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A New Separation, Fractionation and Improving of Abu- Aljeer Asphalt

Tariq Abdul-Jaleel * **Juma Waji Salem **** **Aws Mseer Najres****
***Department of Chemistry - Faculty of Science -University of Anbar**
**** Department of Chemical Engineering- Faculty of Engineering - University of Anbar**

ABSTRACT

The study of springs Abu-Aljeer natural asphalt at proven of al-anbar (Iraq), to find a mean of exploiting the asphalt springs as alternative energy resource, Included two stages The first: Separation the components of asphalt in two ways the first is column chromatography and second represented by extraction- chromatography. The results of separation were 10.20% Asphltene, 89.18% Maltene which separated lately to (Paraffinic 45.23%, Aromatic 28. 39% and Resin 21.66%). The second: Trying to improve the asphalt specifications by using natural materials that are available locally (limestone) with (5, 15, 25, 35%), which have given good results. Rheological properties have been tested, in such average that (virgin Softening was 43^0C, became 56^0C, Penetration was 110 dropped 39 and viscosity was 11355 turnedy other 47231cent-Stok, mm^2\Sec), and flash point (179^0 C became 200 ^{0}C), Fractionated parts of asphalt were characterized by (UV), (FT-IR) spectra. This study also clarified paraffin compounds which represent the main part material and the colloidal state is in Gel phase.

Key Words: Natural Asphalt, Extraction, Fractionation, Improving, Abu-Aljeer

INTRODUCTION

Natural asphalt is a black naturally-occurring material. Evaporation of volatile organic compounds and high contents of asphalting have made it hard and brittle that can be used as a hardener,**[1]** Natural asphalts consists of a mixture of high molecular weight hydrocarbons, and has a large number of carbon up to 150 atoms, with th strength of viscous fluid of special smell, easily soluble, fuses at 10 -71°C, frequently contain mixed mineral matter, and are usually composed in part of nitrogen-sulfur-oxygen heterocyclic compounds and trace metals. Characteristically,

Two form of natural asphalt. The solid kind which in the rocks and called the asphalt rocks. The other in the form of sticky materials, were flow out of the warm sulfur springs, .because of cracking in the earth's crust. The asphalt of Abu Algeer was from this kind. which are flow out from sulfur springs because for

Corresponding Author Email :
Tarik_jm@yahoo.com

Euphrates rift . [2]. Bad rheological properties of natural asphalt making it useless for paving and roofing. The principal components of topped natural asphalt can be determined by fractionation with organic solvents to **Asphaltene** and **Maltenes, [3]**.Solvents separation and fractionation with n-pentane provides the information regarding the details of the individual characterization and reactivity studies various methods are developed to characterize the compositional determination of fractions of asphalt. Chemically the structures of asphalt have two parts the first is Asphaltene which is insoluble in normal heptane, and the other maltenes which are soluble **[4]**. Asphaltene is composed of poly-heterogeneous aromatic rings and substituted by the nitrogen and oxygen and sulfur atoms of high molecular weight, but it is soluble in carbon disulfide CS_2, Maltenes part consists of (Saturated hydrocarbons, aromatics and the Resin) giving the molecules high adhesion, which causes attraction forces between the components of asphalt, **[5]**. Good asphalts have a proper balance of polar and non-polar molecules. The true molecular weight of the non-polar molecules is also important in asphalt performance, especially in low-temperature performance [6]. Asphalts that have too much polar material will be subject to fatigue cracking in thin pavements, brittleness, and thermal cracking. Asphalts that have too much non-polar material, will suffer from fatigue cracking in thick pavements, moisture sensitivity and rutting **[7].** Improving of Asphalts can be made by different methods, **physically** by blending with (polymers, rubbers, waste materials fibers, and mineral salts) and **chemically** by reaction of hydrocarbons chains of asphalts with chemical reagent by substitution reactions such as halogenation. The major types of asphalt products are paving and roofing asphalts**.** Asphalt is also used in asphalt-based paints as protective coatings to prevent corrosion water reservoirs, dams, and sea defence works. **[8]**.

