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Research Paper

Investigation of Long Time Magnetic Nano-particles Stabilization in Aqueous Medium by Different Surfactants

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Abstract

In this study, an experimental investigation on the synthesis method of a ferro-fluid was conducted in order to obtain a highly stable ferro-fluid. For this purpose, various surfactants are applied for colloidal stabilization among which tetra methyl ammonium hydroxide (TMAOH) showed the best performance. Several methods of characterization were chosen to characterize synthesized ferro-fluids such as Zeta potential and the particle size analysis by Dynamic Light Scattering (DLS). Based on the DLS results, the average diameter of Fe_3O_4 nano-particles was about 30 nm. Besides, the Zeta potential analysis was applied to investigate the stability of synthesized ferro-fluids. Moreover, the dynamic viscosity of the ferro-fluid is estimated at four different temperatures (20 ºC, 35 ºC, 50 ºC, 65 ºC). Based on the results, the ferro-fluid viscosity is decreased significantly by the temperature increase. Further, it is obvious that the viscosity of the ferro-fluid is decreased exponentially in terms of shear rate which determines its shear thinning (non-Newtonian) behavior.

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1. INTRODUCTION

Recently, lots of attempts have done to study nanoparticles and nano composites to investigate and fabricate new ones [1-5]. Ferro-fluids are fabricated by Wilson in 1779 for the first time. He made a fluid by tiny particles of iron and water. For many years there was no new idea till 1963, when Steve Papell made a fluid by mixing a magnetic powder and oleic acid. In fact, it was the first real ferro-fluid [6]. The concept of nano-fluid refers to the suspensions including metallic and nonmetallic nano-particles. Nano-fluids are different behaviors in various media and conditions and this leads to important applications in industry. Due to problems in the use of traditional fluids and even micro-fluids, such as sedimentation, erosion, fouling and pressure drop of flow channels; researchers were interested in nano-fluids. At 1993 the idea of nano-fluids was introduced by Choi and Eastman [7] and it led to a revolution in the heat transfer in fluids and founded a new insight to fluid-solid suspension at the nano-scale.[8,9] On the other hand, these new fluids cause many improvements in erosion, corrosion, and sedimentation [10-13]. Rosenweig improved this method for making fluids with higher magnetic strength and higher compression [14]. This method is the base of today's ferro-fluids. The particles size in nano-fluids is between 1 to 100 nm and the most used nano-particles are metallic nano-particles such as Cu, Ag and metallic oxides such as Al2O3 and CuO [15]. In Section 2, the stability of nanofluids and its methods are introduced. In section 3, magnetic fluids are introduced and in section 4, the implemented methods for making nano-particles are presented. In section 5, the criteria for checking the electric charge value, dimensions and stability are presented to evaluate the obtained fluids, and it is shown that one of the methods resulted in a fluid with appropriate characteristics.

2. STABILITY OF NANO-FLUIDS

Nano-fluid is not a simple composition of liquid and nano-particles but due to their high active surface, they tend to be agglomerated which causes sedimentation and reduction of the physical properties of the nano-fluid. So it is a critical factor to distinguish between a nano-fluid and other fluids. Most important parameters in the stability of a nano-fluid are the concentration of nano-particles, the viscosity of the fluid, pH, the type, size and the shape of nano-particles and the ultrasonic time [15].

Preparing the stable nano-particle is a necessary condition to optimize nano-fluid properties. Collection and agglomeration of nano-particles increase the sedimentation probability and decrease the stability of the fluid. The speed of sedimentation of static spherical nano-particle is [16];

$$v = \frac{2R^2}{9\mu}(\rho_p - \rho_1)g \quad (1)$$

Where R is the radius of nano-particles, μ is the viscosity of the fluid, ρ_p is the particles density and ρ_1 is the fluid density. This equation obtains from the balance between gravity, buoyancy, and drag forces on particles.

Considering this rule, the reduction of particle size leads to a decrease in sedimentation rate. When the particles radius passes a critical radius R_c, the Brownian motion leads to sedimentation. Although particles with radius less than R_c will have no sedimentation, but more tiny particles have more active surface, so can agglomerate more. Thus preparing stable nano-fluid needs particles with size less than R_c and methods to prevent them from simultaneous agglomerating. Stability of nano-fluid means that the nano-particles have no agglomeration and sedimentation and the concentration of nano-particles in the fluid remains fixed [17].

