NEW FORMULATIONS FOR HYDRAULIC BIOLUBRICANTS BASED ON EPOXIDIZED VEGETABLE OILS: PASSION FRUIT (*Passiflora edulis* Sims f. *flavicarpa* Degener) AND MORINGA (*Moringa oleifera* Lamarck)

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ABSTRACT

The aim of this work is to develop new biolubricants based on epoxidized vegetable oils, optimizing formulations with an additive package by experimental design. A 2³ factorial design with center points was used to determine optimal conditions for anti-wear, corrosion inhibition, extreme pressure, and antioxidants to be applied with formulations with epoxidized passion fruit and moringa oils. The lubricants obtained were characterized in accordance with DIN 51524 (Part 2 HLP) and DIN 51517 (Part 3 CLP) standards. The epoxidation process improved the oxidative stability and the acid value of both pure vegetable oils. Furthermore, the presence of epoxidized oils improved the solubilisation of the additives, increasing the fluids' performances. Regarding their suitability as lubricants, epoxidized moringa oil with additives presented the best results, followed by epoxidized passion fruit oil with additives, epoxidized moringa oil, epoxidized passion fruit oil, pure moringa oil and, finally, pure passion fruit oil.

KEYWORDS

biolubricant; passion fruit oil; moringa oil; epoxidation; additive package

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1. INTRODUCTION

Over the past few years, public awareness on issues such as environmental pollution and health aspects has increased. In area of lubrication, the major concerns reside in the large amounts of lubricants that reach the environment unduly, most times due to leakages, which are harmful to water, soil, and air. The rise in environmental awareness, demanding high biodegradability and low ecotoxicity of products, is the main driving force behind new technological developments. Thus, biodegradable lubricants can be an option for exploring environmentally sensitive areas.

In 2004, 37.4 million tons of lubricants were consumed worldwide. Of those, 53% were automotive while 32% were industrial lubricants. Among the industrial use, 37% of the lubricants consumed were hydraulic fluids, 7% were transmission oils, 16% cutting fluids, and 9% greases (Mang & Dresel, 2007). The use of biodegradable products such as hydraulic fluids, chain-saw oil, compressor lubricants, and cutting fluids has increased rapidly. New technologies of production are needed in order to enhance the quality of lubricants, and to lower their use in the long term. Meanwhile, regulations in matters such as emission limits, ways of disposal, resources, and environment conservation have become more rigid. Presently, lubricants are required to be assessed on their heat and mechanical capacities, amount of consumption, ecological safety, and biodegradability. These factors have a direct impact on the consumer. It has been predicted that over the next 10-15 years biodegradable lubricants are going to represent 15% of the worldwide production.

Lubricants based on renewable sources such as corn, soybean, and rapeseed oils are already used as an alternative to traditional mineral oil lubricants, with the former having a lower potential of toxicity, and degrading faster than the latter. There are also efforts being made in the genetic engineering field, aiming to produce vegetable oils with better lubricating properties, including genetic modifications of corn and soybean in order to produce oils with a greater amount of oleic acid, consequently, improving oxidative stability (Gairing et al., 1994).

Compared to mineral oils, vegetable oils have better cutting performances. They also extend the useful life of tools used in some industrial activities

and improve surface finishing. Although having many environmental benefits, vegetable oils are more susceptible to degradation by oxidation or hydrolytic reactions. Therefore, the correct selection of vegetable oil and pH control of biolubricant formulations are important issues to be addressed (Woods, 2005).

The performance of lubricants based on different materials, with or without additives, has already been investigated (Cermak et al., 2013; Lawal et al., 2014; Muniz et al., 2008; Paredes et al., 2014; Petlyuk & Adams, 2004). Cottonseed, jatropha, mahua, canola, and sunflower oils have been recently studied as new possible sources of base oil for lubricants (Goud et al., 2006; Madankar et al., 2013; Meyer et al., 2008; Milchert et al., 2010; Paredes et al., 2014). Some promising species such as passion fruit (*Passiflora edulis* Sims f. *Flavicarpa* Degener) and Moringa (*Moringa oleifera* Lamarck) have not yet been investigated for this application. Passion fruit seed oil has a composition of 85% unsaturated fatty acids, of which 69% are linoleic acid and 18 % oleic acid (Ferrari et al., 2004; Lopes et al., 2010). Moringa seeds have around 35% of their weight as oil, with 85% of unsaturated fatty acids, 83.8% of which being oleic acid. All these characteristics are considered desirable for lubricant oil bases (Fahey, 2005; Morton, 1991; Ramachandran et al., 1980).

