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FLOW MODEL SELECTION FOR INVERT EMULSIONS USING DIFFERENT TEMPERATURES

Dr. Muhannad A.R Mohammed
Assistant Prof.
Al-Nahrain University/Chemical Engineering

ABSTRACT:

This research deals with the study of the effect of temperature on the rheological properties (yield point, plastic viscosity and apparent viscosity) of invert emulsions. Twenty seven emulsion samples were prepared; all emulsions in this investigation are invert emulsions when water droplets are dispersed in diesel oil, the resulting emulsion is called water-in-oil emulsion (W/O). The rheological properties of these emulsions were investigated using a couett coaxial cylinder rotational viscometer (Fann-VG model 35 A), by measuring shear stress versus shear rate at different three temperatures (77, 122, and 167) F°. By using the Solver Add in Microsoft Excel®, the Bingham plastic model was found to be the best fits the experimental results. It was found that when temperature increased, it causes a decrease in the rheological properties (yield point, plastic viscosity and apparent viscosity) of emulsions which is due to the change in the viscosity of continuous phase of emulsion that is diesel oil.

Key words: Fluid Flow, Rheology, Emulsions, Non-Newtonian Fluids

الخلاصة :

يهتم هذا البحث بدراسة تأثير درجة الحرارة على الخواص الريولوجية (نقطة الخضوع ، اللزوجة اللدائنية واللزوجة الظاهرية) للمستحلبات .

تم تحضير 27 نموذج مستحلب ، حيث كل المستحلبات المحضرة هي مستحلبات عكسية عندها تكون قطرات الماء منتشرة خلال زيت الديزل والنتيجة تسمى مستحلبات ماء – في – نفط .

تم دراسة الخواص الريولوجية لهذه المستحلبات باستخدام جهاز (Fann – VG – 35A) وذلك بقياس إجهاد القص (Shear stress) المصاحب لكل معدل قص (Shear rate) وباستخدام ثلاث درجات حرارية مختلفة (67, 122 , 167) فهرنهايت.

تم استخدام برنامج مايكروسوفت إكسيل لإيجاد أفضل نموذج رياضي يمثل النتائج العملية وقد وجد إن نموذج بنكهام اللدائني هو الأفضل.

1. NOMENCLATURE :

Symbol	Meaning	Unit
A,B,C	**Parameters in Equation (6)**	**-**
A′,B′,C′	**Parameters in Equation (7)**	**-**
K	**Consistency index of a power law flui**	**-**
K′	**Generalized consistency index**	**-**
n	**Flow behavior index of a power law fluid**	**-**
n′	**Generalized flow behavior index**	**m/s**
α	**Parameter in equation (9)**	**-**
$\dot{\gamma}$	**Shear rate**	**rpm**
η	**Viscosity**	**cp**
η_∞	**Newtonian limiting Viscosity**	**cp**
θ	**Dial reading**	**deg**
τ	**Shear stress**	**lb/100 ft^2**
τ_m	**Shear stress at mean viscosity**	**lb/100 ft^2**
$\tau_\circ$	**Yield stress**	**lb/100 ft^2**
Y_p	**Yield point**	**lb/100 ft^2**
η_p	**Bingham plastic viscosity**	**cp**
η_a	**Apparent viscosity**	**cp**

2. INTRODUCTION:

An emulsion is a system containing two liquid phases, one of which is dispersed as droplets in the other. The liquid which is broken up into droplets is termed the dispersed phase, while the liquid surrounding the droplets is known as the continuous phase or dispersing medium. The two liquids, which must be immiscible or nearly so, are frequently referred to as the internal and external phases, respectively.

Simpson et. al (1961) referred to the materials used in preparing the emulsion, which are bodying agent for oil, primary emulsifier of water in oil, suspension agent, wetting agent and filtration control agent.

Criddle (1960) and Sherman (1968) stated that the principle factors, which influence the flow properties of emulsions, are related to the dispersed phase, continuous phase and the emulsifying agent.

Corins et. al (1976) found that the favorable conditions to form stable water in oil ratio emulsion are: a high oil-water viscosity ratio, a rapid initial adsorption of natural surfactant and at the pH at which the interfacial viscosity greatest.

Menon and Wasan (1988) discussed the various factors affecting the formation and stability of emulsions containing finely divided solids, which are; contact angle, demulsifies concentration, temperature, and solid-liquid interaction.

Kirk-Othemer (1998) discussed that many flow models have been proposed, which are useful for the treatment of experimental data or for describing flow behavior.

Chhabra (1999) stated that one of the most obvious factors that can have an effect on the rheological behavior of a material is temperature. Some materials are quite sensitive to temperature, and a relatively small variation will result in a significant change in viscosity. Others are relatively insensitive. Consideration of the effect of temperature on viscosity is essential in the evaluation of materials that will be subjected to temperature variations in use or processing, such as motor oils, greases, and hot-melt adhesives.

S. Kokal and S. Aramco (2002) stated that crude emulsions are stabilized by rigid interfacial films that form a skin on water droplets and prevent the droplets from coalescing.

The aim of the work is to study the effect of temperature on the rheological properties of invert emulsions, and find the best flow model which best fits to the experimental data.

Flow Models:

Many flow models have been proposed, which are useful for the treatment of experimental data or for describing flow behavior. However, it is likely no given model fits the rheological behavior of material over an extended shear rate range. Nevertheless, these models are useful for summarizing rheological data and are frequently encountered in the literature.

Of the model listed in table (1), the Newtonian is the simplest. The other model can be applied to non-Newtonian materials where time dependant effects are absent.

