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DEVELOPMENT OF CUTTING FLUID FROM FALSE WALNUT OIL USING DESIGN OF EXPERIMENT

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Abstract: In this study, false walnut oil based cutting fluid was formulated for machining alloy of steel materials. The physiochemical properties of the oil as it affects the formulation of cutting fluid were investigated. Emulsifier, biocide, corrosion inhibitor and antioxidant were used as additives in the formulation process using design of experiment method. Design expert software version 7.0 was used to carry out the analysis of variance and optimized the pH value of the cutting fluid formulated. The optimized pH value for the formulated cutting fluid was 8.029 with a viscosity of 2.31 mm^2/s. The optimum value was obtained using 9.35% of emulsifier (A), 0.97% of biocide (B), 10.61% of anti-corrosion (C), and 0.64% of antioxidant (D).

Keyword: ANOVA, cutting fluid, additives, formulation, oil

1. INTRODUCTION

Approximately 85 % of lubricants being used around the world are mineral-based oils (Loredana et al, 2008) and the enormous use of mineral-based oils created many negative effects on environment. The major negative effect is particularly linked to their use, which results in surface water and groundwater contamination, air pollution, soil contamination, and consequently agricultural product and food contamination (Birova et al, 2002). Hence, there is a growing public interest in environmentally friendly lubricants due to awareness of environmental problems associated with conventional mineral oil-based lubricants (Choi, et al, 1997). Even though the toxicity of lubricants is low, their accumulation in the environment may cause damage in the long run. A large proportion of the lubricants pollute the environment either during or after use. In many countries, there are well-defined guidelines and legislations for environmentally friendly lubricants (Zeng et al, 2007). Mineral oil-based lubricants contain many kinds of additives such as antioxidant, antiwear, detergent, dispersant, antifoam, extreme pressure agent, friction modifier, and viscosity improver. Some of these additives are toxic and harmful to human health, wildlife, and environment (Kleinova, et al, 2008). The environmental and toxicity issues of mineral oil-based lubricants and their additives as well as their rising cost related to a global shortage have led to renewed interest in the use of vegetable oils (Lathi and Mattiasson, 2007), such as soybean oil, canola oil, sunflower oil, coconut oil, sesame oil, castor oil, etc. as environmentally friendly lubricants and industrial fluids (Rudnick, 2009). Fox and Stachowiak (2007) observed that low temperature properties and narrow range of available viscosities limit the potential application of vegetable oil as industrial lubricants. To address these limitations, Lou (1996) suggested three methods namely: (i) reformulation of additives, (ii) chemical modification of vegetable-based oils, and (iii) genetic modification of the oil seed crop to enhance the performance of vegetable oil lubricants. Lawal et al (2012) carried out a thorough review on the application of vegetable oil-based cutting fluids in machining ferrous metals. The review highlighted the advantages of cutting fluids and their performances during machining processes with respect to various grades of ferrous metals. In this study, properties of false walnut oil were investigated and the use of design of experiment method was adopted in the formulation of false walnut oil based cutting fluid for machining of alloy of steel materials.

2. MATERIALS AND METHODS

2.1 Materials

The following materials were used in the formulation of cutting fluid from false walnut (*canarium schweinfurtti*) oil sourced from Jiblik, Plateau State in Nigeria. (i) false walnut oil, (ii) distilled water, (iii) additives (emulsifier, biocide, antioxidant and corrosion inhibitor).

2.2 Method

The following methods were involved in the formulation of false walnut oil based cutting fluid.

1. **Properties:** The following physicochemical properties of the false walnut oil were investigated using appropriate method: (i) flash point using flash point tester cup (ii) smoke point using thermometer, (iii) pH value using pH meter, (iv) viscosity @ 40°C using viscometer, (v) iodine value using chloroform, wij's solution, potassium iodide and sodium thiosulphate solution (vi) peroxide value using glacial acetic acid and chloroform (vii) pour point, (viii) free fatty acid using (vix) free fatty composition using gas chromatography.

2. **Formulation method:** The formulation of the cutting fluid involved the use of design of experiment (full factorial method) of four factors at two levels. These levels involved the combination of the upper and lower values which are identified using a (+) and (-) symbols respectively. It therefore, follows that for formulation design, a total of 16 experiments were carried out for the oil, from which the best combination of factors will be determined. The factors and the levels examined according to Muniz et al (2008) except the value of corrosion inhibitor are shown in Table 1.