The aim of this study is to develop an additive package for hydraulic biolubricants using passion fruit seed and moringa seed oils epoxidized with formic acid, employing a 2³ factorial design with center points, where the response surface methodology will be used to obtain the optimal condition.

2. MATERIALS AND METHODS

2.1 Materials

The following chemical reactants were used to produce and characterize the epoxides: chloroform (Quimex, 99%), passion fruit seed oil (*Extrair Óleo Vegetal Ltda.*, Brazil), moringa seed oil (produced locally in the State of Sergipe, Brazil, by cold pressing), hydrogen peroxyde (Synth, 30%), anhydrous sodium sulfate (Vetec, 99%), toluene (Vetec, 99.5%), Wijs solution (Impex), formic acid (Impex, 85%), sodium thiosulfate (Impex, 99.5%), potassium iodate (Impex, 99%), anhydrous sodium

Table 1. Analysed parameters and the respective methods applied.

Parameter	Method	Reference Values	
		DIN 51524 (Part 2 HLP)	DIN 51517 (Part 3 CLP)
Viscosity at 40 °C (mm ² /s)	ASTM D2983 (2004)	80 – 100	≥ 150
Flash point (°C)	ASTM D92 (2002)	205	238
Acid Value (mg de KOH/g of sample)	ASTM D974 (1997)	≤ 2.0	≤ 2.0
Oxidative stability (min)	ASTM D7525 (2009)	-	-
Cooper corrosion	ASTM D130 (2000)	1A	1A
Density at 40 °C (g/cm ³)	ASTM D1298 (1998)	0.88	0.90
Oxirane content (%)	IUPAC (1987)	-	-
Iodine index (mg of I ₂ /g)	AOCS Cd 1-25 (1995)	-	-
Foam Formation	SELUB 09 (2003)	-	-

carbonate (Impex, 99%), benzene (Merc, 99.7%), sodium hydroxide (Impex, 97%), and potassium iodide (Impex, 99%). The commercial hydraulic lubricants used were Tutela Hidrobak VG100 (VG100) and Tutela Baku R150/EP (R150), both paraffinic, produced by Petrona Lubricants International. The additives used for the formulations were antifoam (Liofoam 149), a demulsifier (Liovac OAS700), an anti-wear and corrosion inhibitor (Liovac 3355), and an extreme-pressure additive (Liovac 89ss), all provided by Miracema-Nuodex Ltda. The 3-tert-butyl-4-hydroxyanisole (BHA), purchased from Merck Millipore, was used as antioxidant.

2.2 Preparation and characterization of vegetable oil-based lubricant

The vegetable origin bases were prepared by epoxidation of the vegetable oils. This reaction was used to introduce oxirane rings at the double bonds in the fatty acids chemical structures. This conversion can improve heat and oxidative stabilities, as well as the capacity of lubrication of the vegetable oils. Passion fruit and moringa seed oils were epoxidized with performic acid generated in situ. The reaction was carried out employing a molar rate of 1:1:1 between hydrogen peroxide, formic acid, double bonds for passion fruit seed oil, and 1.5:1:1 for moringa seed oil. The reaction system consisted of a 250-mL reaction flask attached to a reflux condenser, kept under constant stirring (500 rpm) at 30 °C for 3 hours. To avoid the hydrolysis of the vegetable oils, hydrogen

peroxide was added in fractions to the mixture during the first hour of reaction.

The epoxidized oils were analyzed in terms of viscosity (40 °C), flash point, acid value (A.V.), copper corrosion, and density (40 °C). These characterizations were necessary to meet DIN 51524 (Part 2 HLP) and DIN 51517 (Part 3 CLP) standards. Furthermore, to verify the effects of the epoxidation process on the vegetable oils oxidative stability, iodine index, foam formation, and oxirane content were also assessed. The reference values are shown in Table 1.

