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Optimizing Nano Metalworking Emulsions Preparation Using Response Surface Method

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K E Y W O R D S

Metalworking Fluids, Nano emulsions, Response Surface Method

A B S T R A C T

In this work paraffin oil, water and a mixture of surfactants Span20 & Tween20 are utilized for the preparation of the Metalworking Fluids (MWF). A quadratic model was developed by applying the response surface method (RSM) to relate the droplets size and emulsion stability as a response to five independent variables namely the speed and time of mixing, the concentration of the surfactant, Hydrophilic-Lipophilic Balance (HLB) value and pH value. Analysis of variance (ANOVA) was conducted; the results confirm the high significance of the regression model. The predicted values were found to be satisfactory with that experimental value. Mixing speed exerted the highest effect on the droplet size and the stability of the emulsion. The optimum conditions were found to be (the concentration = 4.75 wt.%, time of mixing = 18.12 min, speed of mixing 14998.93 rpm, pH = 10.01 and HLB = 10.87) to attained Nanoemulsion with 2 nm in size and stability of 24 days. Tool wear and surface roughness were studied at a different speed, the results have shown that the wear ratio of the bits for all selected speeds is as follow: using commercial fluid > MWFs. The metallurgical microscope images have shown that, in case using MWFs the surface of cracks between the metals and the tool is more smooth compare with other fluids.

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1. INTRODUCTION

Metalworking Fluids (MWF) is the most important industrial application of oil in water emulsions that are used in the operation of the metal process. MwFs play an important role in lubricating; cooling; corrosion control and chip removal from the cutting zone. In the last years, the Nanoemulsion has been used in many applications especially metalwork fluids, due to their high stability more economically and safety than other types of traditional emulsions [1,2]. Many researchers have found metal work fluids that are based on Nanoemulsion are the perfect solution to improve the properties of the cutting fluids [3,4]. MWFs have typically consisted of mineral oil, water, surfactant and additives. An important property for the emulsion during storage is stability, which indicates the ability of emulsions to change their properties with time [5]. Some factors affect the stability, such as the concentration of emulsion, time of mixing, speed of mixing, HLB for an emulsifier and pH value. The breakdown of the emulsion into the oil and water phase is called destabilization, and its instability comes in several forms such as sedimentation, coalescence, Ostwald ripening and flocculation [6]. There are many studies dealing with the preparation conditions of the emulsions, among them Prinderre (1997) who studied the effect of factorial design methods to decrease the number and time of the different steps required to formulate and estimate stable emulsions [7]. (Tadros et al., 2004) studied the formation and stability of an emulsion produced by using the emulsion inversion point method, and concluded that more stable Nanoemulsion can be obtained with surfactant concentration is about 8 % wt. and the ratio of oil to water is about 20/80 % wt. Liu (2006) studied the stability of paraffin oil in water using (Tween 80/Span 80) surfactant for emulsion production by inversion point manner at various emulsification temperatures [8].

Zinc borate and the lubrication oil additives have been studied to enhance the anti-wear efficiency, perfect film strength, and high resistance temperature [9]. (Khandekar et al., 2011) found that the addition of 1% Al_2O_3 nanoparticles (by volume) influence the properties of cutting fluids to the conventional cutting fluid greatly enhanced its wettability characteristics compared to pure water and conventional cutting fluid [10]. (Al- Sabagh et al., 2012) investigated the impact of adding several percentages of two types of emulsifier (anionic and non-ionic) in order to reach good industrial conditions, coupling agent, stabilizer, biocide, base oil and anti-rust additives, as well as enhancing the stability of the emulsifier system [11]. (Brinksmeier et al., 2015) studied the influence of the required and undesired changes of the MWF properties, where metalworking fluids (MWFs) are used to assure work-piece quality, to decrease tool wear, and to enhance the productivity of the process [12]. The specific chemical structure of an applied MWF should be strongly dependent on the scope of application. Even small changes in the MWF composition can extremely affect the performance of MWFs in the manufacturing processes.

The aim of this work is to prepare metalwork fluids that have special properties depending on the Nanoemulsion for lubrication and cooling application by using homogenizer with high speed.

2. MATERIAL AND METHODS

I. Materials

Liquid paraffin oil is a byproduct from the distillation of East Baghdad petroleum (Viscosity at 40°C = 36.5 cSt, Density of 1.15 gm/ml, Flashpoint > 190°C). Span 20 & Tween 20 were purchased from (Sigma-Aldrich, USA), and deionized water (DW) was used in overall experiments.

II. Nano Emulsion Preparations

For the preparation of O/W Nanoemulsion, the liquid paraffinic oil and deionized water were used as the discontinuous and continuous phase respectively. First, the surfactants with different Hydrophilic-Lipophilic Balance (HLB) are mixed with the paraffinic oil using high-speed Heidolph homogenizers (DIAX 900). Another emulsion formula was separately prepared by homogenizing DW with a mixture of surfactant and paraffinic oil under pH control by adding 0.1N NaOH solution.

III. Particle size determination

The Particle Size of the prepared Nanoemulsion was conducted using Nano Brook 90 PLUS Particle Size analyzer, Brookhaven instruments corporation (USA).

IV. Stability measurement of Nano-Emulsion (NE)

Many methods used to check the stability of an emulsion, among them the determination of the change in the droplet size with time and calculate the ratio of the separated water from the total amount of water in the emulsion [13]. The stability of the emulsion was performed keeping 50 ml of the prepared sample of emulsion at room temperature. The ratio of the separated water volume from the emulsions was reported at a particular time interval.

V. Central Composite Design (CCD)

The response surface method (RSM) is widely used in the design of the experiment (DOE) including several factors where it is required to study the combined effect of the several variables on a response surface [14-16].

In the present work, Design-Expert 7.0.0 software was followed to (DOE) of the formulation of Nanoemulsion with aid of central composite design (CCD) approach. CCD was used to select the number of experiments for the optimum process variables. The overall experiment number (N) was run out as 50 for CCD of five independent variables namely the speed and time of mixing, the concentration of the surfactant, Hydrophilic-Lipophilic Balance (HLB) value and pH of the solution. The selected process variables with their low, central and high level are presented in Table I. While the details of the conducted experiments are shown in Table II.

TABLE I: The independent variables used in the CCD and their levels.

Parameters	Notations	Range and levels		
		Low level	*Central level*	*High level*
Conc. Of surfactant (wt.%)	A	3	6	9
Mixing time (min)	B	10	15	20
Speed of mixing (rpm)	C	5000	12500	20000
pH	D	8	9.5	11
HLB	E	8	10	12

TABLE II: Details of the experiment runs.

