



بسم الله الرحمن الرحيم  
Sudan University of Science and Technology  
College of Graduate Studies  
College of Petroleum Engineering and Technology



**Improvement of Rheological properties of fresh crude oil using**

**Momordica balsamina Plant Extracts**

**Case Study: Hamra - ( Block 2 )**

تحسين الخواص الإنسيابية لخام النفط الطازج باستخدام مستخلص نبات

المومورديكا بلازمينا

(دراسة حالة : حمرا - مربع (2) )

**Master of science in Oil Transportation and Refining Engineering**

**(Transportation and Storage Engineering)**

**By**

**Hana Taha Hussein Al Taher**

**Supervisor**

**Dr.Sumaya Abdelmoniem Mohamed**

**Dr. Mona Abdelmoniem Mohamed**

**2019**



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الاستهلال

قال تعالى:

(ولسوف يعطيك ربك فترضى)

الضحى: الآية (5)

## **Dedication**

To the spirit that taught me how to respect and sincerity of devotion, The pure white heart (my beloved mother) May Allah have mercy on her and make her place the paradise.

To the pure soul of full and high ethic, the Great Heart (my dear Father) May Allah have mercy on him and make his place the paradise.

Who dragged empty cup to teases me a drop of love. To whom who harvest the thorns from my road to guide me through learning (my husband is virtuous) ...

To my sons (Ahmad, Lamar, Talia and Gudy)

To the pure and kind hearts and the innocent souls (my brothers)

And all the gratitude to whom she lighted the candles of the my road, my aunt (Kawthar)

Allah preserve their presence in my life ...

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## Abstract

Petroleum components are complex mixture of various hydrocarbon compounds such as paraffinic, naphthenic, and aromatic compounds. As well as some inorganic components as an impurity which includes sulphur, nitrogen, oxygen, nickel, vanadium, iron and arsenic. According to its composition, oil is classified to paraffinic petrol, an asphaltic base, and a mixed base oil.

Sudanese petrol belongs to the paraffinic type of petrol. It contains a high percentage of paraffin waxes, which influences its rheological properties (viscosity and flow pour point). This property affects directly the transporting process by increasing the cost. So, recently approximately the entire researcher works to find out a suitable natural improver to the rheological properties of the oil.

The use of plants in the field of petrol industry, become the focus of many countries around the world. Some plant products have been used in the treatment of drilling liquids, such as using gum Arabic and guar gum in the treatment of drilling liquids.

This research discusses a new approach for treating the rheological properties of the paraffin Sudanese petrol and the microbial contamination of its draining using *Momordica balsamina* (Cucurbitaceae) fruit and seed extracts.

The authenticated plant material was extracted using two methods: cold maceration method with methanol, and hot continues extraction method using Soxhlet apparatus with different organic solvent in order to increase the polarity (petroleum ether, chloroform, and methanol).

The phytoconstituents of the prepared extracts were tested. The result was reflected the presence of all phytochemical group tested in all extracts except terpenoid was not detected in some seed extract.

The effect of these extracts on the rheological properties of untreated Hamra ore samples (Block B2, a mixture of OGM2 and OGM5) was tested by adding 2000 PPM of the extract to 100 ml of crude sample. Seed petroleum ether extract and fruit chloroform extract were improve best result in treating rheological properties of ore pour point of blank (39°C to 33°C). viscosity improved the sample (plank) at the same (pour point) for the seed extracted by (Petroleum Ether) the new extract production from (1474.69 cp) to 77.13 (cp) where the stored seeds improved its viscosity from (1474.69 cp to 89.79 cp). When used fruits which produced from extracting chloroform newly produced gives the best treatment from using stored fruits where the improved newly fruits produced viscosity for the same sample from (1474.69 cp to 89.98 cp) where the improved stored fruits viscosity from (1474.69 cp to 199.96 cp). the minimum concentration to improve the flow is 1500ppm.

The antimicrobial activity of the prepared extracts was tested against four standard bacterial strains and two standard fungal strains. The result was showed the antimicrobial activity against at least one or more microbial strain tested.

The result of this research was reflected a measurable improvement in the rheological properties of the paraffin Sudanese petrol natural microbial remedy to the microbial contamination of the petrol industry.

## المستخلص

البتترول عبارة عن خليط معقد من المركبات الهيدروكربونية المختلفة منها المركبات البرافينية والمركبات النفثانية والمركبات العطرية . بالإضافة لبعض الشوائب مثل الكبريت والنتروجين والاكسجين وبعض المعادن مثل النيكل والفانديوم والحديد والزرنيخ بهذا التركيب المعقد يصنف البترول الى بترول برفيني اي ذو قاعدة برفينية ، بترول اسفلتي أي ذو قاعدة اسفلتية ، وبتترول مخلوط ذو قاعدة مخلوطة .

البتترول السوداني من النوع البرافيني ، يحتوي على نسبة عالية من الشمع البرافينية ، مما يؤثر ذلك على خواصه الانسيابية (اللزوجة ودرجة الانسكاب) وارتفاع قيمة هذه الخواص يؤثر تأثيرا سلبيا على عملية نقل الخامات بواسطة الانابيب مما يتطلب الامر استخدام عدة معالجات لتحسين الخواص الانسيابية ..

استخدام النباتات في مجالات الصناعة البترولية اصبح تركيز معظم دول العالم حيث يستخدم الصمغ العربي والقوار في معالجة سوائل الحفر.

يتناول البحث اسلوب جديد في معالجة الخواص الانسانية للبتترول السوداني البرافيني باضافة مستخلص نباتي من نبات المومورديكا بلازمينا ..

ثم استخدام طريقتين من طرق الاستخلاص هي : الاستخلاص المستمر)و النقع على بذور وثمار المومورديكا بلازمينا باستخدام جهاز السوكسيلين مع استخدام عدة مذيبات عضوية من أجل زيادة القطبية بتروليوم اثير و الكلوروفوم والميثانول.

تم إجراء اختبارات للمستخلصات وكانت النتائج تعكس وجود جميع الخواص الكيميائية ما عدا التيربونايد لم تتواجد في بعض مستخلصات البذور.

تأثير هذه المستخلصات النباتية على الخواص الانسيابية على عينة من خام هجليج (حمره) من بلوك (ب2) وهي خليط من (OGMs وOGM<sub>5</sub>) مع الاخذ في الاعتبار انها عينة خالية من أي معالجات لتحسين الخواص ، وذلك للتأكد من مدى فاعلية المستخلصات النباتية في معالجة الخواص الانسيابية ، تمت اضافة المستخلصات بنسبة (2000 ppm) الى كل (100 مل من الخام) .

اظهرت النتائج أن مستخلص البذور المستخلص بواسطة (البتروليوم اثير ) وخالصة الثمار المستخلص بواسطة (الكلوروفوم) ، اعطت افضل نتائج في معالجة الخام لتحسين خواصه الانسيابية حيث حسنت (درجة الانسكاب ) للعينة (الخام) من (39c° الى 33c°) وحسنت (اللزوجة) للعينة (الخام) عند نفس درجة (الانسكاب) بالنسبة للبذور الحديثة الانتاج، الناتجة من البتروليوم اثير تعطي نتائج أفضل بالنسبة للبذور حديثة الإنتاج ((cp) 1474.69 - (cp) 77.13) وبالنسبة للبذور المخزنة ((cp) 1474.69 - (cp) 989.79).

وعند استخدام مستخلص الثمار الحديثة الانتاج الناتجة من (الكلوروفوم) تعطي نتائج أفضل من استخدام الثمار المخزنة (cp) 89.98 to (cp) 1474.69 بينما حسنت الثمار المخزنة للزوجة (cp) 1474.69 to (cp) 199.96 . أقل تركيز لتحسين الخواص الانسيابية يمكن إضافته للعينة الخام هي 1500 PPM.

تم اختبار النشاط الميكروبي للمستخلصات على أربعة أنواع من البكتيريا الرئيسية ونوعين من الفطريات وكانت النتيجة أن النشاط الميكروبي المضاد لواحد أو أكثر من أنواع الميكروبات. نتائج هذا البحث تعكس تحسين الخواص الانسيابية للبتترول السوداني البرافيني والتلوث الميكروبي لصناعة البترول.



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## Nomenclature

Symbols	
$\phi$	Porosity
F	Fahrenheit degree
O	Degree
oC	Celsius degree
%	Percentage
abbreviation	
API	American Petroleum Institute
PPD	Pour point Depressant
G.C Ms	Gas Chromatography mass spectrometry.
O.G.M.	Oil Gather Manifold
PPM	part per million
Cp.	Centipoise
Fig	Figure
M <sup>3</sup> /S	Cubic Meter /S

# **CHAPTER ONE**

## **Introduction**

## 1.1 Introduction

Oil is the main source of energy and is one of the most important natural economic resources in the world. The oil industry, like any other industry, has had a simple beginning and has since been developed until it became a giant industry that influences the global economy, and the advances of the human civilization (Abd Al-Aziz Misbah( 2005).

Chemically, oil is a mixture of hydrocarbon compounds which differ in properties, as a result of the difference in the number of the carbon atoms in its molecules, but all of them have in common that they are made up of two elements only; carbon and hydrogen, combined with inorganic compounds as an impurities such as sulfur compounds, nitrogen, oxygen, as well as metals; like nickel, vanadium, iron and arsenic the differ in the petrol composition are lead to a differ in physical properties such as color, density, viscosity, boiling point, molecular weight ( Abd Al-Aziz Misbah (2005). The Hydrocarbon substances that make up oil are divided into three main types: Paraffin compounds - naphthenic compounds and aromatic compounds (Abd Al-Aziz Misbah (2005), Email J - Burcik et al(1994)).

Sudanese crude oil is the paraffinic type; has a paraffinic base; i.e. it has a high content of paraffin wax, influencing its rheological properties (viscosity & flow rate), and therefore affecting negatively the process of transporting crude oil via pipeline ( Kamel Zaied (2015)

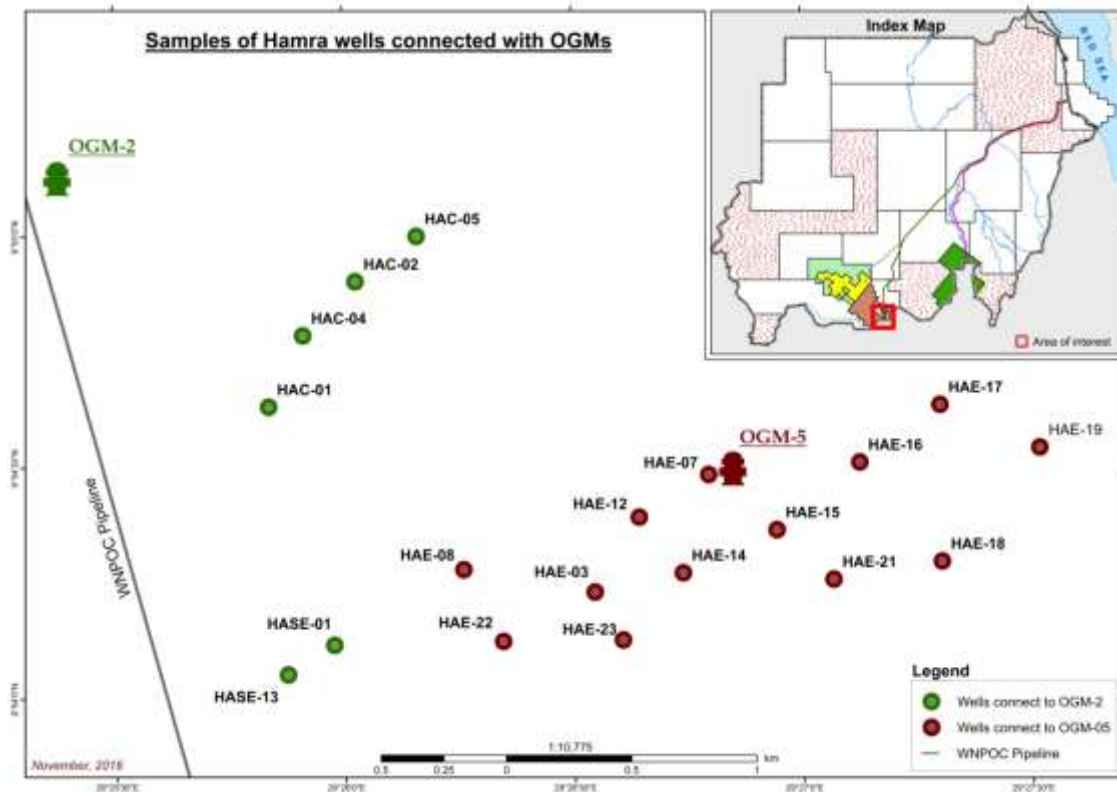
Viscosity and pour point are some of the very important properties of crude oil and oil products; as they strongly affect the process of transporting crude oil via pipelines and pumping oil products in internal combustion engines as well as the behavior of lubrication? oil in equipment and machinery ( Shareef Fathi Al-Shafi'i(2006 )

Several treatments are made to improve the rheological properties including treatment through heating; which are used using two methods: heating method and chemical methods, but each have side effect as an expensive and the hazard to the health. (Elhsarkawy,A.M. Al-Sahhaf T.et –al(1999)(Sadeghazad,a .et al(1998). Other researchers takes a new approach to treating the rheological properties using natural treatment (Gamal E.B.E.L et al. 1989).

The rheological properties that mostly affect the flow in the pipeline during transportation. This research was studied the effect of *Momordica balsamina* parts extract on the rheological properties of the Sudan petrol (Hamar ore sample) with the test of the

remedial effect of these extracts on some pathogenic microorganisms which may develop a resistant due to the petrol daring contamination.

Higilig oil crude are from Profane type , its crude characterized with low rate of sulfur, which makes it safer for the environment, medium density(API) and low mineral content, Hegelig produces ore from several blocks, including Block (B2) which includes many fields including a field which includes many (Hamra) which include many OGM , as ( OGM5, OGM2) and following map include wells include ( OGM5, OGM2).



Figure(1.1) shows (Hamra) Wells (OGM5, OGM2)

## 1.2 Problem Statements:

Sudanese crude oil is the paraffinic type; has a paraffinic base; i.e. it has a high content of paraffin wax, influencing its rheological properties (viscosity & flow rate), and therefore affecting negatively the process of transporting crude oil via pipeline.



### 1.3 Objectives

- 1- Define the optimum method and conditions for the extraction process, and characterize the extracted material.
- 2- Determine the viability and the effectiveness of *Momordica balsamina* seed and fruit the extracted material on treating rheological properties.
- 3- Test the activity of plant crude extracts against some pathogenic selected standard microorganisms

**CHAPTER TWO**  
**Literature Review**

## 2. Literature Review

### 2.1 Petrol

oil is a mixture of hydrocarbon compounds which differ in properties, as a result of the difference in the number of the carbon atoms in its molecules, but all of them have in common that they are made up of two elements only; carbon and hydrogen. There are also some impurities in addition to the hydrocarbon compounds such as sulfur compounds, nitrogen, oxygen, as well as metals; like nickel, vanadium, iron and arsenic. The composition of oil differs between countries, and between oil fields. It also differs in color from light brown to dark black and differs in its natural properties; such as density, viscosity, boiling point, molecular weight as well as other properties. Crude petrol exists in a solid form called kerogen, or in a liquid form and is then called Crude Oil, or in a gaseous form in which case, it's called Natural Gas.(Misbah, 2005)

#### 2.1.1 Classification

Petrol consists of a complex mixture of different hydrocarbon compounds. These hydrocarbon compounds are divided into 3 main types:

**A\ Paraffins:** Which have a high content of paraffinic compounds, these are two groups:

i\ Natural Paraffins: These are found in a higher concentration than their counterparts.

Examples are n-hexane and n-methane. They have the general formula ( $C_nH_{2n+2}$ )

ii\ Isoparaffins: The molecular structure of these compounds is the same as the regular paraffins in the number of carbon and hydrogen atoms, but differ from them in the bonds they have with molecules. Regular paraffins form straight chains with the atoms of carbon and hydrogen while Isoparaffins form branched chains. The difference in the bonds of hydrogen and carbon atoms results in a difference in its natural properties. They follow the same formula as paraffin.

