Egypt. J. Chem. **Vol. 64,** No. 12 pp. 6971 - 6977 (2021)

Egyptian Journal of Chemistry

http://ejchem.journals.ekb.eg/

Study the Rheological and Optical Properties Of Nickel Ferrite /PMMA Nano-Compsite Growth In Crude Oil Via Laser Ablation

Zahraa N. Salman[a]* ,Saif M. Alshrefi[b], Mohammed Hamza Al-Mamoori[c]

[a] *Medical Physics Department, Hilla University College, Babylon, Iraq.*

[b]*Laser Physics Department, College of Science for women, University of Babylon, Hilla, Iraq.*

[c] *College of Materials Engineering, University of Babylon, Hilla, Iraq.*

Abstract

A novel polymeric nanocomposite pour point depressant (PPD), based on Nickel Ferrite and (Polymethyl methacrylate – PMMA) polymer was synthesized and characterized. In this research the sample prepared in tow steps, the first step included prepared the sample to be ablated by using mixing kneading method formed as a Circular tablet contain Nickel Ferrite/PMMA. Then the sample ablated by using Nd:YAG laser with (1064 nm) wavelength,(600 mJ) energy,(6Hz) frequency and (100 pulses) for the tow sample which immersed in dilute Crude Oil (obtained from the Rumaila Oil Field in Southern Iraq). The optical properties of samples showed that the same behaviour but with blue shifting. The rheological properties showed that the viscosity increased as the polymer increased in samples uneven stability in the value of viscosity of the sample has low polymer ratio. The density almost doubled with same ablations' pulses.

Key Words: Crude Oil, Laser Ablation, PMMA Polymer, Absorption, Rheological Properties.

Introduction

PMMA A composite material or (known as a composition material) or (simply composite) is a material made up of two or more constituent materials with significantly different physical or chemical properties that, when combined, produce a material with characteristics that are distinct from the individual components within the finished construction, the constituent components stay separate and distinct.[1] Crude Oil composition variable element (Coal83-87%Carbon, 11-16% Hydrogen , 0-4% Oxygen plus Nitrogen and 0-4% Sulfur). The majority of crude oils have far more hydrogen than coal[2]. Most of them have five to about twenty carbon atoms compounds in petroleum contain. Many of them consist of straight chains of carbon atoms (surrounded by hydrogen atoms) [3]. Aromatic compounds are another type of chemical found in petroleum. They usually have a ring structure and are derivatives of the chemical benzene (C_6H_6). They do have a distinct aroma, but they usually have a negative impact on the environment. Low molecular weight compounds are volatile, evaporating quickly from gasoline at filling stations, for example. Many among them are carcinogenic[4].The ablation process is strongly dependent on the properties of the sample such as composition, thermal diffusivity and absorptivity [5]. Particles generated in the ablation process can have different compositions in terms of size, especially [6]. If there is no transfer efficiency, fractionation occurs because large particles (> 1 m) tend to be lost by gravity or deposited on chamber ablation. Small particles (less than 10 nm) are lost due to diffusion and electrostatic forces [7]. Recently, ferrites are widely studied for their versatile applications [8–10] however; mostly they are used in the magnetic or electric devices, where the high densities are prerequisite. They have also been reported sensitive towards different gases and humidity wherein; low density and nano sized nature is favoured[11]. Nickel Ferrite ($NiFe_2O_4$) has been widely studied as a magnetic material as (spinal) towards reducing gases [12]. Interestingly, nickel ferrite is reported to show sensitivity towards oxidizing gas like chlorine as well as reducing gases like hydrogen sulfide & acetone. Such different responses have been attributed to doping of Pd, Au, Pt, catalytic activities of transition metal ions such as manganese and cobalt[13].A ferrite is a type of ceramic compound composed of iron oxide (Fe_2O_3)

*Corresponding author e-mail: zahraaalhusainy@hilla-unc.edu.iq .; saif_master80@yahoo.com.