Run No.	Conc. (wt.%)	Time (min.)	Speed (rpm)	pH	HLB	Drop. Size (nm)	Stability (Days)
1	3	15	12500	9.50	10	5.8	12
2	6	15	12500	9.50	8	7.8	9
3	9	20	5000	8.00	12	20.5	5
4	3	10	20000	8.00	8	26.2	1
5	6	20	12500	9.50	10	5.9	12
6	6	15	12500	9.50	10	2.6	16
7	6	15	12500	9.50	12	6.5	11
8	3	20	5000	11.00	8	25.8	4
9	9	20	20000	11.00	12	8.7	9
10	6	15	12500	9.50	10	2.6	16
11	3	10	5000	11.00	12	31.8	3
12	3	20	20000	8.00	8	15.2	6
13	6	15	12500	9.50	10	2.6	16
14	6	15	12500	9.50	10	2.6	16
15	9	10	5000	11.00	12	21.8	4
16	9	15	12500	9.50	10	2.9	15
17	9	20	20000	8.00	12	2.3	18
18	9	10	5000	11.00	8	20.7	4
19	3	20	20000	11.00	12	7.6	8

20	9	10	20000	8.00	8	16.6	4
21	9	20	5000	11.00	8	43.2	3
22	6	10	12500	9.50	10	6.9	11
23	3	10	20000	8.00	12	13.1	6
24	3	20	20000	11.00	8	14.1	7
25	3	20	5000	8.00	12	43.5	3
26	9	10	5000	8.00	8	49.2	3
27	9	20	5000	11.00	12	30.8	4
28	6	15	5000	9.50	10	21.3	5
29	3	10	20000	11.00	8	20.1	5
30	9	10	5000	8.00	12	38.9	3
31	9	10	20000	8.00	12	2	20
32	6	15	20000	9.50	10	14.1	9
33	3	10	5000	8.00	8	71.8	4
34	3	10	5000	11.00	8	54.1	3
35	6	15	12500	9.50	10	2.6	16
36	3	10	20000	11.00	12	25.7	6
37	6	15	12500	9.50	10	2.6	16
38	6	15	12500	9.50	10	2.6	16
39	9	20	20000	8.00	8	5.3	12
40	3	20	20000	8.00	12	6.4	11
41	6	15	12500	11.00	10	2.4	18
42	9	10	20000	11.00	8	9.2	9
43	6	15	12500	8.00	10	5.8	13
44	6	15	12500	9.50	10	2.6	16
45	9	10	20000	11.00	12	3.9	15
46	9	20	5000	8.00	8	39	4
47	3	20	5000	11.00	12	50.1	3
48	9	20	20000	11.00	8	6.3	12
49	3	20	5000	8.00	8	32.3	3
50	3	10	5000	8.00	12	46.8	3

3. RESULTS AND DISCUSSION

I. Central Composite Design Models

The quadratic model equations of droplet size and stability were obtained by applying the Design Expert Software.

$$\text{Droplet size} = [2.94 - 2.21 * A + 5.50 * B - 13.11 * C + 1.38 * D - 0.57 * E + 5.52 * A * B + 0.56 * A * C - 0.100 * A * D - 0.81 * A * E - 0.063 * B * C + 6.21 * B * D + 4.54 * B * E + 2.26 * C * D + 0.27 * C * E + 2.16 * D * E + 0.82 * A^2 + 11.49 * B^2 + 14.17 * C^2 + 0.57 * D^2 + 3.62 * E^2] \quad (1)$$

$$\text{Stability} = [13.48 + 1.31 * A - 5.52 * B + 3.38 * C - 0.53 * D + 0.56 * E - 0.62 * A * B + 1.44 * A * C - 0.31 * A * D + 0.50 * A * E + 1.00 * B * C - 1.00 * B * D - 1.13 * B * E - 0.25 * C * D + 1.19 * C * E - 0.81 * D * E$$

Where A, B, C, D, and E are representing the concentration of the surfactant (wt. %), time of mixing (min), speed of mixing (rpm), pH value and HLB value respectively. The positive sign in these equations refers to an increase of independent variables lead to an increase in the response size of the droplet and an increase in the stability of emulsion contrariwise the negative sign refers to the opposite effect.

II. ANOVA Analysis

According to the ANOVA analysis for droplet size and stability of the obtained emulsion, resulting from the models F-values are 13.12 and 13.67, which refer to the two models are significant. There is only a 0.01% chance that a "Model F-Value" of this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicates the model terms are significant. When the values of “Prob > F” are greater than 0.1000 this indicates that the model terms are not significant. In this case, the variables (C, AB, BD, DE and C^2) are significant model terms for droplet size and the variables (A, C, AD, DE, A^2, D^2) are significant model terms for stability [17]. The ANOVA results for the droplet size and the stability of the emulsion models are shown in Table III and IV.

TABLE III: ANOVA regression model for process Analysis of Variance for droplet size.

source	Sum of squares	Df	Mean square	F value	p-value prob>F	
Model	176.89	20	8.84	19.73	< 0.0001	significant
A-Conc.	1.60	1	1.60	3.56	0.0691	
B-Time	0.16	1	0.16	0.35	0.5563	
C-Speed	37.50	1	37.50	83.67	< 0.0001	
D-PH	0.45	1	0.45	1.00	0.3255	
E-HLB	0.35	1	0.35	0.78	0.3848	
AB	2.22	1	2.22	4.96	0.0339	
AC	0.52	1	0.52	1.16	0.2906	
AD	1.355E-005	1	1.355E-005	3.024E-005	0.9957	
AE	0.44	1	0.44	0.98	0.3309	
BC	0.30	1	0.30	0.68	0.4166	
BD	2.51	1	2.51	5.61	0.0247	
BE	1.16	1	1.16	2.59	0.1183	
CD	1.61	1	1.61	3.60	0.0679	
CE	0.38	1	0.38	0.86	0.3618	
DE	1.94	1	1.94	4.33	0.0465	
A^2	0.011	1	0.011	0.024	0.8772	
B^2	0.41	1	0.41	0.91	0.3482	
C^2	10.53	1	10.53	23.49	< 0.0001	
D^2	0.051	1	0.051	0.11	0.7389	
E^2	0.75	1	0.75	1.66	0.2072	
Residual	13.00	29	0.45			
Lack of Fit	13.00	22	0.59	5.43	0.0621	Not significant
Pure Error	0.000	7	0.000			

TABLE IV:ANOVA results from the stability of the emulsions.

