# Viscosity and Ductility improvement of natural Asphalt in (Heet - Anbar) Areas Using Industrial Waste for the Purpose of Recycling.

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### Abstract:

The addition of Phosphogypsum and Cement Kiln Dust granul -sized 100  $\mu$ mboth individually for the weighed (10, 20, 30, 40) wt% to (Heet) natural asphalt (Eyeen Aljeebal) gives an improvement and development in the properties of viscosity and ductility the ideal percentage of this additive was 40%.

The chlorination of Asphalt samples with the addition of Phosphogypsum and cement Kiln Dust with the previous proportions at different times (0.5, 1, 1.52) hours gives better improvement and development in the properties of viscosity and ductility. The favorite time of chlorination is 1.5 hour with 40% addition of the materials combinations. The study shows that the time of chlorination, percentage of addition and mixing temperature had a significant impacton the processes of developing and improving the rheological properties of natural asphalt.

Keywords: natural asphalt, viscosity, ductility, Recycling

# 1 – Introduction

Natural asphalt in(Heet) area is of two types; thefirst issolid and it comes in the form of deposits of rock. It is mixed with A calcite and dolomite rock and is named as asphaltic rocks . The percentage of asphalt in casolid), which flows from ground water with sulfur springs because offractures in the crust of the earth's surface resulting from the crack <sup>(2)</sup> at(Heet – abu aljeer) and extends to Alsumawa region in the south of Iraq . Heet asphalt does not fit for industrial uses because of its poor rheological properties<sup>(3)</sup>. It is black and suffers from oxidation operations andnatural evaporation, leading to an increase in the fraction of asphaltenes; the darkest and stiffest component. The asphalt consists of two main components; asphaltens and maltenes which can be split into (saturated hydrocarbons, aromatics, and resins), in addition to the presence of metal elements such as iron and vanadium  $^{(4),(5)}$ .

In recent studies, the physical and chemical processes are used for the development of natural asphalt to enable it to meet the requirements of industrial use, such as paving, roofing, to prevent moisture and as electrical insulator. Among these operations are aerobic ,anaerobic oxidation and chlorination and the use of the links to increase the adhesive property, such as polymers ,sulfur besides phosphoric and .metal salts. <sup>(6). (7)</sup>.

The chemical composition of asphalt is complex because it contains long hydrocarbons chains and contains condensed aromatic rings and polarized groups. This make asphalt of wide range of properties between the solid and liquid. States being a sticky and flexible substance, this encouraged researchers a long time ago to improve the specifications of the asphalt. In 1920 Abraham<sup>(8)</sup> carried out a study to improve the specifications of asphalt in oxidation method by thermal aging whichchanges the asphalt into a more solid, black, shiny with better rheological properties. The improving process requires that the asphalt accepts the added material and mixes largely with it to the events of the greatest bonding due to reactions of the type chemo-hysics <sup>(9)</sup> Viscosity and ductility properties are affected by the chemical composition of the asphalt. If the asphalt contains a high percentage of aromatics and resins, it is of the type of Sol- type, and entertains high flexibility. When it contains a high proportion of aspheltenes, it becomes of Gel-type, which is less flexible and more polarized and contains voids: because of micelle<sup>(10)</sup>.

The thermal properties of asphalt  $T_g$  are affected when improvement processes are conducted, the transition to the degree of  $T_g$  occurs to the top of the (-20 to + 80) due to increased Linking polarization and the formation of new ionic bonding that make asphalt more viscous and less ductile and resistant to longer operating conditions <sup>(11)</sup>.

The present study aims to improve some of the rheological properties of the viscose type asphalt of Heet area (**Ayeen Ajeebal**), to increase viscosity and ductility in low temperatures and roofing by using harmless and recycled industrial wastes into useful materials and reduce the harm they bring to the environment.

## 2 – Experimental

#### A. Raw materials

Asphalt1c samples from (Heet - Anbar ) region from five main springs. Ayeen Aljeebal spring was the best in rheological properties, Phosphogypsum waste from phosphoric acid plant -Akashat, cement kiln dust from Kubaisa Cement Plant, Fe<sub>3</sub>O<sub>4</sub>of iron filings, and chlorine gas prepared in laboratory by concentrated potassium hvdrochloric acid reaction with permanganate (Fluka). Table (1) shows the specifications of asphalt Eyeen aljebal such as (specific gravity, penetration, softening ... etc.), table (2) specifications of Phosphogypsum and cement kiln dust.

