

## CHAPTER III

### EXPERIMENTAL

#### Materials

- Used marine lubricating oil was obtained from Royal Thai Navy.
- NiO/MoO<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> was commercial available from United Catalyst Inc.
- NiO/WO<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> was commercial available from United Catalyst Inc.
- Raney nickel was commercial available from Merck.
- Universal indicator paper was commercial available from Merck.

#### Apparatus

##### 1) Hydrogenator

The apparatus which consisted of four parts as follow:

##### a) Reactor

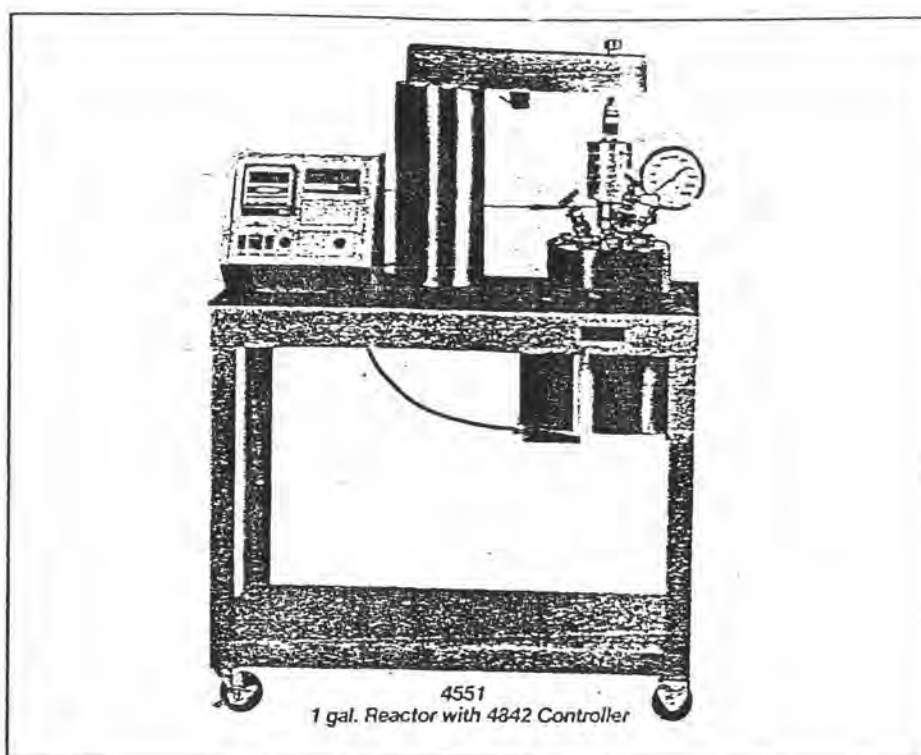
The reactor was a high pressure batch stirred autoclave model 4551 from the Parr Instrumental Company with 3750 cubic centimeters stainless steel 316 cylindrical bomb, split ring closures and bomb heater. The reactor could work in a pressure range of 0-2000 psig and in temperature range of 0-450 °C. The reactor was shown as figure 3.1.

##### b) Reactor fittings

The stirred reactor was equipped with convenient valves and fitting for handling the various functions as shown in Figure 3.2.