Receive Date: 03 June 2021, Revise Date: 22 June 2021, Accept Date: 24 June 2021

DOI: 10.21608/EJCHEM.2021.78910.3857

combined chemically with one or more additional metallic elements. The spinal ferrite structure $MeFe_2O_4$, where Me refers to the metal, can be described as a cubic close-packed arrangement of oxygen atoms, with Me^{2+} and Fe^{3+} at two different crystallographic sites. These sites have tetrahedral and octahedral oxygen coordination (termed as A and-sites, respectively), so the resulting local symmetries of both sites are different. The spinal structure contains two cation sites for metal cation occupancy. There are 8 A-sites in which the metal cations are tetrahedral coordinated with oxygen, and 16 B-sites which possess octahedral coordination. When the A-sites are occupied by Me^{2+} cations and the B-sites are occupied by Fe^{3+} cations, the ferrite is called a normal spinel. If the A-sites are completely occupied by Fe^{3+} cations and the B-sites are randomly occupied by Me^{2+} and Fe^{3+}cations, the structure is referred to as an inverse spinal. In most spinals, the cation distribution possesses an intermediate degree of inversion where both sites contain a fraction of theMe^{2+} and Fe^{3+} cations. Magnetically, spinal ferrites display ferromagnetic ordering. The magnetic moments of cations in the A and B-sites are aligned parallel with respect to one another. Between the A and B-sites the arrangement is anti parallel and as there are twice as many B-sites as A-sites, there is anti moment of spins yielding ferromagnetic ordering for the crystal. The choice of metal cation and the distribution of ions between the A and B-sites therefore, offer a tuneable magnetic system. Spinal ferrite material with $MeFe_2O_4$ type (Me: Fe,Co, and Ni) is group of magnetic materials that have excellent electromagnetic properties so much applied in information storage technology, diagnosis medical, heating and cooling using a magnetic material, anode on the battery, and catalysis[13,14].The utilization of chemical inhibitors such as pour point dispersant (PPD) enhances the flow ability of crude oil at uneven low temperatures. A chemical inhibitor that could be efficient for all the crude oil samples has never been found [14]. A new generation of the PPDs that is based on nanotechnology, including nanocomposites, attracts much attention in recent years. The pour point of an oil is the lowest temperature at which an oil is observed to flow by gravity in a specified lab test. Specifically, the pour point is 3 °C above the temperature at which the oil shows no movement when a lab sample container is held horizontally for 5 seconds. The pour point is an indication of an oil's cold-temperature properties. However, it may not relate to the lowest temperature that the oil will flow to the bearings in a piece of equipment, the lowest operating temperature for a hydraulic oil or the lowest temperature at which gears receive adequate lubrication. Never select a lubricant product based on its pour point alone. The cloud point is approximately the low temperature at which the oil turns cloudy due to the solidification of wax crystals within the oil[15]. Nano-silica, PMMA plus Nickel ferrite ($NiFe_2O_4$) have been very populated recently. Mohammad Ali Kazemi et al. have investigated the performance of polymethyl methacrylate/clay nanocomposite as novel pour point depressant on rheological properties of model waxy crude oil sample. They concluded that the rheological rotational and oscillatory shear tests results revealed that both PMMA and PMMA/clay nanocomposite reduced the yield stress and the gelation point of the waxy oil samples. However, the PMMA/clay nanocomposite effect was significantly higher than its rival (PMMA) [16].

In this study, Nickel Ferrite/PMMA nanocomposites were synthesized by Hexane solvent mixing. In addition, the influences of the neat PMMA/clay Nan composite on the rheological properties of the crude oil density and viscosity were investigated using standard rheological tests.