Table 1: Factors and levels examined in the factorial planning

Factors	Symbol	Level Min	Level Max
Emulsifier	A	(-) 8.0	(+) 12.0
Biocide	B	(-) 0.5	(+) 1.0
Corrosion inhibitor	C	(-) 10.0	(+) 15.0
Anti-oxidant	D	(-) 0.5	(+) 1.0

The influence of the main variables on the output response and their interactions are estimated by mean of the difference between the means of the output responses. The statistical significance resulting from these influences can be determined based on an accepted standard confidence interval of 95%, using the analysis of a normal distribution graph. The output influences or effects of a particular factor variation are used statistically to determine the outputs of a factorial planning. The dependence of the output or response analyzed (Y_r) with the experimental variables (S_i) can be approximated to a polynomial expression:

$$Y_r = C_0 + \Sigma C_i S_i + \Sigma C_{ij} S_i^2 + \Sigma C_{ij} S_i S_{ij} \quad (1)$$

where C_0 is a constant, C_i and C_{ij} are coefficients, S_i is the independent variable and S_{ij} represents the interactions thereof (Montgomery, 2009). The factorial planning consisting of all the various combinations of the factors investigated for the false walnut oil are shown in Table 2.

Table 2: Experimental matrix of the full factorial planning

Experimental run	Factors examined			
	A (%)	B(%)	C(%)	D(%)
1	8	0.5	10	0.5
2	12	0.5	10	0.5
3	8	1.0	10	0.5
4	12	1.0	10	0.5
5	8	0.5	15	0.5
6	12	0.5	15	0.5
7	8	1.0	15	0.5
8	12	1.0	15	0.5
9	8	0.5	10	1.0
10	12	0.5	10	1.0
11	8	1.0	10	1.0
12	12	1.0	10	1.0
13	8	0.5	15	1.0
14	12	0.5	15	1.0
15	8	1.0	15	1.0
16	12	1.0	15	1.0

The formulation of the oil-in-water emulsion cutting fluid from false walnut oil with the following additives (i) emulsifier (tween – 80); (ii) corrosion inhibitor (banana sap); (iii) antioxidant (butylated hydroxytoluene); (iv) biocide (triazine); (v) deionized water. To obtain emulsions with bubble sizes having average diameter of a couple of few micrometers, a powerful homogenizer was improvised using a stirrer attached to a drilling machine chuck. A spindle speed of 1850 rpm was used to stir all the emulsion samples for 10 minutes.

The pH values were determined for each run, this is necessary because any pH value for any of the run that is less than 7.0 is acidic in nature and cannot be used as cutting fluid for alloy of steel materials (Lawal, et al 2013). Hence, pH value in this study is considered as the response or output variable. The stability which was investigated for 24 hours and corrosion resistance of the cutting fluid formulated were equally determined at the Chemical Laboratory of the Department of Chemistry of the Federal University of Technology, Minna according to Alves and Olive (2006). Figure 1 show the flow chat of the mixing process of emulsion of oil-in-water cutting fluid formulation.

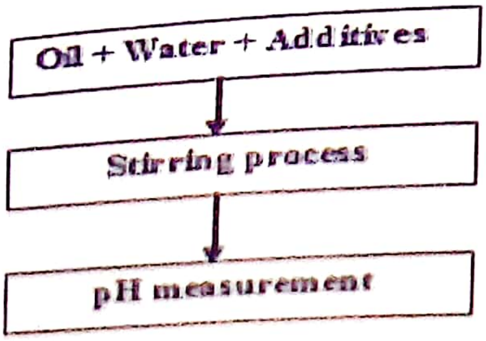

Fig. 1: Flow chat for the mixing process

3. RESULTS AND DISCUSSION

3.1 Properties: The results obtained for all the properties of false walnut oil investigated in this study are shown in Table 3.

Table 3: Physiochemical properties of false walnut oil

S/N	Property	Value
1	Flash point	328°C
2	Smoke point	180°C
3	pH value	7.01
4	Viscosity @ 40oC	$6.82mm^2/s$
5	Iodine value	85.5 g/100g
6	Peroxide value	6.20 mmol/kg
7	Free fatty acid	3.95 mg/KOH/g
8	Pour point	-6°C

The result of fatty acid composition of false walnut oil using gas chromatograph analysis is shown in Table 4. The fatty acid profile indicates the dominant fatty acids in the oil. The false walnut oil consists of 52.85% of unsaturated acids and 43.17% of saturated acids.