The objective of this study was to investigate the virgin Abu- Aljeer asphalts, This paper has a two aims The **first** is extraction and **f**ractionation the chemical composition of asphalt by liquid chromatography methods and study the chemical properties. The solvent deasphaltening is performed using n-pentane solvent into SARA components (Saturates Aromatics, Resins and Asphaltenes). The SARA components are analyzed comprehensively using (FT-IR), UV-vis spectroscopy. The **second** is improving the rheological properties by blending with cheap material .to be used in paving, roofing and industrial. Their results were compared with artificially asphalts such as (Aldaura - Refinery) Iraq.

EXPERIMENTAL SECTION

Raw materials: Sticky asphalt from the Abu- Aljeer area (Ramaadi) .

Chemicals and reagents: Hydrocarbon solvent (C_5, C_6, C_7). Toluene, alcohol**,** carbon tetra chloride (CCL_4). Benzene, from Sigma-Aldrich.. NaOH and H_2SO_4 from Fluka, Co: AL_2O_3, SiO_2 from

Fluka,Co, $CaCO_3$, Attapulgus and Bauxite from Ruttba Geological camp.

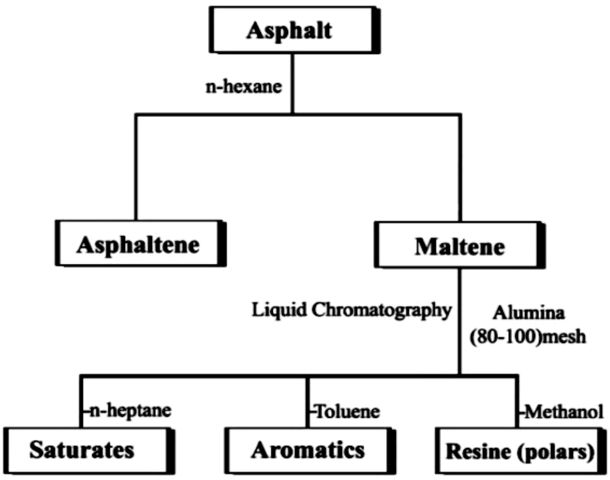
Instrumentation: FT-IR NICOLETIR -100- Spectrophotometer**.** Ultraviolet Spectrometer JENWAY(UK) -6405.Nuclar, Penetrometer Lzy-50/penetration (China).soft meter Ring & Ball Apparatus Lry-35A/softening (China). muffle furnace Carbolite /1200c° (England). Drying oven Sanyo (Japan).

Procedures: Several experiments were carried out in this project. And used standard laboratory equipment

A. Purification of asphalt: In a **500m**l round flask virgin asphalt **(10g**), **500m**l carbon tetra chloride, were shaked for 1.5h, then the mixture was refluxed to **3hrs**. The mixture was cooled at room temperature. And The precipitate of inorganic residual was filtered by ash less filter paper (No. **42**), then washed with sufficient amount of carbon tetra chloride, until the drops coming down which received from the filter paper had become colorless. Precipitate was dryied at 100^0 C for **24**hrs, and weighed to determine the percentage of pure asphalt and insoluble material.

B. Deasphaltening: The Separation of asphaltenes (insoluble) was carried out by solvent precipitate method used hydrocarbon solvent **n- hexane.**

C. Fractionation of Maltenes by Column Chromatography: The soluble part (maltenes) was dried and than using laboratory fractionation column, for fractionated maltens to components by The **SARA** method as shows **Scheme.1,** into groups (Saturated ,Aromatic, Resin and Asphaltine). **ASTM (D3279),** as in reference **[9].**



Scheme (1) Fractionation of asphalt by (SARA) methods

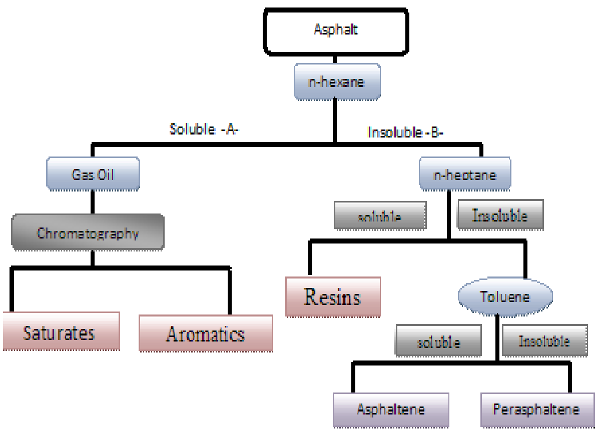
Fractionation by alumina (Al_2O_3): Fractionation of maltens by using alumina was carried out by the same procedure **as in [9]** of every fractionated part of asphalt.