Source	Sum of squares	Df	Mean square	F value	P-value Prop>F	
Model	40.04	20	2.00	17.33	< 0.0001	Significant
A-Conc.	0.85	1	0.85	7.33	0.0113	
B-Time	0.079	1	0.079	0.69	0.4139	
C-Speed	6.57	1	6.57	56.93	< 0.0001	
D-pH	0.14	1	0.14	1.21	0.2798	
E-HLB	0.16	1	0.16	1.36	0.2798	
AB	0.099	1	0.099	0.85	0.2527	
AC	1.58	1	1.58	13.64	0.3633	
AD	0.096	1	0.096	0.83	0.0009	
AE	0.12	1	0.12	1.07	0.3691	
BC	0.37	1	0.37	3.18	0.3099	
BD	0.42	1	0.42	3.66	0.0851	
BE	0.31	1	0.31	2.67	0.0658	
CD	1.551E-004	1	1.551E-004	1.343E-003	0.1132	
CE	1.28	1	1.28	11.10	0.9710	
DE	0.70	1	0.70	6.06	0.0024	
A^2	2.231E-003	1	2.231E-003	0.019	0.0201	

B^2	0.15	1	0.15	1.32	0.8904	
C^2	2.58	1	2.58	22.30	0.2602	
D^2	0.20	1	0.20	1.75	< 0.0001	
E^2	0.57	1	0.57	4.94	0.1966	
Residual	3.35	29	0.12			
Lack of Fit	3.35	22	0.15	5.431	0.0581	Not significant
Pure Error	0.000	7	0.000			

The actual and predicted values of the droplet size and the stability of the emulsion are plotted in Figures (1) and (2) respectively. Good agreements between the actual and predicted values are shown in these figures. The above figures show the experimental values are a good fit with the predicted values that are related to the droplet size and stability.

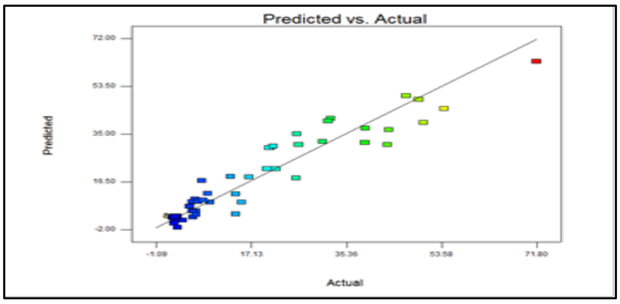


Figure 1: The predicted verses the actual values of the droplet size.

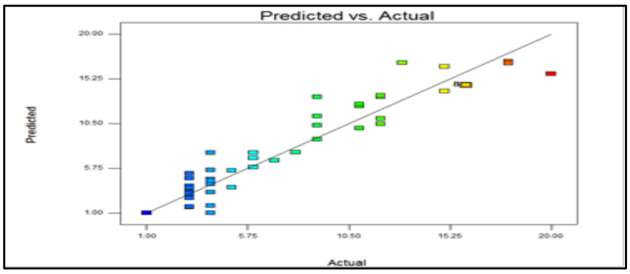


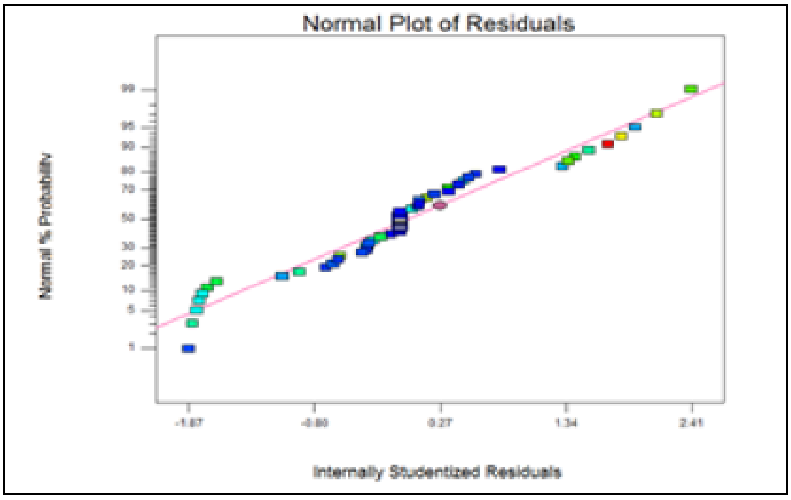
Figure 2: The predicted verses the actual values of the stability.

The suitability of models is examined by design factor R^2; these models gave high R-squared and Adj R-squared values. When the values of (R) are higher than 0.8 this indicates there is good interaction between the response (droplet size and stability) and the independent parameters [18]. The values of the coefficients of determination are close to one, this refers to a good fit of the models with the experimental data and both models are significant, where the difference between the Adj R-squared and the Pred-R squared less than 0.2 [19]. The values of R for two models are shown in Table V.

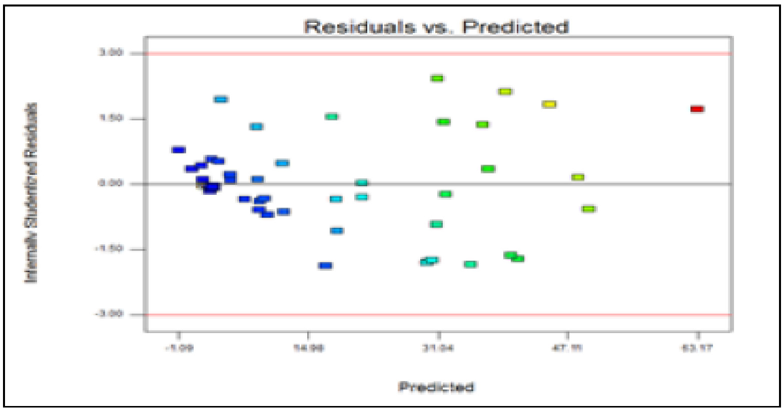
TABLE V: The values of “R" for the two models.

Types of “R"	Droplet size values	Stability values
R-Squared	0.9316	0.9228
Adj R-Squared	0.8844	0.8696
Pred R-Squared	0.7474	0.7219

The shape of probability plots in the normality case was presumed as a straight line composed of combined points with each other. This fact is explained using the plots of normal probability for the droplet size and stability as shown in Figures (3, a) and Figure (4, a) respectively. The authenticity of the models appears in the obtained results from the ANOVA analysis as well as from the examination of the fits versus residuals values, as shown in Figure (3 b) and Figure (4 b) respectively. For the two responses in these Figures, it is found that the modality is decreased in fits as the residuals decreases and increases in the fits as the residuals [20].