#### **B.** Sampling

50 g of natural asphalt - water free (heating asphalt for three hours at a temperature of 150  $c^0$  with stirring) was put in the metal beaker 250 ml. Dry Phosphogypsum (heating for two hours at a temperature of  $100 \text{ c}^0$ ) of a weight 10% was added in the water bath with a constant temperature of 90 c<sup>0</sup> with mechanical stirring for 1.5hr to ensure complete mixing followed up by an optical microscope by preparing thermal microscopic slides. Stirring is stopped when the homogeneous distribution of the sample is observed . This method was repeated with other sampls ( 20,30, 40 .). The same procedure was followed when cement kiln dust was added.

#### **C.** Chlorination of Samples

The asphaltic samples for the purpose of chlorination were prepared as in chlorine gas was added directly (prepared in laboratory) to the asphaltic sample in the presence of a catalyst of iron oxide(  $Fe_2O_3 \ 1\%$ ) with continuous mechanical stirring to ensure exposure of all particles to the reaction of replacement between chlorine and hydrogen. Chlorination processes were carried out at ( 0.5 , 1 , 1.5 ,2) hr period sand after each period of chlorination, the required measuremen

ts for this study were conducted to determine the impact of mixing and chlorination. The process of improvement

#### **D.** Viscosity and ductility test

Viscosity tests were conducted according to the American standard ASTM D 2170 - 86 in oil bath of at constant temperature  $135 c^{0}$ .<sup>(14)</sup> and using viscosity Tube of the canon type.

Ductility tests were carried out according to standard ASTM D113 - 85 at the temperature of 24  $c^0$  and strongly pull at a speed of 5cm / min.<sup>(15)</sup>

#### E. Spectral study

FT-IR spectroscopy device of the type Shimadza Japan was used to locate absorption packages and the amount of creep resulting because of improvement on the asphalt. The samples were in the form of a Film

The highest coefficient of absorbance and high wave length spectroscopy were appointed by using **U.V**visible device of the type **U.V** –visible Cary 100 con Varin Australian

#### **3** - Results and discussion

Asphalt (Eyeen Al-jeebal- Heet) is characterized with specifications which are better in comparison with the other springs in the study area. The asphalt in that spring flows with water in the form of semi-solid viscose, and after the expulsion of water from the asphalt it becomes more solid, black in color and with a mirror form .

addition of Phosphogypsum to the asphalt gives an improvement to the property of viscosity .in such a way that Viscosity increases with the increase in the proportion of the addition. Table( 4) and figure( 1) show the values of resulting viscosity which can be attributed to the factthat the molecules of  $P_2O_5$  in Phosphogypsum 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 cement kiln dust increases viscosity properties because it contains calcium oxide

CaO which is highly effective and has a high capacity to form bonds of adsorption Chemo – Physics type, making it able to configure the bridge link between the hydrocarbon chains in asphalttable (5) and figure (2) show the impact of cement kiln dust addition on viscosity.

The preferred percentage of Phosphogypsum and cement kiln dust addition was  $40\%^{(16),(17)}$ .

When the chlorination of samples was conducted with the addition of Phosphogypsum and cement kilndust in Previous improvement operations and at different times (0.5, 1, 1, 5, 2) hr there was a good improvement in the property of viscosity with increasing time of chlorination table (6) and for the replacement of hydrogen with chlorine to ensure that the reaction with the largest number of particles is done under the ongoing movement to ensure reactions are done under chlorine. Chlorination time was1.5 hours with the addition of 40%; tables (7) ,(8), and figures(3),and(4).

The addition of Phosphogypsum and cement kiln dust to asphalt chlorinated as in the above ratios gives an improvement in ductility values(reduction in stretch). The reason is the hydrogen bonding between Phosphogypsum, cement kiln dust, and asphalt because these materials contain ionic acidity and basic radicals. Also cement kiln dust contains themetal elements that have the ability to form new coordination bonds that increase the strength of linkages with asphalt. figures(5) and (6).