Fractionation by silica gel (SiO_2): Fractionation of maltens by using silica gel was carried out by the same procedure as of the case for fractionation by using alumina. Except that of different % weights of the separated parts.

Fractionation by Binary layers (alumina + silica gel) : A 25g of activated alumina was mobilizes in length 100 cm, diameter **2cm** chromatography column. Then **25g** grams of activated silica gel was also mobilizes on the alumina layer. The fractionation of maltens in a column of binary layers was carried out according to the method of separation of the alumina**..**

Fractionation by extraction – chromatography: In a **250m**l beaker, **5g** of asphalt**,** dissolved in 100 ml n-hexane where for **30** minute. Afterward the mixture was left for **25** minute tesettle down then filtrated. The precipitated part was obtained from filter paper after drying and weighed accurately. Their symbolized **(B)** and solubility part their symbolizes (**A**).

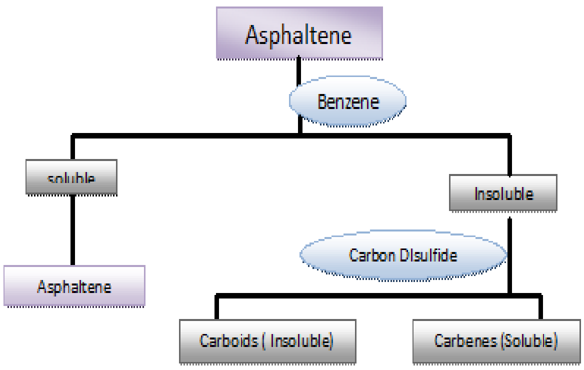
In **250ml** beaker, part **B**, 100ml n – heptane was added then shaking for **30** minute and used of the same previously procedure. Two parts were obtained , soluble their Resin and the other insoluble, which dissolved with toluene to get asphaltene as a soluble part, either insoluble part represents Carbenes and carboieds. Part **A** (soluble part) was distilled, dried and weighed accurately then was dissolved with a few ml of freshly distillated gas oil. Then transfered to fractionating column which packed with alumina **(C)** method, and using gasoil for fractionation of part **A** where carried out according to scheme.**2**.



Scheme 2. Flow chart of fractionation by binary system

Study of Asphaltene: The asphaltene due to has complexes structures, the separated asphalt depending on the solubility and mixing with hydrocarbon solvent such as pentane, hexane, heptane due to dissolving and precipitation. **Scheme.3**

In a **250ml** Erlenmeyer flask, Aspheltene **4g,** benzene 130ml where shaking at room temperature for **50** minutes. Afterward the mixture was left motionless for **15** minute, then filtrated through the use of ashless paper **No (42).** Soluble part were evaporated by (rotary evaporator) to obtained the pure Asphaltene, then drying and weighted. Insoluble part were separation by using carbon disulfide **75ml** according to the previously. Insoluble (precipitate part) Carboieds was drying and weighted. Soluble part were evaporated to obtained carbenes.



Scheme.3. Separation of Asphaltene

G. Improvement of asphalt properties: Improving of asphalt was carried out through a physical blended method. by used Calcium Carbonate (**$CaCO_3$)** which are available in large quantities in the investigated area. In a **500ml** steel beaker, **100g** of Calcium Carbonate was grinded, milled and dried at **9O^{0}C** for 3hrs. Then sieved at radial **125µm**. Then the powder was burned for calcination in a muffle furnace gradually from 300 to1100^{0}C for 3 hrs and CaO are obtained. And their kept in in a desiccator.

In a **100**ml steel beaker, **50g** Asphalt (water free) was deposited in the water bath of constant temperature at **(90^0C)**, **CaO 5%** wt was added and mixed by mechanical stirring for 1hr to ensure complete mixing followed up by an optical microscope to prepare thermal microscopic slides. Stirring is stopped when the homogeneous distribution of the sample is observed. This method was repeated with other samples **(5, 15, 25, and 35) %.** The same procedure was followed when **CaO** powders was added.