1. Experiment &Method

The samples have been prepared into three steps, as following:1\The first step: The formation of $NiFe_2O_4$ ferrite by the solid-state method relies on the following reaction:

$$\mathbf{NiO + Fe_2O_3 \rightarrow NiFe_2O_4}$$

Stoichiometric amounts of nickel oxide (NiO) and hematite (Fe_2O_3) were used as starting reactants. Nickel oxide (99% purity) was purchased from Fisher Scientific and hematite (96% purity) from Sigma-Aldrich. In order to produce a homogeneous mixture for a reaction sample, a mass of (3.2 gm) of nickel oxide and (7.1 gm) of hematite were mixed and thoroughly ground with a pestle in a mortar with the addition of a few drops of ethanol. This grinding was continued until the ethanol had evaporated and the process was repeated four times[17]. The powder was put in Teflon cup (150 ml)sealed then transferred to a Parr reactor (Autoclave-Homemade which modify Series 5500 High Pressure Compact Parr Reactor Model)to a chives the Hydrothermal Methods in Figure (1).The autoclave was placed inside the oven and the temperature of the oven was set to (800°C) for (48 hr.)as in Figure (2

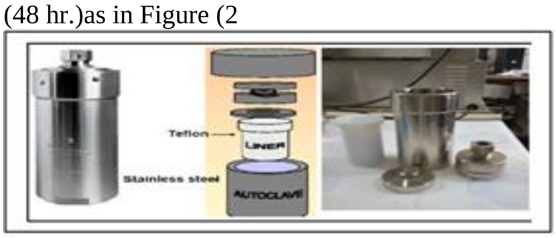

Fig. (1): Schematic of Autoclave Parr Reactor to a chives the hydrothermal method.

Fig. (2): A Photo of the Oven Device

The autoclave was cooled down to room temperature and the result powder inside the Teflon separated from the solution and washed with distilled water several times with Ultrasonic device until the pH of the wash became neutral(pH=7). Then the product was finally dried for (24hr.) at (40– 50°C) in a dust-proof environment.

2\The second step included prepared the sample by using mixing &kneading method formed as a Circular Discs contain Nickle Ferrite "prepared in first step" and (Polymethyl methacrylate – PMMA)"[PMMA-830-IH] was provided by the Korean LG Company with a molecular weight of 149000 Da."polymer, then formed first sample (S1)was contained($NiFe_2O_4$=2 gm) & (PMMA=0.75 gm). Second sample (S2) was contained($NiFe_2O_4$=2 gm) & (PMMA=1.50 gm).Two samples mixed & kneaded with(6 ml Ethanol Alcohol separately, then left to dried at room temperature for 24 hr. It was noted that the dough of sample (S2)(which contains polymer percentage more than sample (S1))was rather soft compared to the sample (S1) (which was more solid).

3\The third step included diluted crude oil by using Hexane (Solvent)with percent(1:3) ml, then the samples ablated by using Nd:YAG laser with (1064 nm) wavelength , (6Hz) frequency and (100 pulses) for the tow samples (S1 & S2)immerse in (4 ml) dilute crude oil, as in Figure (3).The optical and rheological properties where measured. The samples (S1 & S2) compared with blank sample of Crude Oil to illustrate difference of values between the two samples and the crude oil solution sample which considered as (Pure sample).

