DEVELOPMENT OF ASPHALT EMULSIONS INCORPORATING WASTE LUBRICATING OIL

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ABSTRACT

The aim of this study is to develop asphalt emulsions incorporating used lubricating oil (OLUC), as a modifier agent, to produce stable emulsions and resulting in cost-effective and environmentally friendly product suitable for paving applications. A central composite design (CCD) is proposed to reduce the experiment's number. The asphalt emulsions were characterized by Saybolt Furol viscosity, residue by evaporation, settlement and storage stability, sieve test, ductility, and penetration over residue. The results showed that asphalt emulsions, in accordance with all technical specifications, can be produced at 80°C, using 5 minutes as emulsification time and low amounts of surfactant. Moreover, the experiment incorporated OLUC in the composition of asphalt emulsions to obtain new low-cost products for use in paving operations.

KEYWORDS

Asphalt; asphalt emulsion; waste lubricating oil (OLUC); paving; environment

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1. INTRODUCTION

Asphalt bitumen, a by-product of petroleum refining, is considered one of the oldest materials used in construction. It is applied widely in pavement construction due to its thermoplastic, adhesive, and waterproof properties. It is well known that the origin of the crude oil as well as the distillation process affect asphalt properties. According to **Yao et al. (2016)**, asphalt's composition includes mainly carbon (82.9-86.8 wt. %), hydrogen (9.9-10.9 wt. %), heteroatoms, and metals. The large use of this non-renewable material has made it scarce, stimulating the use of waste materials to reduce its consumption (**Asphalt Institute, 2002; Jasso et al., 2015; Li et al., 2015; Sun et al., 2016**).

Asphalt emulsions (AE) are a thermodynamically unstable, but kinetically stable, dispersion of fine asphalt droplets (1-10 μm) in an aqueous phase (oil-in-water emulsion), or of an aqueous phase disperse in an asphalt phase (water-in-oil emulsion). It comprises an emulsifying agent used to stabilize the dispersion and avoid phase separation. The emulsions are prepared in a colloidal mill, an equipment that uses mechanical energy to break the asphalt phase in small droplets (**James, 2006; Kilpatrick, 2012; Mercado et al., 2014; Shen et al., 2013; Takamura & James, 2015**). AE may contain around 40-80 wt. % of asphalt and are classified as anionic or cationic, in accordance with the chemical nature of emulsifying agent used in its composition (**Takamura & James, 2015**). According to **Shen et al. (2013)**, when the asphalt emulsion comes into contact with mineral aggregates, the asphalt droplets coalesce and revert to a continuous film.

The production of asphalt emulsions and the use of modified asphalt binders has become a promising field to researchers, with benefits in several areas of expertise and applications (**Baaj et al., 2013; Croteau & Tessier, 2008; Yang et al., 2014**). The technological advances obtained with these new products have allowed the improvement in quality of paving services. The use of material waste in the composition of new materials is a world trend that has been growing in all branches of economic activity, resulting in cost reduction, resources conservation, construction efficiency increase, quality improvement, and environmental problems' solution.

According to **Kheireddine et al. (2013)**, considerable quantities of used lubricating oils represent an environmental problem. To ameliorate its negative impacts, there's a growing interest in adopting strategies to recovery, recycle, or reuse the waste oil. According to the Annual Report of the Brazilian Ministry of the Environment (**Brasil, 2014**), 1.2 billion liters of lubricating oil were sold during 2014 in Brazil, with only 451.8 million liters being collected for reuse, which corresponds to 37.71 % of the total.

The addition of used lubricating oil (OLUC) to asphalt binder could reduce the amount of solvent used while obtaining asphalt emulsions. The OLUC chemical composition is similar to the one of the asphalt binder. Admitting that there is a degree of compatibility between this waste and the asphalt binder, this work proposes the incorporation of OLUC to new formulations of asphalt emulsions.