H. Measurement of rheological properties:

Penetration Point was measured by using a standard needle which penetrates form to a specified distance and time, according to **ASTM (D5-83),** before and after the improvement. **[10].**

Softening Point was measured according **ASTM (D36-72), [11],** and they represent the fall of the metal ball from a particular height to asphalt disk to certain distance **2.54 cm** under thermal and the weight effect of the ball. Softening Point was measured periodically at different times. And compared their results with that of al-duraa asphalts (petroleum asphalt).

Dynamic viscosity was measured of asphalt before and after the improvement using viscometer type (canon) at **135 C^0**according to the **ASTM (D2170-85).** And by use the following equation**:** **Cst=F × t** **[12].**

Where F is tube diameter and t is the time.

Thermal measurements: Flash and Fire point was measured by using Gluvlande of Open Cup method according to the **ASTM(D92-02a**). **[13].**

J. Coefficients of Asphaltene index I_A., Gaestel index I_G and **Solubility index I_S:**

Coefficient Asphaltene Index **(I_A):**

$$I_A = \frac{Asphaltenes + Resins}{Saturates + Aromatics}$$

Gaestl Index **(I_C): (**represent the resin form**)** was calculated by using the following relationship

$$I_C = \frac{Saturates + Asphaltenes}{Resins + Aromatics}$$

I_S Solubility Index**:** calculated by their eq: **[14].**

$$I_S = \frac{Resins}{Asphaltenes} + \frac{Aromatics}{Saturates}$$

K. Spectroscopy measurement: The spectral study FT-IR were obtained by using NICOLETIR - Spectrophotometer(Anbar- Uinv) , sample was prepared in a film form. Spectroscope (UV-visible) for the asphalt fractional was determining the adsorption group by using UV-visible (Anbar - Uinv).

RESULTS AND DISCUSSION

Fractionation of the natural asphalt of the Abu Aljeer – Anbar/ Iraq, it represents the first study to separate the chemical components of natural asphalt was carried out through SARA technique. Separation is a traditional chromatographic technique which have been extensively used for hydrocarbon groups determination, for instance, the SARA established the relationship between the composition and the stability behavior of the asphalt with the main constituents of the asphalt. The most popular method of chromatography column is the fractionation into different fractions were sequentially eluted by several solvents of different polarity**.**

Several experimental used to separate component the asphalt through SARA process.

purification of asphalt for the purpose of the study by extraction of insoluble materials such (sand and calcite), with carbon tetra chloride, they were of **(0,04% Wt)** and pure asphalt **(99.86% Wt.).**

The asphalt samples were fractionated into **asphaltenes** by value **(11.70 % Wt),** and **maltenes in value of (88.30% Wt)** using n-heptane.

The asphaltenes were precipitated through filtrate by value **(11.70% Wt),** while the soluble **maltenes** were obtained after distillation and drying in value of **(88.30% Wt).**

This separation are carried out by using liquid- solid chromatography and using activated neutral alumina and silica gel as stationary phase and several hydrocarbons as a mobiles phase **,** Identify of ends for separation parts by using of color fixation **[15]. Table1** shows the resin part of the lower percentage Wt **%.**

Table.1 Wt. % of fractionated part of maltens from SARA processes.

Solvent used	Expected of isolated materials	alumina	silica gel	Binary layers
		Wt %	**Wt %**	**W% t**
Heptane	Paraffinic	45.25	43.43	43.96
Toluene	Aromatic	28.39	29.22	31.13
Methanol	Resin	21.66	23.11	21.02
----------	Residue	4.72	4.24	3.90

Separation by using binary layers (alumina + silica gel): fractionation of asphalt has been used by bilayer of common stationary phase alumina and silica gel .

Separation by extraction – chromatography: After separation of asphalt for two parts by using hexane as solvent, one insoluble their asphaltenes, other soluble was evaporated and transfer to alumina column. Chromatography then separated in two parts their compounds which has high paraffin and aromatic component. Afterward evaporated and weighed accurately to determinate **Wt%** ratios of insoluble part was separated for two part one are resin (compounds has a high polarity) and other asphaltenes, then treated with toluene as solvent also was separated for two part. One asphaltens is soluble, other insoluble called carbenes and carboieds. **Table.2.** show the result. Several notes can be observed: **Firstly** the lowest in **% wt ratios** of asphaltenes.