To obtain a proper comparison, *in natura* passion fruit oil, *in natura* moringa oil and two commercial lubricants were also characterized. The commercial lubricants used were Tutela Hidrobak VG100 (VG100) and Tutela Baku R150/EP (R150). These lubricants fully comply with DIN 51524 (Part 2 HLP) and DIN 51517 (Part 3 CLP).

2.3 Preparation of biolubricant formulations

A 2³ factorial design with center points was used for the additivation step of this work to evaluate the best concentrations of anti-wear and corrosion inhibitor (AW), antioxidant (BHA), and extreme pressure additive (EP). The upper and lower limits of EP and AW were those recommended by the manufacturer (Miracema-Nuodex Ltda.). The antioxidant limits were based on the work carried out by Araújo et al. (2009). Both *in natura* and epoxidized oils of passion fruit

Table 2. Additives used for the preparation of biolubricants.

Additive	Symbol	Level		
		Minimum (-)	Maximum (+)	Center Points (0)
Extreme pressure (wt.%)	EP	1.00	5.00	3.00
Anti-wear and Corrosion Inhibitor (vol%)	AW	0.50	1.50	1.00
Antioxidant (wt.%)	BHA	0.10	0.30	0.20
Antifoam (wt.%)	AF	-	-	0.30
Demulsifier (wt.%)	D	-	-	0.15

Table 3. The experimental matrix used in the formulations of new biolubricants.

Assay Nº	Variable / Factor Examined (Levels)		
	EP	AW	BHA
1	-1	-1	-1
2	+1	-1	-1
3	-1	+1	-1
4	+1	+1	-1
5	-1	-1	+1
6	+1	-1	+1
7	-1	+1	+1
8	+1	+1	+
9	0	0	0
10	0	0	0
11	0	0	0

and moringa were additivated, as shown in Table 2. Considering the intended hydraulic application, antifoam, and a demulsifier were also added to the mixture.

Table 3 presents the experimental matrix used in new formulations of biolubricants based on *in natura* passion fruit oil, epoxidized passion fruit oil, *in natura* moringa oil, and epoxidized moringa oil.

After the interpretation of the results presented in Table 3, with their respective response surfaces, the characterization of biolubricants was performed under the best additivation conditions found to the biolubricants of passion fruit fresh oil, epoxidized passion fruit oil, moringa oil in natura, and epoxidized moringa oil, as well as the characterization of commercial lubricants VG100 and R150. It is important to note that all experiments were performed in triplicate, with less than 5% deviation.

3. RESULTS AND DISCUSSION

The results presented and discussed here correspond to the new formulations obtained with passion fruit and moringa vegetable oils, and their epoxidized derivatives.

3.1 Formulation and characterization of hydraulic fluids

Since copper corrosion and foam formation results were the same for all formulations, this effect was left out of the statistical analysis. The variables with statistical significance were: viscosity, acid value, oxidative stability, and flash point. Therefore, Statistica 7.0 was used to generate level curves, allowing the verification of regions where the best conditions for lubricant formulation can be obtained. Figure 1 presents the level curves for the experiments with *in natura* passion fruit oil.

Figure 1. Level curves for *in natura* passion fruit oil formulations. Where: a) Viscosity; b) acid value; c) Oxidative stability; and d) Flash point.

Figure 2. Level curves EPO formulations. Where: a) Viscosity; b) acid value; c) Oxidative stability; and d) Flash point.

Analysing the level curves, one can see that the best conditions for a lubricant formulation with *in natura* passion fruit oil are in the regions where EP

and AW are in their upper limits and BHA in its lower limit. This expectation was confirmed experimentally, yielding a lubricant with a viscosity

Figure 3. Level curves for *in natura* moringa oil formulations. Where: a) Viscosity; b) Acid Value c) Oxidative stability; and d) Flash point.

of 35.30 mm²/s, 1.08 mg KOH/g A.I, a flash point of 224 °C, and 44.34 minutes of oxidative stability.

The level curves were also generated for epoxidized passion fruit oil (EPO), and are shown in Figure 2. The level curves show that the best conditions for a lubricant formulation with EPO are in the regions where EP is in its center point (0), AW in its lower limit (-), and BHA in its upper limit (+). This expectation was confirmed experimentally, yielding a lubricant with a viscosity of 175.43 mm²/s, 0.79 mg KOH/g acid value, a flash point of 204.5 °C, and 204.7 minutes of oxidative stability. However, these results were lower than the value suggested in the level curve for the total acidity value at 3% and higher flash point is 26%. This result represents a great performance as a lubricant oil, meaning that the experimental results exceeded the results predicted in the level curve.