2. MATERIALS AND METHODS

2.1 Materials

The petroleum asphalt cement (50/70 penetration grade) used to obtain asphalt emulsions was supplied kindly by LUBNOR/PETROBRAS (*Lubrificantes e Derivados de Petróleo do Nordeste*, Brazil). It was obtained from a distillation process of crude oils from Fazenda Belém (Aracati, Ceará, Brazil). All experiments used tap water. A quaternary amine salt was used as emulsifying agent (CRQ Produtos Químicos Ltda). Kerosene was used as solvent (Petrobras). Other chemicals used during the experimental were hydrochloric acid (Proquímios, 36.5 %) and an antifoaming agent (Silaex Química Ltda).

In this study, the term "OLUC" refers to the lubricating oil that was collected in gas stations (Natal, RN, Brazil). It represents the lubricating oil collected after usage in automotive engines. A representative sample of OLUC was provided by BQMIL (Brasil Química e Mineração LTDA, Mossoró, RN, Brazil) and it was used as a modifying agent in some asphalt emulsions.

2.2 Preparation of asphalt emulsions

The aqueous and oily phases of the asphalt emulsions were prepared separately, according to

Figure 1. A flowchart describing the methodology adopted in the obtaining of the asphalt emulsions.

the methodology developed by Lima (2012). The aqueous phase was prepared by dissolving the emulsifying agent in an aqueous solution containing HCl and a few drops of the antifoaming agent. After mixing, the aqueous phase was preheated to 60°C. The oily phase was obtained by heating the asphalt cement (110°C) and, after the temperature was stabilized, a mixture of solvent and OLUC was added and homogenized. Finally, the oily phase and the aqueous phase were added simultaneously to a jacketed colloid mill (QFC-REX-1, Meteor) and milled for 5 minutes at 80°C (Figure 1). A 3:7 ratio was maintained between the aqueous and oily phases, respectively, and 5 wt. % excess water were added to compensate losses by evaporation. The composition of the aqueous phase was adjusted according the amount of surfactant required in the experiment. The amount

of asphalt was fixed at 60 wt. % and the amount of OLUC did not exceed 5 wt. % of the overall composition of the asphalt emulsion. The amount of solvent was adjusted according to the quantity of OLUC used in each formulation.

A central composite design (CCD) with four center points was used as response surface methodology to investigate the effects of two factors: surfactant and OLUC percentiles. This variety of design requires 4 levels of each studied factor. Table 1 shows the independent variables, their four levels, and real values adopted in this research. The evaluated responses were Saybolt Furol viscosity (SFV - 50°C), residue by evaporation, and settlement and storage stability. The tests for characterizing the asphalt emulsions were made according to the Brazilian *technical standards*,

Table 1. Codes and levels of the two studied variables.

Symbol	Factor	Level				
		Lowest	Low	Center	High	Highest
		-1.41	-1	0	+1	+1.41
S	Surfactant (wt. %)	0.43	0.60	1.00	1.40	1.57
O	OLUC (wt. %)	1.59	2.00	3.00	4.00	4.41

Axial points: $\pm \alpha = \pm 1.41$

published by Brazilian Technical Standards Association (ABNT-NBR) and evaluated considering a Brazilian specific law (DNIT 165/2013). The number of experimental tests (N) was calculated by using Equation (1):

$$N = 2^n + 2n + C \quad (1)$$

Where, n is the number of factors and C the number of center points. In this study n is equal to 2 and C is equal to 4, consequently, a total of twelve asphalt emulsions were evaluated in this study.

2.3 Characterization of the asphalt emulsions

2.3.1 Sieve test

The sieve test, designed according to ABNT-NBR 14393 (2012), tests asphalt emulsions for non-dispersed asphalt globules. In this methodology, a 1000g sample is poured into a 850 μ m sieve (No. 20). When testing cationic emulsions, the experiments use distilled water to wash the sieve until the wash water runs clear. After rinsing, the sieve and asphalt are dried in an oven at 110° C, until a constant weight. The result was expressed in percentage, calculated according to the equation (2):

$$\% \text{ retained} = \frac{(B - A)}{1000} \times 100 \quad (2)$$

Where:

A = dry weight of the clean sieve, in grams;

B = dry weight of sieve with the residue of asphalt, in grams.

2.3.2 Saybolt Furol viscosity

This test, performed according to ABNT-NBR 14491 (2007), at 50°C, evaluates the consistency of asphalt emulsions. The experiment used a Saybolt Furol viscometer (I-2025, Contenco) for this purpose. It measured the time required, in

seconds, for a 60mL sample to flow continuously through a standard sized orifice (Furol orifice). The results were expressed as Saybolt Furol Seconds (SFS).

