

EXPERIMENTAL  
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## **6. EXPERIMENTAL**

Two vacuum residues ( $VR_s$ ) were delivered, one from Alexandria Petroleum Company and the second from Suez Petroleum Company.

Three solvents, *n*-pentane, *n*-heptane, and ethyl acetate, were used for studying the composition of the two residues. The solvents used are analytical grade from Fluka Chemical Company. IR spectra were recorded on FT-IR spectrometer; model 960 Mo009, ATI Mattson infinity series. Ultraviolet spectra were carried out using UV-VIS spectrometer, model V-570 UV-Vis/NIR spectrometer. Gas Chromatograph was carried out using perkin-Elmer Model 8700. Nickel and vanadium contents were determined using Inductively coupled plasma-atomic emission spectrometer Model Flame Modula, Spectro. Sulphur was determined using X-ray Sulphur Meter Model 3500 S, Oxford. All these analysis were carried out in Egyptian Petroleum Research Institute laboratories.

## **6.1. Determination of Physico-chemical Properties of the Residues and their constituents:**

The physico-chemical properties of the residues and their constituents were carried out according to ASTM and/or IP standard test methods<sup>(138, 139)</sup>. The results of the analysis are given in Tables-2, 4 and 5.

### **6.1.1. Density:**

The density of the residues and their maltenes was determined according to the IP 190 capillary-Stoppard pycnometer method.

API gravity was calculated using the ASTM-IP Petroleum Measurement Tables, according to the ASTM D 1250-IP 200-API 2540 methods<sup>(140)</sup>.

### **6.1.2. Kinematic viscosity:**

The kinematic viscosity of the residues and their maltenes was determined according to the ASTM D 445-IP 71 glass capillary viscometer method.

The kinematic viscosity of the residues and their maltenes was determined at 80 and 100 °C, and then calculated at 50 °C.

### **6.1.3. Pour point:**

The pour point of the residues and their maltenes was determined according to the ASTM D 97-IP 15 method.

### **6.1.4. Sulphur content:**

The sulphur content of the residues and their maltenes and asphaltenes was determined by x-ray fluorescence according to the ASTM D 4294 method.

**6.1.5. Nitrogen content:**

The nitrogen content of the residues and their maltenes, and asphaltenes was determined by modified Kjeldahl method according to the ASTM D 3228.

**6.1.6. Carbon residue content:**

The carbon residue content of the residues and their maltenes was determined according to the ASTM D 524-IP 14 Ramsbottom carbon residue method.

**6.1.7. Wax content:**

The wax content of the residues and their maltenes was determined according to the UOP 46 method<sup>(141)</sup>.

**6.1.8. Ash content:**

The ash content of the residues was determined according to the ASTM D 482-IP 4 method.

**6.1.9. Determination of nickel and vanadium**

The nickel and vanadium concentrations in the studied residues and their maltenes, resins and asphaltenes, were determined through the sulphuric acid wet ashing, dissolution with hydrochloric acid<sup>(142)</sup>, and measurement by the inductively coupled plasma-atomic emission spectrometric technique according to ASTM D 5708.

## **6.2. Fractionation of the Residues into their Constituents**<sup>(143)</sup>:

The studied residues were fractionated into their asphaltenes, maltenes, resins, aromatics, mono-, di-, and polyaromatics and saturates. This is shown in Figure 31.

### **6.2.1. Separation of the Asphaltenes and Maltenes:**

Asphaltenes and Maltenes were separated from the two vacuum residues under studied according to the standard test method IP-143.

The separation of the asphaltenes from Alexandria Vacuum Residue was carried out using *n*-pentane, *n*-heptane, and ethyl acetate as solvents. In the case of Suez Vacuum Residue two solvents only were used, namely *n*-heptane and ethyl acetate because they give the best results in the case of Alexandria Vacuum Residue.

A quantity (20 g) of the residue, was dissolved in the *n*-alkane solvent (1 g : 30 ml) by refluxing for one hour, and then stored in a dark place overnight. The mixture was decanted onto a filter paper (Whatman 42) without agitation, and the residue in the flask was then transferred completely into the filter paper using successive quantities of the hot solvent. The filter paper with its contents was placed in a soxhlet extractor and refluxed with the solvent until becoming colourless. The asphaltenes precipitated on the filter paper were extracted with toluene. Both the *n*-alkane and toluene solvents were distilled off, and the separated maltenes and asphaltenes were dried in an oven to constant weight

### **6.2.2. Separation of the Maltenes into Saturates, Aromatics and Resins**

#### **by Alumina Column Chromatography**<sup>(143)</sup>

Maltenes resulted from deasphalting of the residues were further separated into saturates, aromatics and resins using alumina column chromatography.

A glass column (150 cm × 4 cm i.d.) was packed with 800 g alumina (neutral; 70-230 mesh, Merck) activated overnight at 300° C in a muffle furnace. The column was wetted with *n*-hexane, then 40 g of the maltenes dissolved in the least

amount of *n*-hexane was charged onto the column. The saturates, aromatics and resins were eluted by *n*-hexane, toluene, and methanol-methylene chloride mixture (1:1), respectively. The solvents were distilled off and the separated saturates, aromatics and resins were dried in an oven to constant weight.

### **6.2.3. Separation of the Aromatics into Mono-, Di-, and Polyaromatics by Silica Gel Column Chromatography <sup>(143)</sup>**

Aromatics were further separated into mono-, di-, and polyaromatics using silica gel column chromatography.

A glass column (150 cm × 2 cm i.d.) was packed with 200 g silica gel (100-200 mesh, Merck) activated at 180° C in an oven for 4 hours. The column was wetted with *n*-pentane, then 10 g of the sample dissolved in the least amount of *n*-pentane were charged onto the column.

Monoaromatics were eluted from the column by *n*-pentane-benzene mixture (95:5). After collection the first 100 ml, each 25 ml of the eluted fraction was collected in a weighed flask and the solvent was distilled off, then the separated fraction was dried in an oven to constant weight. The refractive index of each fraction was measured. Monoaromatics were collected from the fractions having refractive index up to 1.53 at 20° C.

Diaromatics were eluted from the column by *n*-pentane-benzene mixture (85:15) using the same procedure used for monoaromatics until reaching the refractive index of 1.59 at 20° C. Polyaromatics were finally eluted from the column by benzene.