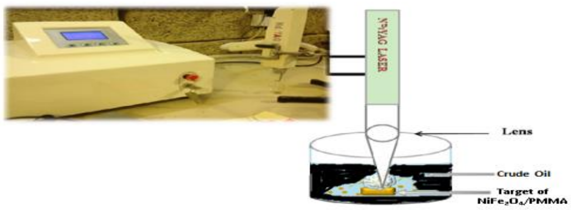

Fig.(3):Experimental set up of laser ablation technique

2. Result and discussion

Figure (4) showed the absorbance of pure, S1and S2 for the prepared samples. The intensity of S1 and S2 decreased compared with the pure sample of crude oil but the intensity of S2 higher than S1 with blue shift in S1 which indicate that the particle size of S1 less than S2. S1is closed to the pure solution that relate to the electron resonance and also because S1 has lower polymer than S2. The nanoparticles of Nickel Ferrite more than that S2. The interaction of laser with crude oil have many probabilities effect on material consist in it so that the behavior different in the case of the same number of pulse. Due to the polymer chain cutting after pulsed with laser so that intensity decreased [18].It is obviously seen from Figure (8) showed an increasing wavelength, the absorbance decreases exponentially to form an strong absorption edge at (500-530 nm), meanwhile, after (570 nm), the absorption value began to decrease to less than (0.5). In other curve (S1) showed a same trend for the absorption edge value in the whole UV–Vis range, which was obviously similar to that of curve (S2). The reason for this phenomenon might be that the introducing of PMMA loaded on the surface of $NiFe_2O_4$, which changed the surface absorption property of composite. The absorption spectrum of (S2) was similar as that of (Pure & S1), which indicated that $NiFe_2O_4$/PMMA (PMMA=0.75 gm) had almost the same response to UV-Vis. light and turning to the left (Blue Shift) as $NiFe_2O_4$/PMMA (PMMA=1. 50 gm) in the whole UV-Vis. band. This phenomenon might be ascribed to the similar UV-Vis. response of PMMA. In a word, composites of $NiFe_2O_4$ with polymer were better UV-Vis. absorption materials compared with the Pure crude oil.

The transmittance showed in Figure (5),S1 is higher than S2 and pure which indicates behaviours transmittance increased as the absorbance decreased, means that S1 sample make solution more transparent to the wavelength in visible region. The transmission decreases as content of PMMA increases. On the other hand the pure crude oil has high transmittance. Due to the formation of layer of covalent bonds formed between polymer chains and $NiFe_2O_4$ that decrease the transmitting of the incident light particularly at the shortest wavelengths. This means that the electrons are emphatically connected to their atoms through covalent bonds and the breaking of electron linkage and moving to the conduction band require photon with high energy[19].

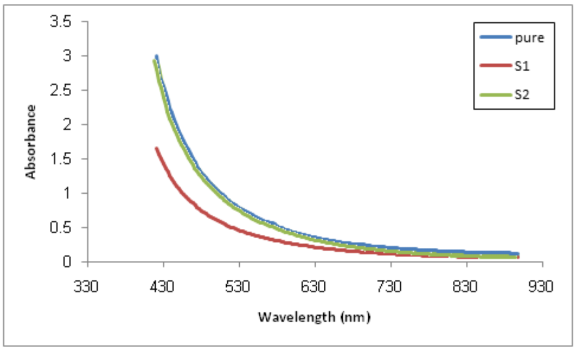

Figure 4 The spectrum of absorbance of pure , S1 and S2 samples.

Considering optical property of $NiFe_2O_4$/PMMA nanocomposites, transmission of each sample at visible light region was determined. Crude oil offered the highest transmission. It could give 73%T. Although transmission of visible light was slightly decreased in samples prepared, it was still high as 89%T. Highly optical clarity of $NiFe_2O_4$/PMMA nanocomposites was due to disorderly exfoliated morphology leading the $NiFe_2O_4$ nanoparticles in PMMA were relatively smaller than wavelength of visible light [19-21]. However, the $NiFe_2O_4$/PMMA nanocomposites prepared (S2:PMMA=1.50 gm) showed dramatic increase of %T as compared to those (S1:PMMA=0.75 gm). This was due to the $NiFe_2O_4$nanoparticles were not exfoliated and tended to highly aggregated in PMMA as shown in figure.

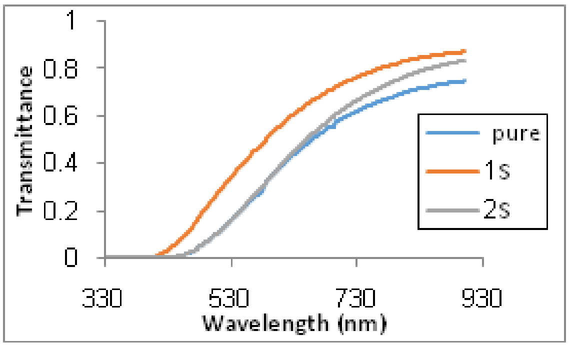

Figure 5 The spectrum of transmittance of pure , S1 and S2 samples.