The Bingham plastic model, $\tau - \tau_\circ = \eta \quad \dot{\gamma}$, is the two parameter model which has been widely used as a model for non-Newtonian fluids such as drilling fluid and emulsion. Fluids that exhibit Bingham Plastic behavior are characterized by a yield point (τ_o) and a plastic viscosity (η_p) that is independent of the shear rate. The presence of a yield stress means that a certain critical shear stress must be exceeded before flow can begin. If the fluid exhibits Newtonian flow after the yield value is exceeded, it is called a Bingham Plastic fluid.

In Fann VG- viscometer 35A plastic viscosity in (cp), the yield point in (lb/100 ft^2) and apparent viscosity in (cp) can be determined from:

$$\eta_p = \theta_{600} - \theta_{300}$$

$$Y_p = \eta_p - \theta_{300}$$

$$\eta_a = \frac{\theta_{600}}{2}$$

Where;

θ_{600} = Dial reading at 600 rpm.

θ_{300} = Dial reading at 300 rpm.

3. EXPERIMENTAL WORK:

Emulsions Used:

All emulsions prepared in this investigation are invert emulsions when water droplets are dispersed in oil the resulting emulsion is called water- in - oil emulsion (W/O). Generally, W/O-emulsions are preferred when a large amount of oil is desired.

Diesel oil was used as the external phase of all samples being tested, and distilled water was used as the dispersed phase with powdered sodium chloride to provide the salinity of the water phase.

Additives which were used as follow a soluble oil additive as emulsifying agent, barite as weighting material, lime to provide alkalinity, organophilic clay to suspend the weight material, and finally asphaltic material to provide emulsion stability at high temperature, and gel strength.

Sets of Experiments:

Twenty seven experiments were performed to study the effect of temperature on rheological properties (plastic viscosity, yield point and apparent viscosity) of emulsions and to find the best fits model by using the Solver Add in Microsoft Excel. Experiments were performed at different three temperatures (67, 122 and 167) °F.

Experimental procedure:

It was found that the following mixing procedure included sequence of additives and mixing periods gave a best results in preparing emulsions as shown below:-

1. 475 ml diesel oil was measured in a beaker and placed in the Hamilton Beach cup.
2. 25 ml of water was measured in beaker.
3. Add salt (NaCl) to beaker of water.
4. Add desired concentration of emulsifier (soluble oil additive) concentrate drop by drop to the diesel oil in the Hamilton Beach cup while mixing, and mix for 60 minutes.
5. All powder materials and additives were weighted by using electronic balance, some of these materials are changed in tests and the others are constant as shown in table below:
6. Slowly add lime additive, and mix for 30 minute.
7. Slowly add the required volume of brine while mixing and mix for 45 minutes.
8. Slowly add asphaltic material additive, and mix for 30 minutes.
9. Slowly add organophilic clay additive, and mix for 30 minutes.
10. Slowly add the total amount of barite as slow as possible to obtain the required final percentage volume of barite. Then mix the final mixture for 30 minutes.
11. The prepared solution was kept at rest at room temperature for 24 hour prior to conducting the rheological measurements.

Fann Viscometer model 35 A testing procedure:

Twenty seven emulsion samples were tested using the Fann viscometer model-35 A. These samples included three various volume percentages of barite, with three different volume percentages of emulsifier and three different concentrations of asphaltic material.

The emulsion samples were tested for rheological properties under three different temperatures of (77 °F, 122 °F and 167 °F), at each temperature the rheological properties were measured.

Before any test, the viscometer must be calibrated for both shear rate and shear stress to be insuring that it will give accurate results.

The sample cap was filled with emulsion sample prepared at 25 °C (77 °F) to the scribed line, and the rotor was immersed to the proper immersion depth.

The instrument was operated at 300 rpm for three minutes to equalize the temperature of the bob, rotor and emulsion.

The instrument switched to 600, 300, 200, 100, 6, and 3 rpm and the dial reading was recorded.

Repeat the above procedure to the emulsion sample after heated to 50 °C (122 °F), and then after heated to 75 °C (167 °F).

4. RESULTS AND DISCUSSION:

Flow Model Selection:

The selection of the flow model that best fit the rheological behavior is useful for treating experimental data or for describing flow behavior.

In this work a method, which is proposed by Faith A. Morrison (2005) is used. This method uses the Solver Add-in in Microsoft Excel® to optimize the solution.

The basic outlines of this method include:

- Arranging the experimental data in the Excel spreadsheet. By using two columns, one for shear rate and the other for shear stress.
- Create a column that has a predicted value of shear stress calculated from a considered flow model. Since it has not know the values of any model parameters, will be started with some guesses.
- Create a new column for the square of the deviation between the actual shear stress and the predicted value. Add up all the values in the error column and put that value in a cell.
- The Solver function in the Excel® is set up to minimize the error cell mentioned above.
- Solver will replace our initial guesses with optimized values.
- Solver allow us to put constrains on the ways in which it manipulate the parameters may be negative.

Applied the experimental data which prepared in 25 °C (77 °F) by using Add-in Microsoft Excel® and the result was the best by using Bingham plastic model.

By using Add-in Microsoft Excel® the experiments at 25 °C (77 °F) will be taken because the experiments at 122 °F and 167 °F give the same results, which is the Bingham plastic model best fits the experimental results.

The results by using the Add-in Microsoft Excel® are shown in tables (3) to (11).

5. EFFECT OF TEMPERATURE ON RHEOLOGICAL PROPERTIES OF EMULSIONS:

The results of over all tests are presented in figures (1) to (9), which are plot of (yield point, plastic viscosity and apparent viscosity) versus temperature. From these figures, one can notice that the increase in temperature will decrease the rheological properties (yield point, plastic viscosity and apparent viscosity), due to the change in the viscosity of the continuous phase of emulsions which is diesel oil.