The level curves for *in natura* moringa oil are presented in Figure 3. One can observe that the best additive concentrations to be used with *in natura* moringa oil are those in which EP and BHA additives are in their upper limits, while AW is in its lower limit. These observations were confirmed

experimentally, yielding a lubricant with a viscosity of 49.42 mm²/s, 0.44 mg KOH/g of acid value, a flash point of 216 °C, and 55.2 minutes of oxidative stability.

The level curves were also generated for epoxidized moringa oil (EMO) formulations, and are shown in Figure 4. It can be observed that the best concentrations of additives to be used in formulations with EMO are on the regions where BHA is in its upper limit, EP in its center point, and AW in its lower limit. The confirmation rendered a lubricant with viscosity of 83.50 mm²/s, 0.24 mg KOH/g of acid value, 211 °C of flash point, and 311.80 minutes of oxidative stability. However, it is worth noting that these results were higher than those suggested in the level curves for the total acidity value by 30%, flash point 5%, and oxidative stability around 40%. The level curves do not effectively represent the values observed experimentally, however, the performance of the biolubricant was higher compared with the other results found in its planning matrix, concluding that the percentages of selected additives provide superior performance to the base oil.

Figure 4. Level curves for formulations with epoxidized moringa oil. Where: a) Viscosity; b) acid value; c) Oxidative stability; and d) Flash point.

Table 4 presents the results of the characterizations obtained for the biolubricants of passion fruit oil *in natura* (PFO) and with additives (PFOA), epoxidized passion fruit oil (EPFO) and with additives (EPFOA), moringa oil *in natura* (MO) and

with additives (MOA), and epoxidized moringa oil (EMO) and with additives (EMOA), as well as the characterizations of commercial lubricants VG100 and R150.

Table 4. Characterization of the biolubricants based on passion fruit oil *in natura*, epoxidized passion fruit oil, moringa oil *in natura*, epoxidized moringa oil, and commercial lubricants.

Parameter	Passion Fruit Oil				Moringa Oil				Commercial Lubricants	
	<i>in natura</i>		Epoxidized		<i>in natura</i>		Epoxidized		VG100	R150
	PFO	PFOA	EPFO	EPFOA	MO	MOA	EMO	EMOA		
Viscosity at 40 °C (mm ² /s)	31.78	35.3	185.65	175.43	44.88	49.42	80.37	85.5	99.95	150.04
Flash Point (°C)	228	224	190	204.5	204	216	230	211	244.5	195
Acid Value (mg de KOH/g)	1.12	1.08	0.83	0.77	0.41	0.42	0.2	0.24	0.6	1.1
Oxidative Stability (min)	7.5	44.47	16.89	204.7	28.27	55.2	24.57	311.80	1670.26	931.16
Copper Corrosion	1A	1A	1A	1A	1A	1A	1A	1A	1A	1A
Density at 40 °C (g/cm ³)	0.91	0.91	0.91	0.91	0.90	0.90	0.93	0.94	0.88	0.90
Oxirane Oxygen (%)	-	-	5.55	5.55	-	-	4.08	4.08	-	-
Iodine Value, WIJS (mg de I ₂ /g)	130.7	132.3	15.62	17.02	71.9	73.9	6.18	8.18	-	-

From Table 4, one can observe that the iodine value decreases after the epoxidation of *in natura* oils, while the amount of oxygen as oxirane increases. These results confirm that the process of epoxidation for both *in natura* oils occurred (Adhvaryu & Erhan, 2002; Petrovic et al., 2002; Dinda et al., 2008; Meyer et al., 2008; Milchert et al., 2010; Mushtaq et al., 2013). It is observed that the presence of the additive increases the viscosity of the *in natura* passion fruit oil without additives (PFO) and the *in natura* moringa oil without additives (MO), and that after the epoxidations, this increase also occurs. However, with the additive, the epoxidized passion fruit oil with additives (EPFOA) had a slight decrease in viscosity if compared to the epoxidized passion fruit oil without additives (EPFO), whilst for the epoxidized moringa oil with additives (EMOA) the opposite happened. It is also observed that the viscosity of the lubricant VG100 is the closest to the EMOA, while the R150 fluid has a viscosity that is the closest to the epoxidized passion fruit oil with additives (EPFOA). The results of EMOA fall within the requirements of DIN 51524 (Part HLP 2), whereby the viscosity must be between 80 and 100 mm²/s, while EPFOA is within the limit of DIN 51517 (Part 3 CLP), whereby the viscosity must be equal to or greater than 150 mm²/s.

