3 MATERIALS AND METHODS

3.1 Introduction

For this chapter, there is an explanation of the material used and detailed procedure that were used to run this experiment to achieve the objective of this research. Summarization of the experimental flow chart related to experimental procedure is shown in Figure 3-1.

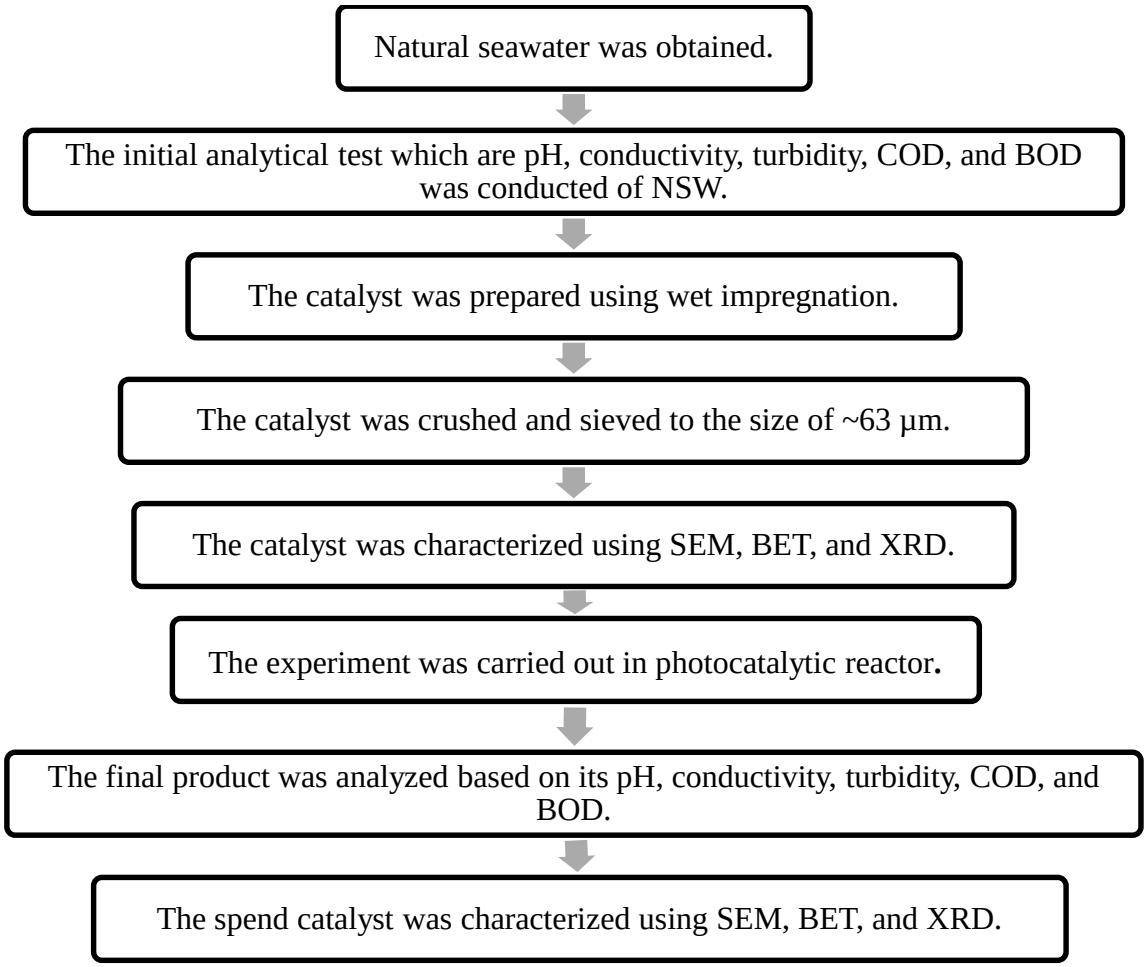

Figure 3-1: Flow diagram of experimental procedure.

3.2 Material

For this research, the raw materials used for photocatalytic reaction preparation were natural seawater (NSW) and palm oil fibre ash (Biomass ash). While the chemicals used for these research were Titanium Dioxide (TiO_2) catalyst, Nickel Nitrate Hexahydrate ($Ni(NO_3)_2.6H_2O$), and Ferum (III) Nitrate nonaydrate ($Fe(NO_3)_3.9H_2O$).

3.2.1 Natural Seawater (NSW)

The NSW sample from Teluk Cempedak Kuantan, Pahang. The sample was stored at 4 °C in the refrigerator.

3.2.2 Oil palm fiber ash (Biomass ash)

Oil palm fiber ash was obtained from Felda Lepar Hilir 3 palm oil mill, Gambang, Kuantan, Pahang. It was used as a catalays supporter for TiO_2.

3.2.3 Chemical

The chemical used was TiO_2 powder with 99% purity, Nickel Nitrate Hexahydrate ($Ni(NO_3)_2.6H_2O$) with 99% purity, Iron(III) Nitrate Nonahydrate ($Fe(NO_3)_3.9H_2O$) with 99% purity. These three chemical was obtained from Sigma-Aldrich Malaysia.

3.3 Preparation of Solution and Catalyst

3.3.1 Preparation of NSW

For the natural seawater, the seawater was obtained from Teluk Cempedak, Kuantan, Pahang. The NSW was stored in a refrigerator at 4 °C. The volume of the NSW used for each experiment was depend on the weight ratio of 1:400 which represent the weight of catalyst and the weight of the NSW respectatively.

3.3.2 Preparation of Catalyst

For this research, three type of catalyst were produced via wet impregnation. The detail of the catalyst is illustrated in Table 3-1. The solution was stirred by using hot plate at 80˚C for four hours. The solution was then dried overnight at 100˚C in the oven. After that, the solution was calcined at 500˚C for four hours in the furnace. The solution was then crushed and sieved to the size of ~63µm.

Table 3-1: The weight ratio of the three catalyst.

Catalyst	**Weight Ratio (%)**		
	TiO_2	**Biomass Ash**	**Metal(Nickel/Ferum)**
No metal loading	50	50	0
Nickel loading	45	45	10
Ferum loading	45	45	10

3.3.3 Characterization of Catalyst

The fresh and used catalyst were characterized by using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Brunauer Emmett Teller (BET). The catalyst was characterized by using XRD to determine the crystal structure of the catalyst. While SEM was used to determine the catalyst's surface mophology. BET surface analysis was used to measure the total specific surface area, pore size and pore volume of the catalyst.

3.3.3.1 Scanning Electron Microscopy (SEM)

SEM (CAEL ZEISS) was used for catalyst characterization and surface morphology analysis. A high resolution SEM coupled to an energy dispersive X-tray spectrometer system was producing various signals contained the information regarding the sample’s surface topography and composition. Then the samples were mounted on a stub of metal with adhesive, coated with 40 - 60 nm of metal such as Gold/Palladium and then were observed in the microscope.