With a view to Derjaguin-Landau-Verwey-Overbee theory (DLVO), stability of nano-particles in a fluid can be calculated from total attraction forces and repulsion forces act on the particles. Totally, there are four forces between molecules: (a) Van Der Waals forces, (b) magnetic bipolar forces (if the particles are magnetic), (c) Electrostatic forces (for particles with surface electric charges), (d) Steric forces (for particles coated by polymers and surface activators). If total repulsion forces dominant attractive forces, the fluid will be stable, otherwise the particles will attract each other and the agglomeration and consequently the sedimentation will occur.

The fundamental mechanisms affect stability of colloids based on different types of repulsion, have two categories; 1- Space repulsion and 2- Electrostatic repulsion. Figure 1 denotes these repulsions.

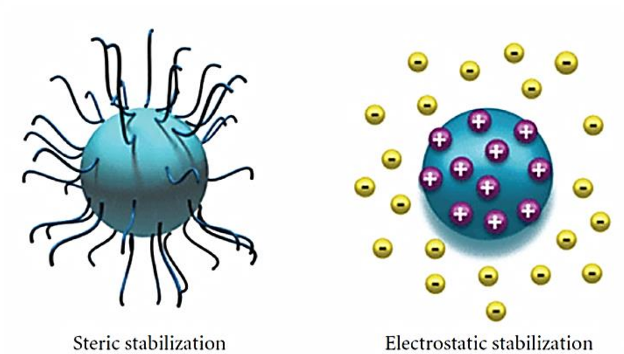

Fig.1. Space and electrostatic repulsions [18]

There are three ways of stability of nano-fluids. Each of these methods individually or together can improve the stability of nano-fluids. In the following we will introduce these methods briefly;

2-1-Surfactant addition

The surfactant addition is an affordable and economic method to stable a nano-fluid. Active surface material has remarkable effects on the surface characteristics system. A hydrophilic polarizing head and a hydrophobic tail (usually a hydrocarbon chain) are present in these materials. Active surface materials can be 4 groups according to combinations of the hydrophilic head:

a) Non-ionic, which have no charged group in the hydrophilic head.
b) Anionic, which have a negative charge.
c) Cationic, which have a positive charge.
d) Amphoteric, which the charge of the hydrophilic head is negative or positive.

To choose a suitable active surfactant, should be considered if the base fluid is a polarized one, the amphoteric surfactant will be used, otherwise soluble in oil surfactant will be used [18]. Also, care must be taken in the use of these materials because excessive presence of them in the nano-fluid changes the properties of the nano-fluid and has influence on mass and heat transfer.

Adding surfactant materials to the nano-fluid may cause some problems such as spume and reduction of thermal conductivity. Also the destruction of the bond between the surfactant and the nano-particle at temperature above 60 ^{o}C results in losing the stability of the nano-fluid. [19]

2-2-PH control of nano-fluid

The stability of a nano-fluid depends directly to its electro-kineticall properties. In such a way that if the charge density at the surface of the nano-particle is high, due to an electrostatic repulsive force, nano-particles will be stable in the fluid. [19]

2-3-Ultrasonication

To enhance nano-fluids stability, ultrasonic vibrators can be used. Two aforementioned methods are used in this direction by modifying the surface of nano-particles, but here, ultrasonic waves lead to poor surface bonds between nano-particles, so it breaks agglomerating and increases the stability of the nano-fluids. [19]

3. MAGNETIC FERRO-FLUID

Ferro-fluid (a combinations of the words ferromagnetic and fluid) or magnetic fluid solution, is a fluids that becomes highly magnetic against a magnetic field. [20]

It is a colloidal mixture ferro-fluid which is composed of nanoscale ferromagnetic particles or suspended ferrimagnetic particles in a carrier fluid (usually an organic solvent or water). Each small particle is coated with a surfactant to prevent clumping. When this fluid exposed to a strong magnetic field, ferromagnetic particles form large clumps, so that large magnetic particles can be separated from the homogenous mixture. The attraction between nano-particles is so weak that the Van Der Waals force is enough to avoid clumping or agglomerating. [20]

Ferro-fluids usually don't maintain their magnetization in the lack of an external magnetic field. So that, they are classified as super paramagnets instead of ferro magnets. [21]

Ferro-fluids and magneto rheological fluids (MRF) vary in particles size. [21] The particles in ferro-fluids are usually nano-particles which are suspended in the fluids by Brownian motion and they do not sediment under usual circumstances. While particles in MR fluids are mostly at the micrometer scale which are too heavy for Brownian motion and so settle over time. Therefore, ferro-fluids and MR fluids are used in really different applications. [21]