Figure (6) show reflectance spectrum of three samples, samples have the same value but there is different in reflect wavelength because of S1 has nanoparticle smaller than S2in visible region, the pure solution has reflect in ultra violet region which shifting after ablation to the visible region . The shifting causes because the density of nickel in S1 more than in S2 and metals are reflect light because of free electrons [19]. It is well known that the ratio of octahedral coordinated (Ni^{2+}) to tetrahedral coordinated (Ni^{2+}) depends on the groundwork method. Therefore, reflectance measurement can be a useful tool for investigating the communication of $NiFe_2O_4$nanoparticles with the supporting structure [13,18-21]. The reflectance spectrum shows a $NiFe_2O_4$/PMMA band and a high number of bands that probably arose from the presence of nickel species in both the octahedral and tetrahedral coordination sites. The curves are characterized by a broad band in the range of (450-530 nm) and multiple edges bands in the range of (670–750 nm). Ni(II) ions with the electron configuration of ($3d^8$) have three spin-allowed electronic transitions and two spin-forbidden transitions in the UV-V is. range. Since the bands for nickel in the octahedral coordination sites correspond to higher energy than those for nickel in the tetrahedral sites, the modification can be attributed to the decrease in bands of nickel ions in the octahedral sites. Furthermore, the broadening of band representing the octahedral and tetrahedral sites occupied by the nickel species depended on the method used to prepare the nanoparticles[13,14,18].

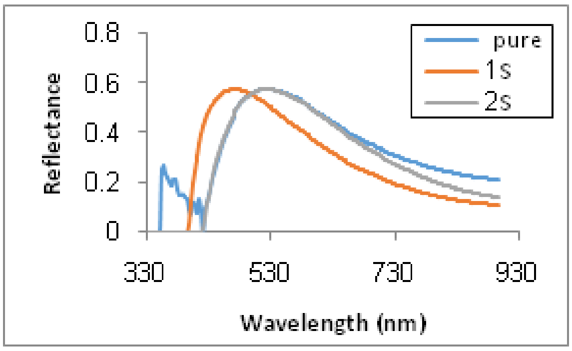

Figure 6 The spectrum of absorption coefficient of pure, S1 and S2samples.

Extinction The relation between the absorption coefficient (α) and the wavelength was represented in Figure (7)of S1, S2 and pure samples, for the prepared samples. The absorption coefficient increase as the absorbance increased but there red shifting after ablation compared with the pure. S1 has nanoparticle in size less than S2, because S1 has blue shift compared with S2 for the same number of pulses. The absorbance of the all samples changed with decreasing PMMA amount. The high values of absorbance can be attributed to the formation of $NiFe_2O_4$/PMMA nanoparticles transfer in the crude oil by laser ablation[18].The absorption coefficient can be calculated by Beer–Lambert's law as:

$I=I_0\, e^{-\alpha t}$ **and** $\alpha=2.303\log(A/t)$

Where:

(t) is the thicknesses of the film in (cm), (α) is the absorption coefficient $(cm)^{-1}$, whereas log (I/I_0) represents the absorbance (A).

It is shown that an increase in the values of the absorption coefficient with the increasing of PMMA content at (430-630 nm) which indicates the crystalline nature of the samples. The absorption coefficient α $(cm)^{-1}$ value is constant and small edge at (580 nm) which means that the possibility of electron transition is little because the incident photon energy is not sufficient to transfer the electron from the valence band to the conduction band ($h\upsilon<E_g$). While at high photon energy the absorption coefficient value is bigger and hence a great possibility for electron transitions[18-20].

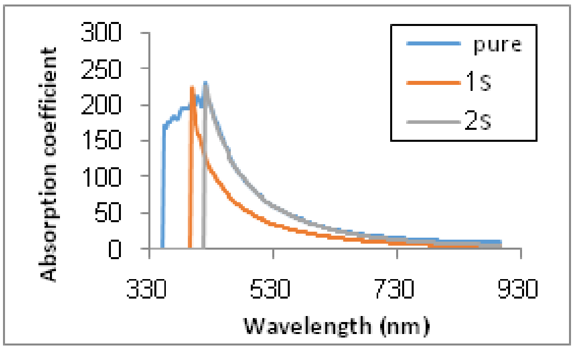

Figure 7 The spectrum of extinction coefficient of pure , S1 and S2 samples.