2.3.3 Settlement and storage stability test

This test, made according to ABNT-NBR 6570 (2010), indicates the emulsion's stability in storage. In this test, a 500mL sample of the asphalt emulsion was kept at rest, in an graduated cylinder, during five days. After this storage time, two 55mL sample aliquots, one from the top and other from the bottom of the cylinder, were taken and transferred, separately, to two beakers previously weighed. Then, the residue by evaporation of each sample was determined (see item 2.3.4), being the settling and storage stability (S_{SST}) determined by using Equation (3):

$$S_{SST} (\%) = B - T \quad (3)$$

Where T and B are the average percentage of asphalt residue from top and bottom, respectively.

2.3.4 Residue by evaporation

The residue by evaporation was determined according to ABNT-NBR 14376 (2007). This method consists of heating an asphalt emulsion sample, under constant stirring, until total evaporation of volatile compounds (water and solvent). During the procedure, the sample is weighed at every 5 min, until a constant weight is obtained and, visually, a mirrored surface is observed. The results, expressed as percentage of asphalt residue (R), are determined by using Equation (4):

$$R (\%) = \frac{C - A}{(B - A)} \times 100 \quad (4)$$

Where, A is the weight of the test set, B is the weight of the test set with the sample, and C is the weight of the test set with the asphalt residue.

Table 2. Asphalt emulsions compositions and results of characterization tests.

Emulsion	Surfactant (wt. %)	Water (wt. %)	OLUC (wt. %)	Solvent (wt. %)	R ₁ (SFS)	R ₂ (wt. %)	R ₃ (wt. %)
DNIT 165/2013	-	-	-	-	20 - 400	min., 62	max., 5
AE1	0.60 (-1)	34.1	2 (-1)	8	72	59.1	3.49
AE2	1.40 (+1)	33.3	2 (-1)	8	62	58.3	3.87
AE3	0.60 (-1)	34.1	4 (+1)	6	40	59.0	1.71
AE4	1.40 (+1)	33.3	4 (+1)	6	139	58.7	3.36
AE5	0.43 (-α)	34.3	3 (0)	7	42	59.8	8.85
AE6	1.57 (+α)	33.1	3 (0)	7	170	58.9	3.68
AE7	1 (0)	33.7	1.59 (-α)	8.41	42	58.0	3.45
AE8	1 (0)	33.7	4.41 (+α)	5.59	56	59.0	4.83
AE9	1 (0)	33.7	3 (0)	7	108	63.4	1.01
AE10	1 (0)	33.7	3 (0)	7	114	59.4	1.36
AE11	1 (0)	33.7	3 (0)	7	111	59.7	2.80
AE12	1 (0)	33.7	3 (0)	7	104	60.0	2.22

Fixed composition: asphalt (60 wt. %), HCl (0.3 wt.%), antifoaming agent (0.15 g), water (5 wt.% excess);
R₁ (SFS): viscosity, in Saybolt Furol Seconds; R₂: residue by evaporation; R₃: Settlement (results in module).

2.3.5 Penetration over residue

The penetration test was performed in accordance with **ABNT-NBR 6576 (2007)**. This test measures the depth of penetration of a standard needle subjected to a load of 10 g for 5 seconds over an asphalt residue sample, at 25°C. A universal penetrometer (1/10mm, I-2050, Contenco) was used for this purpose and the result is expressed in millimeters (mm).

2.3.6 Ductility test

The ductility was determined according to **ABNT-NBR 6293 (2001)**. The test expresses the ability of asphalt to be extended and is run by molding a briquette of asphalt residue under standard conditions and dimensions. After that, the asphalt briquette is submerged into a water bath, at 25°C, during 30 minutes and, soon after, subjected to traction in a ductilometer (I-4201, Contenco), with 5cm/min speed rate. The sample is elongated until rupture, with the result expressed in centimeters.

3. RESULTS AND DISCUSSIONS

Table 2 shows the compositions of the 12 asphalt emulsions obtained in accordance with the central composite design (CCD) and the results of viscosity (R₁), residue by evaporation (R₂), and settlement and storage stability (R₃).