Often in the synthesis process of ferro-fluids, magnetic particles are coated by various materials that exhibit two important functions; First, they are used as wetting agents, that means by introducing strong molecular attraction between themselves and the molecules of the carrier fluids, a uniform fluid is obtained and they prevent the particles agglomeration even under strong magnetic gradients. Secondly, their lower specific weight than magnetic particles causes the average specific weight of coated particles to be less than the specific weight of uncoated ones and so reaches the suspension limit in the carrier fluid, thus forming a stable colloidal fluid. [22]

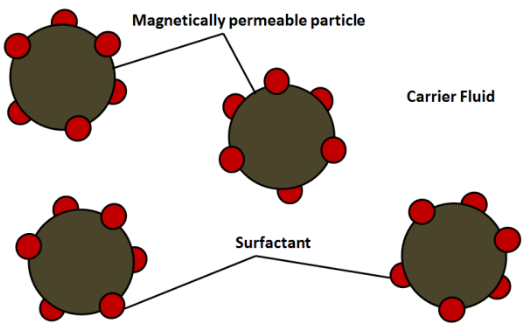

Fig.2. Magnetic nano-particles with surfactant

4. METHODS FOR MAKING NANO PARTICLES

Various methods are used to synthesize nano-particles [23]. Here, Iron oxide nano-particles was synthesized using deionized water , tetra methyl ammonium hydroxide N(CH3)4+ OH-, 37%HCL ,25%NH4OH, Iron (III) chloride , Iron (II) chloride, Cetyl tremethyl ammonium bromide (CTAB). Different methods were applied to synthesize which are briefly mentioned below;

1- We prepared toning solution in paraffin with weight percentages of 2, 4, 8, 10, and then we placed it in a high power sonicator. After sonication, we tested material with a frequency meter.

2- We prepared toning solution in paraffin with weight percentages of 0.5, 2, 4, 8, and then we placed it in a high power sonicator. After sonication, we tested material with a frequency meter.

3- We prepared ordinary toning solution in water with weight percentages of 0.2, 0.5, 1, and then we placed it in a high power sonicator. After sonication, we tested material with a frequency meter.

4- We prepared iron oxide nano-particle solution in water with weight percentages of 0.2, 0.5, then we added 0.01 g of CTAB and we placed it in a high power sonicator. After sonication, we tested material with a frequency meter.

5- We prepared iron oxide nano-particle solution in water with weight percentages of 0.1, 0.2, then we added 0.003 g of CTAB and we placed it in a high power sonicator. After sonication, we tested material with a frequency meter.

6- 0.1g of iron oxide nano-particles was dissolved in 20 mL of water and 10mL of acid. Then 0.02 g of Iron chloride was added while the solution was on the mixer. The container was sealed by aluminum foil and the nitrogen tube was immersed in the solution through a small hole. Then 10 mL of the base was mixed with 40 mL distilled water and were added slowly to the solution. The color of the solution changed from yellow to brown. Adding the base was continued till the color of solution changed to black. By placing a magnet under the container, magnetic particles settled and the liquid spilled on top of it. Afterwards we added 0.5 mL tetra ethyl ammonium to the black solid and stirred the solution well. Then we tested material with a frequency meter.

7- 2 g of iron (III) chloride was dissolved in 20 mL of water and 10mL of acid. Then 1g of Iron (II) chloride was added while the solution was on the mixer. Then 10 mL of the base was mixed with 40 mL distilled water and were added slowly to the solution. The color of the solution changed from yellow to brown. Adding the base was continued till the color of solution changed to black. By placing a magnet under the container, magnetic particles settled and the liquid spilled on top of it. Afterwards we added 0.5 mL tetra methyl ammonium hydroxide to the black solid and stirred the solution well. Then we tested material with a frequency meter. The experiment was also performed at temperature of 40 ^{o}C and 90 ^{o}C, but the best results were obtained at the room temperature.