**B\ Naphthenes:** Oil has a lower content of naphthenes than paraffins such as Cyclopentane ( $C_nH_{10}$ ), Cyclohexane ( $C_6H_{12}$ ), The general formula of these compounds is  $C_nH_{2n}$

**C\ Aromatics:** Oil has some aromatic compounds in low amounts, that differ according to the nature of the oil and the region where the oil fields are. Examples of such compounds are: Benzene ( $C_6H_6$ ), Toluene ( $C_7H_8$ ), and Ethylbenzene ( $C_8H_{10}$ ) with general formula  $C_nH_{2n-6}$  .(Misbah 2005; Hameed *et al*; 1994 and Burcik 1994).

Owing to the contrast in the proportion of hydrocarbon compounds that make up oil, as well as the proportion of impurities in it depending on the layer carrying the oil and its depth, or the oil field, It has become difficult to categorize oil based on an accurate chemical analysis of its components. It is, however, now categorized according to the main type of hydrocarbon compounds that make it up. Based on this approach, oil can be categorized into the following types:

**i\ Paraffin base crude:** It is oil that has paraffins forming a great proportion of its composition. When filtered, it leaves a solid substance, which is wax. This type of crude is considered one of the best types of crude oils as some of the most important light oil derivatives are extracted from it in large amounts such as; benzene, kerosene, as well as heavy wax, which is extracted in lower amounts. Sudanese crude oil belongs to this type of crude.

**ii\ Asphalt base crude:** It is a type of crude that naphthenes make a great proportion of its composition. When filtered, it forms a solid substance which is asphalt. This type of oil is generally not considered a good type of heavyweight oil low price products, such as fuel oil (which is used as an alternative to coal), and asphalt in greater amounts than light products like benzene and kerosene, which are extracted in comparatively lower amounts.

**iii\ Mixed base crude:** This type of crude is a blend of the previous two types; the paraffin and asphalt types in more or less the same proportions. Extracts from this type include gas oil, diesel, and lubrication oil. (Misbah, 2005; Mohammed , 2005; and Burcik, 1994).

## **2.2. Rheological Properties**

Paraffin oil contains a high amount of waxes; which influence the rheological properties, viscosity, and pour point. Which in turn, affect negatively the process of transporting crude via pipelines and storage tanks.

### **2.2.1 Viscosity**

Viscosity is the resistance of a liquid to pouring. It is the friction between the molecules of the liquid when they are moved or are under the effect of an external force. It is also defined as the Dyn force exerted on an area unit on one of two horizontal planes the distance between which is one unit, one plane is fixed and the other is mobile, moving one unit, with the space between the two planes being full of the viscous fluid. (Al-Shafi'I, 2006)

Viscosity is one of the most important properties of crude oil and oil products. It strongly affects the process of transporting crude oil via pipelines and pumping fuel in internal combustion engines and lubrication oils in equipment and machinery.

**2.2.1.1 Causes of viscosity:** according to Al-Shafi'I, 2006; and Misbah, 2005,

i\ The cohesion forces between molecules of the fluid: The greater cohesive forces between molecules of the fluid were, the higher its viscosity was, and vice versa.

ii\ The number of molecules that comprise the crude: The higher the number of molecules, the less effect the temperature had on the crude, for example: Glycol, which contains two hydroxide groups (OH – CH<sub>2</sub> – CH<sub>2</sub> – OH) can form more viscous bonds than alcohol (CH<sub>3</sub> – CH<sub>2</sub> – OH) which has one hydroxide group. (it is noted how the more hydroxide groups there are, the more viscous the crude is.)

iii\ Random movement of the fluid molecules and between the different layers of the fluid: Experiments were carried out by Newton whose aim was to determine the viscosity of a fluid using two boards; one fixed, and the other mobile, between which he put a viscous fluid. He then categorized fluids based on viscosity to:

A\ Newtonian fluids: Are fluids whose viscosity appears only when moved.

B\ Non-Newtonian fluids: Are characterized by stress and capillary stress when static.

### **2.2.1.2 Types of Viscosity**

**i/ Dynamic viscosity:** Is the relationship between the shear stress and acceleration. It is constant in Newtonian fluids, and changes in proportion with acceleration in non-Newtonian fluids. It is represented by the symbol ( $\mu$ ) and is measured by (Centipoise).

**ii/ Kinematic Viscosity:** Is the viscosity of the fluid measured in centipoise and divided by the density. It is represented by ( $\gamma$ ) and its unit of measurement is (Stoke).

$$\gamma = \mu / \rho$$

Where:  $\gamma$   $\equiv$  Kinematic viscosity,  $\mu$   $\equiv$  Dynamic viscosity,  $\rho$   $\equiv$  Density (Kg/m<sup>3</sup>)

**iii/ Conventional viscosity:** Is the ratio between the passing time of a volume of a liquid and the passage time of the same volume of distilled water at temperature (20o C)

Represented by the symbol  $\mu^-$

Viscosity's main measurement units are: The principal unit of dynamic viscosity is ( N.m<sup>2</sup> / s<sup>2</sup>), While kinematic viscosity's unit is (m<sup>2</sup>/s)

There are other secondary units known as: Poise: for measuring dynamic viscosity, Stoke: for measuring kinematic viscosity.

Main units can be converted into secondary units as follows:

1 poise = 0.1 (N.s/m<sup>2</sup>) ;                      1 stoke = 10<sup>-4</sup> (m<sup>2</sup>/s)

In practical applications, these units are used:

Centistokes = 0.01 stokes;                      Centipoise = 0.01 poise

While in some European countries, (Engler) units are used. Also, in the UK, (Redwood) units are used. In the US, (Saybolt). But these units are increasingly falling into disuse. (Al-Shafi'I, 2006; and Misbah, 2005)

### **2.2.1.3 Factors that affect viscosity**

#### **i\ Temperature:**

The viscosity of a crude is closely related to temperature as temperature and viscosity are inversely proportionate; viscosity is lower at higher temperatures and higher in lower temperatures. Strong bonds between atoms results in an increase in viscosity and crude are heated, temperature increases the space between these atoms and consequently in a fall in viscosity, and thereby facilitating the transportation of the crude via pipelines. The decrease in viscosity due to heating depends on the substances that make up the crude. Which means that viscosity does not fall to the same degree in all of the crudes because it depends primarily on the nature of the composition of the crude as well as other factors (Al-Shafi'I, 2006; Misbah , 2005; and Mohammed ,2005)

#### **ii\ pressure:**

The effect of pressure on hydrocarbons which form part of the crude varies as it has been noted that paraffinic hydrocarbons are affected less notably by pressure, whereas naphthenic as well as aromatic hydrocarbons' viscosity are highly affected by pressure. It has also been found that the effect of pressure on the viscosity of the crude is not as great as the effect of temperature. ( al- Yaari,2011; and Haq,1981)

#### **iii\ The amount of dissolved gas:**

The viscosity of crude oil decreases as the amount of gas dissolved in it increases i.e. viscosity is inversely proportionate to the amount of dissolved gas.

### **2.2.2. Pour point**

Pour point is one of the rheological properties that affect the transportation of crude oil via pipelines, and is defined as:

“The lowest temperature at which the crude starts to flow.”. That is to say, that the crude oil should always have a higher temperature than the pour point when it is being transported. Otherwise, it will not move and will require higher pressure for it be moved. Pour point is measured in Fahrenheit (° F) or Celsius (° C). Pour point is an indicator of

paraffinic or aromatic substances that are in the crude. It is higher in crude oils that are high in paraffinic and aromatic substances and lower if paraffinic and aromatic substances are lower (Al-Shafi', 2006; Misbah, 2005; and Machado, 2001)

## **2.3 Treatment of rheological properties**

### **Treatment of rheological properties**

- **Chemical treatment**

This method depends on adding assistance materials of Polymers and Surface stimulants to the oil crude with high viscosity in low concentration that led to improve the oil behavior and decrease the pour point, and obstacles the polymers growth and bind it without linked the polymers together.

The method aimed to improve the protection the building of pipeline, to keep the specific viscosity economically from power consumption, the assistance material possessed the ability to reduce the degree pour point and viscosity and the primary stress of running in case of work and sport working for long period, and help the polymers to formed smellers and did not liked with together. E.G. the chemical laboratory proved that any temperature less than the degree centigrade of pour point, so if we did not add polymers and surface stimulants the oil crude will be Intertwined and lumped. But when we add polymers and surface stimulants, the flow will be free and the viscosity will be fixed. ( J.O Adelya, K.K Salma et al (2009), ( J.R. Becker (1997), ( Knox, T et al, (1962).

### **Friction Reduction Method**

#### **Drag reduction additives**

Drag-reducing agents, or drag-reducing polymers (DRP's), are additives in pipelines that reduce turbulence in a pipe. They increase the pipeline capacity by reducing turbulence and therefore allowing the oil to flow more efficiently. A polymeric drag reducer has been tested in an 8-in. diameter crude oil pipeline. The test segment was 28 miles long. At the normal flow velocity in the 8 in a line of about 6 ft/sec, drag reductions of 16, 21 and 25 percent were obtained at polymer concentrations of 300, 600 and 1,000 volume ppm. A series of tests was run to determine the effect of flow velocity on drag reduction. Drag reduction decreased as the flow velocity decreased. An additional test in a 12-in. The pipeline, 32 miles long, supported the results obtained in the 8-in. pipeline. In none of the tests did the polymer appear to degrade or lose effectiveness because of turbulent shear in the lines.

Tests were run for the polymer and various polyisobutylenes in a 1-in.-ID laboratory pipe polyisobutylenes in a 1-in.-ID laboratory pipe viscometer. These tests covered a range of concentrations and flow velocities. The results obtained in the 1-in. line showed the same general characteristics as those obtained in the large pipelines. An equation is presented that correlates pipelines. An equation is presented that correlates the 8-in. and 12-in data as a function of flow velocity and polymer concentration.

- **Mechanical Treatment:**

- **The Pour point depression mechanism**

- The pour point of a liquid is the temperature at which it becomes semi-solid and loses its flow characteristics. High pour point phenomena in waxy crudes have been observed (high paraffin content crude oils; which are typically found in crude derived from a larger proportion of plant material). There are differences in crude oils that do and do not respond to pour point depressants.

- Microfiltration** is used to separate the waxes that precipitate out at the various temperatures in waxy crude oils. These waxes, which can be composed of saturates, aromatic, polar and asphaltenes, vary in molecular weight and composition with their temperature of precipitation in a waxy crude. Waxes that precipitate out of the crude oil solution at temperatures above the pour point are of higher molecular weight and have a high asphaltene content. that precipitate at lower temperatures have lower molecular weights and contain mostly saturates. Detailed chemical analysis showed that the temperature where asphaltenes are no longer associated with the waxes represents the maximum degree of pour point depression obtainable. (D.S. Schuster et al.) ( AJIENKA, K,A et al (1990).

- **Mix and Dilution method:**

- **Dilution:**

- Dilution can be used as a means to reduce the high viscosity of heavy crude oils to help with their transportation using solvents and gas condensate. The viscosity of two Iranian heavy crude oils was measured by mixing with solvents at different temperatures. Dilution of both oil samples with toluene and heptane resulted in viscosity reduction. However, their effect became less significant at higher concentrations of diluent. Because of forming hydrogen bonds, adding methanol to heavy crude oil resulted in higher viscosity. By adding condensate, the viscosity of each sample reduced. Gas condensate had a greater impact on heavier oil; however, at higher temperatures, its effect was



reduced. Diluting with naphtha decreased heavy oil viscosity in the same way as n-heptane and toluene. Besides experimental investigation, different viscosity models were evaluated for prediction of heavy oil/solvent viscosity. It was recognized that Lederer model is the best one. Experimental study of Iranian heavy oil viscosity reduction by diluting with heptane, methanol, toluene, gas condensate and naphtha, Amir Hossein Saeedi Dehaghani, Mohammad Hasan Badizad)

### **The In-situ upgrading method**

Studies on the application of transition-metal catalysts for heavy oil or bitumen in-situ upgrading were conducted in the absence of a porous medium, mainly measuring the characteristics of heavy oil in reaction with metal ions at static conditions with the help of a magnetic stirrer. Metal species in ionic form are not soluble in the oil phase. Therefore, metal particles, as inhomogeneous catalysts, are considered in this paper. Furthermore, dynamic tests in porous media are needed to clarify the injection possibility of the metal particles and their effect on the in-situ upgrading of heavy oil. Injection of metal particles may deteriorate the recovery process by damaging porous media because of attractive forces such as van der Waals and electrostatic forces between particles and porous rock. A better understanding of these forces and their importance in the retention of particles is required. In this paper, the catalysis effect of pure nanometer-sized nickel during steam-injection application was compared with that of an industrial catalyst such as micron-sized Raney nickel. The changes in the viscosity, refractive index, and asphaltene content were measured after each test to analyze the catalysis effects. Nickel nanoparticles showed a better catalysis compared with Raney nickel. The approximate optimum concentration of the catalysts was determined. Then, the catalysis effect of nickel nanoparticles was studied in the presence of sand pack as a porous medium. The results showed accelerated catalysis in presence of the sands. Also, nickel nanoparticles improved the oil recovery factor. The next phase of this paper studies the injectivity and transport of nickel particles. The injected suspension was stabilized by use of xanthan gum polymer and ultrasonication. The effect of solution pH, which controls the magnitude of the repulsive electrostatic forces, was clarified. Stabilization of the metal particles' suspension was studied at different pH values through zeta-potential measurements. Also, the zeta potential of the recovered suspensions was studied to confirm the stability of the suspension during travel through the porous medium. Depending on the size, particles carry different charges and

have different settling velocities. Therefore, the stabilization pH and dispersant concentration were different from one sample to another. The results of the injectivity tests confirmed the lower retention and better injectivity of nanoparticles in comparison with micron-sized particles. [Yousef Hamed Shokrlu, Tayfun Babadagli]

### **In-Situ Upgrading of Heavy Oils and Bitumen by Propane Deasphalting: The Vapex Process**

Many of the Lloydminster-type heavy oil reservoirs are unsuitable for thermal recovery by steam injection because they are relatively thin and the heat losses to the under- and overburden are excessive. To overcome this difficulty, a new approach has been proposed which utilizes the injection of saturated hydrocarbon vapors at approximately reservoir temperature. There are several modifications of the basic principle of development in which the amount of extraneous heat injected varies from none to moderate, and the vapor chamber is maintained at reservoir temperature to 30C above it. This flexibility allows the approach to be followed for the recovery of both conventional heavy oils and for bitumen. The method, termed "Vapex", utilizes two horizontal wells and is closely related to the SAGD process but with the steam chamber replaced by a chamber containing hydrocarbon vapor near its dew point. The quality of bitumen or heavy oil can be improved substantially by in situ deasphalting with propane vapor. The removal of asphaltenes reduces the viscosity of the produced oil and creates an oil that is more easily refined. [ I.J. Mokrys, R.M. Butler (U. of Calgary)

- **Water treatment:**

#### **Emulsification**

Oil-water emulsions are fine dispersions of oil in water (O/W) or of water in oil (W/O), with drop sizes usually in the micron range [1, 2]. In general, emulsions are stabilized by surfactants. In some cases, multiple emulsions such as water in oil in water (W/O/W) or oil in water in oil (O/W/O) can be found. Emulsions can be stabilized by other species, provided that they adsorb at the oil-water interface and prevent drop growth and phase separation into the original oil and water phases. After adsorption, the surfaces become viscoelastic and the surface layers provide stability to the emulsion. ( D. Langevin, S. Poteau, I. Hénaut and J.F. Argillier) .