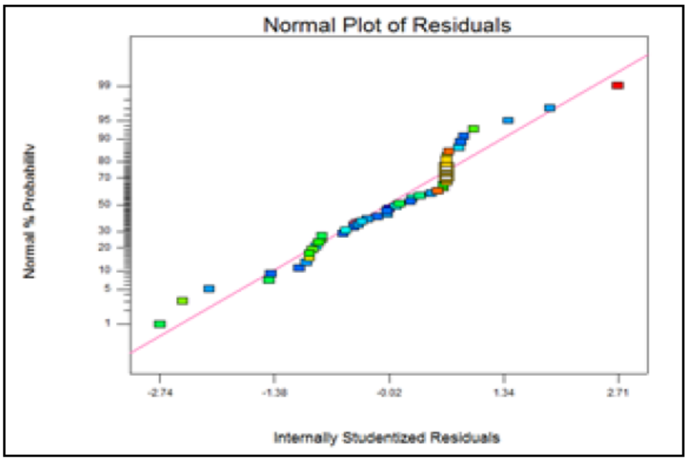


a)

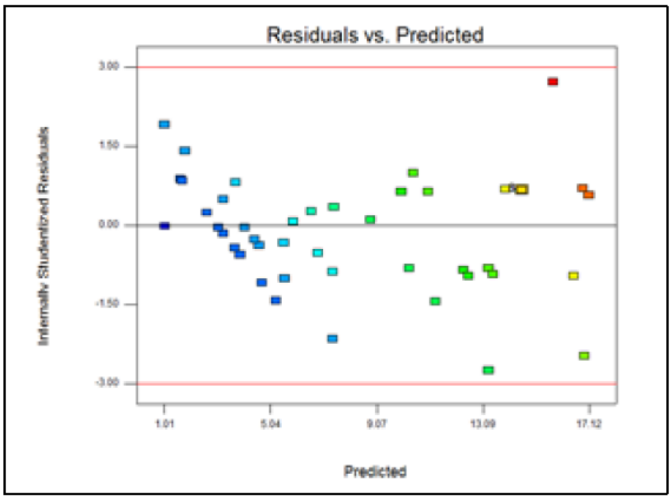


b)

Figure 3: (a) Normal probability and (b) Residual vs. predicted for the droplet size.



a)



b)

Figure 4: (a) Normal probability and (b) Residual vs. predicted of the stability.

III. Estimation of the Optimum Conditions

The main objective of the expert design program is to know the best conditions of the independent variables that can be used in this work, such as the concentration of surfactant, HLB value, speed of mixing, time of mixing and pH value to get smaller droplet size and higher stability for the Nanoemulsion that used as cutting fluid. These parameters have to be in ranges leading to the droplet size gets to a minimum value, and the "stability" gets to a maximum value. The decrease in droplet size with the concentration of surfactant is shown in Figure (5a). The size of the droplet is decreased with increasing the surfactant concentrations from 3 to 9 wt%. Figure (5b) shows the influence of droplet size is not significant with an increase in the time of mixing from 10 to 30 min. The droplet size decreases with an increase in the speed of mixing from 5000 to 20000 rpm, as shown in Figure (5.c), where Figure (5.d and 5.e) show a medium influence of the droplet size with both pH and HLB.

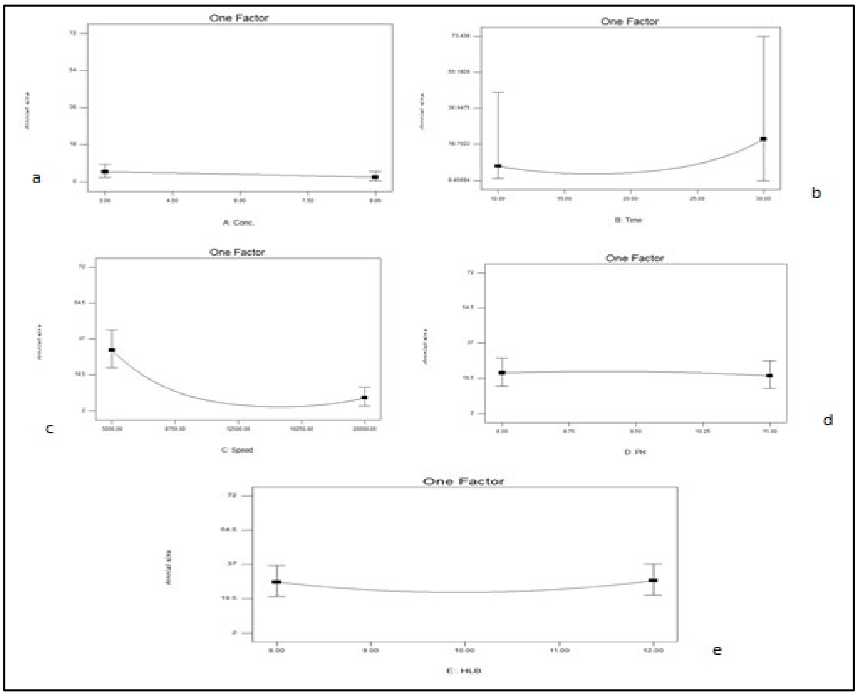


Figure 5: Influence of independent variables (a) Effect of surfactant concentration (b) Effect of time (c) Effect of speed on droplet size (d) pH value (e) HLB value on the droplet size.

The software Design-Expert 7.0.0. was used and the size of the minimum droplets of 2 nm and maximum stability was attained with high desirability = 1 at the following optimum conditions: concentration of surfactant = 4.75 %, time of mixing = 18.12 min, speed of mixing 14998.93 rpm, pH =10.01 and HLB =10.87, as shown in the Figure (6). Under these conditions, three experimental runs were conducted and droplet size was measured and found to be 1.9 nm, which is in good agreement with the predicted value (as appears in Figure 7 and Table VI).

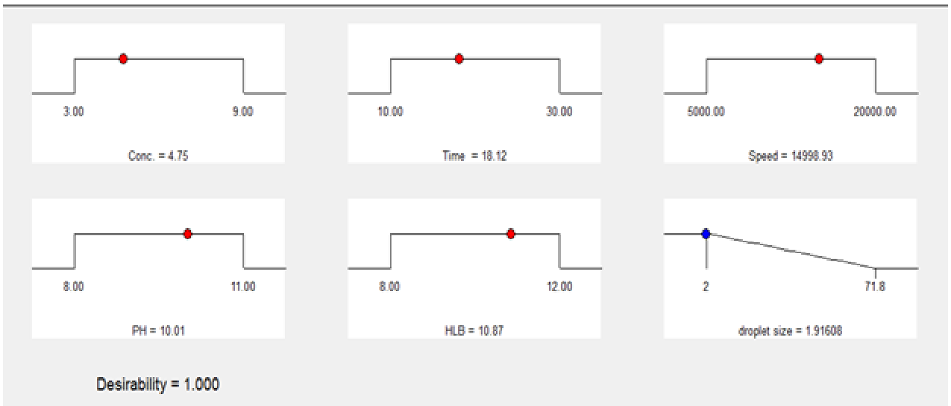


Figure 6: Optimum conditions for preparation Nanoemulsion.