Temperature causes reduction in the viscosity of diesel oil and in the friction forces between the solid particles in the emulsions mixtures, which will result in a noticeable decrease in plastic viscosity of emulsion samples, also the yield point decreased due to reduction in the attraction forces between the solid particles in the emulsions since the distance between these particles becomes larger. Apparent viscosity decreased due to these effects in the emulsion mixture. In other meaning, it may be said that temperature causes an expansion of sample.

It was found that the concentration of asphaltic material which was 35.5 gm was effective for emulsion samples of 0.46 % vol. of barite and 2.4 % vol. of emulsifier to be stable at temperatures up to 167 °F, no flocculation was observed. Since the values of (plastic viscosity, yield point and apparent viscosity) decreases gradually as temperature increased.

The same effect of 35.5 gm of asphaltic material was obtained on emulsion samples of 0.65 % vol. of barite. The emulsion samples of 0.84 %. Vol. barite follows the same behavior.

These samples which were prepared with emulsifier concentration of 2.4 % by volume, it was found that 2.4 % emulsifier sufficient to coat all the water droplets. Insufficient emulsifier concentration will result in a break down of emulsion with increasing temperature that will lead to a free water phase in the emulsion, which causes water-wetting of solid constituents in the emulsions, especially barite. Water-wetting of barite causes it to be settled, that will increase the rheological properties.

The concentrations of 35.5 gm of asphaltic material and 2.4 % emulsifier were the best to be selected in preparing emulsions samples with vols. % of barite (0.46, 0.65, and 0.84), with out thermal flocculation up to 167 °F.

Although, the emulsions prepared were in satisfactory conditions at temperatures up 167 °F with 35.5 gm asphaltic material, it was decided to increase the concentration of asphaltic material to 40.5 and 45.5 gm, with three different vols. % of barite to demonstrate its effect on rheological properties of emulsions with temperature. Increasing the asphaltic material to 40.5 and 45.5 gm of samples of 0.46 % vol. of barite, resulted in better behavior of rheological properties with temperature. This is due to the low solubility factor of asphaltic material which is related to the temperature and will always be present in the fluid as a tacky undissolved solid and will work continuously as a plastering agent, thus it improves emulsion behavior, especially at high temperatures and reduces chances of flocculation.

A similar effect of 40.5 and 45.5 gm of asphaltic material was obtained on samples of 0.65 % vol. of barite. For 0.84 % vol. of barite, also 40.5 and 45.5 gm tend to improve the behavior of rheological properties.

The emulsifier concentration was increased to 3.2 % and 4 % by volume to establish it's effect on rheological properties of emulsion under temperature. It was noticed that this increase has no appreciable effect on the values of plastic viscosity, yield point and apparent viscosity but it resulted in a stronger emulsion with better tolerance to temperature.

6. CONCLUSIONS:

1. All emulsions prepared in this investigation are invert emulsions when water droplets are dispersed in oil the resulting emulsion is called water-in-oil (W/O) emulsions .
2. The increase in temperature will decrease the rheological properties (yield point, plastic viscosity and apparent viscosity) of emulsion .
3. By using the Solver Add-in-Microsoft Excel, the Bingham plastic model was found to be the best fits the experimental results.

Table (1) Flow equations for flow models (Chhabara & Kirk).

Flow model	Flow equation	Eq. No.
Newtonian	$\tau = \eta \dot{\gamma}$	**…(1)**
Bingham plastic	$\tau - \tau_{\circ} = \eta_p \dot{\gamma}$	**…(2)**
Power law	$\tau = k\dot{\gamma}^n$	**…(3)**
Modified power law	$\tau - \tau_{\circ} = k\dot{\gamma}^n$	**…(4)**
Casson fluid	$\tau^{1/2} - \tau_{\circ}^{1/2} = \eta_{\infty}^{1/2}\, \dot{\gamma}^{1/2}$	**…(5)**
Robertson-Stiff	$\tau = A \ (\dot{\gamma} + C)^B$	**…(6)**
Modified Robertson-Stiff	$\tau - \tau_{\circ} = A'(\dot{\gamma} + C')^B$	**…(7)**
Williamson	$\eta = \eta_{\infty +} \dfrac{(\eta_{\circ} - \eta_{\infty})}{1 + \dfrac{\lvert\tau\rvert}{\tau_m}}$	**…(8)**
Cross	$\eta = \eta_{\infty} + \dfrac{(\eta_{\circ} - \eta_{\infty})}{1 + \propto \dot{\gamma}^n}$	**…(9)**

Table (2) Constant materials for all tests.

Material	Weight (gm)
Lime (CaO)	23.7
Organophilic clay	2.37
NaCl (salt)	7

Table (3) Model selection for Emulsions using Add-in Microsoft Excel at room temperature 77 °F (25 °C) with (Barite = 0.46 , Emulsifier = 2.4) Vol. %

Weight of Asphaltic material (gm)	Sum of Square Error			
	Newtonian Eqn. (1)	Power Law Eqn. (3)	Bingham Eqn. (2)	Casson Eqn . (5)
35.5	2.1942	0.1924	0.0037	0.0538
40.5	2.2632	0.1540	0.0079	0.0518
45.5	2.2336	0.1492	0.0167	0.0663

Table (4) Model selection for Emulsions using Add-in Microsoft Excel at room temperature 77 °F (25 °C) with (Barite = 0.46 , Emulsifier = 3.2) Vol. %

Weight of Asphaltic material (gm)	Sum of Square Error			
	Newtonian Eqn. (1)	Power Law Eqn. (3)	Bingham Eqn. (2)	Casson Eqn . (5)
35.5	2.1763	0.1684	0.0042	0.0438
40.5	2.2652	0.1545	0.0086	0.0531
45.5	2.3441	0.1443	0.0174	0.0644