Table.2 Wt% of fractionated part by extraction – chromatography methods

Solvent Used	Expected isolated materials	Wt%
Gas Oil	Paraffinic	42.12
Gas Oil	Aromatic	34.9
n-heptane	Resin	10.34
n-heptane	Asphaltene	9.68
Toluene	Perasphaltene	0.0
-----------	Residue	2.78

Secondly increase in **% Wt.** ratios of aromatic and decrease in **%** Wt. ratios of Resin. This is due to the asphaltenes can working to cover their outer surface with resin particles to produce micelles. This can form bonds with aromatic compounds and lead to increase the %Wt. ratios of aromatic part. **[15]**

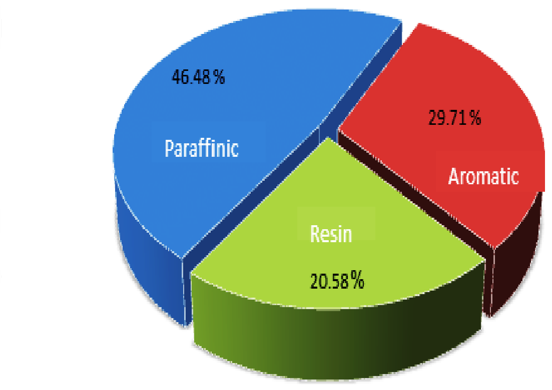


Figure.1. Average of Wt. % fractionated parts of maltenes in different columns

Form Fig .1. The weighted ratios of the asphalt Abu- aljeer area are consists of paraffin in mainly and **Fig.2.** Indicate the Wt % of fractionations part in different column and the residual.

Asphaltene Study: The purpose of this method to disintegration of conglomerate understandings their structure. This study was out carried by using solubility and precipitate methods to search for carbenes and carboieds which insoluble in organic solvents, but carbenes are dissolves in **CS_2**. The Abu- Aljeer asphalt of does not contain carbenes and carboieds in their structure,

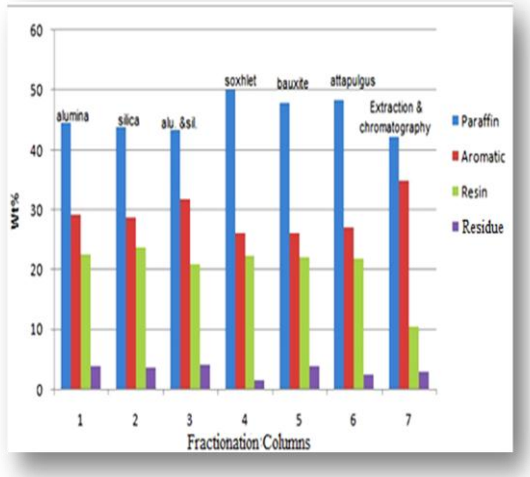


Figure. 2. Wt % of fractionated part for asphalt in different columns

Rheological Study: addition of Calcinated Calcium Carbonate **(CaO)** to the asphalt in **(5, 15,25, 35) %.** gives an improvement to the property of Softening and Penetration. generally has been observed that Softening was directly proportional with added of **(CaO)**. Also in such a way that penetration decreases with the increase in the proportion of the addition. **(CaO). Table 8. Fig3. Fig 4.**. Which can be attributed to the fact that the molecules of **(CaO** has the ability to form new bonds and links with polar groups in the natural asphalt which increase the overlap and the formation of bonds to form bridges between the association of hydrocarbon chains of high molecular weight. Also, the addition of **(CaO)** powders increases viscosity properties because calcium oxide **CaO** which is highly effective and has a high. capacity to form bonds of adsorption Chemo – Physics making it able to configure the bridge link between the hydrocarbon chains in asphalt [8] **Table.3. Fig.5.**

Table.3. Values of rheological properties

NO	% CaO added	Softening C^0	Penetration ,mm,100 mg,25 C^0 ,5sec	Viscosity mm ^{2}/Sec,135 C^0 ,centi-Stoke
1	0	43	110	11355
2	5	45	87	29276
3	15	48	63	38213
4	25	52	56	42821
5	35	56	38-39	47231
Duraa	-----------	49- 58	40-50	-----------

