CHAPTER III
EXPERIMENTAL

3.1 Materials

3.1.1 API Separator Sludge (American Petroleum Institute Separator Sludge)

API separator sludge is obtained from an API separator as shown in Figure 3.1 in the wastewater treatment plant of PTT Public Company Limited, Thailand. Within the sludge matter, it contains high amount of solid and oil mixed in emulsion form. "As-received" sample appears to be black, high in viscosity and high volatiles.

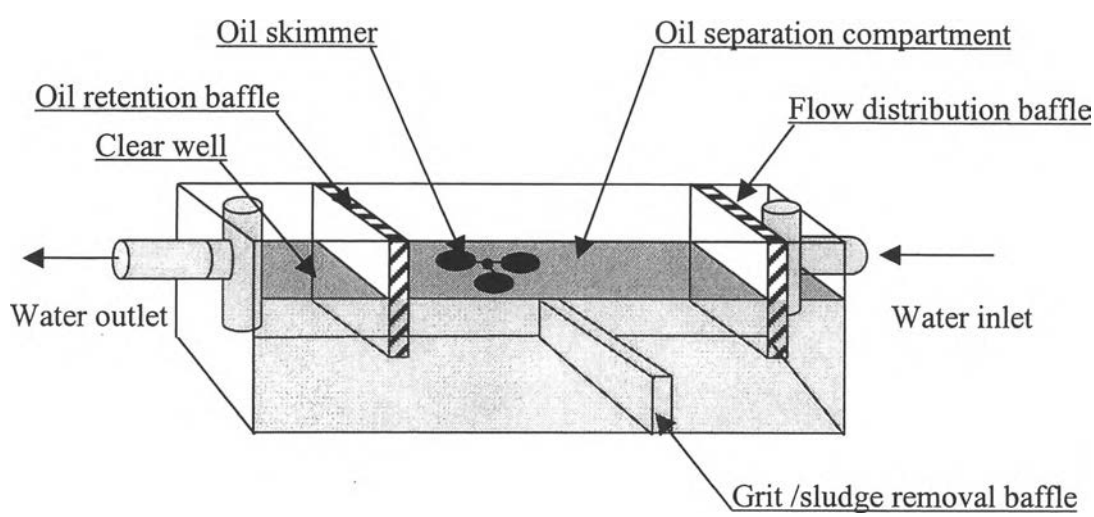

Figure 3.1 Schematic diagram of the simplest API separator system (Conference on Urban Runoff Management, 1993).

3.1.2 Nitrogen and Helium

Nitrogen and Helium with 99+% was used in Thermogravimetric analyzer and Mass spectroscopy, respectively. These two types of gas were used to control pyrolysis condition of dried API separator sludge sample.

3.2 Equipment

3.2.1 Centrifugal Machine

Centrifuge machine (Model 4236) of ALC International Co, Ltd was used to separate phases of "As-received" sample contained in centrifugal tube of 50 ml.

3.2.2 Vacuum Oven

Sanyo Gallenkamp vacuum oven Model OVA031.xx1.5 was used to dry the "As-received" sample by control temperature at 110°C for 9 hrs.

3.2.3 Thermogravimetric Analyzer (TGA) Instrument

DuPont TG-1000 thermogravimetric analyzer was used to collect the data of sample weight loss during pyrolysis of dried sludge sample at all range of setup temperature.

3.2.4 TPDRO and Mass Spectroscopy

TPDRO 1100 of Thermo Finnigan Co., Ltd. was connected with Omnistar™ Mass spectrocopy. TPDRO 1100 was used to pyrolyze the dried API sludge sample while Omnistar™ Mass spectrocopy was collected the pyrolysis products after pyrolysis from TPRDO 1100 at the same time. This system is powerful tool to study the basic of pyrolysis of an API sludge behavior.

3.3 Experimentals

3.3.1 Sample Preparation

Before analyzing, water was removed from the as-received sample by a centrifuge machine with 4000 rpm for 30 min. under atmospheric pressure and room temperature. Amounts of separated phases were measured for its composition. Then, a vacuum oven at temperature 110°C dried solid part for 9 hours. The dried sludge was kept in a clean and dried desecrator.

Dried sludge sample was analyzed by ASTM D3172-89 (Proximate Analysis) to get the raw data consisted of Ash%, Volatile matter%, Fixed carbon%, and Moisture%. Heating values of sample were obtained by a bomb calorimeter followed by ASTM D2015.

3.3.3 Thermogravimetric Analysis (TGA) Analysis

The pyrolysis of the API separator sludge were carried out in a DuPont TG-1000 thermogravimetric analyzer with 5, 10 and 20°C · min^{-1} heating rates from room temperature to 700°C under the nitrogen atmosphere. Approximately, 10 to 20 mg of the dried sludge were used for each experiment. The sample was loaded in a platinum plan in the heated zone of TGA. A small thermocouple probe (type K) measured the temperature in the vicinity of the sample. The experimental results of thermogravimetric analysis (TGA) and differential thermogravimetric analysis (DTG) were simultaneously recorded. In this work, each experiment was repeated at least twice. The TGA system is shown in Figure 3.2.