Table (5) Model selection for Emulsions using Add-in Microsoft Excel at room temperature 77 °F (25 °C) with (Barite = 0.46 , Emulsifier = 4)Vol. %

Weight of Asphaltic material (gm)	Sum of Square Error			
	Newtonian Eqn. (1)	Power Law Eqn. (3)	Bingham Eqn. (2)	Casson Eqn . (5)
35.5	2.1891	0.1654	0.0038	0.0435
40.5	2.2747	0.1512	0.0098	0.0533
45.5	2.3502	0.1419	0.0171	0.0633

Table (6) Model selection for Emulsions using Add-in Microsoft Excel at room temperature 77 °F (25 °C) with (Barite = 0.65 , Emulsifier = 2.4) Vol. %

Weight of Asphaltic material (gm)	Sum of Square Error			
	Newtonian Eqn. (1)	Power Law Eqn. (3)	Bingham Eqn. (2)	Casson Eqn . (5)
35.5	2.1834	0.2278	0.0278	0.1020
40.5	2.2523	0.2032	0.0360	0.1041
45.5	2.2743	0.1948	0.0319	0.0986

Table (7) Model selection for Emulsions using Add-in Microsoft Excel at room temperature 77 °F (25 °C) with (Barite = 0.65 , Emulsifier = 3.2) Vol. %

Weight of Asphaltic material (gm)	Sum of Square Error			
	Newtonian Eqn. (1)	Power Law Eqn. (3)	Bingham Eqn. (2)	Casson Eqn . (5)
35.5	2.1812	0.2450	0.0326	0.1156
40.5	2.2714	0.1876	0.0265	0.0896
45.5	2.2783	0.1940	0.0328	0.0991

Table (8) Model selection for Emulsions using Add-in Microsoft Excel at room temperature 77 °F (25 °C) with (Barite = 0.65 , Emulsifier = 4) Vol. %

Weight of Asphaltic material (gm)	Sum of Square Error			
	Newtonian Eqn. (1)	Power Law Eqn. (3)	Bingham Eqn. (2)	Casson Eqn . (5)
35.5	2.2041	0.2188	0.0236	0.1340
40.5	2.2820	0.1806	0.0248	0.0857
45.5	2.3065	0.1783	0.0316	0.0923

Table (9) Model selection for Emulsions using Add-in Microsoft Excel at room temperature 77 °F (25 °C) with (Barite = 0.84 , Emulsifier = 2.4) Vol. %

Weight of Asphaltic material (gm)	Sum of Square Error			
	Newtonian Eqn. (1)	Power Law Eqn. (3)	Bingham Eqn. (2)	Casson Eqn . (5)
35.5	2.2246	0.1864	0.0157	0.0736
40.5	2.2625	0.1936	0.0258	0.0918
45.5	2.2760	0.1917	0.0286	0.0946

Table (10) Model selection for Emulsions using Add-in Microsoft Excel at room temperature 77 °F (25 °C) with (Barite = 0.84 , Emulsifier = 3.2) Vol. %

Weight of Asphaltic material (gm)	Sum of Square Error			
	Newtonian Eqn. (1)	Power Law Eqn. (3)	Bingham Eqn. (2)	Casson Eqn . (5)
35.5	2.2329	0.1837	0.0164	0.0739
40.5	2.2601	0.1822	0.0188	0.0791
45.5	2.2813	0.1914	0.0306	0.0965

Table (11) Model selection for Emulsions using Add-in Microsoft Excel at room temperature 77 °F (25 °C) with (Barite = 0.84 , Emulsifier = 4)Vol. %

Weight of Asphaltic material (gm)	Sum of Square Error			
	Newtonian Eqn. (1)	Power Law Eqn. (3)	Bingham Eqn. (2)	Casson Eqn .(5)
35.5	2.2480	0.1787	0.0162	0.0727
40.5	2.2606	0.1877	0.0220	0.0852
45.5	2.2907	0.1838	0.0282	0.0914