TABLE VI:The result of the three runs.

Run	Eff. Diameter (nm)
1	1.6
2	2
3	2
average	1.9

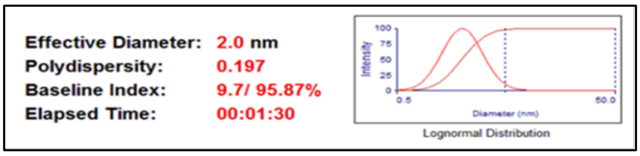


Figure 7: Diameter measure by DLS based on the optimum conditions.

IV. Influence of Independent Parameters on the Droplet Size and Stability of Emulsion

The five independent variables and their interaction have different influences on the size of droplets. The percentage of contributions of each variable were shown in Figure (8). It was found that the speed of mixing (C) has the highest effect followed by the combined effect of the time of mixing with pH value (BD), the concentration of surfactant with the time of mixing (AB) and the square term of mixing speed (C^2) in addition to other variables which have the lowest effect. The percentage contributions (PC) are calculated according to Equation (3), where (S) represent the value of each term from ANOVA analysis (A, B, C, D, E, AB, AC, AD, AE, BC, BD, BE, CD, CE, DE, A^2, B^2, C^2)

$$\mathrm{pc} = \frac{s}{\sum s} * 100 \qquad (3)$$

S: Value of each term from ANOVA analysis

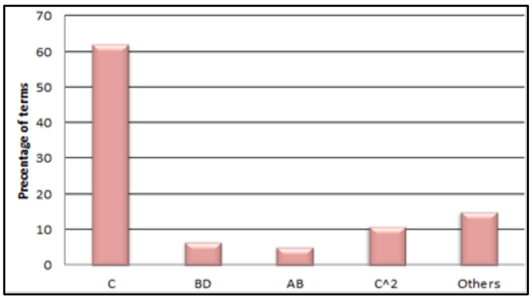


Figure 8: The percentage of contributions of independent parameters that affect the droplet size of the emulsion.

Figure (9) shows the effect of the independent variables and their combined effect on emulsion stability. The same trend was found, where the mixing speed had the highest effect on the stability of the emulsion while the other parameter as follows: the concentration of surfactant (A), the combined effect of HLB with the speed of mixing (CE) and the square of the speed mixing and HLB value.

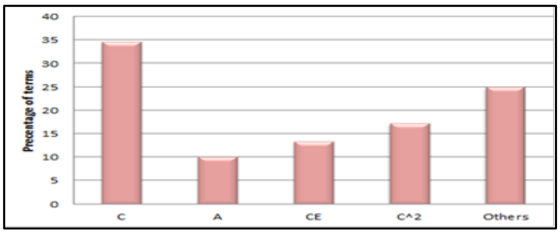
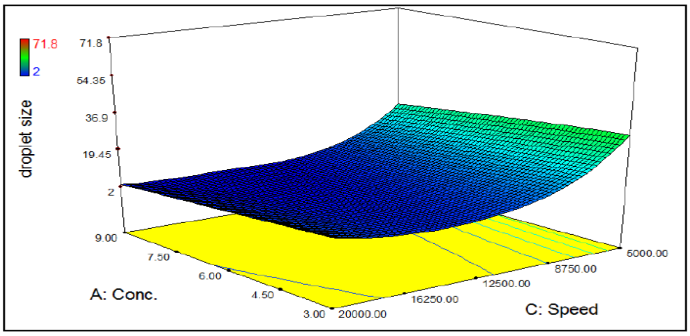


Figure 9: The percentage contributions of the independent parameters that affect the stability.

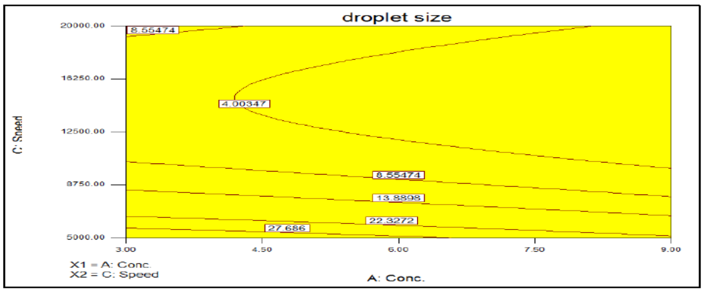
According to the stability of the emulsion shape, Figure (9) explain the distribution of the variables from the highest effect to the lowest. In the same case of droplet size, the speed of mixing (C) has the highest effect on stability. This leads to set the influence of the important parameter on the two responses besides to concentration of surfactant (A). Other variables such as the combined influence of HLB value with the speed of mixing (CE) and square value for speed and HLB.

V. Effect the concentration of surfactant on the droplet size

Surfactant is an important parameter that can enhance the interfacial films around the droplets. However, the lipophilic and hydrophilic emulsifiers are aligned with each other, this result is leading to strengthening the emulsifier film when using the mixture surfactant [21]. Figures (10, a, and b) show the 3D and 2D contour plots of the interaction effect of surfactant concentration with the speed of mixing, while the other parameter is constants. It can be seen that the size of the droplet decreases, as the concentration of surfactant increases from 3 to 9 wt.%. This behavior occurs because the high concentration of surfactant in the bulk is reducing the surface tension between paraffin oil and water, which is sufficient to allow rapid diffusion and adsorption of the surfactant to the newly formed and more stable droplets. Similar findings were reported by Kentish et al. [22].



a)



b)

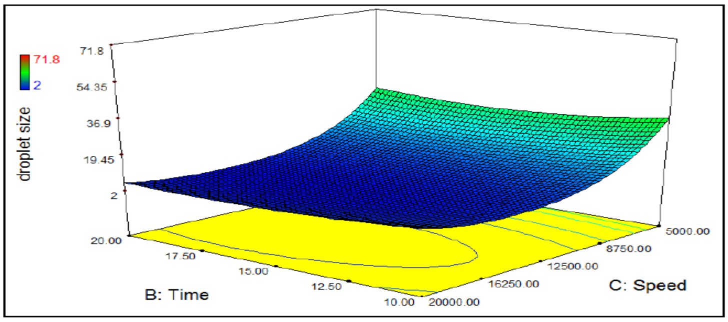
Figure 10: The effect of surfactant concentration and mixing speed on the droplet size (a) 3D plot and (b) 2D contour.