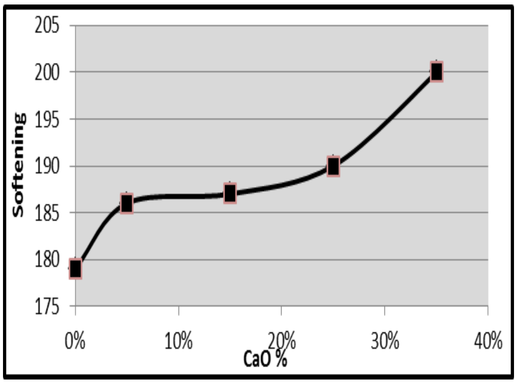


Figure. 3. Relationship between softening and CaO%

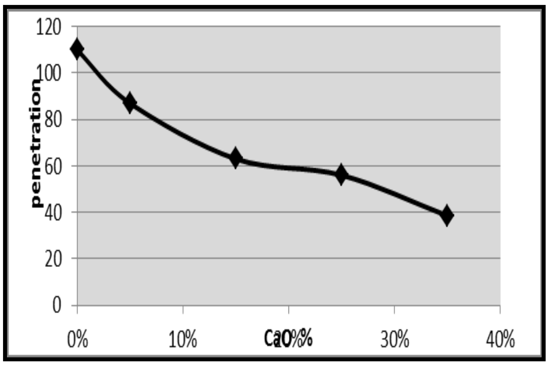


Figure. 4. Relationship between penetration and CaO%

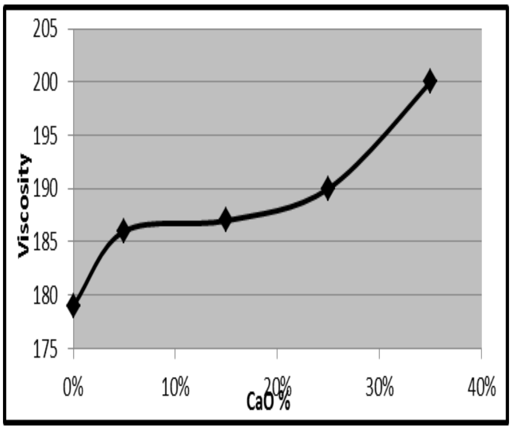


Figure. 5. Relationship between viscosity and CaO%

Improvement of thermal properties: Flash and fire point are increase when calcium oxide CaO added and the optimum value was %35 by weight which lead to increase in degree of the links in the asphalt molecules and also work to receipt dissipate in the heat in the asphaltic body.**Table.4. , Fig 6.**

Table.4 Values of flash and fire point of asphalt

NO	% CaO added	Flash Points	Fire Points c^o
1	0	179	186
2	5	186	192
3	15	187	200
4	25	190	204
5	35	200	216
Duraa	---	240	-------

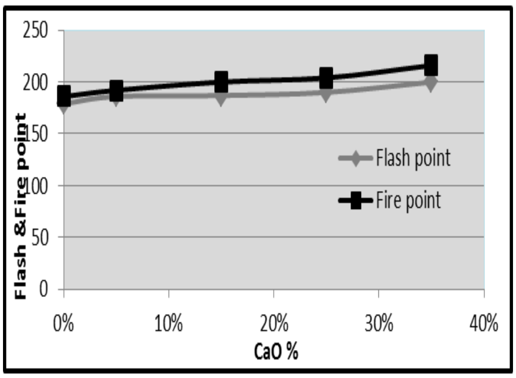


Figure. 6. Relationship between flash and fire point and CaO%

Colloidal system of Asphalts: The study of colloidal stability was carried out by using four columns in the same solvent to calculate three factors**:** $\mathbf{I_A}$ **Index** This is the change rate of Asphalten contant. $\mathbf{I_G}$ the **Gaestel Index** which reflects dispersing capability of Maltens on Asphaltenes**.**