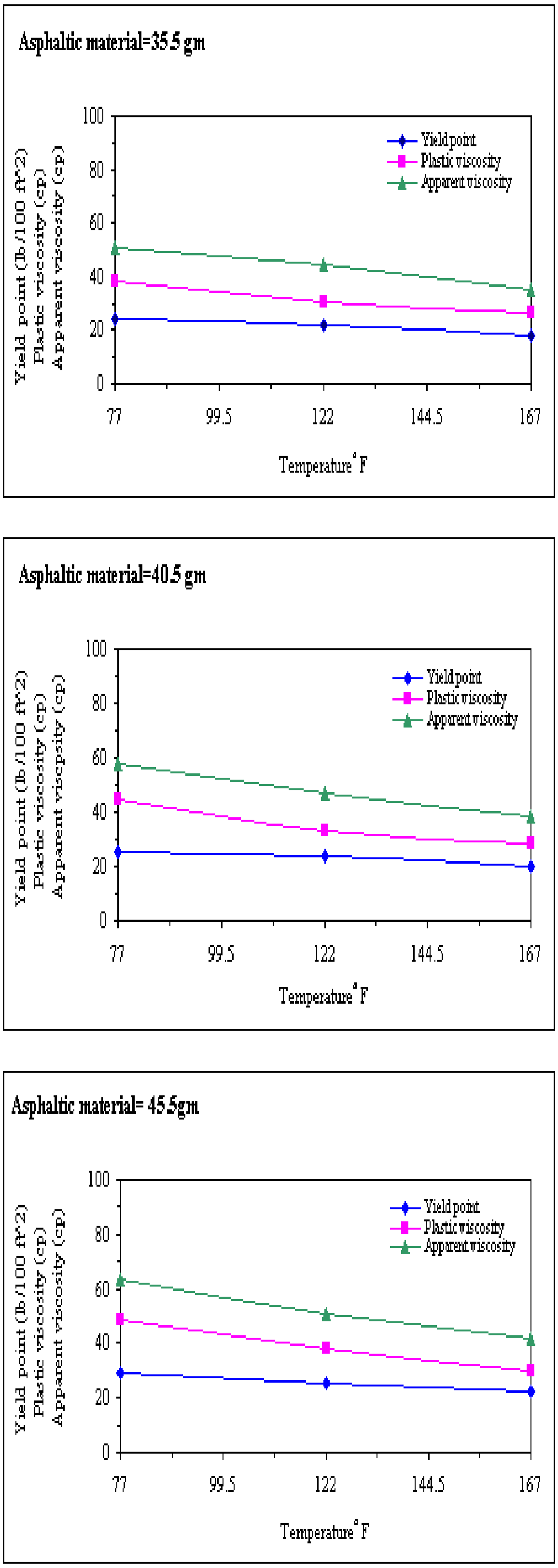


Figure (1) Effect of Temperature on Rheological properties of Emulsions with (Barite = 0.46 , Emulsifier = 2.4) Vol. %

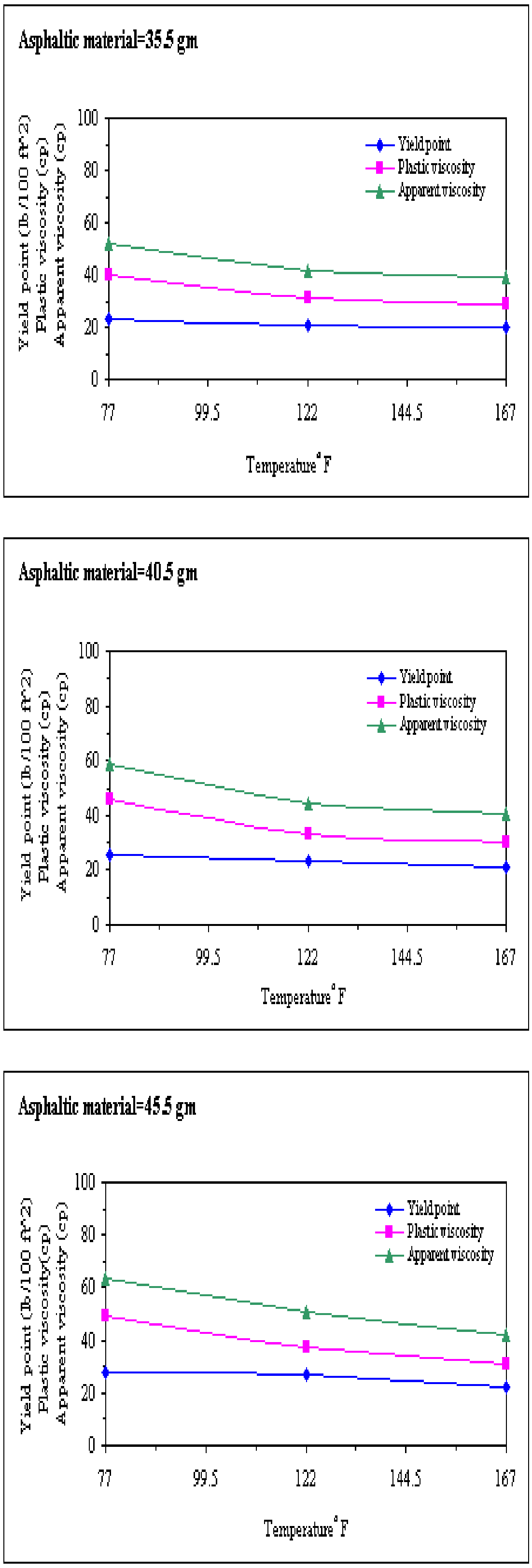


Figure (2) Effect of Temperature on Rheological properties of Emulsions with (Barite = 0.46 , Emulsifier = 3.2) Vol. %