To comply with standards DIN 51524-2 (Part HLP 2) and DIN 51517-3 (Part 3 CLP) requirements, the lubricants must have acid values less than 2.0 mg KOH/g of sample, as shown in Table 1. The results of all fluids are within this specification, as seen in Table 4. Passion fruit oil has the highest acid values if compared to other fluids, but there was a decrease in the acid value with additives, and also after epoxidation. Moringa oil reached the lowest acid values, whilst the epoxidized moringa oil without additives (EMO) presented a decrease of about 50% of its acid value in comparison to the MO. However, after the addition of the epoxidized oil there was an increase in the acid value. Still, in Table 4, the temperature of the *in natura* passion fruit oil with additives' (PFOA) flash point (FP) decreased by 2% in comparison to the PFO. This also occurred when the PFO was epoxidized, decreasing its FP by about 15% after epoxidation (EPFO). Additionally, the EPFO had an increase in FP of about 7% after the additive, indicating a higher affinity of the chemical additives when the oil is epoxidized. The moringa oil behaved differently, in that the FP increased by 6%

for the *in natura* moringa oil with additives (MOA), and by 13% after epoxidation (EMO).

However, when this epoxidized moringa oil was treated with additives, a drop of about 10% in its FP was observed. The fluids EPFO, EPFOA, and MO had a FP below 205 °C, but the fluids PFO, PFOA, MOA, EMO, and EMOA had a FP higher than the one required by the DIN 51524 (Part 2 HLP) standard. Out of all the oils studied, only the VG100 had a FP higher than 238 °C, which is the minimum limit stated by the DIN 51517 (Part 3 CLP) standard.

Although the oxidative stability (ASTM D 7525) is not a parameter required by DIN 51524-2 (Part 2 HLP) and DIN 51517-3 (Part 3 CLP) standards for the classification of lubricants, this parameter was determined to confirm that the process of epoxidation in the presence of an antioxidant additive improves the behavior of vegetable-based fluids. It is important to highlight that the value of the oxidative stability of PFO after the epoxidation process (EPFO) presented an increase of 145%. Moreover, the EPFOA had an oxidative stability 12 times higher than that of the EPFO. This demonstrates that the additives have an improved chemical affinity with the epoxidized oil and, consequently, enhance the qualities of the fluid as a lubricant. The oxidative stability of the commercial fluids was superior to the one of the fluids studied because of their mineral origin, being chemically stable to the formation of free radicals, which promotes the oxidation of the fluids (Salimon et al., 2010; Nagendramma & Kaul, 2012). The *in natura* moringa oil (MO) had an increase of 95% in its oxidative stability with additives (MOA), but this same oil had a 40 % drop after the epoxidation process (EMO). However, the EMO increased about 11 times its oxidative stability after treatment with additives (EMOA). This indicates that the epoxidized oil has a better affinity with the antioxidant additive, thereby improving the qualities of the fluid as a lubricant.

The results of fluid characterizations based on passion fruit and moringa oils showed that the process of epoxidation with subsequent chemical treatment with additives improved the oil's physicochemical characteristics, especially in relation to oxidative stability and acid value.

4. CONCLUSIONS

This work demonstrates that novel biolubricants, with satisfactory properties, were obtained successfully using epoxidized passion fruit and moringa oils as templates. The epoxidation process improved the oxidative stability and the acid value of the oils. Moreover, the epoxidized oils have a better compatibility with additives, resulting in increased performance of fluids. In terms of performance as a lubricant, epoxidized moringa oil with additives (EMOA) can be considered the best among all tested fluids, being followed by epoxidized passion fruit oil with additives (EPFOA), epoxidized moringa oil without additives (EMO), and epoxidized passion fruit oil without additives (EPFO), in this order.

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