None of the experiments 1 to 5 showed good results compared to the frequency of stable samples of magnetic fluid. However, the results of experiment 6 were slightly similar to stable samples due to the re-synthesis and stabilizing of magnetic iron oxide nano-particles. But experiment 7 provides better results. In order to study and analyze the sample accurately, various tests have been performed and the results will be presented bellow;

5. DISCUSSION

To survey the stability we used Zeta potential measurement, which shows the magnitude of the electric charge on the surface of nano-particles. Basically, streaming potential measurements proceed in such a way that the solid particles are compressed into a diaphragm, in which a liquid flowing at a constant speed experiences a pressure drop. The movement of the liquid causes part of the electrochemical double layer to shear off, creating a potential difference that is picked up by measuring electrodes. This voltage, which is measured at both electrodes, is proportional to the zeta potential of the particles. After calibration with standard materials, the measurement signal is output as streaming potential or zeta potential. According to previous studies, Zeta potentials greater than 30 mV are considered as the stability criterion of nano-fluids.[24,25] In this study the sample number 7, which showed a good frequency compared to stable samples, has a Zeta potential value of 40 mV, which is shown in figure 3. The sample inserted in a synthesized pH and other pHs using a small amount of acid to evaluate Zeta potential.

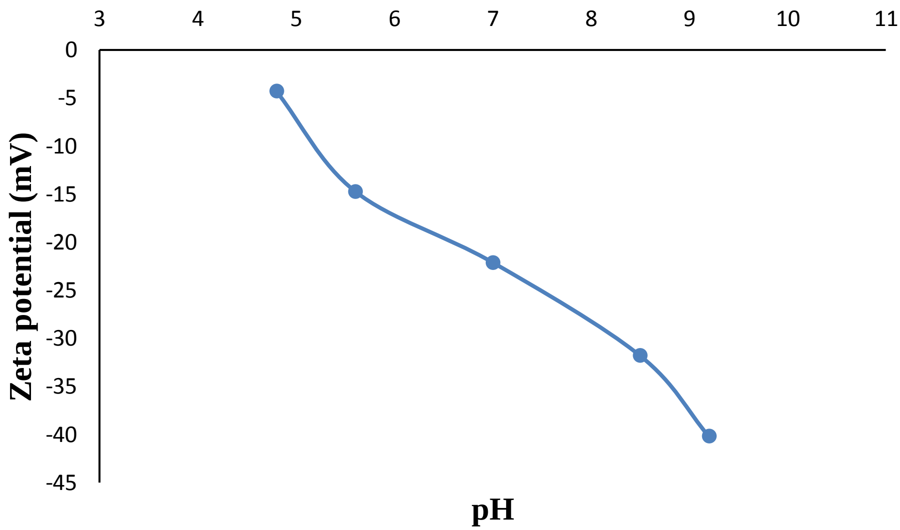

Fig. 3. Zeta potential measurement values in terms of pH changes

One method of measuring particle size is dynamic light scattering (DLS). The mechanism of light scattering is used by DLS to provide a measure of the size of particles i.e. a laser light passes through colloidal solution and then, the modulation of the intensity of scattered light as a function of time is analyzed. Also DLS is used for measuring the particle size of dispersing colloidal samples to survey the formulations stability, and to find aggregation or agglomeration [26]. Therefore, in order to evaluate the size of synthesized nano-particles in the base fluid after stability, the particle size distribution test i.e. (DLS) was used. As shown in figure 4, the average particle size is 30 nm, which is an acceptable value for long time stability [27].

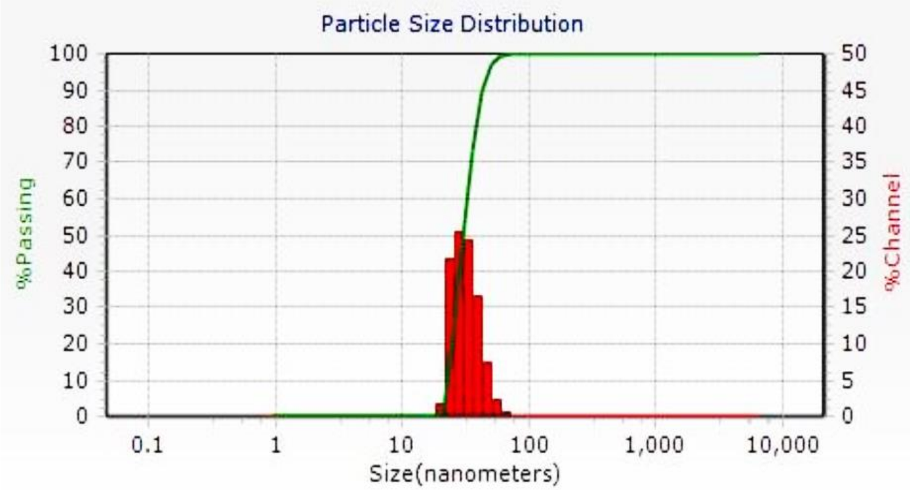

Fig.4. Magnetic nano-fluid particles size distribution

The survey of rheological behavior of the nano-fluid sample is the most important step of this study. Ferrofluids viscosity is one of the important issues of rheology. Newtonian behavior of a nano fluid can be expressed by following equation;