Chlorination also increases the polarization in the asphalt because of the strong inductive effect of chlorine which increases the ionic strength linking bonding by radicals from Phosphogypsum and radicals in the cement kiln dust that further increase the improvement in ductility properties. Figure (7) and figure (8). The small graule- size of the added particles from industrial waste leads to a reduction in the free size and potential voids in building asphalt or what is so called molecular packing asphalt to be another factor to improve the properties of asphalt<sup>(18)</sup>. Also FT-IR spectra after improvement showed a changein absorption packaged compared with the reference asphalt forms (9) and (10). The revealed evidence of stretch absorption of packets in (2920) cm<sup>-1</sup>and of packet (2925) cm<sup>-1</sup> to represent saturated hydrocarbon. Moreover They reveal an absorption of the c = c in cm(1630) cm<sup>-1</sup>. The package absorption of the (c = 0) shows a package to absorb aromatic substitutes and other packages group to absorb methylene CH<sub>2</sub> - CH<sub>2</sub>. Generally, FT-IR spectra show a physically change and another chemical one.

UV spectra in the natural asphalt in Heatgave one package limited to a range between (280-260) nmwhen the absorption was 2.53. This transition is due to the excitation of the type .

 $(n^{\star} \rightarrow \pi)^{\&} (n^{\star} \rightarrow \sigma)^{\text{or between the atoms of}}$ 

carbon and oxygen of the carbonyl group of chromoform returning to the absorbing group (C = O  $\,$ 

 $(CH3)_2$ ) which is absorbed in the(280nm, 190nm) or chromoform (CH<sub>2</sub> = CH-CH = CH-CHO), which is absorbed at 263nm. When Phosphogypsum and cement kiln dust 40% were added to the asphalt with chlorine, a double pack and second pack is signaled(370nm, 320nm, 280nm) in three adsorptions(5, 4.8, 2.5), 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. figure (11), and (12) **4** – **Conclusions** 

Asphalt of (Ayeen Al-jaabel), Heet region, responded to the viscosity and ductility improvement operations by adding Phosphogypsum and cement kiln dust by 40%. Which The chlorination of asphalt with the addition of a waste in the above process improves of ductility and viscosity, and is commensurate with the percentage of chlorine replaced with hydrogen.

The used of waste in this study gives a good picture to reduce environmental pollution. and the current study was able to recycle harmful waste materials to useful ones.

The improvement process of the ductility and viscosity was effected by chlorination time, and percentage of waste addition and mixing temperatures.

Asphalt	SP.G	Referen	Pent.	Soft.	FlashPo	Visco.	Ductl.	Igin.	PH
		ces			int			Point	
Ayeen jeebal	1.089	12	122	44	183	1010cst	83	195	6.4

SP.G = Specific gravity, pent.= Penetration, soft= Softening, Visco.= Viscosity, Ductl.= Ductility,

Filler	Sp.G	Referenc	PH	$P_2O_5$	CaSO <sub>4</sub>	MgO	SiO <sub>2</sub>	CaO	Fe <sub>2</sub> O <sub>3</sub>	$Al_2O_3$
		es								
Phosphogypsu m	2.58	13	5.8	1.90	94%	-	3.5	-	-	-
Kiln Dust	1.79	13	12.04	-	_	3.3	4.5	49%	2	5.2

#### Table (2) Specifications of Phosphogypsum and cement kiln dust

## Table (4) degree of viscosity and ductiletyfor asphalt non chlorination plus Phosphogepsum

NO	Additive %	degree of viscosity 135C <sup>0</sup> , CST	degree of Ductility ( 25 C <sup>0</sup> , 5 cm)
1	0	10101	83
2	10	27110	37
3	20	35232	30
4	30	40281	27
5	40	46501	27

#### Table (5) degree of viscosity and ductility for asphalt non chlorination plus Kiln Dust

NO	Additive % degree of viscosity 135C <sup>0</sup> , CST		degree of Ductility ( 25 C <sup>0</sup> , 5 cm)		
1	0	10101	83		
2	10	28937	72		
3	20	37120	54		
4	30	40881	47		
5	40	46521	28		

NO	Time Chlorination	degree of viscosity	degree of Ductility	
		135C <sup>0</sup> , CST	$(25 \text{ C}^0, 5 \text{ cm})$	
1	0	10101	83	
2	0.5 h	10663	80	
3	1 h	11772	93	
4	1.5 h	12249	112	
5	2 h	13924	112	