Table 4: Fatty acid composition of false walnut oil

S/N	Acid type	Form	Value (%)
1	Palmitic	C16:0	36.26
2	Palmitoleic	C16:1	3.80
3	Stearic	C18:0	6.91
4	Oleic	C18:1	2.26
5	Linoleic	C18:2	46.79
6	Others		3.98
7	Total saturated		43.17
8	Total unsaturated		52.85

3.2 Formulation of cutting fluid

The value of the pH of a cutting fluid is very important in the determination of whether the fluid will be corrosive when used for alloy of steel materials during machining process. It also indicates whether the fluid will lead to health hazard (causing skin disease) to the operators as well as having the potential to control biological growth in the cutting fluid. The pH value should be fairly above the neutral point of 7.0 but should not exceed 9.5 as it becomes very caustic and not desirable to the machined surface or the machine operator [13]. Therefore, pH value was used as response or output variable because of its effect on the material and health implications to the operator. The pH value for each run of the factorial plan of formulated oil-in-water emulsion is shown in Table 5. The analysis of pH data obtained for the sixteen experimental runs of the formulations were carried out using design expert software (design expert version 7.0).

Table 5: The pH values of the 2^4 full factorial planning

Experimental run	1	2	3	4	5	6	7	8
pH	8.1	7.8	8.2	7.9	7.7	7.7	7.9	7.9
Experimental run	9	10	11	12	13	14	15	16
Ph	7.8	7.7	7.9	7.9	7.9	7.8	8.0	8.0

The result of the analysis of variance (ANOVA) of the pH values in Table 6 shows that the model was very significant with only a probability of 0.04% of the Model F-value occurring as a result of noise in the experiment. The emulsifier (A), the bocide (B), and the anti-corrosion agent (C) had significant effects on the pH of the formulations while the antioxidant (D) had minimal effect on the pH of the formulation

Table 6: Analysis of variance (ANOVA) for the pH value model

Source	SS	DF	MS	P	Prob < F	Remark
Model	0.29	9	0.032	25.78	0.0004	significant
A	0.040	1	0.040	32.00	0.0013	significant
B	0.090	1	0.090	72.00	0.0001	significant
C	0.010	1	0.010	8.00	0.0300	significant
D	0.0025	1	0.0025	2.00	0.2070	insignificant
AC	0.022	1	0.022	18.00	0.0054	significant
AD	0.010	1	0.010	8.00	0.0300	significant
BC	0.0025	1	0.0025	2.00	0.2070	insignificant
CD	0.090	1	0.090	72.00	0.0001	significant
ACD	0.022	1	0.022	18.00	0.0054	significant
Residual	0.0075	6	0.00125			
Cor Total	0.30	15				

The F-value of 25.78 for the model implies that the model is significant. There is only a probability of 0.04 chance that the "Model F-value" this large could occur due to noise. If the Prob>F value is very small, that is, less than 0.05, for any individual term, then the term in the model have a significant effect on the anticipated response. Otherwise, the term in the model is insignificant. Also, the interactions between the emulsifier (A) and the anti-corrosion (C), between the emulsifier (A) and the antioxidant (D), between the anti-corrosion (C) and the antioxidant (D), and between the emulsifier (A), anti-corrosion (C) and antioxidant (D) have significant effect on the pH value. The Predicted R-squared of 0.8207 generated by the model is in reasonable agreement with the "Adjusted R-squared of 0.9370. The Adequate Precision measured the signal to noise ratio and must be greater than 4 to be desirable. The value of 17.889 obtained indicates an adequate signal from the experimental results. The prediction model for predicting the value of pH value of a given formulation based on values of the various factors at specified levels and their interactions is given by:

$$pH = 7.89 - 0.050A + 0.075B - 0.025C - 0.012D + 0.037AC + 0.025AD + 0.012BC + 0.075CD - 0.037ACD \quad (2)$$

Figure 2 show the plot of predicted values versus the associated noise or error in the experimentation. The points which are evenly dispersed around the central horizontal axis fell within ±3 standard deviations. Statistically, this implies that the probability of the predicted values lying within three standard deviations in either direction from its standard mean is 99.74%. In other words, practically all values of the predictions are confined in a confidence interval of six standard deviations.