c) Automatic temperature controller

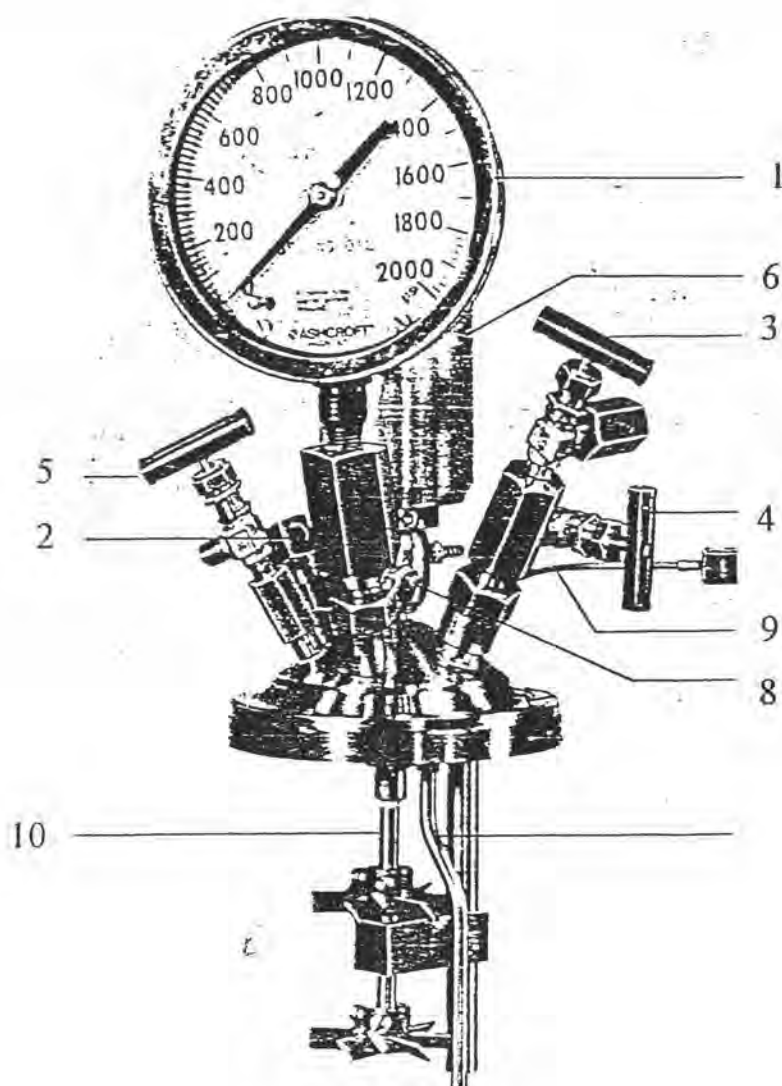
The controller used was model 4842 PID controller from Parr Instrumental Company. It was operated with a dual thermocouple. There were various enhancement modules to assist in monitoring and controlling the temperature, pressure, and stirring speed. The range of stirring speed could be adjusted in the range of 0-1000 rpm.



**Figure 3.1** Floor stand reactor

d) Gas controller system

The system consisted of a hydrogen tank with a pressure regulator (0-200 psig).



- |  |                                    |
|--|------------------------------------|
| 1. a pressure gauge                                      | 2. a safety rupture disc           |
| 3. a gas inlet valve                                     | 4. a liquid sampling valve         |
| 5. a gas release valve                                   | 6. a stirrer magnetic drive system |
| 7. a water cooling channel                               | 8. a thermocouple                  |
| 9. a dip tube  |                                    |
| 10. a stirring shaft with 6-blade turbine type impellers |                                    |

**Figure 3.2** Reactor Fitting

2) Viscometer

The apparatus model K-234A from Hochler Instrumental Co.,Inc. was used.

3) Colorimeter

The fisher ASTM Colorimeter was used.

4) Sulfur Analyzer

The apparatus model SLFA-800 from HORIBA was used.

5)  $^{13}\text{C}$ -NMR spectrometer

The NMR spectrometer model AC-F 200 from Bruker was used.

6) Thermogravimetric Analyzer

The TGA model NETZSCH STA 409 C was used.

**Procedure [49,50,58]****A. Effect of Catalyst Types on Hydrogenation of Used Oils.**

The used lubricating oil was charged into the reactor and the required quantity of catalysts were added. The reactor was then closed. After a thermocouple was inserted into a sturdy thermowell, connecting the stirring motor, and passing water into the cooling channel. Before run the process all oxygen gas in the reactor was removed by purging the system with some partial pressure of hydrogen gas (about 10-20 psig) for 2 minutes.