$\mathbf{I_S}$ **Index** this is the Susceptibility of **Solubility** Asphalt. **Table.5.** Show the **result** of calculated colloidal system. The calculated values of $\mathbf{I_A}$ **Index** and $\mathbf{I_G}$ **Index**, in silica gel and alumina column are convergent with the calculated values by **Attapulgite** and **Bauxite** column. And the $\mathbf{I_G}$ values are more clears than values of $\mathbf{I_A}$ these values are similar to the results of the researcher Oyekunle from lakooz – University / Nigeria **[17].**

And the values of $\mathbf{I_S}$ **index** was **(2.5185)** within the following ranges:

Is < 4 asphalt is gel
4<Is>9 asphalt is sol-gel If
Is > 9 asphalt is sol

The resulting value of Abu - Aljeer Asphalt lies within the range of the colloidal gel.

Table.5. Values of I_A Index and I_G Index of colloidal system in differential column

I_C	I_A	Resin	Aromatic	Paraffin	Asphalten	Columns
1.0936	**0.4860**	**1.0065 gm**	**1.3004 gm**	**1.9827 gm**	**0.5403 gm**	**Alumina**
1.2482	**0.4610**	**0.9833 gm**	**1.1639 gm**	**2.1400 gm**	**0.5403 gm**	**Bauxite**
1.0666	**0.4931**	**1.0573 gm**	**1.2835 gm**	**1.9564 gm**	**0.5403 gm**	**Silica gel**
1.2356	**0.4498**	**0.9766 gm**	**1.2103 gm**	**2.1620 gm**	**0.5403 gm**	**Attapulgi**

FT-IR and U.V study: U.V Spectral shows the normal strong package for natural asphalt at 250nm This transition is due to the excitation of type (**π → π*) for (- C = C)**in aromatic ring and weak package of type **(n →** π*) at 340nm,for(-N=N-) , (C=O).**Fig.7.** When asphalt improving by **CaO,** Spectral show**:**

Spectral of **U.V** show two package, First at **295nm** to a (- **C** = **C**) for aromatic of poly rings, second peak at (400 nm) are due to calcium oxide where the addition of the fillers led to the appearance of transitions similar to charge transfer. This indicates a change in the structure of natural asphalt.**Fig.8.**

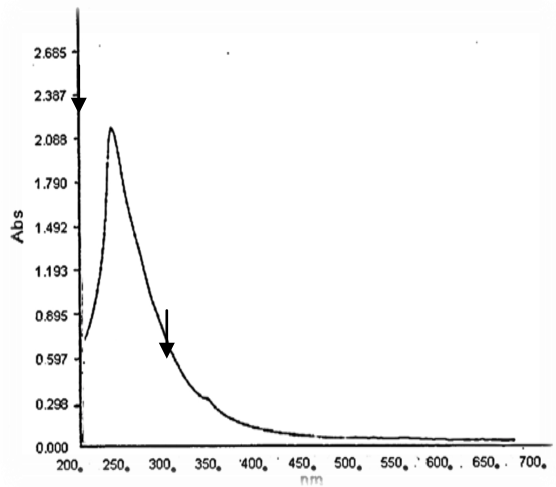


Figure 7. U.V Spectra for virgin asphalt**.**

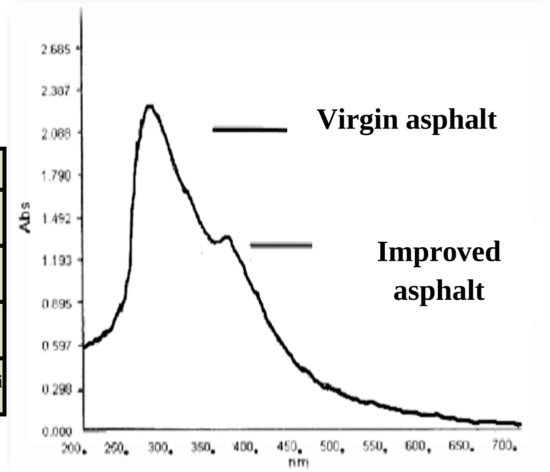


Figure. 8. U.V Spectra for improving asphalt.