$$\tau = \mu\gamma \quad (2)$$

While γ, μ and τ are shear tension, dynamic viscosity and shear stress, respectively. As shown in figures 5 to 8, the viscosity values decreased exponentially in terms of shear rate at various temperatures, indicating the non-Newtonian behavior of the nano-fluid. As it be expected for non-Newtonian materials and can be seen from these figures, the viscosity values generally depends to the shear rate values. This fluid is a shear thinning fluid because the viscosity decreases as the shear rate increases. The increase of one or two viscosity values may be a measurement error.

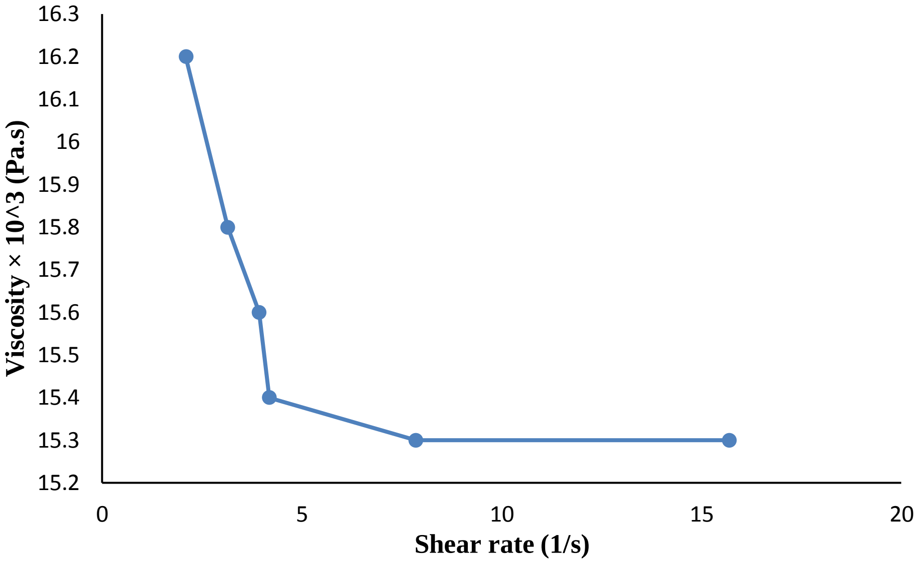

Fig.5. Viscosity values in terms of shear rate of the nano-fluid at a temperature of 20 ºC.

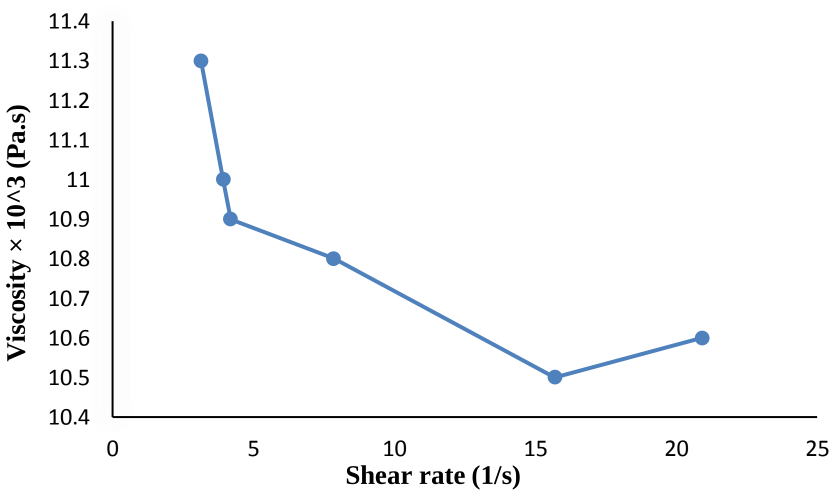

Fig.6. Viscosity values in terms of shear rate of the nano-fluid at a temperature of 35 ºC.