#### **Stability and separation of water-in-oil emulsions:**

A study by Antonio Luzaiadio Bucu Andrewhose main objectives were as follows: to rank crude oil samples in terms of creating stable emulsions; to assess the effect of the

brine pH on emulsion stability; to investigate the influence of different organic acids on emulsion stability; and to determine the efficiency of an electric separator in removing water droplets from a flowing organic liquid. Seven crude oil samples from different sources such as A, C, H, M, P, U, and V were used to investigate the water-in-crude-oil emulsion. Two crude oil blends were also used. Brine solution comprising 4 wt% NaCl and 1 wt% CaCl<sub>2</sub> was used. In the study, the gravity settling, critical electric field (CEF) and centrifuge test methods were used to estimate the emulsion stability created by the crude oil and crude oil blend samples. The experiments were carried out at 60°C. In the gravity test method, the brine pH, stirring speed, stirring time and water-cut (the fraction of water in the emulsion) were changed in 2IV-1 factorial design. The parameters for the centrifuge and CEF test methods were selected on the basis of the gravity test method. The crude oil samples were ranked in terms of creating a stable emulsion in the following order V, U, P, H, A, M, and C. The crude oil blends created more stable emulsions than their respective constituents. The ranking order of the crude oil samples did not correlate to asphaltenes, resins, wax or total acid number (TAN). There was a good correlation between the test methods used. There was an increase and decrease in the brine pH when different crude oil samples were in contact with the brine. It is believed that the structure of the surfactants present in crude oil may explain the emulsion-forming characteristics of different crude oil deposits around the world. To account for the effect of organic acids on emulsion stability, different organic acids were used. In this case, a mixture of equal volumes of heptane and toluene (here referred to as heptol) was used as the model for crude oil. The brine solution composition was the same as the one used in the crude oil experiments. Equal volumes of heptol and brine were mixed for a period of time and then separated. The brine pH was changed from acidic to basic. In this regard, gas chromatography and liquid chromatography were used to analyze the concentration of the acids in the brine and heptol samples. It was found that the partitioning coefficient for acids containing a straight-chain hydrocarbon moiety decreased with an increase in molecular weight. However, the partitioning coefficient depended on the structure of the acid. The presence of a benzene ring in the organic acid increased the partitioning coefficient. Organic acids with rings created an interface layer when the heptol sample was mixed with the basic brine solution. This confirmed that the emulsion of water and crude oil starts with the formation of a film, and it also provides insight into the formation of naphthenate soap. It is believed that the naphthenic acids that cause stable emulsions

have rings. More organic acids should be tested. It is recommended that the interaction of asphaltenes, resins, and naphthenic acids should be investigated at different pH levels, temperatures and pressures. The separation of water droplets from a flowing organic liquid was carried out using a direct current (D.C.) electric separator. The separator used centrifugal forces and a D.C. electric field to enhance the removal of water drops from a flowing organic liquid. For this, vegetable oil, crude oil blend, and heptane were used as the continuous phase. The experiments were carried out at room temperature (for heptane and vegetable oil) and at 70°C (for vegetable oil and crude oil blend). The flow rate to the separator was kept constant. The separator removed water droplets from flowing organic liquids. A maximum of 97% (at 100 V) of water droplets was removed from the heptane liquid; a maximum of 28% (at 100 V) of water droplets was removed from the vegetable oil at 70°C and 5% (at 100 V) of water droplets was removed from the crude oil blend. The D.C electric field enhanced the efficiency of the separator in removing water droplets. The break-up of the droplets is suspected to decrease the efficiency of the separator. This separator can easily be installed into existing process lines and does not require much space. However, further improvements are needed in the design of this separator. Emulsions created in the petroleum industries are quite complex to deal with. The identification of the structure of the components in crude oil is a matter that still has to be investigated. An improvement in the techniques may lead to a better understanding of the cause of the ultra-stable emulsion encountered in the petroleum and related industries. (Hossein Saeedi Dehaghani, Mohammad Hasan Badizad), (D. Langevin, S. Poteau, I. Hénaut and J.F. Argillier), ( Antonio Luzaidio Buco Andre)

### **Core-annular flow**

The emerging energy efficient technology in the field of high viscous oil transportation is a water-lubricated transport of heavy oil, known as core annular flow or CAF. In this method of transportation, a water film surrounds the oil core and acts as a lubricant so that the pumping pressure necessary for the lubricated flow is comparable to the one for water alone. The water fractions are typically in the range of 10-30%. Many theoretical studies, laboratory, and field tests have been carried out and have shown that the configuration of core-annular flow applied to heavy oils is stable. However, only two industrial examples of this technology are known:

- the 38.6 km Shell line from the North Midway Sunset reservoir to the central facilities at Ten Section (California), line that was operated during 12 years;

- the 55 km pipe from San Diego to Budare (Venezuela) used for transporting Zuata heavy crude oil (9.6 API°) .

The main problem of that technology is that the oils tend to adhere to the wall, leading to restriction and an eventual blockage of the flow system. This kind of difficulty can be exacerbated during a shutdown operation allowing stratification of oil and water phases and requiring a large restart pressure. (S. Ghosh, et al.)( Joseph, D., Bai, R., Chen, K.P. and Renardy, R., (1997))( Joseph, D. and Renardy, Y. (1993))( Oliemans, R. and Ooms, G. (1986))Guevara, E., Gonzalez, J. and Nuñez, G. (1998)

- **Viscosity Reduction Heating , Method**

### **Viscosity Reduction**

A study by Basma & Ali concluded that “Heating had a dramatic effect on the heavy crude viscosity, but it failed to achieve a practical level”. Another study by Anand et al, India, stated that Crude oil from Rajasthan Oil Fields is having high wax content, with pour point in 40°C-45°C range and viscosity in the range of 93.5-288 Cp (centipoise) at 42oC temperature. It poses challenges not only for processing such crude oils in old refineries but, also in the transportation of such crude oils to refineries situated far away from Rajasthan oil fields through conventional pipelines. Transportation of Rajasthan crude oil is feasible, without any quantity restrictions, through heated oil pipeline; however, such pipelines require higher capital as well as operating costs.

As viscosity decreases very rapidly with increasing temperature, heating is an attractive method for improving the flow properties of heavy crude oils. It is always cited as a possible process to reduce the viscosity of heavy oils A famous example is the Alyeska pipeline in Alaska which transports crude oil at approximately 50°C. However, the design of a heated pipeline is not easy as it involves [3] many considerations: expansion of the pipelines, a number of pumping/heating stations, heat losses etc. Other significant issues are the high costs and the greater corrosion rate of the internal pipe due to the temperature. Moreover, a recent study showed that heat treatment could induce changes in the colloidal structure of the crude oils and worsen their rheological properties. (Basma M. Yaghi & Ali Al-Bemani), Gerez, J.M. (1996), (Anand Kumar Tiwari, Sanjay Kumar Jha, Randhir Kumar), (Guevara, E., Gonzalez, J. and Nuñez, G. (1998)), (Meyer, R.F. (1998)), ( Nunez, G.A., Rivas, H.J. and Joseph, D. (1998)), ( Yaghi, B. and Al-Bemani, A. (2002)), (Evdokimov, I.N., Eliseev, D.Yu., and Eliseev, N.Yu. (2002)) ( Albino Mukhametshinea , et al (2013).

**Viscosity Reduction Method approaches:**

The approaches used for reducing the viscosity of a heavy crude include heating, blending with a light crude and with kerosene, and forming oil-in-water emulsions. Heating had a dramatic effect on the heavy crude viscosity, but it failed to achieve a practical level; consequently, blending the heavy crude with either light crude or kerosene was attempted and further reduction was achieved, but substantial amounts of these expensive diluents are required. Alternatively, emulsion formation was carried out, and it was established that a practical level of reduction is achievable with 70-75% oil content, in the high shear rate range, and at 30-50°C. The effect of temperature on the viscosity of the crude oil mixtures and emulsions can be fitted on the American Society for Testing and Materials (ASTM) double-log model with an average deviation of 7.2 - 9.4. ( Basma M. Yaghi & Ali Al-Bemani)

## ***2.5 Momordica balsaming***

### **2.5.1 Taxonomy**

Kingdom – Plantae

Division – Angiosperms

Subdivision – Eudicots

Class – Rosida

Order – Cucurbitales

family – Cucurbitaceae

Genus – Momordica

Species – Balsamina

### **2.5.2 Botanical Description**

Momordica Balsamina, is a tendril-bearing herbal annual vine. It is found in tropical areas in Africa, Asia, and Australia. It is also grown in home gardens. The plant usually grows in the wilderness in Africa and Asia. It has been obtained in the US. The plant belongs to the gourd family (cucurbitaceae) which includes cucurbits, melons, and squashes in addition to the genus 'Momordica'. The plant has thin stems and is tendril-bearing, reaching in height about 5 meters. Its leaves are lobed (5-7 lobes), circular, waxy leaves reaching in length about 12 cm. The plant is self-pollinating as both male and female flowers exist in the plant. The flowers are yellow. The fruit, reaching in length about 4-6 (inches), has a green color that changes into yellow when ripe – it is oval, oblong in shape that splits into three lobes when ripe. The seeds are found inside the fruit surrounded by the red-colored core.

### **2.5.3 General uses**

The plant has a wide spectrum of medicinal and nutritional values and has been used as a traditional folk medicine in many countries. The leaves, fruits, seeds, and bark of the plant contains resins, alkaloids, flavonoids, glycosides, steroids, terpenes, cardiac glycoside, saponins having various medicinal importance viz. anti-HIV, anti-plasmodial, shigelloidal, anti-diarrheal, anti-septic, anti-bacterial, anti-viral, anti-inflammatory, anti-microbial, hypoglycemic, antioxidant, analgesic and hepatoprotective properties. The therapeutic agent 'Momordin' is capable of inhibiting the growth of HIV and other viruses. The leaves are also important source of nutrients having 17 amino acids with adequate mineral composition like potassium, magnesium, phosphorus, calcium, sodium, zinc, manganese and iron. It also helps to combat the problem of micronutrient



deficiencies in soil and high value of protein and fat with low fibre content. High potassium content is a good source for the management of hypertension and other cardiovascular conditions. This plant is being promoted as a protein supplement for cereal-based diets in poor rural communities. The commercial exploitation of this plant for biopharmaceuticals and nutraceuticals are some of the prospective future potential of this wild herb. [Thakur GS, Baq M, Sanodiya BS, Bhadouriya P, Debnath M, Prased GB, Bisen PS] ,(Gamal E.B.EL et al (1989)



### **Photographs (2.1) plant *Momordica Balsamina***

#### **1.2.3 Extraction techniques of medicinal and aromatic plants**

Extraction, as the term is used pharmaceutically, involves the separation of medicinally active portions of plant or animal tissues from the inactive or inert components by using selective solvents in standard extraction procedures. The products so obtained from plants are relatively impure liquids, semisolids or powders intended only for oral or external use. (Amita P., and Shalini T., 2014)

#### **The basic parameters influencing the quality of an extract are:**

1. Plant part used as starting material
2. Solvent used for extraction
3. Extraction procedure

#### **Plant material**

Plant based natural constituents can be derived from any part of the plant like bark, leaves, flowers, roots, fruits, seeds, etc. i.e. any part of the plant may contain active components.

#### **Choice of solvents**

For Successful determination of biologically active compounds from plant material is largely dependent on the type of solvent used in the extraction procedure.

#### **A property of a good solvent in plant extractions includes:**



1. Low toxicity
2. Ease of evaporation at low heat
3. Promotion of rapid physiologic absorption of the extract
4. Preservative action
5. Inability to cause the extract to complex or dissociate

**The factors affecting the choice of solvent are:**

1. Quantity of phytochemical to be extracted
2. Rate of extraction
3. Diversity of different compounds extracted
4. Diversity of inhibitory compounds extracted
5. Ease of subsequent handling of the extracts
6. Toxicity of the solvent in the bioassay process
7. Potential health hazard of the extractants

**Extraction Procedure:**

The general techniques of medicinal plant extraction include maceration, infusion, percolation, digestion, decoction, hot continuous extraction (Soxhlet), aqueous-alcoholic extraction by fermentation, counter current extraction, ultrasound extraction (sonication), supercritical fluid extraction, and distillation techniques (water distillation, steam distillation, phytonic extraction (with hydro fluorocarbon solvents). For aromatic plants, hydro water and steam distillation), hydrolyticmaceration followed by distillation. (sukhdev S. H. *et al*, 2008)

**Hot Continuous Extraction:**

In this method, the finely ground crude drug is placed in a porous bag or “thimble” made of strong filter paper, which is placed in chamber E of the Soxhlet apparatus . The extracting solvent in flask A is heated, and its vapors condense in condenser D. The condensed extractant drips into the thimble containing the crude drug, and extracts it by contact. When the level of liquid in chamber E rises to the top of siphon tube C, the liquid contents of chamber E siphon into flask A. This process is continuous and is carried out until a drop of solvent from the siphon tube does not leave residue when evaporated. The advantage of this method, compared to previously described methods, is that large amounts of drug can be extracted with a much smaller quantity of solvent. This effects tremendous economy in terms of time, energy and consequently financial inputs. At small scale, it is employed as a batch process only, but it becomes much more economical and

viable when converted into a continuous extraction procedure on medium or large scale. (sukhdev S. H. *et al*, 2008)

### **Maceration**

In this process, the whole or coarsely powdered crude drug is placed in a stoppered container with the solvent and allowed to stand at room temperature for a period of at least 3 days with frequent agitation until the soluble matter has dissolved. The mixture then is strained, the marc (the damp solid material) is pressed, and the combined liquids are clarified by filtration or decantation after standing. Circulatory extraction

### **Infusion**

Fresh infusions are prepared by macerating the crude drug for a short period of time with cold or boiling water. These are dilute solutions of the readily soluble constituents of crude drugs.

### **Digestion**

This is a form of maceration in which gentle heat is used during the process of extraction. It is used when moderately elevated temperature is not objectionable. The solvent efficiency of the menstruum is thereby increased. (sukhdev S. H. *et al*, 2008)

### **This extraction process has significant advantages:**

- i) A unit quantity of the plant material can be extracted with much smaller volume of solvent as compared to other methods like maceration, decoction, percolation.
- ii) CCE is commonly done at room temperature, which spares the thermolabile constituents from exposure to heat which is employed in most other techniques.
- iii) As the pulverization of the drug is done under wet conditions, the heat generated during comminution is neutralized by water. This again spares the thermolabile constituents from exposure to heat.
- iv) The extraction procedure has been rated to be more efficient and effective than continuous hot extraction.

### **Advantages of the Process**

- Unlike other processes that employ high temperatures, the phytonics process is cool and gentle and its products are never damaged by exposure to temperatures in excess of ambient.
- No vacuum stripping is needed which, in other processes, leads to the loss of precious volatiles.

- The process is carried out entirely at neutral pH and, in the absence of oxygen, the products never suffer acid hydrolysis damage or oxidation.
- The technique is highly selective, offering a choice of operating conditions and hence a choice of end products.
- It is less threatening to the environment.
- It requires a minimum amount of electrical energy.
- It releases no harmful emissions into the atmosphere and the resultant waste products (spent biomass) are innocuous and pose no effluent disposal problems.
- The solvents used in the technique are not flammable, toxic or ozone depleting.
- The solvents are completely recycled within the system.

**CHAPTER FOUR**  
**Materials and Methods**

### 3. Materials and Methods

#### 3.1 Material

##### 3.1.1 Chemicals and solvents

Table (3.1) Chemicals and solvents

Items	Source
Petroleum ether	Apha chemicals Made in India P – ether (60 – 80°C)
Chloroform	Alpha chemicals Made in India CH Cl <sub>3</sub> (62° C).
Methanol	Alpha chemicals Made in India MOOH (63° C)
Toluene	LabaChemiePut, India
Ethyl Acetate	S.D. Fine – Chem, Ltd, Mumbai
Methanol	LabaChemiePut, India
Hydrochloric acid (Dilute)	LabaChemiePut, India
Wagner’s reagent	LabaChemiePut, India
Hager’s reagent	Alpha Chemicals, India
Fehling A@ B Solution	Oxford Lab Chem, India
Sodium Hydroxide	LabaChemiePut, India
Ferric Chloride	
Benzene	
Ammonia	
Chloroform	
Lead Acetate	
Copper Acetate	
Nitric Acid	
Alcoholic $\alpha$ -naphthol	LabaChemiePut, India
H <sub>2</sub> SO <sub>4</sub>	LabaChemiePut, India
Distilled water	Distiller (BIBBY Scientific Ltd, UK)

### 3.1.2 Equipments and Instruments

Table (3.2) Equipments and Instruments

Items	Manufacture
Soxhlet Apparatus	
Rotary apparatus	
Brookfield	Brookfield DV – III Ultra Programmable Rheometer
Wax content	Rotary vacuum Evaporator RE 100
Density	Anton Paar Densitometer DMA 4500 M
Water content	Heater + Flask 1000 ml Trab + Condenser
Pour point	Cloud and pour test cabinet ASTM D 97 D2500 P592
Gas Chromatography – Mass spectroscopy	G C. MS- QP2010 Ultra

### 3.1.2 Row petrol samples

Case Study Hamra- ( Block 2 ) mixture of (OGM<sub>2</sub>+OGM<sub>5</sub>)

### 3.1.3 Plant material

*Balsamina Momordica* fruits were collected from Aloubid during autoum 2016 and authenticated by Dr. Yahia Suleiman at the herbarium of Medicinal and Aromatic plants & Traditional Medicine Research Institute, National Center for Research, Khartoum Sudan.

