for 1 hour at heating rate 10 °C/min and the resultant charred PKS was separated from impurities before proceed into activation step. Charred PKS was store in airtight container to prevent moisture absorption.

3.3.2 Activation of Charred PKS using Different Activation Agents

In this study, four activation agents were used to activate PKS char that is (1) KOH (2) NaOH (3) H_2SO_4 and (4) $ZnCl_2$. Activation agents used were commonly used as activation agents to produce ACs. As-prepared charred PKS produced from previous

work was treated with different impregnation ratio (0.5, 1.0 1.5 and 2.0). Impregnation ratio is defined as the ratio of mass activation agents to mass of charred PKS. Charred PKS was introduced into 50% m/v solution of activation agent for 24 h by sonication process. The impregnated charred PKS was then dried at 105 °C in an electrical oven to remove moisture then carbonized in an electric furnace at heating rate of 10 °C/min and the final temperature of 500 °C with holding time of 2 hours. The summary of carbonization condition was illustrated in Table 3.1. The resultant ACs were washed repeatedly with 1 M HCl and then rinsed with hot deionized water until pH 8-7 was obtained then dried at 105 °C. The ACs were then stored in airtight bottle samples to prevent moisture absorption. Resultant ACs then named X-Y where X was the activation agent used and Y was the impregnation ratio of the activation agent used. The example of activation condition was shown in Table 3.2.

Table 3.1: Synthesis condition of ACs.

Start Material	Activation agent	Impregnation time	Activation temperature	Activation time
Charred PKS	KOH NaOH H_2SO_4 $ZnCl_2$	24 hours	500 °C	2 hours

Table 3.2: Example of activation condition in different impregation ratio.

Mass of charred PKS (g)	Impregnation ratios	Mass of activation agent (g)	Volume of deionized water (ml)	Designated name
15	0.5	7.5	7.5	KOH-0.5
	1.0	15.0	15.0	KOH-1.0
	1.5	22.5	22.5	KOH-1.5
	2.0	30.0	30.0	KOH-2.0