FT-IR spectra of virgin asphalt show the evidence of stretching absorption of peak in **3443 cm^{-1}** which corresponds to O – H, ofr phenol and alcoholic. And peak at **2854-2925** cm^{-1} which belongs to methyl group for saturated C – H, peak at **1639cm^{-1}** due to Rocking C=C in plane of aromatic ring besides two sharp peak at 1459cm^{-1} and 1376cm^{-1} because of C – H bending for saturated CH_3- , --CH_2 , 1252cm^{-1} to C = O for carboxylic and esters , **722 cm^{-1}** a peak outer of plane (OOP) for **C – H** aromatic

FT-IR spectra of improved asphalt by added CaO with % 35.The observed change of characteristic peak pattern of new asphalt which was similar that with same position of virgin but different with as intensity. Compare of the absorption peaks change with the reference of virgin asphalt due to formation H – bond after added CaO. Peak at **854cm^{-1}** to $CO^{-}_{3,}$ $_{485}cm^{-1}$ which corresponds to trace element (V, Fe, Ni, Si, Ca). **Table 6. Fig 9. [18]**

Table.6. Values of adsorption peaks for FT-IR Spectral to improving Asphalt

NO	Group	Asphalt	Asphalt+add	NO	Group	Asphalt	Asphalt+add
1	$O\text{-}H_{st}$	3443	3443	7	$C\text{-}O_{st}$*	1252	1252
2	$C\text{-}H_{st}$	2925-2854	2927-2854	8	$=C\text{-}H_{oop}$	722	722
3	$C=O_{st}$	------	1743	9	CO_3^-	------	854
4	$C=C_{ro}$	1643	1643	10	X	------	485
5	$C\text{-}H_{be}$	1459	1459	11			
6	$C\text{-}O_{st,m}$	1376	1376	12			

Carboxylic acids , esters , ether // *alcohols, phenols //oop = out of plane //st = Stretching ro=rock , x =Trace Element

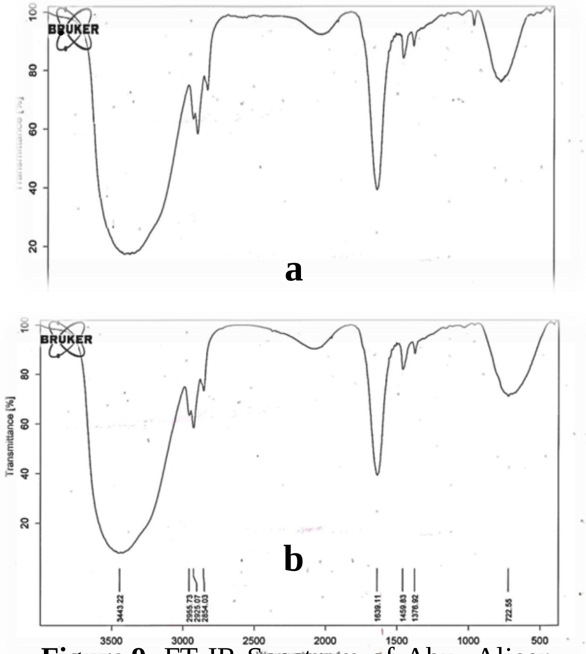


Figure.9. FT-IR Spectrum of Abu- Aljeer before (b) and after (a) improving

CONCLUSIONS

Our investigation of Abu- Aljeer natural asphalt is a novel study carried out by using this several processes such solvent extraction, column chromatography, soxhlet method, rheological, thermal studies and spectral. Where explained: Small amounts of insoluble materials, such as limestone, sand and minerals were founded. Also Asphaltens part percentage is lower than maltens part **1: 10**. **SARA** process for fractionation of maltene revealed that they contained more aliphatic compounds than resin, aromatic or asphaltene. The rate of acids and organic bases, has been also lowered the weight ratios obtained of the separated parts of the asphalt (Abu-Aljeer) as similar the weighted ratios obtained from materials of asphalt oils of Kirkuk and Qayyarah Mosul. The asphalt of Abu Aljeer - Anbar cannot

be used in paving and roofing, while improved asphalt were used in paving, roofing and industrial purposes.
But after treatment, the specifications of the improved asphalt were similar to that of Al- Dauraa refinery. Natural asphalt of Abu- Aljeer successfully separated and characterization its fractions were the first results obtained and show that the Abu Aljeer asphalt is rich at paraffinic compounds,

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