According to the results presented in Table 2, it is possible to observe that the values of viscosity (R₁) are in the range of 40 - 170 SFS, which allows one to classify the obtained AEs as medium setting ones. In relation to the residue by evaporation (R₂), the obtained values are between 60 -58 wt.%, i.e., values close to the percentage of asphalt binder used in its composition. One can observe that only AE9, with 63.4 wt.%, is in accordance with Brazilian legal specification (**DNIT 165/2013**). When evaluating the results for settlement and storage stability (R₃) one found that only AE5 presented a value above 5 wt.%.

Figure 2 shows the Pareto chart for viscosity (R₁), considering 95% confidence interval ($p < 0.05$ - $R^2 = 0.939$). The Pareto charts for R₂ and R₃ were

Figure 2. Pareto chart at 95% confidence level showing the effect of the viscosity.

Figure 3. Response surface and level curves for viscosity (R_1).

not shown because these effects and its interactions were not significant, assuming a 95% confidence interval. For viscosity, one can notice that only the amounts of surfactant and OLUC had statistically significant effects, as well as the interaction between them. It was observed that an increase in the quantity of surfactant used in the production of AEs increased the values of viscosity. On the other hand, when the amount of OLUC was increased, an opposite effect was observed, i.e., a reduction in viscosity values was obtained. Therefore, for viscosity (R_1), the effect of the surfactant factor was positive while the effect of OLUC factor was negative. One can also observe a significant linear interaction effect between these two factors.

The regression coefficients that were significant in the adopted confidence interval (95%) were used to obtain Eq.5, a second-order mathematical model that describes the behavior of viscosity. This model is valid only when using the coded values adopted for the factors, according Table 1 (-1.41, -1.0, 0.0, +1.0, and +1.41).

$$R_1 = 33.75 S + 8.1 O - 1.438 S^2 - 29.94 O^2 + 27.25 SO + 109.2 \quad (5)$$

Table 3. Composition of the asphalt emulsions obtained in the optimization study.

Emulsion	Surfactant (wt. %)	OLUC (wt. %)	CAP (wt. %)	Solvent (wt. %)
DNIT 165/2013	ND	ND	ND	ND
AE13	0.60	4.41	60	5.59
AE 14	0.60	4.41	62	3.59
AE 15	0.60	4.41	64	1.59
AE 16	0.60	6.00	64	0
AE 17	0.70	6.00	64	0
AE 18	0.80	6.00	64	0
EMAM	ND	ND	ND	ND

Water (5% excess); ND (not determined)

Table 4. Results of the characterization tests for optimized asphalt emulsions.

Emulsion	Sieve test (wt. %)	Viscosity (SFS)	Residue (wt. %)	Settlement (wt. %)	Penetration (mm)	Ductility (cm)
DNIT 165/2013	máx., 0.1	20 - 400	mín., 62	máx., 5	4.0 – 15.0	mín., 40
AE13	0	28	60.4	6.44	6.37	> 120
AE 14	0	31	60.2	0.16	6.30	113.6
AE 15	0	38	60.9	0.14	5.80	116.5
AE 16	0	35	62.2	4.57	8.24	> 120
AE 17	0	23	63.4	1.45	6.47	88.5
AE 18	0	25	63.0	5.12	6.03	76
EMAM	ND	18	64.3	ND	3.85	> 120

SFS: Saybolt Furol Seconds; ND (not determined); Settlement (results in module)

To check the representativeness of the model obtained, the analysis of variance (ANOVA) was performed. The quadratic model obtained to describe the behavior of R_1 was significant ($p < 0.05$) and predictive, because the F calculated was 4.2 times greater than the F tabled. Therefore, Equation (5) can be used to carry out predictions about the viscosity of the asphalt emulsions within the limits established in this study. The correlation coefficient (R^2) obtained for this model was 0.939. This means that 93.9 of the total variation around the average value can be explained by the regression.

Observing the surface and level curves shown in Figure 3, it is possible to notice a relevant interaction influence of the amounts of surfactant and OLUC on viscosity. From the analysis of the level curve, one can suggest, as optimal working range, asphalt emulsions formulations comprising

1.0 - 1.6 wt.% surfactant and 3.0 - 4.4 wt.% OLUC. For R2 and R3, the response surfaces and level curves were not shown because the regression models were not significant at $p < 0.05$.