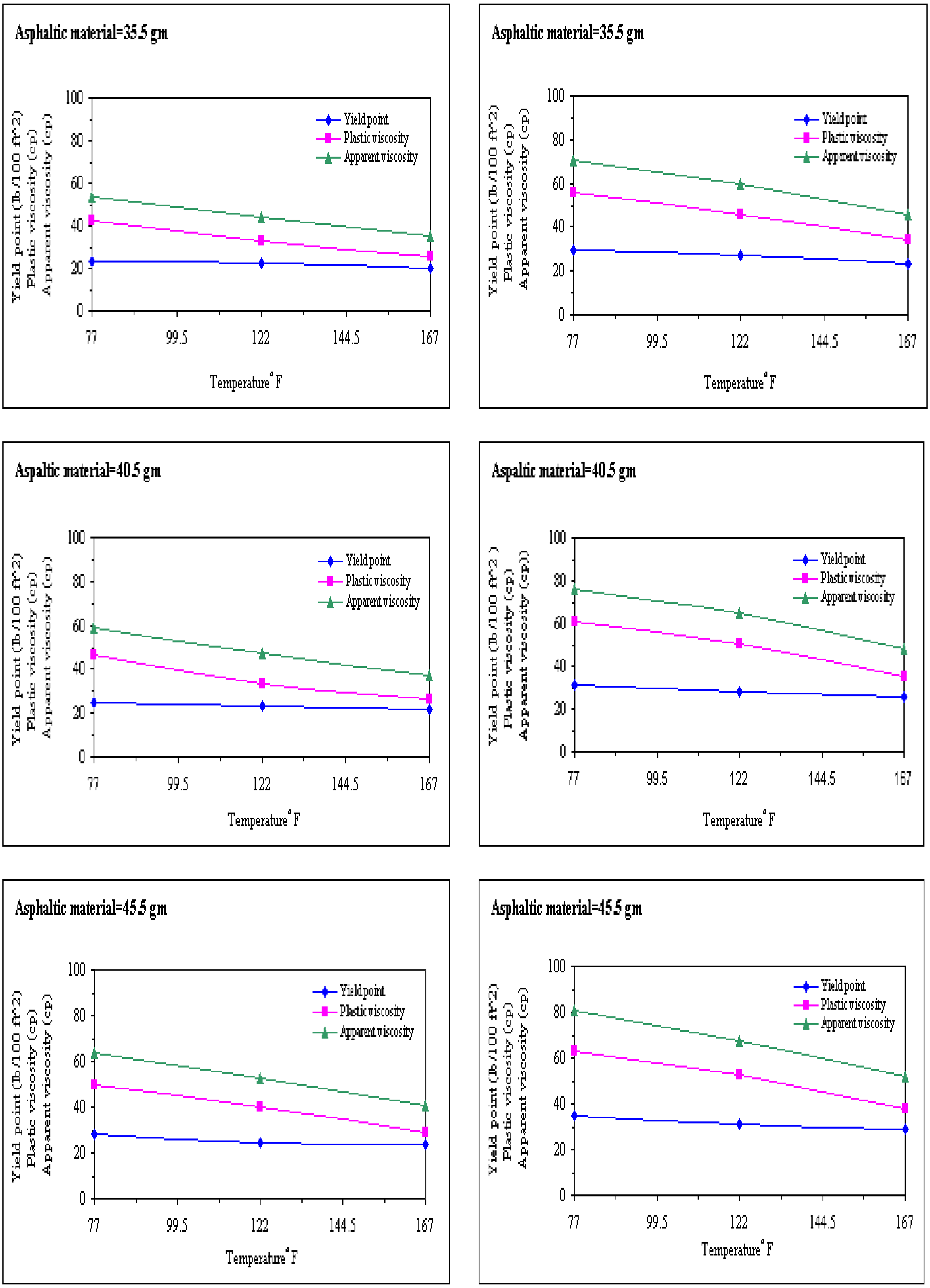
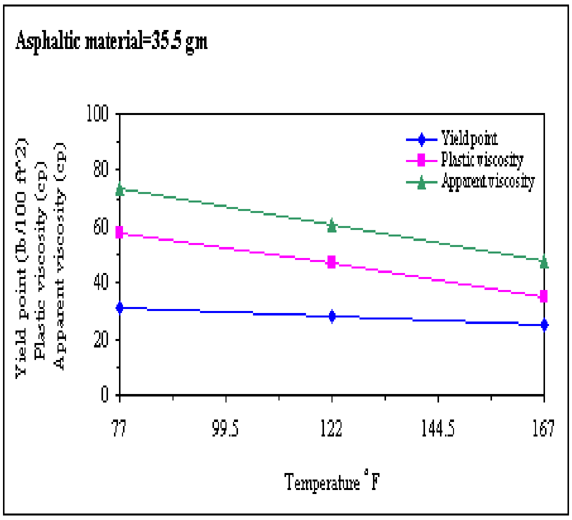
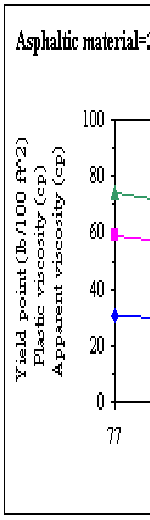
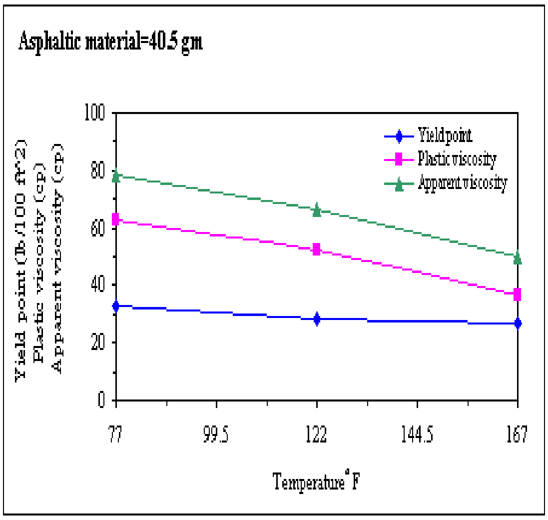


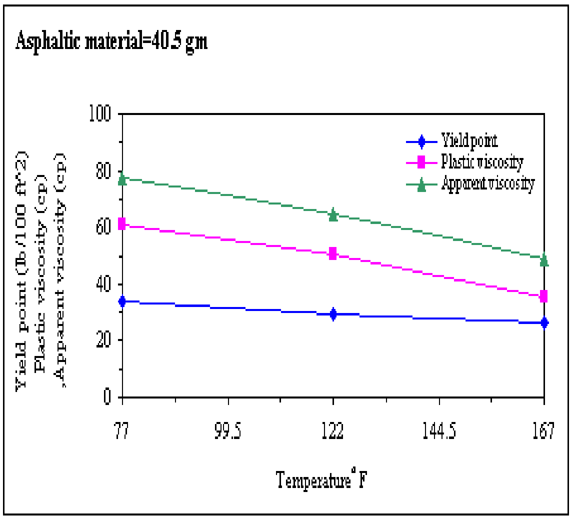
Figure (4) Effect of Temperature on Rheological properties of Emulsions with (Barite = 0.65 , Emulsifier = 2.4) Vol. %