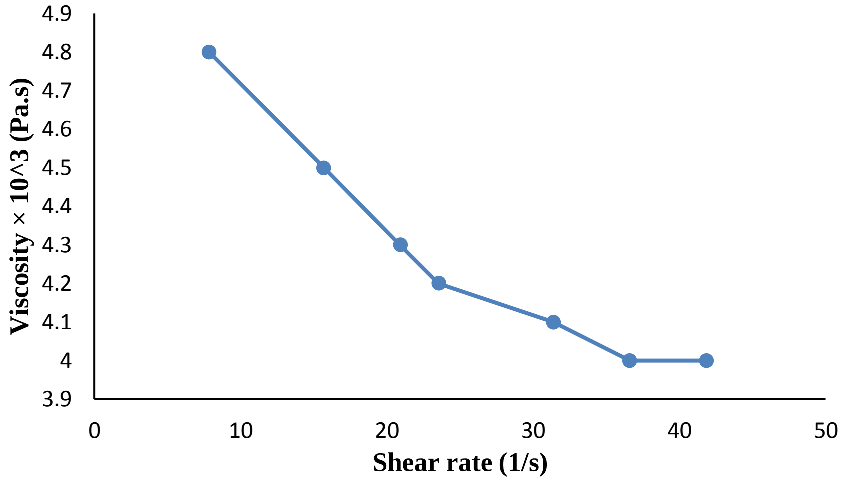

Fig.7. Viscosity values in terms of shear rate of the nano-fluid at a temperature of 50 ºC.

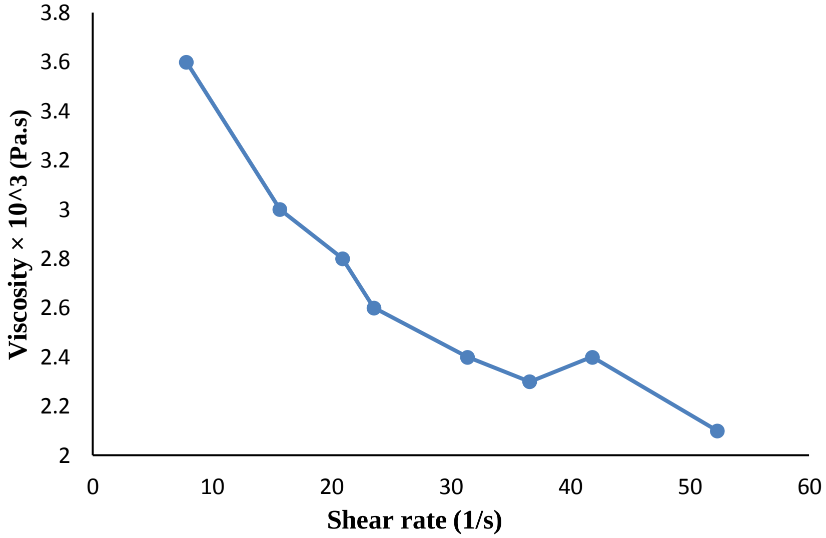

Fig.8. Viscosity values in terms of shear rate of the nano-fluid at a temperature of 65 ºC.

By fitting data, an appropriate equation corresponding to the viscosity changes in terms of concentration at different temperature was found. In the following, the general form of the equation is given and the fitting data in table 1 is presented.

$$\mu = ae^{b\dot{\gamma}} + ce^{d\dot{\gamma}} \quad (3)$$

TABLE1

Obtained coefficients from fitting corresponding to the viscosity changes in terms of concentration at different temperatures

Temperature	a	b	c	d	R^2
20 °C	3.564	-0.6007	15.18	0.000503	0.9731
35 °C	32.64	-1.348	10.88	-0.00156	0.9464
50 °C	4.799	-0.01445	0.4159	-0.02862	0.995
65 °C	2.121	-0.09065	2.65	-0.00421	0.9818

This fit equation will be used in the future experience to synthesize similar stable ferro fluids by nano-particles.

6. CONCLUSION

In this study, various surfactants are applied for colloidal stabilization among which tetra methyl ammonium hydroxide (TMAOH) showed the best performance. According to the DLS results, the average diameter of Fe_3O_4 nano-particles was about 30 nm which is appropriate for long time stability. Also the ferro-fluid viscosity is decreased significantly by the temperature increase. The rheological behavior survey showed that ferro-fluid viscosity depends completely to shear rate and decrease with it so this magnetic nano-fluid is a non-Newtonian sample. So a magnetic nano-fluid with the desired size and stability was synthesized.

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