### 3.1.4 Microorganism

Table (3.3) Microorganism

Bacterial strains	
<i>Bacillus subtilis</i>	NCTC8236
<i>Staphylococcus aureus</i>	ATCC25923
<i>Escherichia coli</i>	ATCC25922
<i>Pseudomonas aeruginosa</i>	ATCC27853
Fungal strains	
<i>Aspergillus niger</i>	ATCC9763
<i>Candida albicans</i>	ATCC7596

## 3.2 Methods

### 3.2.2 Preparation of row petrol sample

A mixture of (OGM2 – OGM5) wells in (Hamra) field. Taken on the date 19 April 2016).

### 3.2.3 Preparation of plant material

The authenticated plant fruit was washed under the shade by spreading over burlap sacks for 12 days to constant weight. The seeds were removed from the dry fruit manually, then each part was grinded to coarse particle, and stored in a container for further use.



Photo ( 4.1) Dry Fruit



Dry Seeds

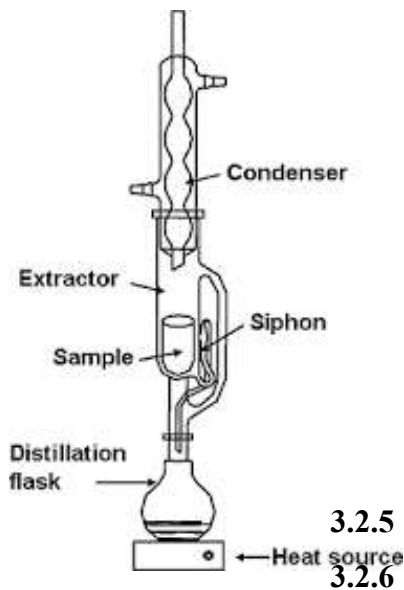
### 3.2.4 Preparation of plant crude extract

#### 3.2.4.1 Continuous Extraction:

A 100g weight of the coarse particles of the plant material was exhaustively extracted using Soxhlet apparatus with different organic solvents in order of increasing polarity: petroleum ether, chloroform, and methanol. Each extract was filtered and evaporated under reduced pressure using Rotary evaporator individually. The percentage of different extract yield were then calculated and tabulated. The different extracts were preserved in refrigerator till used. (Harborne, 1984)

#### 3.2.4.2 Maceration:

A 100 weight of the coarse particles of the plant material was macerated with methanol for 72hrs at room temperature. The mixture was filtered and evaporated under reduced pressure using Rotary evaporator. The organoleptic and the percentage of different extract yield were tabulated. The different extracts were preserved in refrigerator till used. (Harborne, 1984)



3.2.5  
3.2.6

Photo (4.2) Soxhlet apparatus

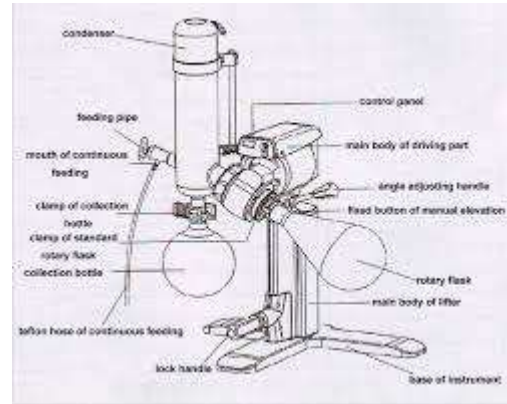


image showed Rotary apparatus

### 3.2.7 Test of rheological properties

- 1\ Density measurement using a densitometer, is a device that measures the API and the density of the sample.
- 2\ Measuring the pour point: is a test that measures the lowest temperature at which the crude can flow.
- 3\ Measuring the wax content: is a test that measures the percentage of wax in the crude sample. (rotary vacuum evaporator RE 100)
- 4\ Measuring the water content: which is a test that gives the percentage of water in the crude oil sample. (device: heater + 100 ml flask + trab + condenser) (illust.
- 5\ Measurement of dynamic viscosity: using Brookfield device: is a test that measures the change in viscosity along the change in temperature from (30o – 75o C)

#### 3.2.7.1 Viscosity

The crude sample is heated in a water bath adjusted to (75 – 80o C). The sample's temperature is then checked to ensure it is above the (WAT) and previously mentioned extracts are respectively injected (at a concentration of 2000 ppm of each extract.)

Samples are inserted consecutively into Brookfield's device to measure their dynamic viscosity (30o – 75o C). Each sample is inserted separately.



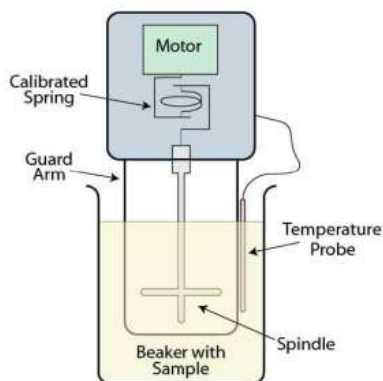


Photo . (4.3) Brook FIED

### 3.2.7.2 Pour point

The pour point test and the dynamic viscosity tests repeated to determine the best result obtained with different concentrations; pour point for ( blank) sample, and in addition to (2000 PPM) and (500 ppm –1500 ppm) for each 100 ml of crude oil, and that is to determine the least concentration for the plant extract that improves the crude's properties. (Omnia Abdalla Eltgani Ali (2016)),(Kamel Zaied (2015))

### 3.2.8 Qualitative test

#### 3.2.8.1 Phytochemical screening

Phytochemical screening was conducted for the extract using the method described by Prashant (2011)

#### **Test for Phenol:**

To 2 ml of the prepared extract, three drops of ferric chloride solution were added. Formation of the bluish green or red colour indicates the presence of phenols.

#### **Test for Tannin:** two methods were used

**(a) Ferric chloride test:** 10ml of distilled water was added to 3 ml of ethanol extract, 3 ml of 5% w/v ferric chloride solution was added. The blue black colour indicated the presence of tannins.

**(b) Gelatin test:** 3ml solution of ethanol extract, aqueous solution of gelatin and sodium chloride were added. A white buff coloured precipitate indicates the presence of tannins

#### **Test for Flavonoid:** two methods were used

(a) **Lead acetate Test:** 3ml of solution of ethanol extract, 3 ml of lead acetate solution was added. A white precipitate indicates the presence of flavonoid.

(b) **Alkaline Reagent:** 3ml of solution of ethanol extract was treated with few drops of sodium hydroxide solution formation of intense yellow colour, which becomes colourless on addition of dilute HCL indicates the presence flavonoids.

#### **Test for Saponin:**

The test solution was shaken with water. Copious lather formation indicates the presence of saponin.

**Test for Steroid:** two methods were used

(a) **Liebermann- Burchard Test:** to 2 ml of the test solution, a few drops of chloroform, 3 - 4 drops of acetic anhydride and one drop of concentrate sulphuric acid were added. Appearance of purple colour, which changes to blue or green colour, shows the presence of steroid.

(b) **Salkowski Test:** Few mg of residue of each extract was taken in 2 ml of chloroform and 2 ml of concentrated sulphuric acid was added along the side of the test tube. The test tube was shaken for few minutes. The development of red color in the chloroform layer indicates the presence of sterols

#### **Test for carbohydrates:**

**Molisch's Test:** A few mg of the test extract was placed in a test tube containing 0.5 ml of water, and it was mixed with two drops of Molisch's reagent (dissolving 10 g of  $\alpha$ -naphthol in 100 ml of 95 % alcohol). 1 ml of concentrated sulphuric acid was added along the side of the inclined test tube, so that the acid formed a layer beneath the aqueous solution without mixing with it. If a red brown ring appears at the common surface of the liquids, sugars are present.

**Test for Alkaloids:** two methods were used

(a) **Mayer's test:** To 2 ml of crude extract, two drops of Mayer's reagent were added along the sides of test tube. Appearance of white creamy precipitate indicates the presence of alkaloids.

(b) **Wagner's test:** A few drops of Wagners reagent are added to few ml of plant extract along the sides of test tube. A reddish- brown precipitate confirms the test as positive.

#### **Test for Glycosides**

(50 mg) of extract were hydrolyzed with concentrated hydrochloric acid for 2 hours on a water bath, filtered and the hydrolyzed extract was subjected to the following tests.

### **Borntrager's test:**

To 2 ml of filtered hydrolyzed extract, 3 ml of chloroform is added and shaken, chloroform layer is separated and 10% ammonia solution was added. A pink colour indicates presence of glycosides.

### **Test for Diterpenes:**

#### **Copper acetate test:**

0.5 g of the extract was dissolved in water and treated with 10 drops of copper acetate solution, formation of emerald green colour indicates presence of diterpenes.

### **3.2.8.2 Gas chromatography mass spectroscopic**

The petroleum ether crude extract of *Momordica balsamina* seeds was subjected to gas chromatography-mass spectrometry analysis (Shimadzu QP2010). The GC column was a DB-5MS (30 m in length, 0.25  $\mu\text{m}$  in thickness, 0.25 mm). The carrier gas was helium, with the pressure of 81.9 KP and flow rate of 1.33 mL/min. The injector temperature was 250 ° C and the split ratio was 1:50. The temperature was programmed in the range of 60-240 ° C increasing at 3 ° C/min. The mass spectra were recorded in the electron ionization mode, with ionization energy of 70eV. The temperature of the ion source was 250 ° C (Vidotto et al. 2013)

### **3.2.9 Antimicrobial activity**

Antimicrobial activity was studied by agar –diffusion method. Each inoculate of the test organisms (1ml ) was poured into sterile petri –dish. Media ( 45°C) was poured into petri dishes (20 ml). Then it was left to stand. Cups of 8 mm diameter were removed using cork porer. The cups were filled with the prepared plant extracts using sterile micro-pipette. Plates were then incubated up side down at 37°C for 24 hrs. .The sensitivities of the test organisms to the plant extracts were indicated by clear zones of growth inhibition around the cups containing the plant extracts and diameter of the clear zone was taken as an index of the degree of sensitivity (Kavangh, 1972).

**CHAPTER FOUR**  
**Results and Discussion**

## 4 Results and Discussion

### 4.1 Results

#### 4.1.2 Yield percent and organoliptic properties of crude extracts

The metabolites from the plant were extracted by methanol. As represented in table (4:1) the maximum yield was obtained from fruit using soxhlet.

**Table (4:1)Yield Percent and organoliptic properties of crude extract**

<b>Plant part</b>	<b>Method extraction</b>	<b>Plant extract</b>	<b>Yield %</b>	<b>Organoliptic properties</b>
Seed	Soxhlet	Petroleum ether	6.92	Lower polarity
		Chloroform	3.42	Medium polarity
		Methanol	2.48	High polarity
	Maceration	Methanol	3.175	High polarity
Fruit	Soxhlet	Petroleum ether	1.12	Lower Polarity
		Chloroform	0.56	Medium polarity
		Methanol	19.73	High polarity
	Maceration	Methanol	16.823	High polarity

#### 4.1.2 Crude oil properties with and (with crude oil key)

Table (4.2) Crude oil properties with and (with crude oil key).

<b>Test of rheological properties</b>	<b>POUR POINT(C<sup>0</sup>)</b>	<b>Density(g/ml)</b>	<b>API</b>	<b>S.G</b>	<b>Wax content(g)</b>	<b>Water content(ml)</b>
<b>Result</b>	<b>39</b>	<b>0.85525</b>	<b>33.79</b>	<b>0.85610</b>	<b>24.55</b>	<b>62</b>

### 4.1.3 Effect of extracts on rheological properties

#### Viscosity and pour point.

Table (4.3) viscosity for crude oil with and without additives at the same doses (2000ppm) for the stored seeds and fruits .

Temperature	Viscosity (CP)								
	BLANK	Plant Extract							
		A1	A2	A3	A4	B1	B2	B3	B4
75	59.99	64.99	94.98	54.99	54.99	144.97	49.99	49.99	39.99
74	59.99	74.98	99.98	54.99	64.99	149.97	54.99	54.99	44.99
73	59.99	84.98	99.98	59.99	69.99	154.97	54.99	64.99	49.99
72	64.99	89.98	99.98	59.99	74.98	159.97	44.99	74.98	64.99
71	64.99	99.98	104.98	54.99	79.98	164.96	39.99	89.98	74.98
70	64.99	104.98	104.98	54.99	84.98	164.96	39.99	89.98	79.98
69	64.99	104.98	104.98	54.99	84.98	169.96	39.99	99.98	89.98
68	69.99	109.98	109.98	54.99	89.98	169.96	34.99	99.98	89.98
67	69.99	109.98	109.98	54.99	94.98	174.96	34.99	109.98	94.98
66	69.99	119.97	109.98	54.99	99.98	179.96	34.99	109.98	99.98
65	69.99	119.97	114.98	54.99	99.98	179.96	34.99	114.98	99.98
64	74.98	124.97	114.98	54.99	104.98	179.96	34.99	124.97	104.98
63	74.98	124.97	119.97	54.99	109.98	189.96	29.99	129.97	104.98
62	74.98	129.97	119.97	54.99	109.98	189.96	34.99	129.97	114.98
61	74.98	134.97	124.97	59.99	114.98	194.96	34.99	134.97	119.97
60	79.98	139.97	129.97	59.99	119.97	199.96	29.99	139.97	119.97
59	89.98	149.97	149.97	59.99	129.97	204.96	29.99	154.97	124.97
58	114.98	154.97	179.96	59.99	159.97	209.96	29.99	169.96	139.97
57	139.97	179.96	204.96	59.99	179.96	219.95	29.99	184.96	144.97
56	154.97	194.96	214.95	59.99	199.96	239.95	29.99	199.96	154.97
55	169.96	214.95	234.95	64.99	214.95	249.95	29.99	214.95	164.96
54	184.96	234.95	259.94	64.99	234.95	259.94	29.99	229.95	174.96
53	199.96	254.95	274.94	64.99	254.95	274.94	34.99	249.95	189.96
52	219.95	269.94	299.94	69.99	269.94	289.94	34.99	269.94	194.96
51	239.95	294.94	319.93	69.99	299.94	304.93	34.99	284.94	214.95
50	269.94	319.93	349.93	74.98	334.93	334.93	39.99	319.93	234.95
49	304.93	344.93	389.92	79.98	374.92	364.92	39.99	359.92	254.95
48	349.93	384.92	434.91	84.98	419.91	394.92	39.99	389.92	269.94
47	389.92	409.91	484.90	89.98	474.90	424.91	44.99	439.91	304.93
46	449.90	454.90	554.88	99.98	544.88	484.90	49.99	489.90	344.93
45	529.89	489.90	644.86	114.98	629.87	554.88	49.99	569.88	389.92
44	614.87	554.88	744.84	134.97	714.85	609.87	59.99	664.86	444.91
43	714.85	619.87	874.81	164.96	829.82	699.85	69.99	739.84	514.89
42	839.82	674.86	1034.78	199.96	974.79	799.83	89.98	869.81	594.87
41	1014.78	759.84	1239.74	244.95	1149.75	879.81	104.98	1019.78	689.85

40	1214.74	869.81	1509.68	319.93	1364.71	1019.78	134.97	1204.74	814.83
<b>39</b>	<b>1474.69</b>	<b>989.79</b>	<b>1834.61</b>	<b>419.91</b>	<b>1654.65</b>	<b>1204.74</b>	<b>199.96</b>	<b>1349.71</b>	<b>969.79</b>
38	1824.61	1144.76	2274.51	654.86	2009.57	1459.69	249.95	1614.66	1164.75
37	2324.50	1344.71	3024.35	929.80	2474.47	1819.61	354.92	1994.57	1439.69
36	3274.30	1599.66	3839.18	1339.71	3379.28	2369.49	539.88	2569.45	1994.57
35	4364.07	1944.59	4878.96	1909.59	4374.07	3249.31	849.82	3399.27	2684.43
34	5358.86	2244.52	5323.86	2664.43	5358.86	4584.02	1259.73	4634.01	3734.20
33		2824.40		5288.87			2164.54		5263.88
32		3854.18					3134.33		
31		4888.96					4119.12		
30							4858.96		

Key: A1=petroleum ether extract of seeds. A2=chloroform extract of seeds.A3=ethanol  
A4= Methanol Extract

B1=petroleum ether extract of fruits.B2=chloroform extract of fruits.B3=ethanol extract  
B4 = Methanol Extract .