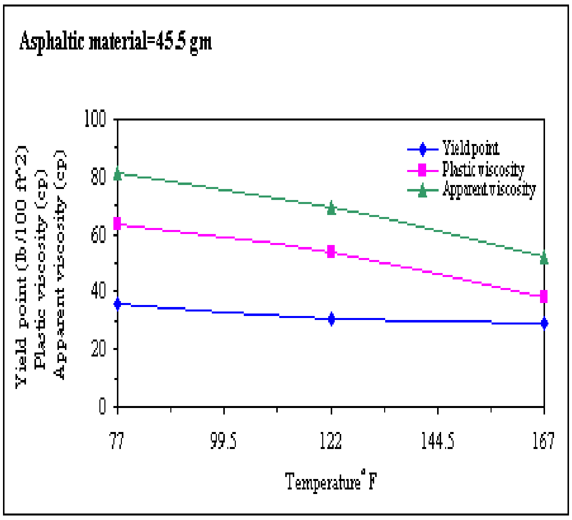
Figure (3) Effect of Temperature on Rheological properties of Emulsions with (Barite = 0.46 , Emulsifier = 4) Vol. %











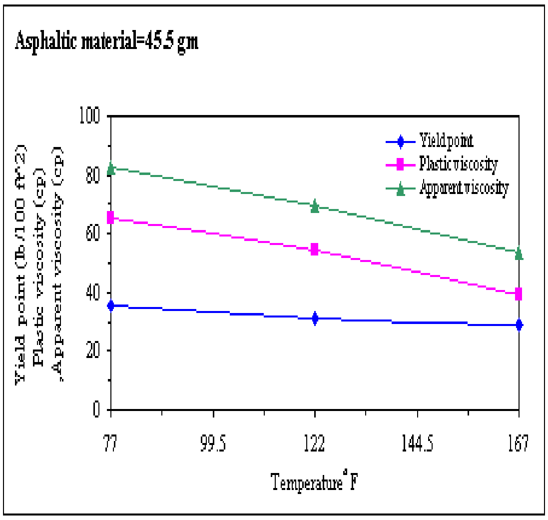


Figure (5) Effect of Temperature on Rheological properties of Emulsions with (Barite = 0.65 , Emulsifier = 3.2) Vol. %

Figure (6) Effect of Temperature on Rheological properties of Emulsions with (Barite = 0.65 , Emulsifier = 4) Vol. %

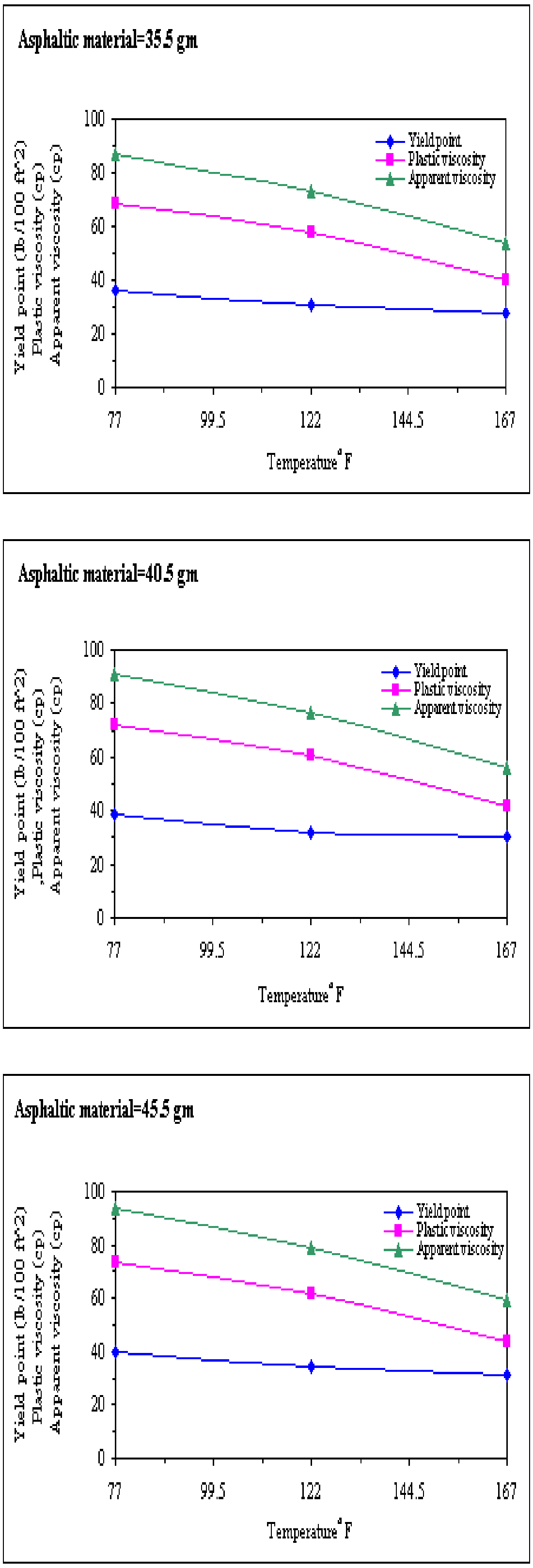


Figure (7) Effect of Temperature on Rheological properties of Emulsions with (Barite = 0.84 , Emulsifier = 2.4) Vol. %