VI. Effect of speed and time of mixing on the droplet size

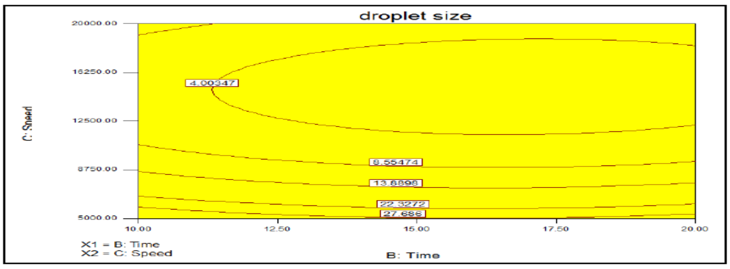
The speed and time of mixing directly influence the dispersion of oil in water. Figure (11, a. b) shows the interaction effect of time and speed of mixing on the droplet size. This Figure reveals that the increase in speed from 5000 to 20000 rpm with increasing the time from 10 to 20 min have led to decreasing the droplet size to 2 nm. The same results were obtained by Gardouh et al., [23]. This is due to the increase of mixing speed, is generating a shear force that breaks the coalescence and also leads to better droplet breakup. This is necessary to disperse the oil phase in the water phase and leading to amplifies the mechanism of rupture, which leads to a decrease in the droplet size [24].

VII. Influence of pH on the droplet size of the emulsion

Figure 12 shows the interaction effect of pH with speed on the droplet size. It can clearly be seen that when pH is increased from 8 to 11 the size of the droplet is decreased to 2 nm at pH 10. The same results were obtained by Ashrafizadeh and Hoshyargar [13] and Ozturk [25]. This is because the surface tension of emulsion is rapidly decreasing at the alkaline solutions, where the high negative charge of the oil/water interface is leading to the electrostatic repulsion between the droplets, which is strong enough to overcome the interactive interaction between the droplets.

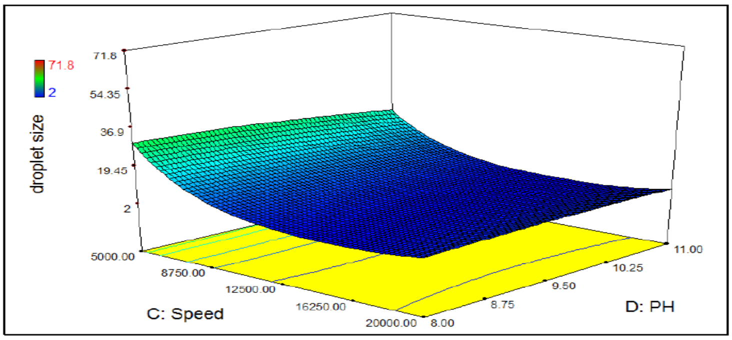


a)

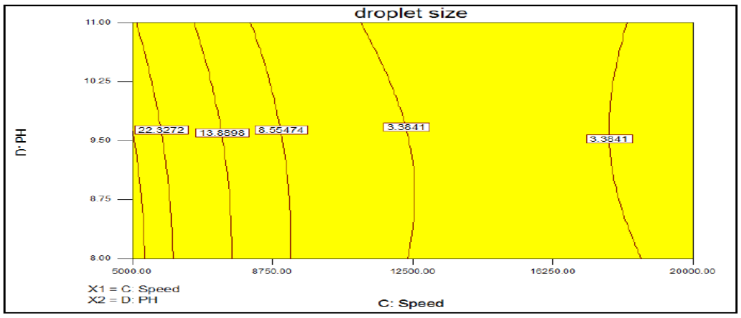


b)

Figure 11: The interaction effect of the time and speed of mixing on the droplet size (a) 3D plot and (b) 2D contour.



a)

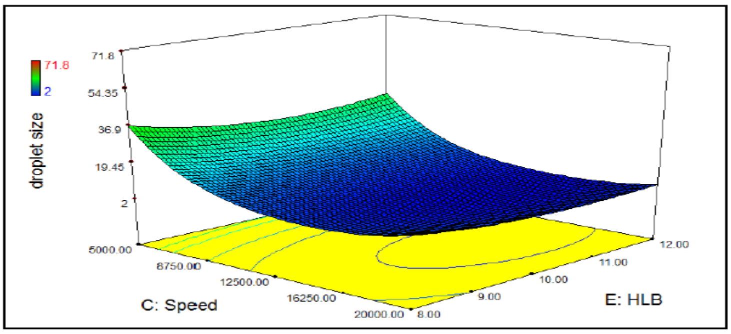


b)

Figure 12: The interaction effect of pH with speed on the droplet size (a) 3D plot and (b) 2D contour.

VIII. Influence of HLB on the droplet size of the emulsion

The HLB value of the surfactant is an important variable in the emulsion preparation because the type of the formed emulsion is strongly dependent on this value. Thus, producing emulsion with specific properties required to select a suitable HLB ratio. Figure (13. a, b) shows the combined effect of HLB and mixing speed on the droplet size of the emulsion. It can clearly be seen that when the ratio of HLB increases from 8 to 12 an oil-water emulsion is formed with Nano size of droplets about 2 at HLB equal to 10.87. The parallel line in the 2D contour Figure indicates the absence of the interaction effect of HLB value and mixing speed on the size of droplets.



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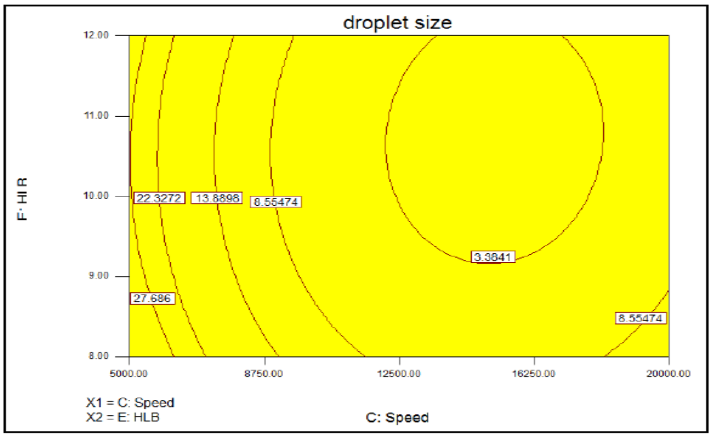
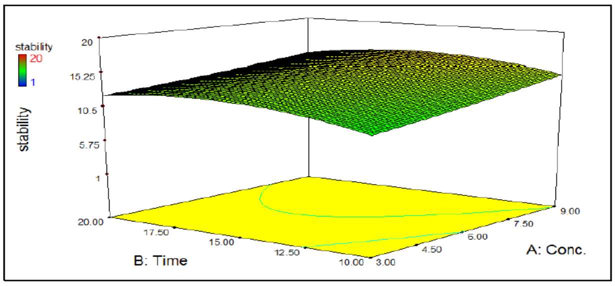


b)