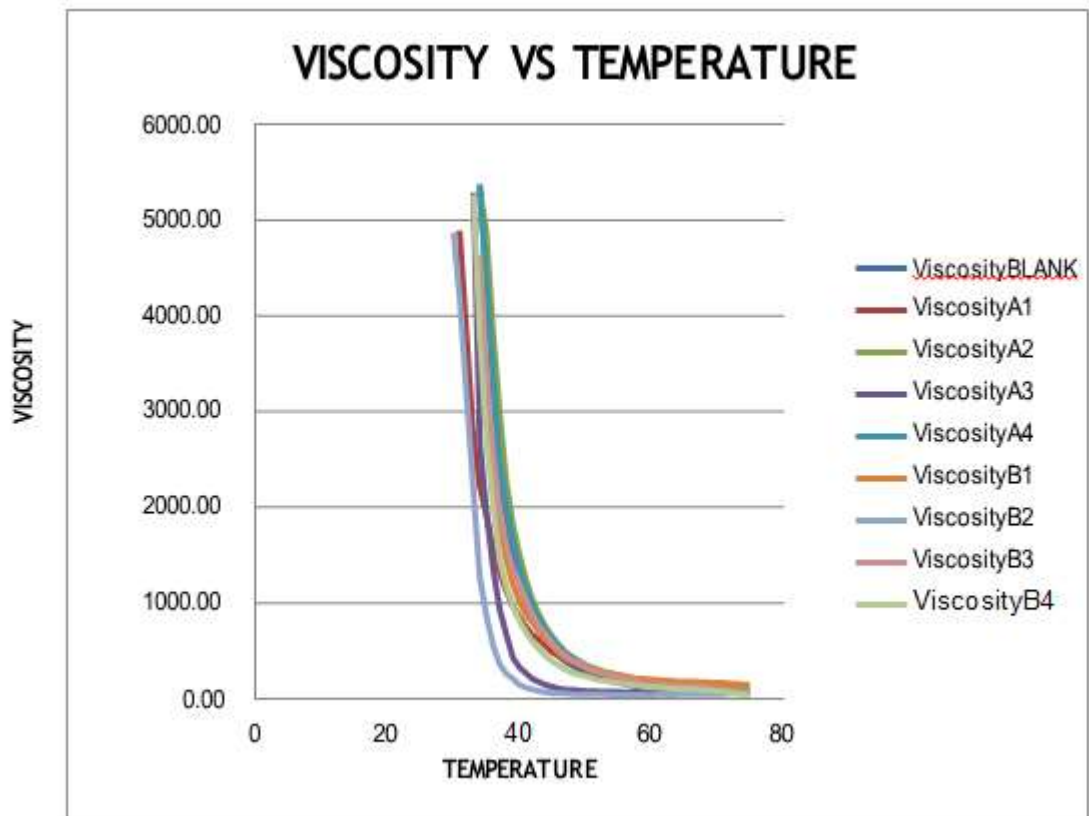


Table (4.4) Pour point for crude oil with and without additives at the same doses (2000ppm) for stored seeds and fruits

<b>Pour point Test extract at (2000PPM)</b>	<b>° C</b>
<b>A1</b>	<b>33</b>
<b>A2</b>	<b>36</b>
<b>A3</b>	<b>39</b>
<b>A4</b>	<b>39</b>
<b>B1</b>	<b>36</b>
<b>B2</b>	<b>33</b>
<b>B3</b>	<b>36</b>
<b>B4</b>	<b>39</b>

Key: A1=petroleum ether extract of seeds. A2=chloroform extract of seeds.A3=ethanol  
A4= Methanol Extract

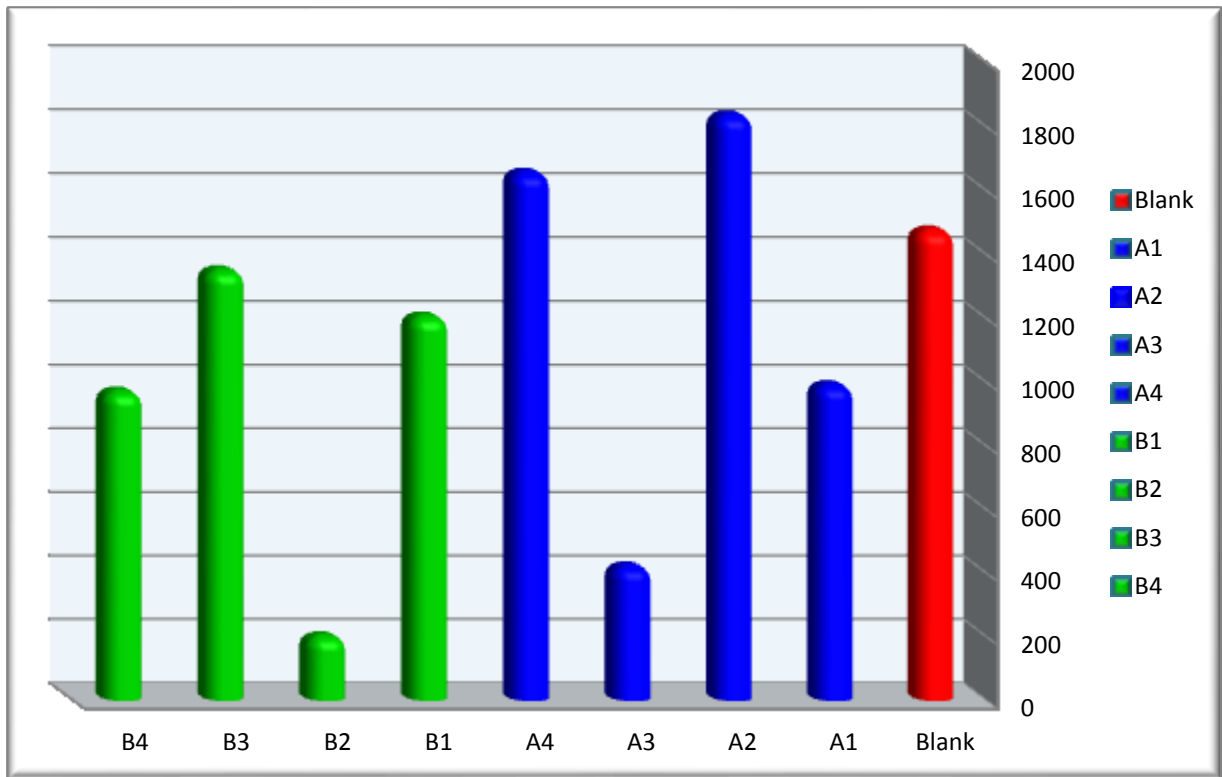
B1=petroleum ether extract of fruits.B2=chloroform extract of fruits.B3=ethanol extract  
B4 = Methanol Extract .



**Figure (4.1) 2000ppm viscosity vs 2000ppmTemperature (A1. A2. A3.A4) and (B1. B2. B3.B4) (oil crude key ) plant.**

Key: A1=petroleum ether extract of seeds. A2=chloroform extract of seeds.A3=ethanol A4= Methanol Extract

B1=petroleum ether extract of fruits.B2=chloroform extract of fruits.B3=ethanol extract B4 = Methanol Extract .



**Figure (4.2) Pour point blank at 39 C<sup>o</sup>**

**For blank and extract of stored seeds and fruits**

Key: A1=petroleum ether extract of seeds. A2=chloroform extract of seeds.A3=ethanol  
A4= Methanol Extract

B1=petroleum ether extract of fruits.B2=chloroform extract of fruits.B3=ethanol extract  
B4 = Methanol Extract .

**Table (4:5) viscosity for crude oil with and without additives at the same doses(2000ppm) for newly productive seeds and fruits**

Temperature	Viscosity (CP)						
	BLANK	Plant Extract					
		A1	A2	A3	B1	B2	B3
75	59.99	12.85	4.28	12.85	8.57	4.28	12.85
74	59.99	12.85	12.85	12.85	8.57	8.57	12.85
73	59.99	12.85	12.85	12.85	8.57	8.57	12.85
72	64.99	12.85	12.85	12.85	8.57	8.57	17.14
71	64.99	12.85	12.85	12.85	8.57	8.57	17.14
70	64.99	12.85	12.85	12.85	8.57	8.57	17.14
69	64.99	12.85	12.85	12.85	8.57	12.85	17.14
68	69.99	12.85	12.85	12.85	8.57	12.85	17.14
67	69.99	12.85	12.85	12.85	8.57	12.85	17.14
66	69.99	12.85	12.85	12.85	8.57	12.85	17.14
65	69.99	12.85	12.85	12.85	12.85	12.85	17.14
64	74.98	12.85	12.85	17.14	12.85	12.85	17.14
63	74.98	12.85	12.85	17.14	12.85	12.85	21.42
62	74.98	12.85	17.14	17.14	12.85	12.85	21.42
61	74.98	12.85	17.17	17.14	12.85	12.85	21.42
60	79.98	12.85	17.14	17.14	12.85	12.85	21.42
59	89.98	12.85	17.14	17.14	12.85	12.85	21.42
58	114.98	17.14	17.14	17.14	12.85	17.14	21.43
57	139.97	17.14	17.14	17.14	12.85	17.14	21.43
56	154.97	17.14	17.14	17.14	12.85	17.14	21.44
55	169.96	17.14	17.14	17.14	12.85	17.14	21.44
54	184.96	17.14	17.14	17.14	12.85	17.14	21.44
53	199.96	17.14	21.42	21.42	12.85	17.14	25.71
52	219.95	17.14	21.42	21.42	12.85	17.14	25.71
51	239.95	21.42	21.42	21.42	12.85	21.42	25.71
50	269.94	21.42	21.42	25.71	17.14	21.42	34.28
49	304.93	21.42	21.42	25.71	17.14	21.42	38.56
48	349.93	21.42	25.71	34.28	17.14	21.42	34.28
47	389.92	21.42	25.71	34.28	21.42	21.42	47.13
46	449.90	25.71	25.71	42.85	21.42	25.71	42.85
45	529.89	29.99	34.28	42.85	25.71	25.71	51.42
44	614.87	34.28	42.85	55.7	34.28	25.71	59.99
43	714.85	34.28	42.85	72.84	34.28	29.99	72.84
42	839.82	42.85	47.13	111.4	47.13	34.28	81.41
41	1014.78	51.42	59.99	119.97	47.13	38.56	94.27

40	1214.74	59.99	68.56	124.26	64.27	59.99	111.4
39	<b>1474.69</b>	<b>77.13</b>	<b>103.98</b>	<b>141.4</b>	<b>92.41</b>	<b>89.98</b>	<b>137.11</b>
38	1824.61	98.55	107.12	171.39	94.27	89.98	167.11
37	2324.50	124.26	141.4	205.67	124.26	102.84	209.96
36	3274.30	162.82	184.25	265.66	167.11	107.12	269.94
35	4364.07	244.23	257.09	321.36	235.66	111.4	355.64
34	5358.86	338.5	372.78	441.33	321.36	128.54	484.18
33		484.18	561.31	625.58	509.89	162.82	681.28
32		728.42	882.67	916.95	831.25	209.96	994.07
31		1251.16	1516.82	1371.14	1366.85	321.36	1598.23
30		2099.55	2515.18	2048.13	2185.25	462.76	2339.5
29		2099.36	3689.21	3230.74	3372.14	741.27	3594.95
28		4117.68		4284.8		1066.92	
27						1731.06	
26						2720.85	
25						4121.98	

Key: A1=petroleum ether extract of seeds. A2=chloroform extract of seeds.A3=ethanol

B1=petroleum ether extract of fruits.B2=chloroform extract of fruits.B3=ethanol extract

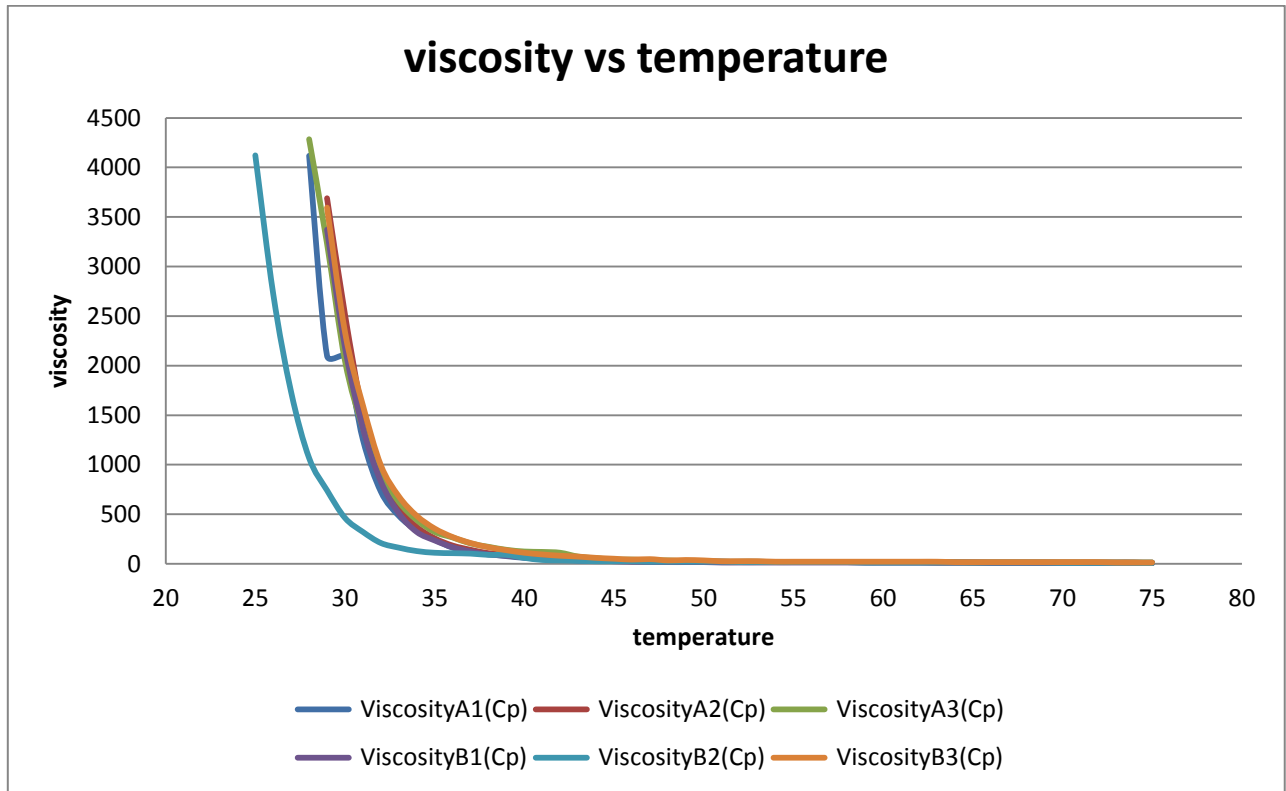


Figure (4.3) viscosity vs 2000ppm temperature 2000ppm Temperature (A1. A2. A3.) and (B1. B2. B3.) (Blank ) plant

Key: A1=petroleum ether extract of seeds. A2=chloroform extract of seeds.A3=ethanol  
 B1=petroleum ether extract of fruits.B2=chloroform extract of fruits.B3=ethanol extract

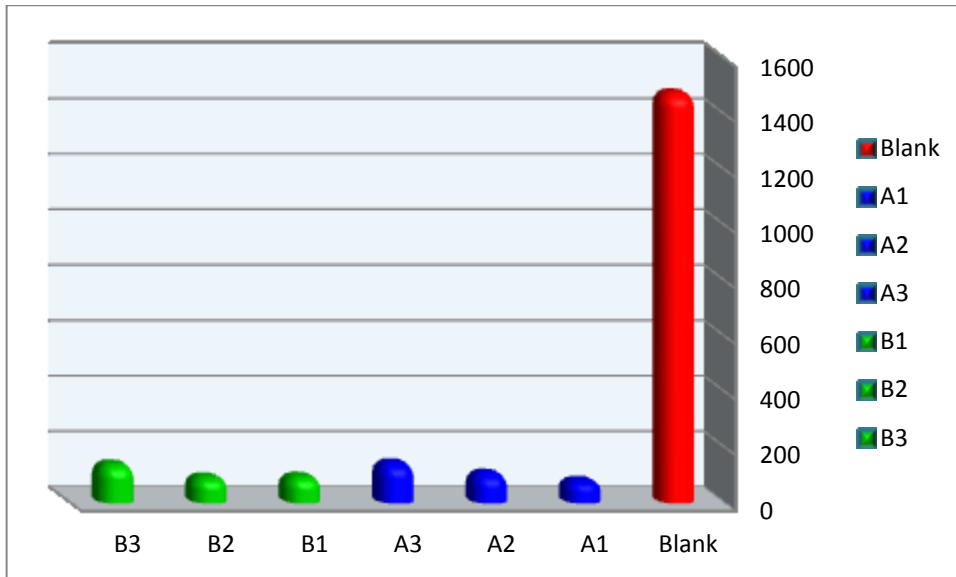


Figure (4.4) Pour point at 39 C<sup>o</sup> between the blank sample and the extractions of the fresh seeds and fruits

Key: A1=petroleum ether extract of seeds. A2=chloroform extract of seeds.A3=ethanol  
 B1=petroleum ether extract of fruits.B2=chloroform extract of fruits.B3=ethanol extract

Table (4:6) Viscosity profile for crude oil with and without one type additive (A1) at different doses (2000,1500,500,ppm).