Extinction coefficient showed in figure (8) of the prepared samples. It increased as the absorbance increased but red shift happened after ablation. The extinction coefficient falls rapidly with increasing wavelength until, in the (530-680 nm) region. Crude oil except for a few narrow bands shown by zig-zag lines at (350-415 nm)region. Wavelengths which are shared by the polyethylene chamber windows, have very little spectral absorption here. By using laser ablation technique described earlier, larger drop curve was obtained for the (S2) sample. Because of the larger and broader curve distribution for these samples, a smaller peak extinction is found in the visible, but extinction by scattering is enhanced at longer wavelengths.

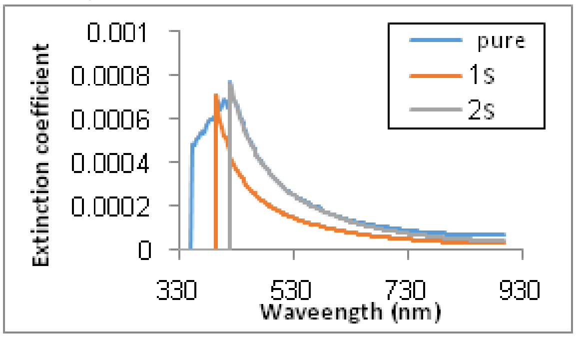

Figure 8 The spectrum of extinction coefficient of pure , S1 and S2 samples.

Figure (9) showed refractive index of S1,S2 and pure samples which have high refractive index with value 10& S1 has blue shift compared with S2.The extinction coefficient falls rapidly with increasing wavelength until, in the (530-680 nm) region.

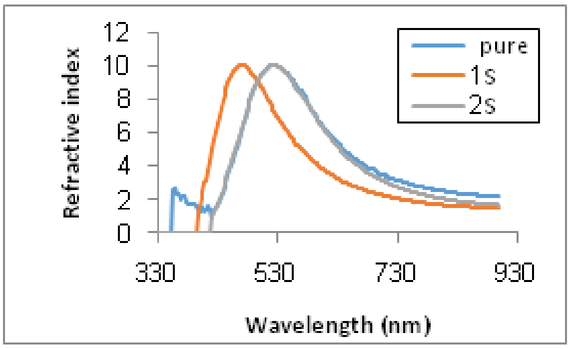

Fig. (9):The spectrum of refractive index of pure , S1 and S2 samples.

Figures (10) and (11) showed the viscosity of (S1) and (S2) respectively. Noticed that S1 has viscosity lower than (S2) because (S2) have polymer rather more than (S1).Also noticed that (S1)has stability than in (S2) which decreased as time interval increased which indicate that S2 has degradation after ablation because the density of nanoparticle lower than S1 which make S2 have weak structure[20,21].

The high viscosity of heavy crude is attributable to (1) their high molecular weight components, which become entangled and aggregated at low temperature and (2) the formation of ordered structures in the liquid phase. Heating heavy crude oils destroys the ordered structures in the liquid phase and reduces the viscosity. Figures (10 & 11) illustrates the effect of room temperature on the crude oil under investigation. One can see that the oil behaves as a fairly Newtonian fluid, becoming more pronounced as the temperature increases. It can also be seen that the temperature has a dramatic effect on the crude oil viscosity. Heating the crude from (20 to 25 °C), for example, reduces the viscosity from approximately (15000 to 11000 mPas), while heating it to 50°C reduces its viscosity to (<1300 mPas). Our aim is to test the crude's viscosity to a normal level at a temperature not exceeding (25 °C).Higher pour point reduction is achieved with the $NiFe_2O_4$/PMMA nanocomposite, and the efficiency is increased by increasing the PPD concentration. The pour point of the untreated crude oil (Pure). Better performance of the PMMA/clay nanocomposite is observed in comparison with the PMMA in the reduction of the pour point.