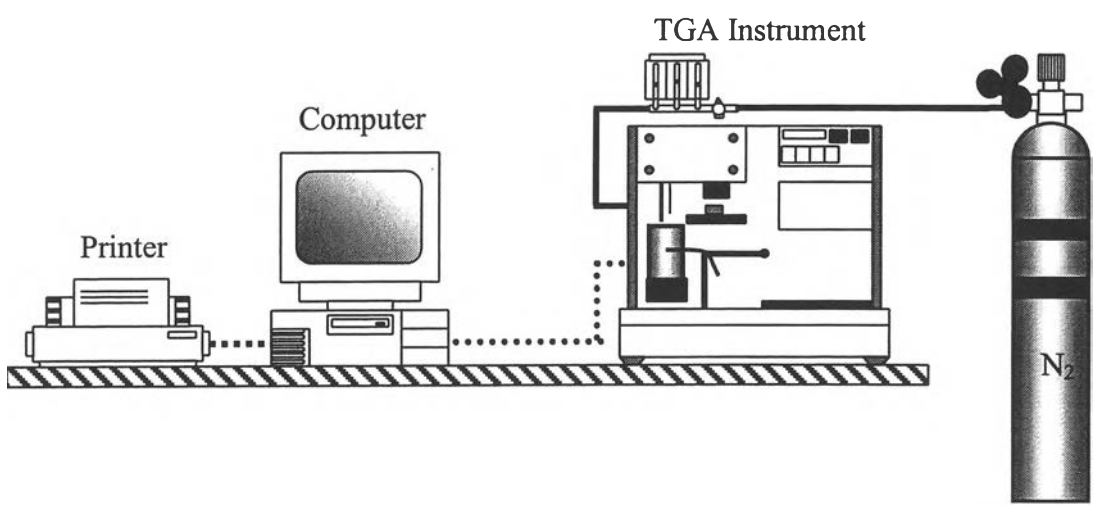

Figure 3.2 The thermogravimetric analyzer (TGA) system.

3.3.4 TPDRO and Mass Spectroscopy

As widely used as a means to analyze products from the thermal decomposition, an OmnistarTM mass spectrometer (MS) was employed here. The dried API sludge sample of 50 mg was held between the quartz wool plugs placed at the bottom of quartz tube of TPDRO 1100 instrument. The purpose of using quartz wool was to prevent the explosion of sludge during receiving heat and to distribution of temperature to sludge matter. The operating temperature was around 100 to 700°C with the heating rate of $10°C \cdot min^{-1}$. Helium with the flow rate of 25 $cc \cdot min^{-1}$ was used as the carrier gas and was pass through the reactor until desired temperature was reached. The products from pyrolysis leaving the reactor with helium gas were passed directly through an OmnistarTM mass spectrometer (MS) as shown in Figure 3.3. To standardize the MS signal, ion mass calibration is carried out prior to the analysis of each experiment. Selected ion monitoring (SIM) mode was employed to collect the ion intensity (eV) of gaseous products.

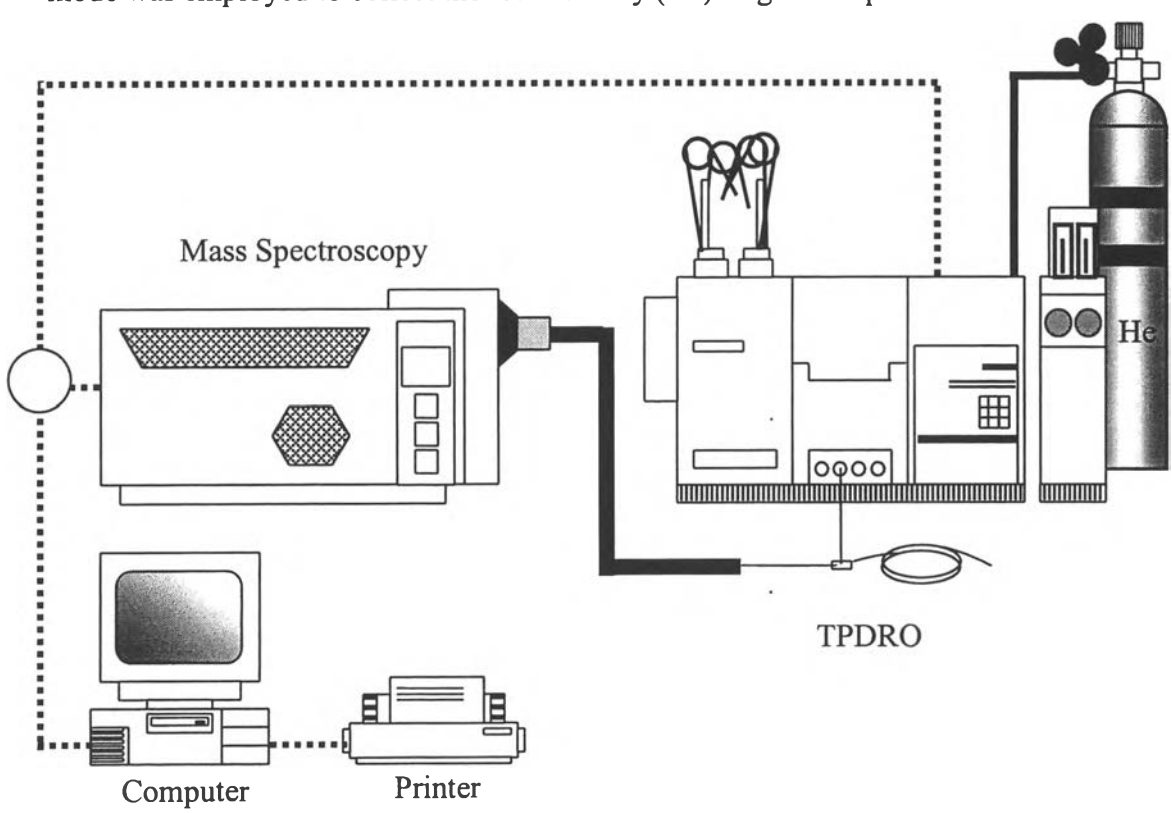

Figure 3.3 TPDRO and Mass spectroscopy system.