Temperature C°	ViscosityA1 (2000ppm)(Cp)	Viscosity A1 (1500 PPM)Cp	Viscosity A1(500PPM)Cp
75.00	12.85	17.14	17.14
74.00	12.85	17.14	17.14
73.00	12.85	17.14	17.14
72.00	12.85	17.14	17.14
71.00	12.85	17.14	17.14
70.00	12.85	17.14	17.14
69.00	12.85	17.14	17.14
68.00	12.85	17.14	17.14
67.00	12.85	17.14	17.14
66.00	12.85	17.14	17.14
65.00	12.85	17.14	17.14
64.00	12.85	17.14	17.14
63.00	12.85	17.14	17.14
62.00	12.85	17.14	21.42
61.00	12.85	17.14	21.42
60.10	12.85	17.14	17.14
59.00	12.85	17.14	21.42
58.00	17.14	21.42	17.14
57.00	17.14	17.14	21.42
56.00	17.14	21.42	21.42
55.00	17.14	25.71	21.42
54.00	17.14	21.42	21.42
53.00	17.14	21.42	21.42
52.00	17.14	25.71	25.71



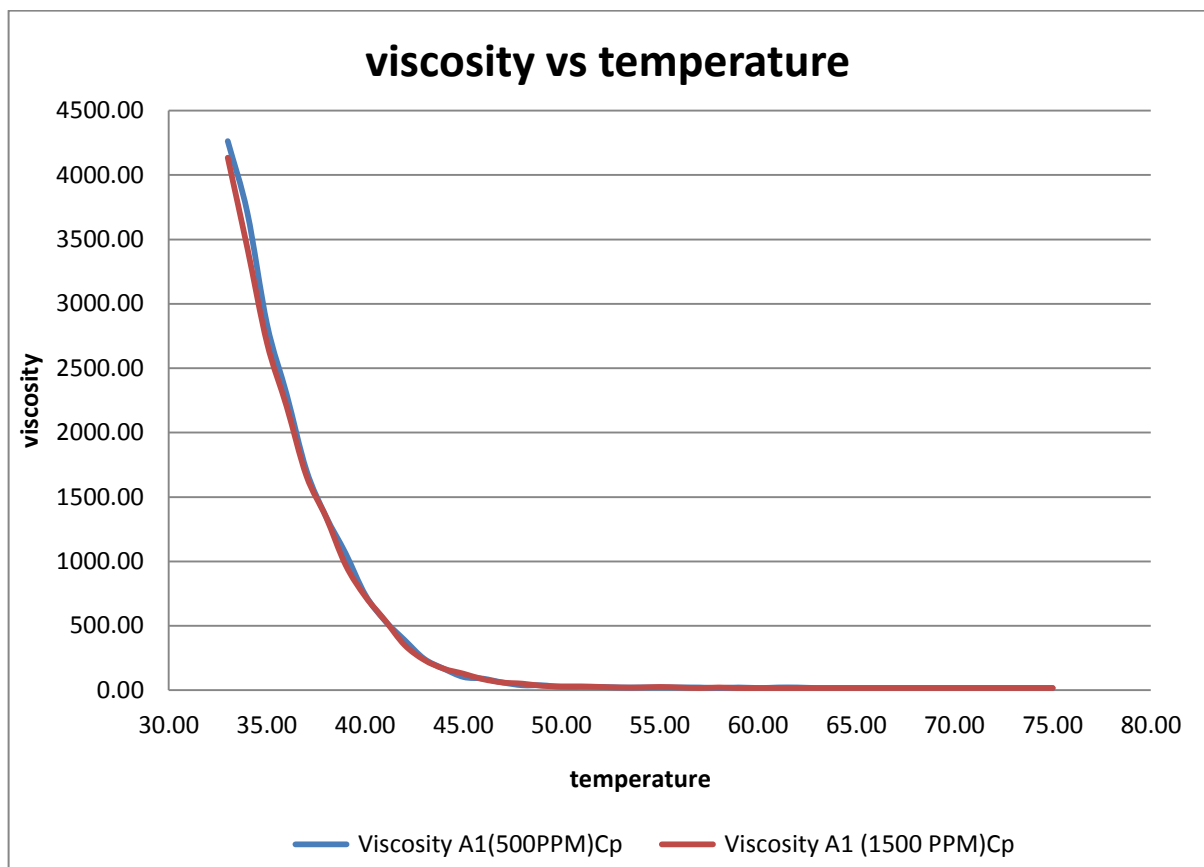
51.00	21.42	29.99	25.71
50.00	21.42	29.99	25.71
49.00	21.42	34.28	38.56
48.00	21.42	51.42	38.56
47.00	21.42	59.99	59.99
46.00	25.71	85.70	89.98
45.00	29.99	128.54	102.84
44.00	34.28	167.11	167.11
43.00	34.28	235.66	244.23
42.00	42.85	351.35	389.92
41.00	51.42	544.17	539.88
40.00	59.99	728.42	741.27
<b>39.00</b>	<b>77.13</b>	<b>972.65</b>	<b>1062.63</b>
38.00	98.55	1345.43	1349.71
37.00	124.26	1666.79	1709.64
36.00	162.82	2202.39	2296.65
35.00	244.23	2695.14	2840.82
34.00	338.5	3432.12	3706.35
33.00	484.18	4134.83	4263.38
	728.42		
	1251.16		
	2099.55		
	2099.36		
	4117.68		

**Key: A1=petroleum ether extract of seeds.**

**Table (4-7) pour point for crude oil with and without one type of additive (A1) at different doses(2000,1500,500ppm)**

Sample	POUR POINT(C <sup>0</sup> )
Blank	39
A1 (2000 PPM)	33
A1 (1500 PPM)	39
A1 (500 PPM)	39

**Key: A1=petroleum ether extract of seeds.**



**Figure (4.5) viscosity vs Temperature (A1.2000ppm), (A1 15000ppm) (A1500ppm)**

**Key: A1=petroleum ether extract of seeds.**

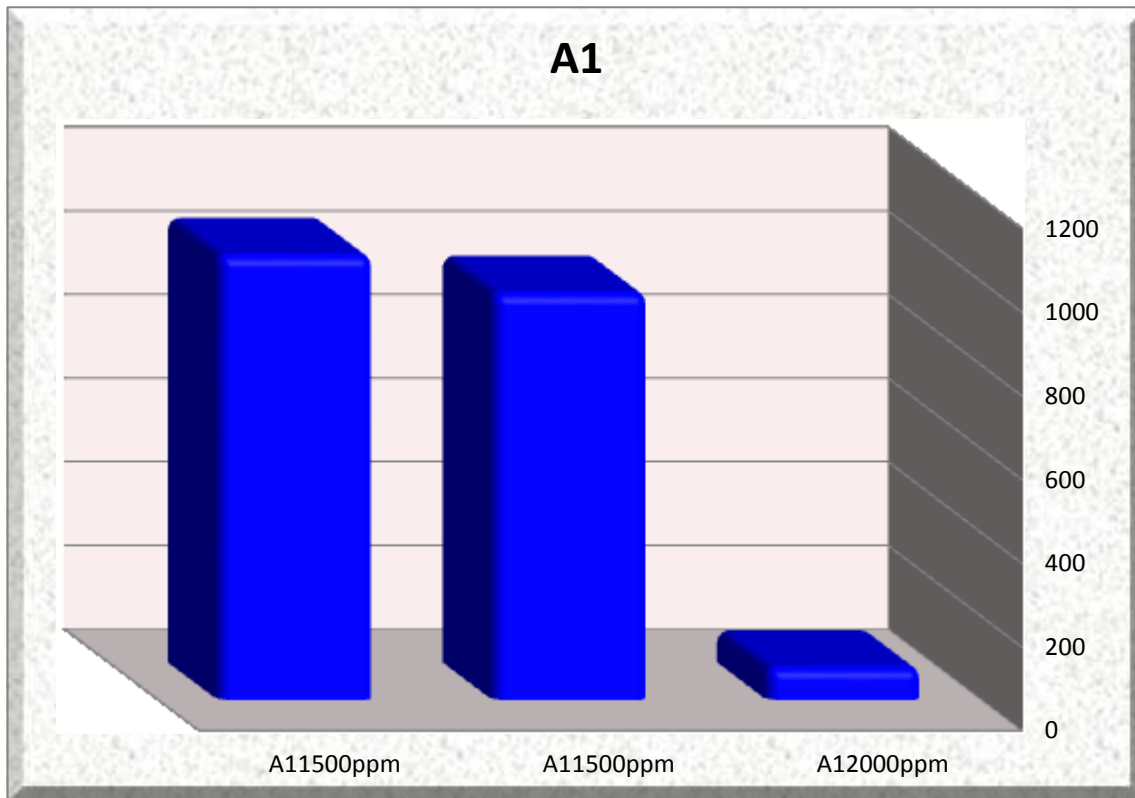


Figure (4.6) Pour point at 39 C<sup>o</sup> between the extractions of (A1.2000ppm), (A1 15000ppm) (A1500ppm) -

Key: A1=petroleum ether extract of seeds.

Table (4:8) viscosity for crude oil with and without one type of additives (B2) at the different doses(2000,1500,500ppm)

Temperature(C <sup>o</sup> )	ViscosityB <sub>2</sub> 2000(Cp)	ViscosityB <sub>2</sub> 1500(Cp)	ViscosityB <sub>2</sub> 500(Cp)
75.00	4.28	12.85	12.85
74.00	8.57	12.85	12.85
73.00	8.57	12.85	12.85
72.00	8.57	12.85	17.14
71.00	8.57	12.85	17.14
70.00	8.57	12.85	17.14
69.00	12.85	12.85	17.14
68.00	12.85	12.85	21.42

<b>67.00</b>	<b>12.85</b>	<b>12.85</b>	<b>21.42</b>
<b>66.00</b>	<b>12.85</b>	<b>12.85</b>	<b>21.42</b>
<b>65.00</b>	<b>12.85</b>	<b>12.85</b>	<b>21.42</b>
<b>64.00</b>	<b>12.85</b>	<b>17.14</b>	<b>21.42</b>
<b>63.00</b>	<b>12.85</b>	<b>17.14</b>	<b>21.42</b>
<b>62.00</b>	<b>12.85</b>	<b>17.14</b>	<b>21.42</b>
<b>61.00</b>	<b>12.85</b>	<b>17.14</b>	<b>25.71</b>
<b>60.00</b>	<b>12.85</b>	<b>17.14</b>	<b>25.71</b>
<b>59.00</b>	<b>12.85</b>	<b>17.14</b>	<b>25.71</b>
<b>58.00</b>	<b>17.14</b>	<b>17.14</b>	<b>25.71</b>
<b>57.00</b>	<b>17.14</b>	<b>17.14</b>	<b>25.71</b>
<b>56.00</b>	<b>17.14</b>	<b>17.14</b>	<b>25.71</b>
<b>55.00</b>	<b>17.14</b>	<b>17.14</b>	<b>25.71</b>
<b>54.00</b>	<b>17.14</b>	<b>17.14</b>	<b>25.71</b>
<b>53.00</b>	<b>17.14</b>	<b>17.14</b>	<b>25.71</b>
<b>52.00</b>	<b>17.14</b>	<b>21.42</b>	<b>29.99</b>
<b>51.00</b>	<b>21.42</b>	<b>21.42</b>	<b>29.99</b>
<b>50.00</b>	<b>21.42</b>	<b>21.42</b>	<b>34.28</b>
<b>49.00</b>	<b>21.42</b>	<b>25.71</b>	<b>34.28</b>
<b>48.00</b>	<b>21.42</b>	<b>25.71</b>	<b>42.85</b>
<b>47.00</b>	<b>21.42</b>	<b>25.71</b>	<b>47.13</b>
<b>46.00</b>	<b>25.71</b>	<b>25.71</b>	<b>51.42</b>
<b>45.00</b>	<b>25.71</b>	<b>29.99</b>	<b>55.70</b>
<b>44.00</b>	<b>25.71</b>	<b>34.28</b>	<b>68.56</b>
<b>43.00</b>	<b>29.99</b>	<b>34.28</b>	<b>77.13</b>
<b>42.00</b>	<b>34.28</b>	<b>42.85</b>	<b>89.98</b>
<b>41.00</b>	<b>38.56</b>	<b>55.70</b>	<b>115.69</b>
<b>40.00</b>	<b>59.99</b>	<b>68.56</b>	<b>137.11</b>
<b>39.00</b>	<b>89.98</b>	<b>89.98</b>	<b>167.11</b>

<b>38.00</b>	<b>89.98</b>	<b>124.26</b>	<b>214.24</b>
<b>37.00</b>	<b>102.84</b>	<b>179.96</b>	<b>278.51</b>
<b>36.00</b>	<b>107.12</b>	<b>244.23</b>	<b>372.78</b>
<b>35.00</b>	<b>111.40</b>	<b>381.35</b>	<b>531.32</b>
<b>34.00</b>	<b>128.54</b>	<b>612.73</b>	<b>784.12</b>
<b>33.00</b>	<b>162.82</b>	<b>934.09</b>	<b>1199.74</b>
<b>32.00</b>	<b>209.96</b>	<b>1525.39</b>	<b>1838.18</b>
<b>31.00</b>	<b>321.36</b>	<b>2275.23</b>	<b>3020.78</b>
<b>30.00</b>	<b>462.76</b>	<b>3839.18</b>	<b>4387.64</b>
<b>29.00</b>	<b>741.27</b>		
<b>28.00</b>	<b>1066.92</b>		
<b>27.00</b>	<b>1731.06</b>		
<b>26.00</b>	<b>2720.85</b>		
<b>25.00</b>	<b>4121.98</b>		

**Key : B2=chloroform extract of fruits.**

Table (4.9) Pour point for crude oil with and without one type of additives (B2) at different doses (2000,1500, 500,ppm).