In fact the viscosity is directly opposite with the density. It is proved that the viscosity is a bit influenced with the presence of homogeneous catalyst. Viscosity is defined as the internal resistance to flow of any fluid which means the greater viscosity; the greater is the resistance to flow[22,23].

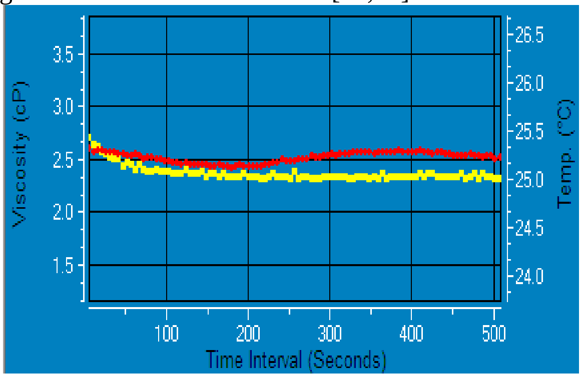

Fig. (10):The viscosity as a function of temperature of different time interval of S1 sample.

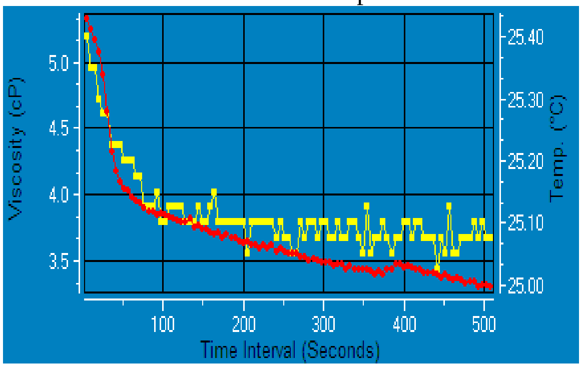

Fig. (11):The viscosity as a function of temperature of different time interval of S2 sample.

Table 1(1) shows the values of density for S1 and S2, where the density of S1 is higher than the density of S2 because of nanoparticle in size of S1 smaller than S2 which filled the vacancies between the liquid chains so the density increased.

Table (1) : Illustrate the values of density for S1 & S2 samples**.**

Samples	Weight (gm)	Volume (cm^3)	Density (gm/cm^3)
S1	0.664	0.5	1.268
S2	0.4534	0.5	0.812

3. Conclusion

Nanoparticles (NPs) exhibited exceptional outcomes when applied in various sectors including oil and gas industries. The harshness of the reservoir situations disturbs the effective transformations of the NPs in which the nano-particles tend to agglomerate and consequently leads to being attached on chemical bonds with organic crude oil compounds. Hence, Electromagnetic-Assisted nano fluids (such as ablated by laser) are very consequential in supporting the effective performance of the nano producing process &determine the nature of the alternate influence between (NPs) and the crude oil. The ablation process is heavily influenced by sample parameters like composition, thermal diffusivity, and absorptivity. The interaction of a laser with crude oil has a variety of effects on the material, resulting in diverse behavior for the same amount of pulses. Because of polymer chain was cut after being pulsed with laser, the intensity was reduced in absorbance. The viscosity decreases with increasing percentage of polymer addition .Also, the preparation samples have stability, which decreased with increasing time interval, which indicates the role of ablation due to the density of nanoparticles formed. Application of nanoparticles in thermal oil recovery processes presents several advantages, exhaustive investigations on improving synthesis method of nanomaterials and expansion of applications of existing nanomaterials need to be carried out.$NiFe_2O_4$/PMMA composites had disparity in optical properties performance by the response to UV–Vis. light. The results showed an increase absorption coefficient, extinction coefficient and refractive index with decreasing the percentage ratio of PMMA polymer. It is proved that the viscosity is a bit influenced with the presence of homogeneous catalyst .Finally, successful implementation of NPs to thermal oil recovery processes will facilitate the application of nano materials in the oil industry.

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