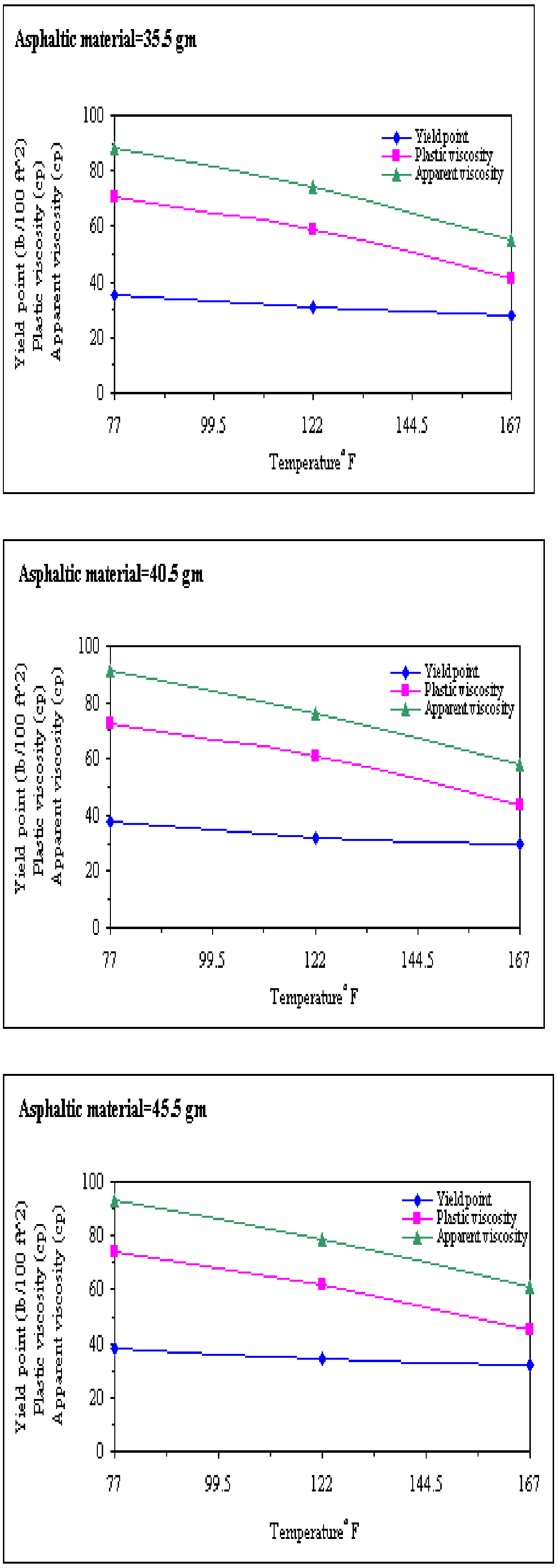
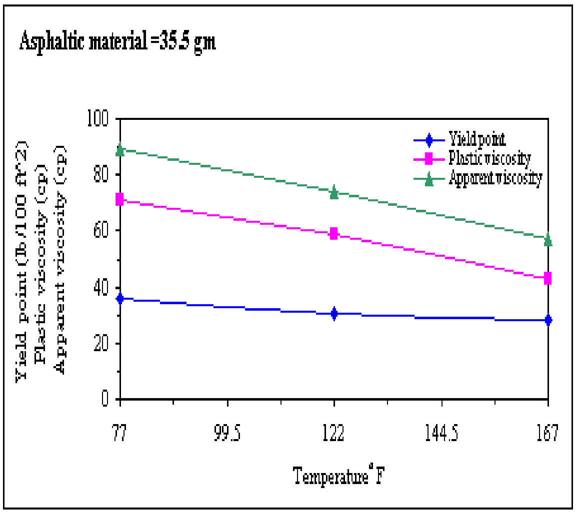
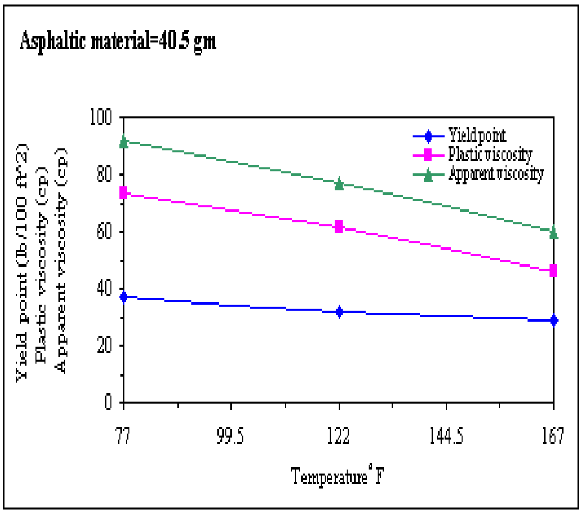


Figure (8) Effect of Temperature on Rheological properties of Emulsions with (Barite = 0.84 , Emulsifier = 3.2) Vol. %





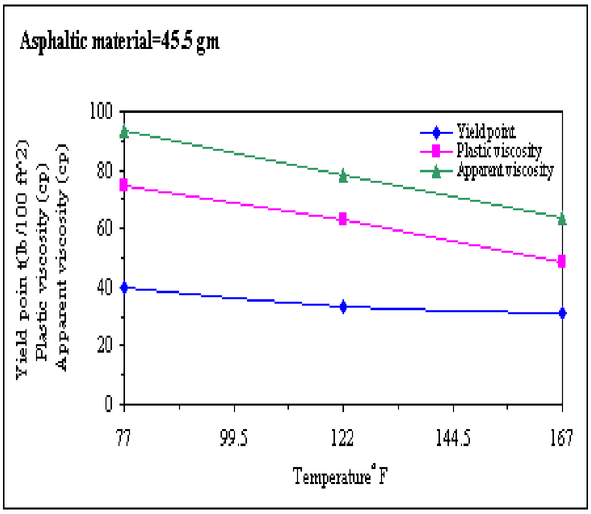


Figure (9) Effect of Temperature on Rheological properties of of Emulsions with (Barite = 0.84 , Emulsifier = 4) Vol. %

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