Sample	Pour point (c <sup>o</sup> )
BLANK	39
<b>B<sub>2</sub>2000 PPM</b>	33
<b>B<sub>2</sub>1500 PPM</b>	36
<b>B<sub>2</sub>500 PPM</b>	39

**Key : B2=chloroform extract of fruits.**

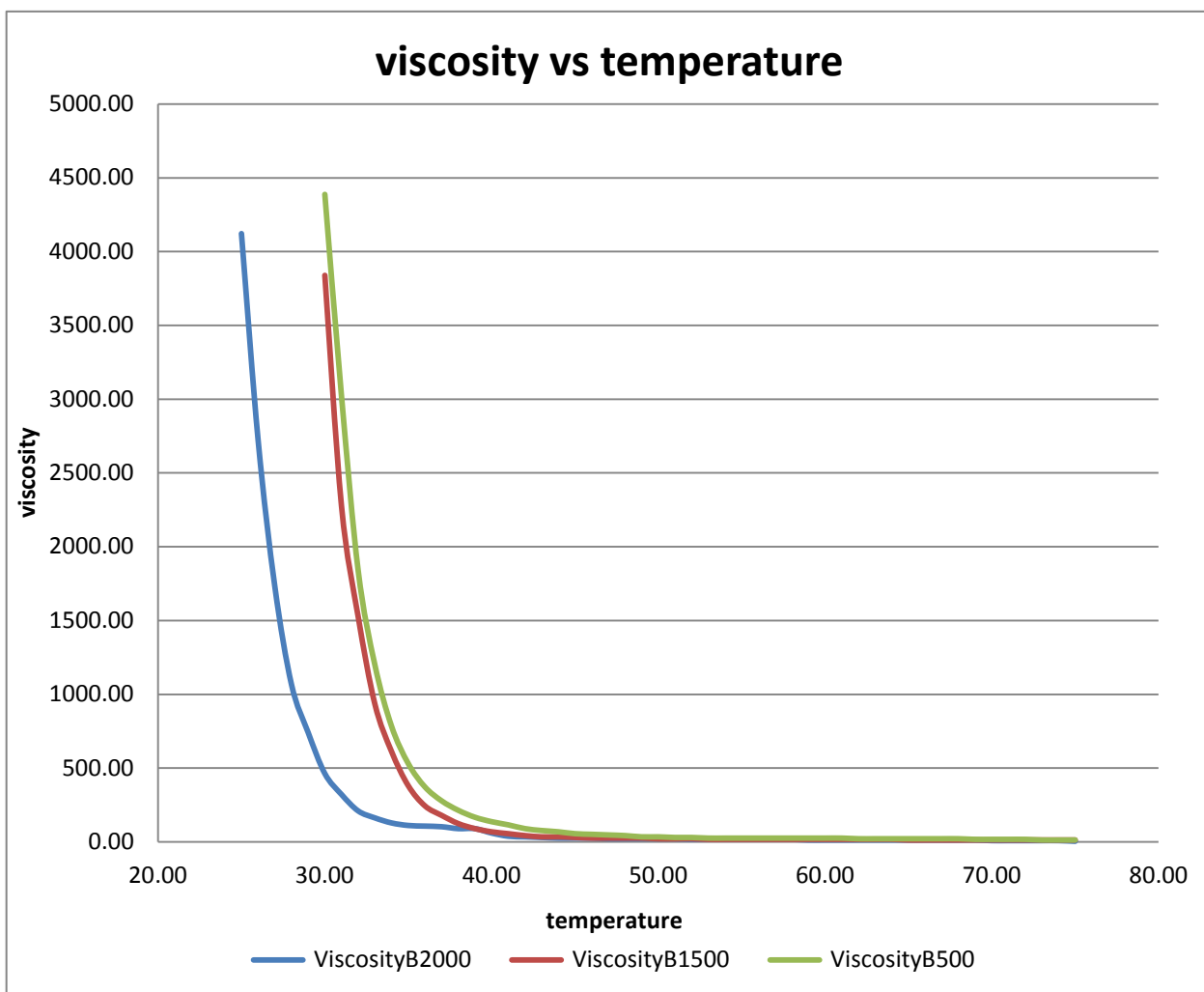


Figure (5.7) viscosity vs Temperature (B2) 2000ppm, 1500 ppm, 500 ppm B2(500ppm) .

**Key : B2=chloroform extract of fruits.**

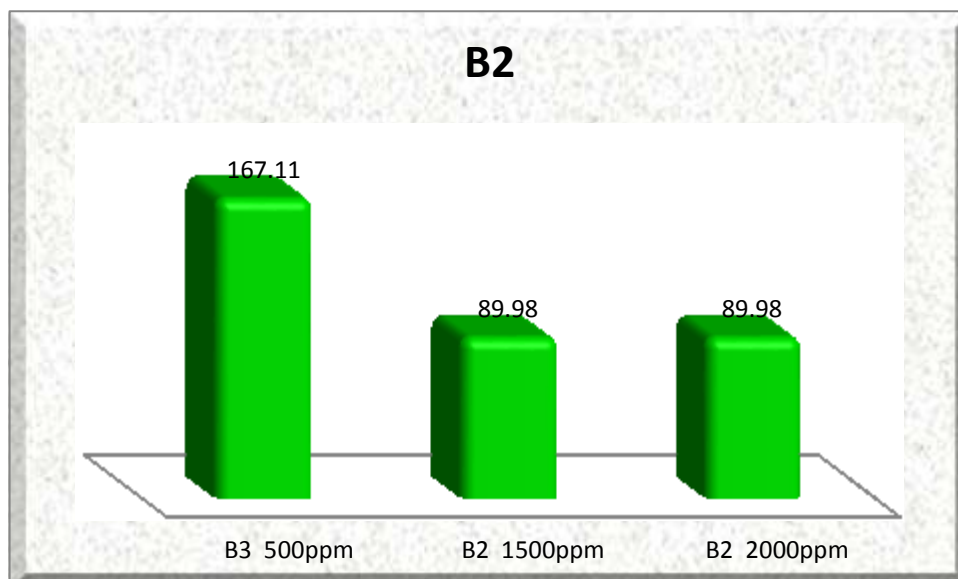


Figure (4.8) pour point at 39 C<sup>o</sup> the comparison between the extractions of (B2.2000ppm), (B2 1500ppm) (B2 500ppm) - at pour point

#### 4.1.4 Qualitative chemical test

##### 4.1.4.1 General Phytochemical screening

Table (4:10): Phytochemical screening of fruit and seed extracts

Phytochemical Group	Reagents	Seed extracts			Fruit extracts		
		A1	A2	A3	B1	B2	B3
Alkaloid	Wagner	+	+	+	+	+	+
	Hager	+	+	+	+	+	+
Carbohydrates	molish	+	+	-	+	+	-
	Fehling	+	+	-	+	+	+
Glycoside	Modified bontriger	+	+	+	+	+	+
Saponin	Forth test	+	+	-	+	+	+
Phytosterol	salkowiske	+	+	+	+	+	+
	Libermann	+	+	+	+	+	+
Phenol	Ferric chloride	-	-	+	-	-	+

<b>Flavanoide</b>	<b>Alkaline test</b>	+	+	+	+	+	+
	<b>Lead acetate</b>	+	+	+	+	+	+
<b>Protein and Amino acid</b>	<b>Xanthoproteic</b>	+	+	+	-	-	-
<b>Diterpenes</b>	<b>Cupper acetate</b>	+	-	+	+	-	+

Key: A1=petroleum ether extract of seeds. A2=chloroform extract of seeds.A3=ethanol

B1=petroleum ether extract of fruits.B2=chloroform extract of fruits.B3=ethanol extract of fruits.(+) detection of secondary metabolite, (-) no detection of secondary metabolite



### 5.1.4.2 GC- MS Analysis chromatography mass spectroscopic

Table (4.11) Constituent of plant extract (A1)

#### Peak Report TIC

Peak#	R. Time	Area	Area%	Name
1	5,944	560,283	0.21	Octaonic acid, methyl ester
2	6,986	651,658	0.24	2,4-Nonadienal, (E,E)
3	7,273	218,648	0.08	2,4-Nonadienal,
4	10,194	2,508,709	0.94	Nonanoic acid, 9-oxo methyl ester
5	11,586	87,973	0.03	Nonanedioic acid, dimethyl ester
6	13,580	568,215	0.21	Methyl tetradecanate
7	14,654	239,478	0.09	Pentadecanoic acid, methyl ester
8	15,490	224,960	0.08	9-Hexadecanoic, methyl ester(Z)
9	15,691	40,838,372	15.28	Hexadecanoic, methyl ester
10	15,831	1,266,906	0.47	7,10-Hexadecadienoic acid, methyl ester
11	16,657	554,731	0.21	Hexadecanoic acid, 15-methyl, -methyl ester
12	17,341	34,770,520	13.01	9,12-Octadecanoic acid(Z-Z)-methyl ester
13	17,390	39,536,195	14.79	9-Octadecanoic acid(Z)-methyl ester
14	17,607	44,706,158	16.72	Methyl stearate
15	18,630	79,333,871	29.67	Gamma. – Linolenic acid, Methyl ester
16	18,756	5,082,644	1.90	Methyl 9, cis, 11 trans.t.13.trans-octadecanoic acid
17	19,003	2,099,766	0.79	Cis-7-10,13,16-docosatetraenoic acid, methyl ester
18	19,155	1,519,562	0.57	Cis-1-Eicosenoic acid, methyl ester
19	19,353	2,865,984	1.07	Eicosanoic acid, methyl ester
20	19,408	677,462	0.25	Tricyclo[20.8.0.0(7,16)] triacontane,1(22).]
21	20,365	739,298	0.28	2-Ethyl-5-methyl-octahydrocyclopenta(b)
22	20,423	497,538	0.19	2-4 dodecadienal
23	20,565	1,081,589	0.40	2-4 dodecadienal, (E,E)-
24	20,605	732,562	0.27	Bicycle[2.2.1]heptan-2-o;,1.3.3-trimethyl
25	20,849	4,989,980	1.87	6-Methyl –bicyclo(4.2.0)octan-7-ol
26	20,971	463,286	0.17	docosanoic acid. methyl ester
27	22,474	535,927	0.20	Tetracosanoic acid, methyl ester
		267,352,275	100.00	

Table (4.12) Constituent of plant extract (B2)

## Peak Report TIC

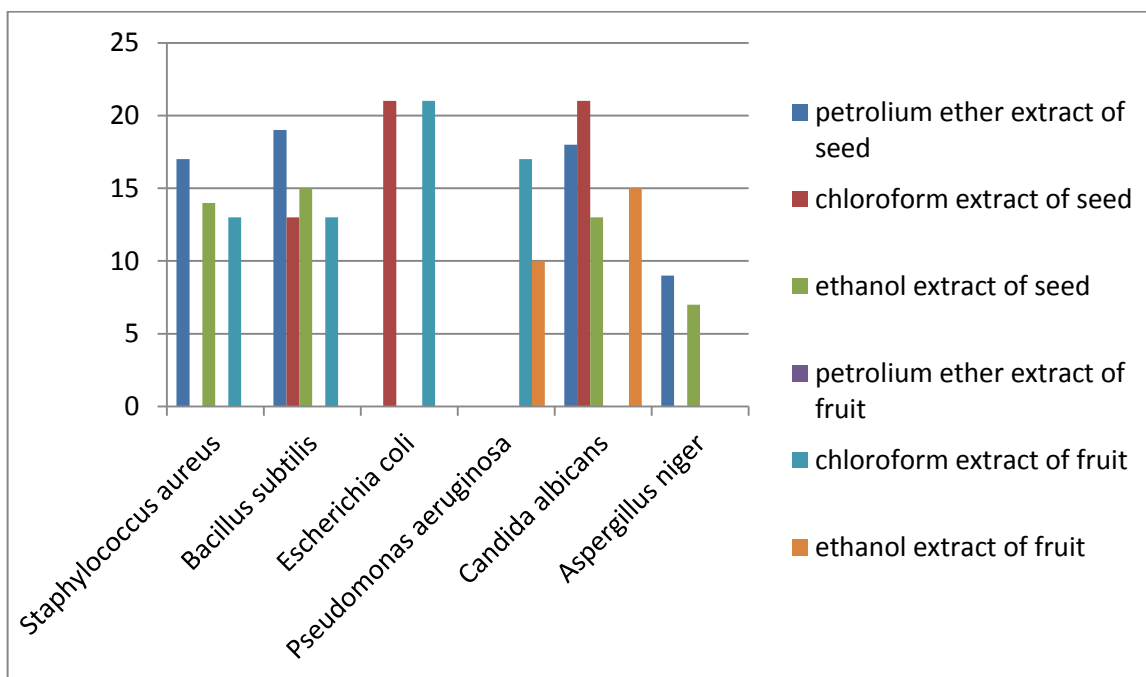
Peak#	R. Time	Area	Area%	Name
1	3,230	114117	0.12	2,5- Dimethylhexane-2,5-dihydroerixude
2	3,421	285051	0.30	1-methoxy -2-propyl acetate
3	3,490	348362	0.37	2.4-Nonadienal, (E,E)1-Methoxy-2 propyl acetate
4	3,556	137510	0.14	Ethanol, 2-[2-(2-butyl-2butyl ester
5	3,808	73970	0.08	Acetic acid, diethyl-
6	5,162	1079073	1.14	Propanoic acid, 2-methyl-,propyl ester
7	5,362	440797	0.46	Malic Acid
8	5,492	343847	0.36	Pyrazine, tetramethyl-
9	10,928	119065	0.13	Trans-, beta,-Ionone
10	11,595	282984	0.30	2(4H) – Benzofuranone, 5,6,7,7a-tetrahydrolic
11	11,752	299129	0.32	Dodecanoic acid
12	12,081	84585	0.09	3,5,9-Undecatrien-2-one, 6,10-dimethyl-
13	13,051	177808	0.19	2-(2-Hydroxyhex – 1 – enyl)-3-methyl-5,6-dil
14	13,486	280114	0.30	Tetradecanal
15	14,002	1955414	2.06	Hexadecanoic acid
16	15,674	713763	0.75	Hexadecanoic acid, methyl ester
17	15,894	609623	0.64	Cis-9- Hexadecanoic acid
18	16,115	39529602	41.67	n- Hexadecanoic acid
19	16,338	1136300	1.20	Hexadecanoic acid – methyl ester
20	17,791	12804504	13.50	Oleic Acid
21	17,813	8564988	9.03	9,12,15- octadecatrienoic acid,( Z,Z,Z)
22	17,967	10485741	11.05	Octadecanoic acid
23	19,163	3762359	3.97	Stearic acid , 2 hydroxy-1-methylpropyl
24	20,678	3347820	3.53	Linolenic acid, 2-hydroxy-1-(hydroxyl methyl)
25	20,808	1099245	1.16	Eicosanic acid
26	20,887	1504913	1.59	Hexadecanote acid, 2- hydroxyl – 1 – (hydroxymethyl )
27	22,285	1007721	1.06	9- Octadecenoic acid, 2-hyrdoxy – 1 – (hydroxyl methyl)
28	22,346	745783	0.79	3-Tetradecenoic acid,
29	22,444	523522	0.55	Octadecanoic acid, 2,3-dihydroxypropyl
30	23,206	746609	0.79	Squalene
31	23,348	2264576	2.39	Ergost – 5 – en-3 – ol, (3 , beta)
		<b>94868895</b>	<b>100.00</b>	

#### 4.1.4 Antimicrobial activity of the crude extracts

**Table (4:13) the antimicrobial activity of *Momordica balsamina* fruit and seed extracts at concentration 20mg/ml.**

<i>Crude extract or fraction</i> (20mg/ml)	<i>MIZD (mm)</i>					
	<i>Bacterial strain</i>				<i>Fungal strains</i>	
	<i>S.a</i>	<i>B.s</i>	<i>E.c</i>	<i>Ps.a</i>	<i>C. a</i>	<i>A. n</i>
A1	17	19	–	–	18	9
A2	–	13	21	–	21	–
A3	14	15	–	–	13	4
B1	–	–	–	–	–	–
B2	13	13	21	17	–	4
B3	–	–	–	6	16	–

S.a = Staphylococcus aureus    B.s= Bacillus subtilis    E.c = Escherichia coli    Ps.a= Pseudomonas aeruginosa , C.a = Candida albicans , A.n = Aspergillus niger



**Figure (4:9) the antimicrobial activity of *Momordica balsamina* fruit and seed extracts at concentration 20mg/ml.**

## 4.2 Discussions

The study used the extraction of seeds and fruits of *Momordica Balismina* stored and newly produced, the results show when used seeds (A) (produced from extracting Petroleum Ether) the newly produced that give the best treatment of crude oil from stored seeds, where newly production seeds are improved (viscosity) for the sample (blank) at the same pour point which 39°C to 33°C is viscosity from (1474.69 cp) to 77.13 (cp) where the improved seeds its viscosity from (1474.69 cp to 989.79 cp) (table (5.5)). When used fruits (B2) which produced from extracting chloroform newly produced gives the best treatment from using stored fruits where the improved newly fruits produced viscosity for the same sample from (1474.69 cp to 89.98 cp) where the improved stored fruits viscosity from (1474.69 cp to 199.96 cp) (table 5.3). From these results showed that to get best results it is better to use seeds (A1) and fruits (B2) newly produced, because storage affected the secondary plant metabolites.