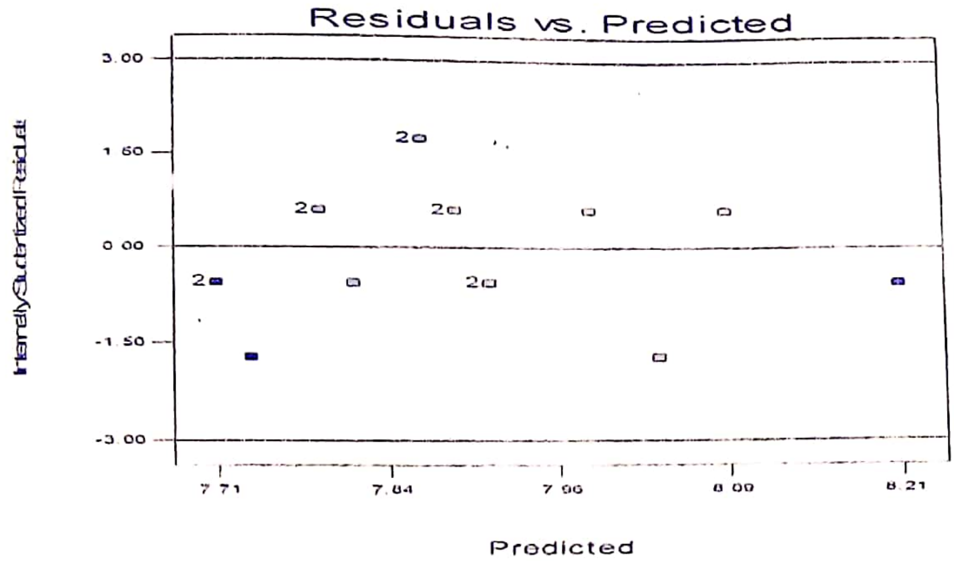

Figure 2: Residual plot for checking errors

Figure 3 show the contour plot representing the interaction between the emulsifier (A) and anti-corrosion (C) and the resultant pH values when the biocide (B) and the antioxidant (D) are kept at their minimum values of 0.5% each

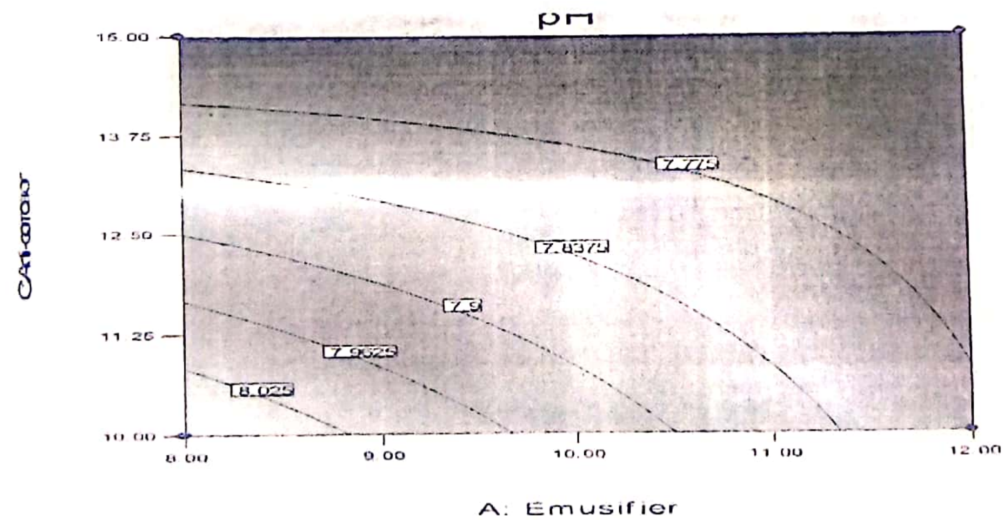

Figure 4: Contour plot

The contour plot shows that the maximum pH value of 8.025 is obtained along the contour line joining the emulsifier value of 8.80% and the anti-corrosion value of 10.75%. The pH value decreased to 7.775 as the values of both additives approach their maximum values of 12% and 15% respectively. It can be observed that both additives do not have much effect on the pH values of the formulation at their maximum values but rather around their minimum value. The formulation of the false walnut oil cutting fluid was optimized using the pH model of equation (2) generated by the design expert software. The optimized pH value for false walnut oil cutting fluid obtained was 8.029 with a viscosity of 2.31 mm^2/s. The pH value of

8.029 is within the acceptable range for machining alloy of steel (Lawal et al, 2013)

4. CONCLUSION

Oil-in-water emulsion cutting fluid using false walnut oil was formulated using design of experiment. The optimization by desirability function has been performed to obtain the optimum pH value condition for the development of cutting fluid using appropriate additives. The optimum value was obtained using 9.35% of emulsifier (A), 0.97% of biocide (B), 10.61% of anti-corrosion (C), and 0.64% of antioxidant (D). The adjusted R^2 (0.9370) obtained was sufficient to build other prediction models.

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