3.1 Optimization of the emulsification process

In this study, the AEs were prepared using the same methodology used in the previous study (item 2.2) and a 3:7 ratio between the aqueous and asphalt phases was maintained, respectively. However, the compositions of the aqueous and oily phases were modified according the compositions shown in Table 3. The amounts of antifoaming agent and acid were fixed at 0.15g and 0.3 wt.%, respectively.

Table 4 shows the results of the characterization tests. One can observe that none of the AEs

presented asphalt residue retained on the 850 μ m sieve (No. 20), indicating an appropriate grinding during emulsion production and absence of non-dispersed asphalt globules.

Considering the viscosity results, one can observe that the obtained values are between 23 and 38 SFS, which approached to the viscosity value of a commercial cationic medium setting asphalt emulsion (18 SFS), kindly supplied by EMAM Asfaltos (Pacatuba, CE, Brazil). On the other hand, there was a significant difference between viscosity values obtained for AEs of the CCD experimental design (Table 2) and the results for the optimized ones (Table 4), whereas it was possible to increase the amount of asphalt binder and reduce (or even eliminate) the presence of solvent.

In relation to the residue by evaporation, AE16, AE17, and AE18 reached the minimum percentage established by Brazilian legal specification. These emulsions have 64 wt.% asphalt binder in its compositions, indicating that the amount asphalt is the predominant factor to reach the minimum specified value. It is important to notice that these emulsions were made without the use of a solvent.

The results presented in Table 4 for settlement and storage stability indicate the presence of stable AEs, except for AE13 (6.44%). The lowest percentage of asphalt (60 wt.%), OLUC (4.41 wt.%), and surfactant (0.6 wt.%); and the maximum quantity of solvent (5.59 wt.%) were used in the formulation of this emulsion. These factors are in some ways associated and, therefore, it is necessary a certain domain of the process seeking to balance the influence between these variables.

The test of penetration over residue measures the consistency of asphalt binder. In this study, the results were in accordance with Brazilian legal specification, ranging from 5.8 to 8.2 mm. The ductility test expresses the ability of asphalt to be extended and, as one can observe in Table 4, all emulsions presented results above the minimum established by **DNIT 165/2013** (Table 4), which is 40 cm.

At last, AE16, AE17, and AE18 showed the best results, being AE17 the top one, especially due to its low viscosity, high content of asphalt residue, and absence of solvent, which implies an effective cost reduction in the final product.

4. CONCLUSIONS

This work investigated the effect of the addition of a waste lubricating oil (OLUC) in asphalt emulsions. The experimental design for evaluating the amounts of surfactant and OLUC in the production of asphalt emulsions allowed one to conclude that it is possible to obtain stable emulsions. Despite using 60 wt.% cement asphalt in emulsion formulations, none of them presented the asphalt residue by evaporation in accordance with Brazilian law (**DNIT 165/2013** - 62 wt.%). The evaluation of new formulations, using 64 wt.% cement asphalt, showed that it is possible to incorporate 6 wt.% OLUC and produce stable asphalt emulsions, with low amounts of surfactant (0.7 wt%) and without solvent, in accordance with the Brazilian regulation. This study showed a new alternative for the reuse of OLUC, mainly in places where the re-refining practice is not possible. The incorporation of OLUC to asphalt binder is an environmentally friendly practice that adds value to oil waste, reducing the costs of asphalt emulsions.

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NOMENCLATURE

ABNT - NBR - Brazilian Technical Standards Association
 AEs - Asphalt emulsions
 ANOVA - Analysis of variance
 Br - Bromine
 C - Carbon
 CAP - Petroleum asphalt cement
 CCD - Central composite design

DNIT - National Department of Transport Infrastructure
 H - Hydrogen
 HCl - Hydrochloric acid
 L - Linear
 N - Nitrogen
 O - Oxygen
 Q - Quadratic
 SAQ - Quaternary amine salt
 SFS - Saybolt Furol Seconds
 SFV - Saybolt Furol Viscosity
 OLUC - Used Lubricating Oil

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