From the extract results it is found that seeds (A1) and Fruits (B2) has high polarity where we can use water to extract great quantity of extractions.

A1 and B2 extracts can not be determined as polar or non-polar components. The results of GC-MS analysis, any components of this extract increased more than 9%, is effected the treatment of the rheological properties. These components can be prepared in the lab.

From the results of (phyto chemical) for seeds (A1) and Fruits (B2) it found that its include a high rate of secondary plant metabolites. Also its internal contents is similar as alkaloids, carbohydrates, glycosides, saponins, phytosterols and flavonoids.

The results of Antimicrobial activity it has been found that extractions may treat the polluted microbial environment and the bacteria found in the effected water by oil when it erected in soil in oil fields.

When compared the research's results with the treatment results that done in the field, it found that the target of the pour point is less than 25°C. and viscosity is less 170 cp. The results that collected by the research in compared with the results of the fields is acceptable.

The research results gave a suitable results because the sample is extracted from OGM only one, where the treatment that done in the field for a great group of OGM includes

a crude with different levels of viscosity and pour point where treatment done in the field in one PDF which all ores of the field are collected in the central unit.

# **CHAPTER FIVE**

## **Conclusion & Recommendation**

## 5.1 Conclusion

Viscosity and Pour point Sawa This plant was stored or modernized because it contains many secondary exploits that affect the wax network in the crude which is its role in controlling the flow of crude

## 5.2 Recommendation

- Continue on the side of the extraction of plants to address the problems facing the petroleum industry. Continuing to conduct several tests on different types of crude with different properties to determine the effect of the extract of *Momordica Blasimina* on the materials that have different waxy structures .
- Search for other methods that can be use for extractions as Aqueous Alcoholic Extraction by Fermentation, Counter-current Extraction, Ultrasound Extraction (Sonication), Supercritical Fluid Extraction.
- It is also recommended to test physiological properties of extractions.
- We must made GCMS for all extractions that we get to interpreted the ability of A1 and B2 to differentiate why are always better than other extractions.
- Antimicrobial activity of the crude extracts, gave positive results in addressing pollutions, we can prepared with water pollutions that accompany with petroleum.

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# Appendix (1)

Stages and growth of Momrdica Balsming plant



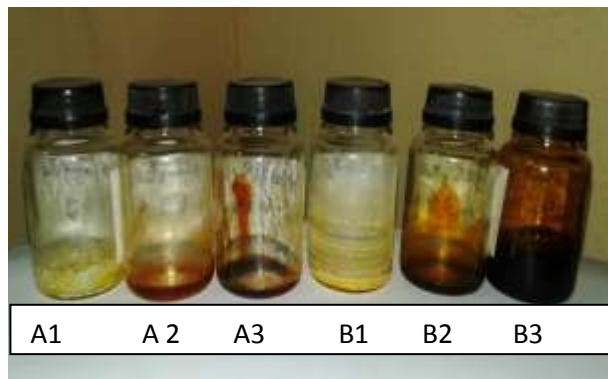
**plant Momordica Balsaming fruits and seeds separately .**



Fruits

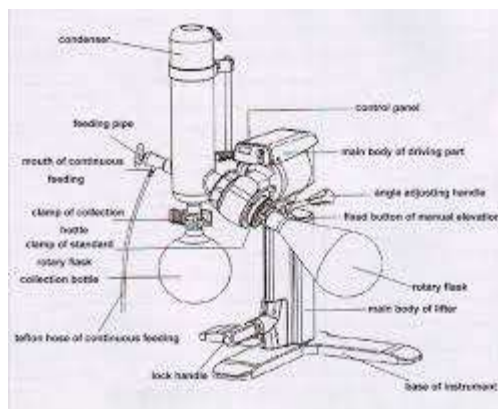
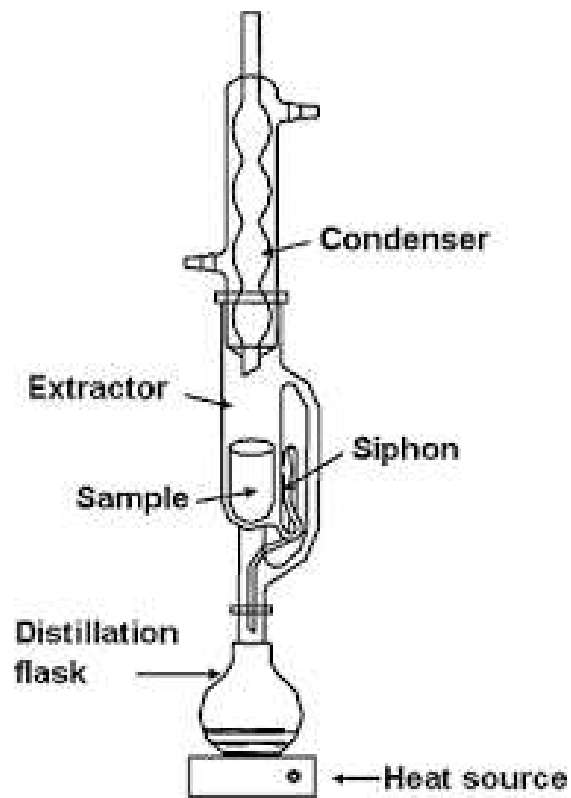
Seeds

**Extracted samples:**



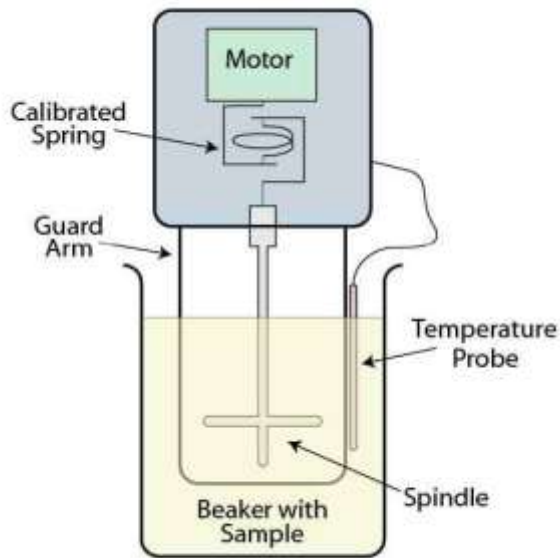
## Appendix (2) Apparatuses

### Soxhlet



Rotary Apparatus.

**Brook FIED :**



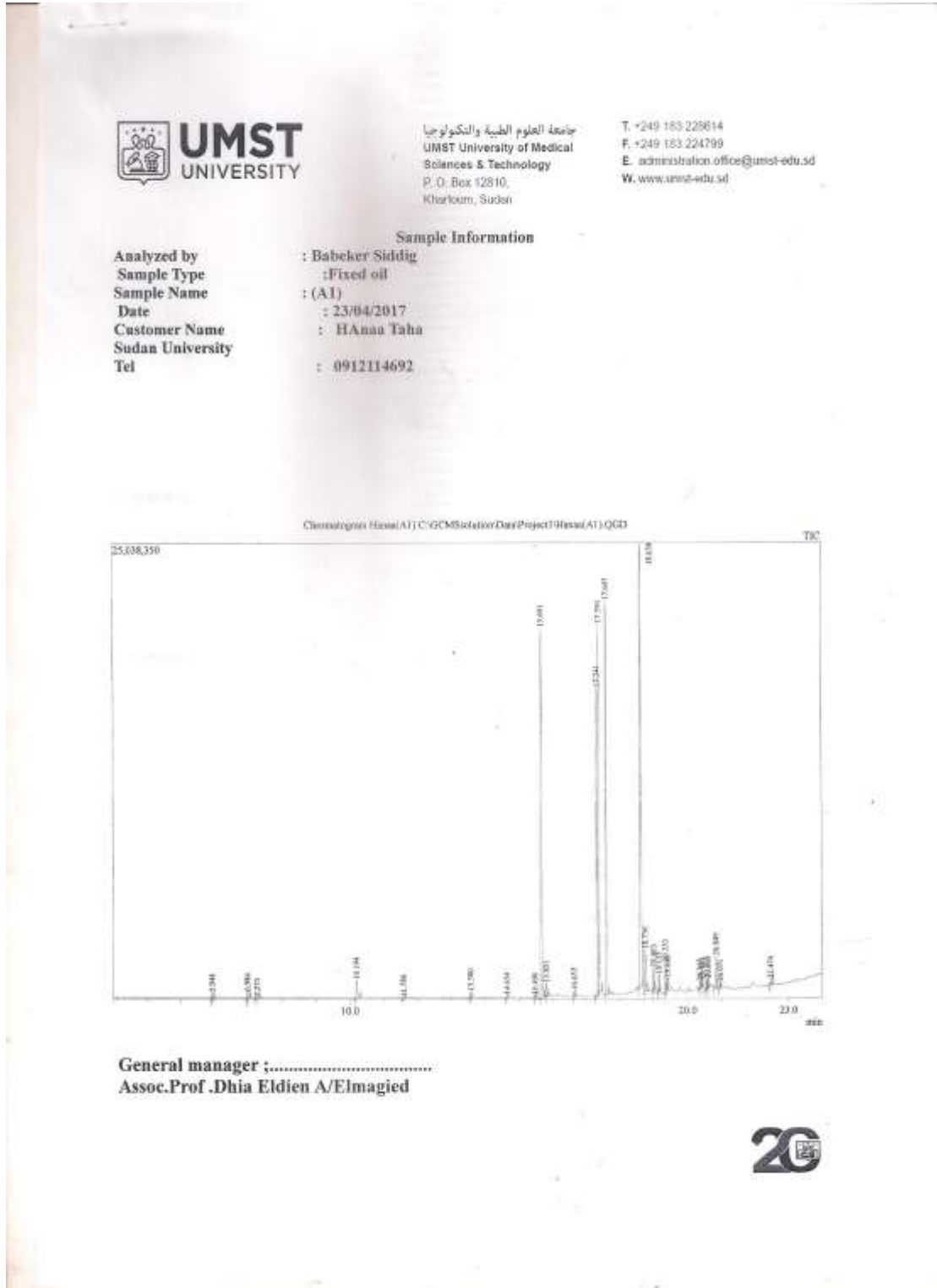
**Gas chromatographs:**



# Appendix ( 3)

An official annex came from competent )

peak Report TIC the plant Extract (A1)







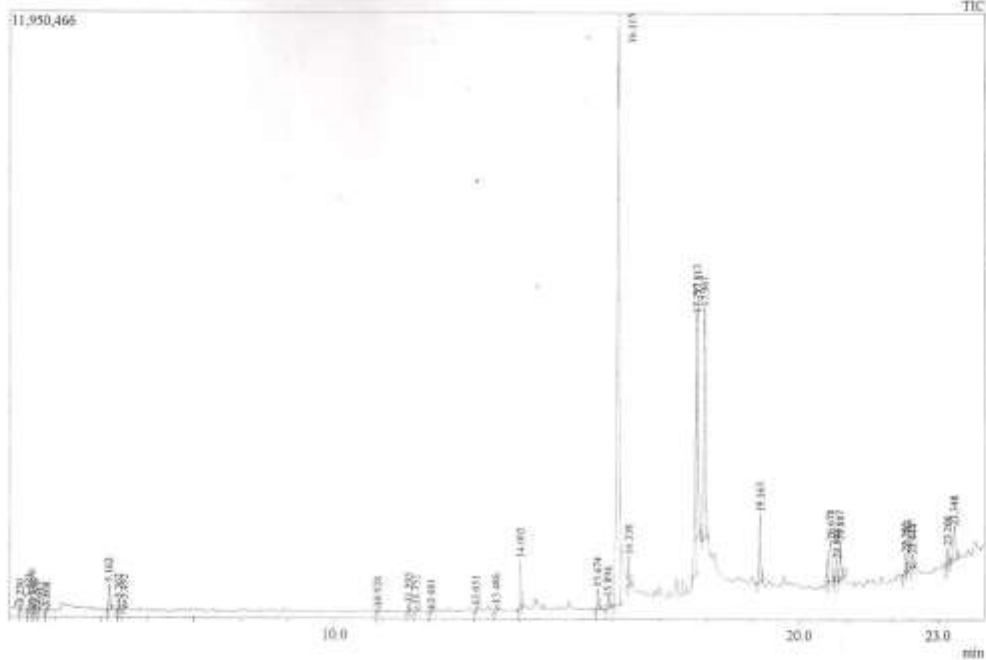
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W. www.umst-edu.sd

**Sample Information**

Analyzed by : Babeker Siddig  
Sample Type : Plant Extract  
Sample Name (B 2)  
Date 22/02/2017  
Customer Name Hanna Taha  
University of Sudan  
Tel 0912114692

Chromatogram B 2 C:\GCMSolution\Data\Project\B 2.QGD



General manager ;.....  
Assoc.Prof .Dhia Eldien A/Elmagied





Method

[Comment]

----- Analytical Line 1 -----

[AOC-201]

# of Rinses with Presolvent	:	2
# of Rinses with Solvent(post)	:	2
# of Rinses with Sample	:	2
Plunger Speed(Suction)	:	High
Viscosity Comp. Time	:	0.2 sec
Plunger Speed(Injection)	:	High
Syringe Insertion Speed	:	High
Injection Mode	:	Normal
Pumping Times	:	5
Inj. Port Dwell Time	:	0.3 sec
Terminal Air Gap	:	No
Plunger Washing Speed	:	High
Washing Volume	:	5ul
Syringe Suction Position	:	0.0 mm
Syringe Injection Position	:	0.0 mm
Use 3 Solvent Vial	:	1 vial

[GC-2010]

Column Oven Temp.	:	60.0 °C
Injection Temp.	:	300.00 °C
Injection Mode	:	Split
Flow Control Mode	:	Linear Velocity
Pressure	:	100.2 kPa
Total Flow	:	50.0 mL/min
Column Flow	:	1.61 mL/min
Linear Velocity	:	46.3 cm/sec
Purge Flow	:	3.0 mL/min
Split Ratio	:	1.0
High Pressure Injection	:	OFF
Carrier Gas Saver	:	OFF
Splitter Hold	:	OFF

Oven Temp. Program	Rate	Temperature(°C)	Hold Time(min)
-	-	60.0	0.00
-	10.00	300.0	0.00

< Ready Check Heat Unit >

Column Oven	:	Yes
SPLI	:	Yes
MS	:	Yes

< Ready Check Detector(FTD) >

< Ready Check Baseline Drift >

< Ready Check Injection Flow >

SPLI Carrier	:	Yes
SPLI Purge	:	Yes

< Ready Check APC Flow >

< Ready Check Detector APC Flow >

External Wait	:	No
Equilibrium Time	:	3.0 min

[GC Program]

[GCMS-QP2010 Ultra]

IonSourceTemp	:	200.00 °C
Interface Temp.	:	280.00 °C
Solvent Cut Time	:	2.50 min
Detector Gain Mode	:	Relative
Detector Gain	:	0.88 kV +0.00 kV
Threshold	:	0

[MS Table]

--Group 1 - Event 1--  
Start Time :3.00min  
End Time :24.00min  
ACQ Mode :Scan  
Event Time :0.30sec  
Scan Speed :2000  
Start m/z :40.00  
End m/z :550.00  
  
Sample Inlet Unit :GC  
  
[MS Program]  
Use MS Program :OFF



Date 21.2 2016

To whom it may concern

This to certify that the plant materials were taxonomically authenticated by Yahya Suliman Mohamed at the herbarium of Medicinal and Aromatic Plants & Traditional Medicine Research Institute (MAPTRI), National center for Research, Khartoum, Sudan, and voucher herbarium samples were deposited there for further future reference.


Botanical name: *Morinda zosaming* L.


Family: Capparidaceae

Local name: Alyeir

Name: Hanna Taha Hussin

Sudan University Of Science & Technology

  
Dr. Reem Hassan Ahmed  
Head department of Taxonomy &  
Phytochemistry

  
Prof. Yehia Ahmed Mohamed  
Director of Medicinal and Aromatic  
Plants Research & Tradition Medicine &  
Institute (MAPTRI)