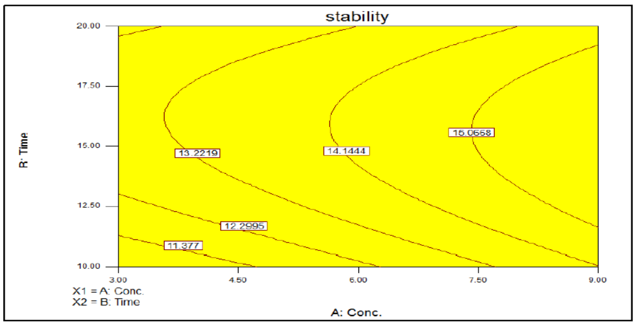
Figure 13: The interaction effect of HLB value and mixing speed on the droplet size(a) 3D plot and (b) 2D contour.

IX. Effect of surfactant concentration on the stability

Stability is an important property of the emulsions. To enhance it there are many methods that can be used, one of them is to increase the concentration of surfactant. This increase is leading to reduce creaming, flocculation phenomenon and decrease the droplet size. The Nano-size is more kinematical stable, which improves the stability of the emulsion, this agrees with the results of Kumar and Mandal [26]. The slow change in the droplet size with time as a result of increasing the range of surfactant is leading to enhance stability [27]. Figure (14. a, b) shows the effect of surfactant on Nano-emulsion stability.



a)

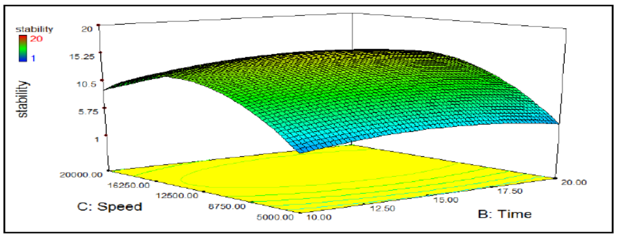


b)

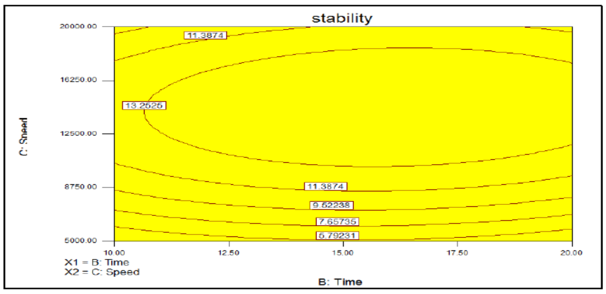
Figure 14: The influence of surfactant concentration and time of mixing on the stability of emulsion (a) 3D plot and (b) 2D contour.

X. Effect of speed and mixing time on stability

Figure (15. a, b) shows the combined effect of speed and mixing time on the stability of the emulsion. It can be seen that increasing the mixing speed increases the stability of the emulsion. This is because the raise of mixing as a result of applying high mechanical energy is leading to break the large droplets into small droplets, which will cause the time of separation of NE to be longer, and thus the system will be more stable [28].



a)

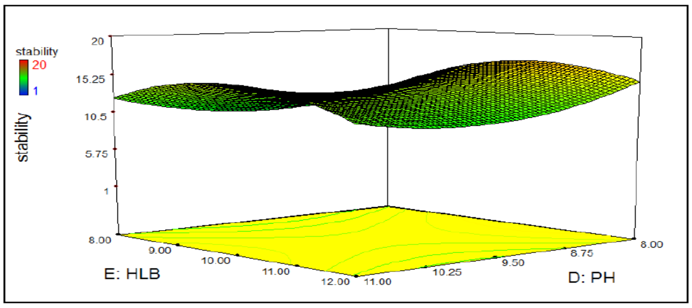


b)

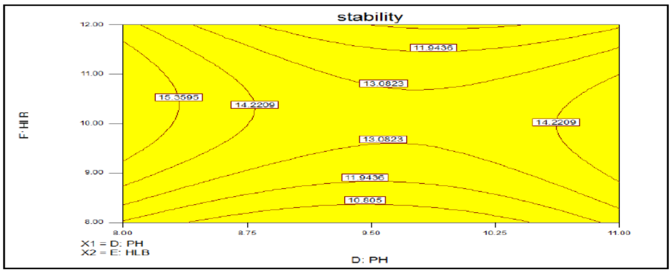
Figure 15: The influence of speed and time of mixing on the stability of emulsion (a) 3D plot and (b) 2D contour.

XI. Effect of pH and HLB values on the stability of the emulsion

The pH of the continuous phase is related to the electrostatic stability of the Nano-emulsion. It prevents the droplets from becoming too close to each other. This is called the energy barrier, so the decline in electrostatic repulsion reduces this barrier, which will not be enough to prevent the droplet to aggregate [29]. Figure (16. a and b) show the influence of pH and Hydrophilic-Lipophilic Balance HLB values on the stability of the emulsion. It can be noticed the increase in the pH is increasing the stability of the emulsion; this is due to the surface charge of the droplets may produce repulsive force with the other droplets versus flocculation and coalescence. This finding agrees with the results in the literature [30]. According to the HLB value, as the HLB increase to 9. The stability of emulsion will be increased, this is because the system will be a more viscous and smaller size of droplets [26].



a)



b)

Figure 16: The influence of pH and HLB values on the stability of the emulsion (a) 3D plot and (b) 2D contour.