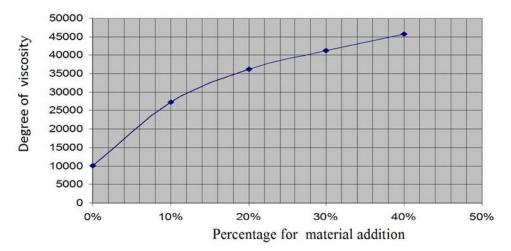
Table (6) degree of viscosity and ductility for asphalt chlorination

Table (7) degree of viscosity and ductility for asphalt chlorination 1.5h plus Phosphogepsum

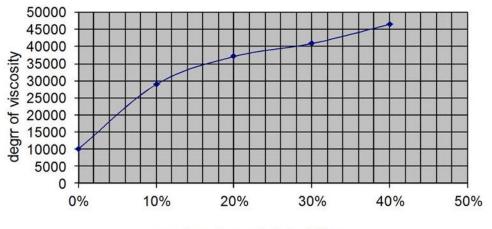
NO	Additive	degree of viscosity	degree of Ductility ( $25 \text{ C}^0$ , 5 cm)		
	%	135C, C <sup>0</sup> ST			
1	0	12249	112		
2	30	45461	36		
3	40	47121	25		

Table (8) degree of viscosity and ductilety for asphalt chlorination 1.5h plus Kiln Dust

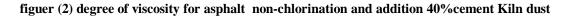
NO	Additive	degree of viscosity	degree of Ductility
	%	135C <sup>0</sup> , CST	$(25 \text{ C}^0, 5 \text{ cm})$
1	0	12249	112
2	30	42315	50
3	40	46567	35

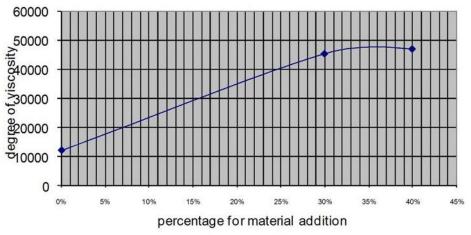


figuer (1) degree of viscosity for asphalt non-chlorination and addition 40%Phosphogypsum

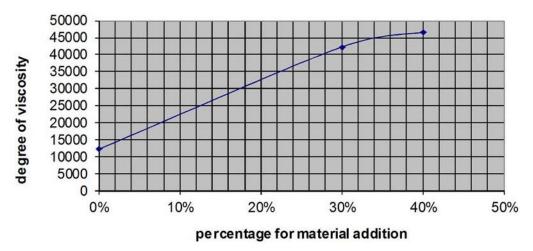


percetage for material additon

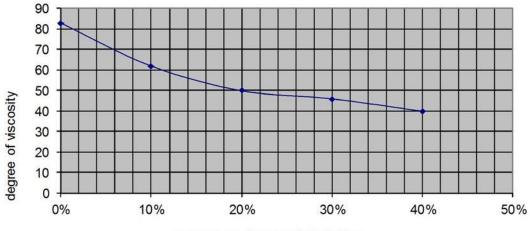




figuer (3) degree of viscosity for asphalt chlorination1.5 h and addition40% Phosphogypsum

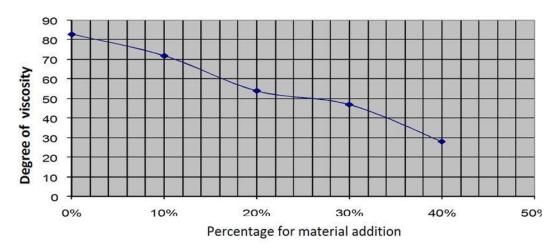


figuer (4) degree of viscosity for asphalt chlorination 1.5 h and addition40% cement Kiln dust

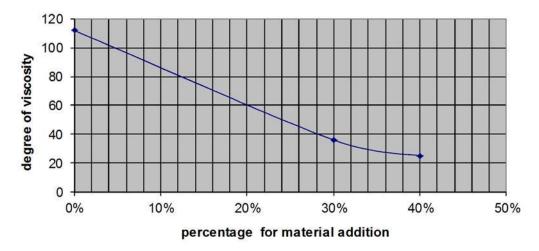


percentage for material addition

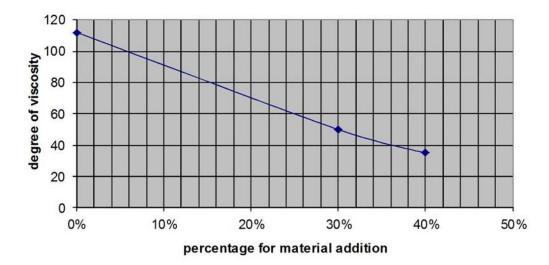
figuer (5) degree of ductility for asphalt non-chlorination and addition40%Phosphogypsum