To achieve the catalytic hydrogenation conditions, temperature, time were set by the step controller, whereas the pressure of hydrogen gas was adjusted by the gas regulator. All tests were run under conditions below:

weight of used oil	=	300 g
reaction temperature	=	350 °C
pressure	=	500 psig
reaction time	=	30 minutes
stirring speed	=	300 rpm
concentration of catalyst	=	40 %
catalyst used	=	R-Ni
		NiO/MoO <sub>3</sub> /Al <sub>2</sub> O <sub>3</sub>
		NiO/WO <sub>3</sub> /Al <sub>2</sub> O <sub>3</sub>

## **B. Determination of Physical and Chemical Properties of Hydrogenated Oils.**

### Physical Properties :

1. Color, visual (ASTM D 1500)
2. Pour Point (ASTM D 97)
3. Kinematic Viscosity (ASTM D 445)
  - @ 40 °C
  - @ 100 °C
4. Viscosity Index (ASTM D 2270)
5. Flash point (ASTM D )
6. Sulfur Content (ASTM D )

### Chemical Properties :

1. The percentage of oxidation compounds and oxidation point were determined by Thermogravimetric Analyzer.
2. The composition (%Cp, %Cn, %Ca) of oils were determined by <sup>13</sup>C-NMR method.

### **C. Effect of Time on Hydrogenation of Used Oils.**

This step, used oil was treated under conditions below:

weight of used oil	= 300 g
reaction temperature	= 350 °C
reaction time	= 60, 30, 20, 10 minutes
stirring speed	= 300 rpm
pressure	= 500 psig
concentration of catalyst	= 40 %
catalyst used	= NiO/WO <sub>3</sub> /Al <sub>2</sub> O <sub>3</sub>

### **D. Determination of Physical and Chemical Properties of Hydrogenated Oils.**

All properties were done as in section B.

### **E. Effect of Concentration of Catalyst on Hydrogenation of Used Oils.**

This step, used oil was treated under conditions below:

weight of used oil	= 300 g
reaction temperature	= 350 °C
reaction time	= 10 minutes
stirring speed	= 300 rpm
pressure	= 500 psig
concentration of catalyst	= 40, 35, 30, 20 %
catalyst used	= NiO/WO <sub>3</sub> /Al <sub>2</sub> O <sub>3</sub>

### **F. Determination of Physical and Chemical Properties of Hydrogenated Oils.**

All properties were done as in section B.



### **G. Effect of Time on Hydrogenation of Used Oils.**

This step, used oil was treated under conditions below:

weight of used oil	= 300 g
reaction temperature	= 350 °C
reaction time	= 60, 30, 20, 10 minutes
stirring speed	= 300 rpm
pressure	= 500 psig
concentration of catalyst	= 40 %
catalyst used	= NiO/MoO <sub>3</sub> /Al <sub>2</sub> O <sub>3</sub>

### **H. Determination of Physical and Chemical Properties of Hydrogenated Oils.**

All properties were done as in section B.

### **I. Effect of Concentration of Catalyst on Hydrogenation of Used Oils.**

This step, used oil was treated under conditions below:

weight of used oil	= 300 g
reaction temperature	= 350 °C
reaction time	= 10 minutes
stirring speed	= 300 rpm
pressure	= 500 psig
concentration of catalyst	= 40, 35, 30 %
catalyst used	= NiO/MoO <sub>3</sub> /Al <sub>2</sub> O <sub>3</sub>

### **J. Determination of Physical and Chemical Properties of Hydrogenated Oils.**

All properties were done as in section B.

**K. Effect of Time on Hydrogenation of Used Oils.**

This step, used oil was treated under conditions below:

weight of used oil	= 300 g
reaction temperature	= 350 °C
reaction time	= 30, 20, 10 minutes
stirring speed	= 300 rpm
pressure	= 500 psig
concentration of catalyst	= 40 %
catalyst used	= Raney-Ni

**L. Determination of Physical and Chemical Properties of Hydrogenated Oils.**

All properties were done as in section B.

**M. Effect of Concentration of Catalyst on Hydrogenation of Used Oils.**

This step, used oil was treated under conditions below:

weight of used oil	= 300 g
reaction temperature	= 350 °C
reaction time	= 10 minutes
stirring speed	= 300 rpm
pressure	= 500 psig
concentration of catalyst	= 40, 35, 30 %
catalyst used	= Raney-Ni

**N. Determination of Physical and Chemical Properties of Hydrogenated Oils.**

All properties were done as in section B.