XII. Nanoparticle Dispersions in Nano- Emulsion

The main role of Metalworking fluids (MWFs) is to reduce wear and friction of moving surfaces, in addition, to dissipate heat. These properties can be improved by added additives [31]. In the literature, the size of NPs in the range of 2-120 nm and a purity of 99% was added to the MWFs [32]. In this work, CuO with an average particle size of 20-40 nm was added to the NE. The CuO nanoparticles were dispersed in NE in concentrations of 0.1-0.4 wt%. The addition of the CuO is conducted as found by Wu *et al.,* [33]. The commercial and prepared MWFs with added nanoparticles as an additive were investigated using a turning machine. A turning machine is used in the forming of the metals to get the required shapes. The forming process was carried out by removing the part of the metals using different cutting pens by turning them around the axis of metal with the cutting pen moving in a linear motion. Figure 3.8 shows the turning machine. The main piece in the turning machine is the tool pit, which is designed with different shapes. Figure 3.9 shows the tool bit used in the turning machine. Wear measurement was done by weighing carbide pit samples before and after the tests. Table VII shows the wear ratio of the bits using cutting speeds (260, 370 and 540 m/min) and three types of MWFs. This ratio is calculated from the following equation:

Wear ratio% = (Weight of carbide bits before the test - Weight of carbide bits after the test)/ (Weight of carbide bits before test).

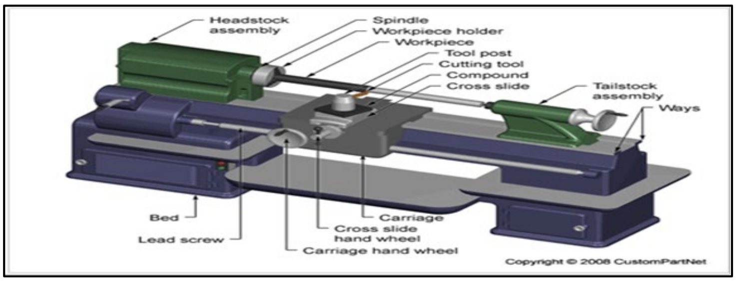


Figure 17: Turning Machine.

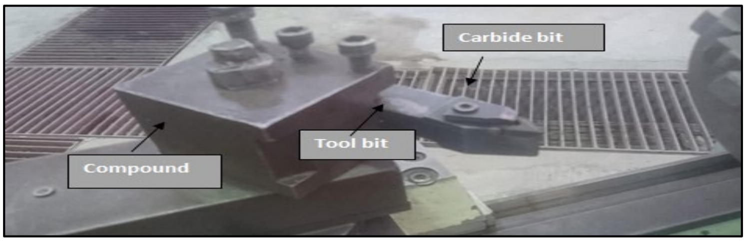


Figure 18: Acutting tool in the turning machine.

TABLE VII: The wear ratio for the three tests.

Test No	Speed of cutting	Wear ratio%		
		Commercial MWFs	*NE- MWFs*	*NE with NPs-MWFs*
1	260	20.9	8.3	6.2
2	370	25.1	9.3	7.76
3	540	38.6	9.6	8.56

According to Table VII, the wear ratio of the bits is as follows for all the speeds: using commercial fluid > Nano-emulsion without CuO > Nano-emulsion with CuO. These results could be due to the use of Nano-fluids, which act as a third body between the workpiece and the cutting tool which reduce the fraction and temperature formation in the cutting zone this finding is agreed with the results in the literature [34]. During the machine operation, the fraction occurs between the metal and the cutting tool leads to wear the surface and scratches the cutting tool. This reduces the effectiveness of the work, which requires shutting the machine down. The metallurgical microscope was used to observed the worn surface of the test sample to study the effect of the cutting fluids on the surface. Figure (4.20) shows the formation of the cracks as a result of fractions. In the image (c) a silverpoint on the surface refer to the area of cracks were seen and a large area of cracks occurs when using commercial fluids, this is because of the high fraction between the workpiece and cutting tool. Those areas are gradually reduced to a few points on the surface when using Nano-emulsion without CuO, as can be seen in image (a). Using the Nano-emulsion with CuO, image (b), the small droplet makes the surface smoother, which facilitates the sliding of the two surfaces on each other and reduces friction in the cutting zone. In order to reduce these points, Nano-particles were mixed with the base oil, which will eliminate these points and works as a third body that decreasing the fraction and makes the bits suitable for many uses. These results are agreed with the literature [33].

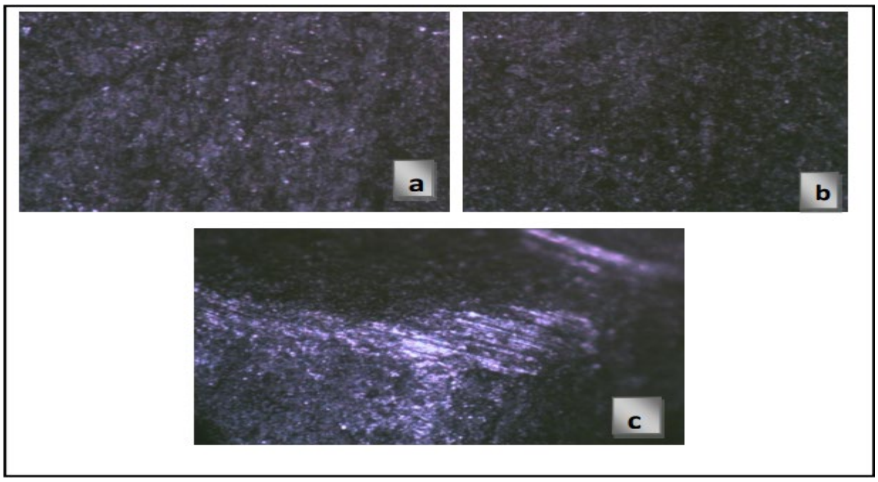


Figure 19: Picture of bits surface by metallurgical microscope for (a) using Nano-emulsion without NPs (b) using Nano-emulsion with NPs (c) using commercial fluids.

4. CONCLUSION

The results of this work which aimed to formulate useful Metalworking fluids (MWFs), from paraffin oil, the mixture of surfactant (Span20 and Tween20) and water at high homogenization speed would reveal the following:

1) The CCD method was applied to develop a quadratic model for the droplet size and stability of the emulsion. The predicted values were calculated using the quadratic models and they were in good agreement with experimental findings.
2) The droplet size of Nano-emulsion decreases when increasing the speed of mixing, the concentration of the surfactant, the time of mixing, and pH value with the moderate value of HLB.
3) Mixing speed exerted the highest effect on the droplet size.
4) The optimum conditions were found to be (the concentration = 4.75 wt.%, time of mixing = 18.12 min, speed of mixing 14998.93 rpm, pH = 10.01 and HLB = 10.87) to attain Nano-emulsion with 2nm in size.
5) The operation of the three types of fluids shows decreasing in the wear of cutting tools from 38.6 wt.% to 9.6 and 8.56 wt.% when compared with the investigated commercial fluid, Nano-emulsion and Nano-emulsion with CuO Nanoparticle, respectively.
6) Reduce the roughness of the carbide bit when using Nano-emulsion with and without Nano-particle.

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