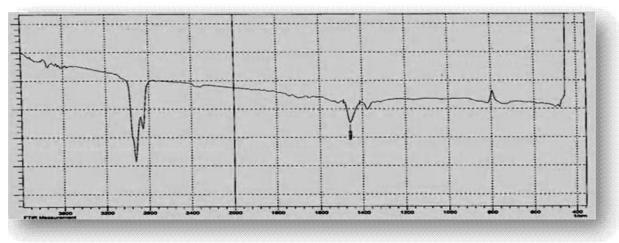
figuer (6) degree of ductility for asphalt non-chlorination and addition40% cement Kiln dust



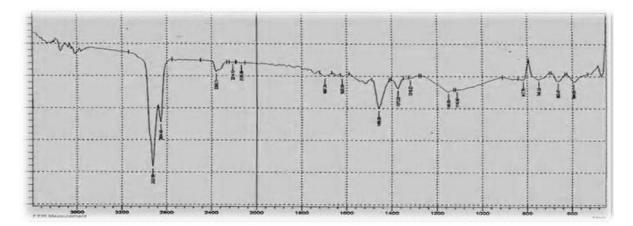
figuer (7) degree of ductility for asphalt chlorination1.5 h and addition 40% Phosphogypsum



figuer (8) degree of viscosity for asphalt chlorination1.5 h and addition40% cement Kiln dust



figuer (9) FT- IR Spectra for virgin asphalt



figuer (10) FT- IR Spectra for asphalt chlorination 1.5 h addition 40% Phosphogypsum

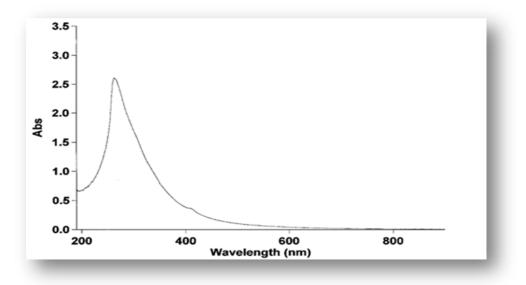


figure (11) U.V Spectra for virgin asphalt

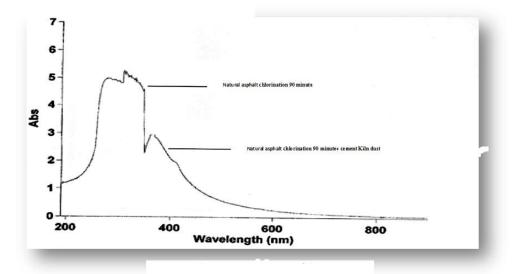


figure (12) U.V Spectra of natural asphalt chlorination 1.5 hr & addition 40% Cement Kiln dust

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# تطوير الخواص اللزوجية والمطية للإسفات الطبيعي منطقة (هيت – ألأنبار) باستخدام نفايات صناعية

# لغرض تدويرها

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الملخص

ان إضافة الفوسفوجبسم وغبار فرن الأسمنت بحجم حبيبي100µ كلا على انفراد وبالنسبة الوزنية ( 10 , 20 , 30 , 40 الى أسفلت هيت الطبيعي (عين الجبل) يعطى تحسنا وتطويرا في خاصيتي اللزوجة والمطية وكانت النسبة المثالية لهذه الإضافة 40% .

كما أن كلوزة نماذج الإسفلت المضاف اليها الفوسفوجبسم وغبار فرن بالنسب السابقة وبأزمان مختلفة ( نصف ساعة ، ساعة ، ساعة ونصف ، ساعتان) يعطى تحسنا وتطويرا إضافيا على خاصيتي اللزوجة والمطية وكان زمن الكلورة المفضلة ساعة ونصف وبنسبة إضافة 40 % من كلا المادتين. وأوضحت الدراسة أن زمن الكلورة ونسبة الأظافة ودرجة حرارة المزج لها تأثير مهم على عمليات تطوير وتحسين الخواص الريولوجية للإسفلت الطبيعي.

كلمات مفتاحيه : الإسفلت الطبيعي ، اللزوجة ، المطية ، تدوير ، نفايات صناعية