Wax Recovery from Cameroon Petroleum Refinery Conditioned Oily Sludge

P. Ze Bilo'o, G. A. Mouthe Anombogo, C. C. Ndjeumi, and M. B. Ngassoum

***Abstract* — As all petroleum refineries, the Cameroon National Petroleum Refinery generates oily sludge that have to be treated. A certain number of researches have already been conducted on it in the sense of oil content recovery before disposal. The conducted study had the objective of wax precipitation from the recovered oil fraction of oily sludge. The experimental design on three parameters (Acetone/Hexane ratio named Solvent, Solvent/Hydrocarbons extract ratio, and Contact time) allow us to reveal that 2,6-Dimethylundecane (C13) and Tetracosane (C24) are the only compounds that are extracted no matter the extraction conditions. Also, it was shown that the most abundant compounds with concentrations ranged from 49 to 225 ppm and from 22 to 105 ppm respectively for 2,6-Dimethylundecane (C13) and Tetracosane (C24). Nonadecane and Pristane are absent in extract n°1 (8:1; 4:1; 90) and n°4 (10:1; 6:1; 90) respectively. Phytane is completely absent from all extracts. The extraction condition that gave the highest amount of wax is the extraction n°10 (9:1; 4:1; 105).**

***Key words* — Cameroon, GC-FID, Oily sludge, Paraffins, Wax recovery.**

I. Introduction

The oily sludge generated by petroleum refinery is made up of solid particles, water, and hydrocarbons []1-[5] and of poisonous, carcinogenic or mutagenic compounds such as polycyclic aromatic hydrocarbons [6]-[9]. An annual generation estimation of 30,000 tons of oily sludge is the result of the functioning of a medium size refinery processing about 12,000 to 15,000 m3/day of crude oil [10]. Oily sludge have gone through various treatment methods. These methods can be classified as hydrocarbon recovery or hydrocarbon destruction. For hydrocarbon destructive methods, we can find incineration [11], pyrolysis [12]-[14], landfilling [10], [15], composting [16], [17], the use of microorganisms [2], [18] [,19]. Research conducted for hydrocarbon recovery are for example those applying solvent extraction [20]-[22], oily sludge conditioning [23]-[25], surfactant [26], ultrasound [27], freezing and thawing [28], and Carbon dioxide extraction [29].

The composition of the hydrocarbon fraction of oily sludge varies from one refinery to another, and also from one oily sludge to another depending on the type of refined crude oil. Previous analysis made on oily sludge of the National Petroleum Refinery of Cameroon [4], [5] showed the high content of aliphatic hydrocarbons with high molecular weight which can be called wax. Some other analysis done on the hydrocarbon fraction of the oily sludge showed that it has some physicochemical properties similar to the diesel fuel [30]. Our survey is orientated toward the oily sludge of the National Petroleum Refinery of Cameroon. The objective of this work is to carry out wax recovery from the hydrocarbon fraction of this oily sludge.

II. Material and Methods

A. Material

The sample of oily sludge used was provided by the National Petroleum Refinery of Cameroon and had an average constitution of 66% of water, 2% of fine particles and 32% of hydrocarbons. The hydrocarbons fraction was extracted from the oily sludge on the best extraction conditions as described by Ze and Ngassoum [22] by using the gasoline. It's from this extract that the wax is recovered since it was shown that the gasoline fraction of 70–110 °C highly extract aliphatic hydrocarbons of high molecular weight [22]. The extracted hydrocarbon fraction of oily sludge will be called Paraffinic Hydrocarbons extract.

B. Methods

1) Diesel fuel dewaxing

Previous studies [30] showed that the hydrocarbon fraction of oily sludge from the National Petroleum Company has some physicochemical properties similar to the Diesel fuel which is known having high amount of paraffins. In order to define optimum dewaxing operating conditions, a dewaxing experimental design was applied on Diesel fuel. The used extraction parameters are Contact time, Solvent/Diesel fuel ratio and Acetone/Hexane ratio.

2) Paraffinic hydrocarbons extraction

Based on previous studies of Ze [30], the extraction methods used for paraffinic hydrocarbons extraction are those described by Ndjeumi *et al.* [20] and Ze and Ngassoum [22].

3) Paraffinic hydrocarbons extract dewaxing

In order to build-up the extracted paraffinic hydrocarbons dewaxing experimental design, previous dewaxing kinetic experiments were realized on diesel fuel with three parameters: Contact time, Acetone/Hexane ratio named

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Solvent and Solvent/Hydrocarbons extract ratio. The mass of 3 g of diesel fuel were used for each experiment.

The oily sludge dewaxing was conducted following the factorial composite centered plan with two levels and two tests at the center. This method was chosen due to the reduction of the number of manipulations, the study of the various interactions between the studied parameters and the graphic representation of results as response surface of models.

Once the experimental design ready, the applied dewaxing protocol as presented in the Fig. 1 below was applied.

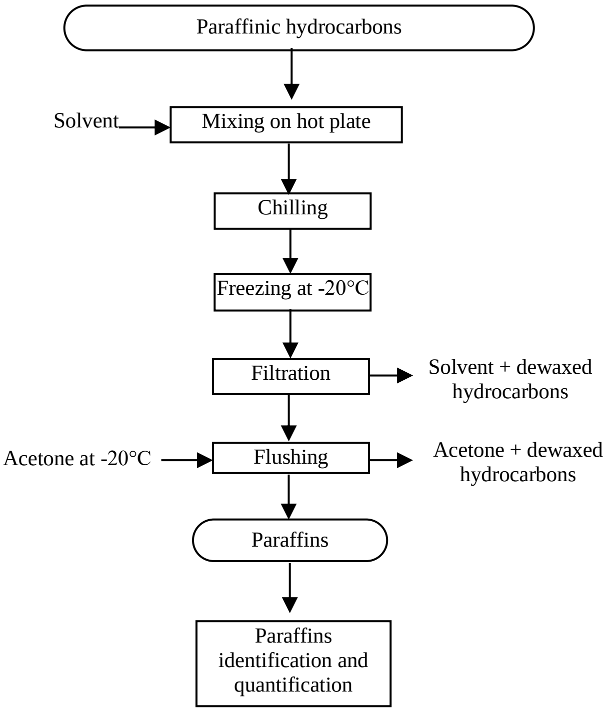


Fig. 1. Dewaxing protocol.

The paraffins identification and qualification were realized as described by Ndjeumi *et al*. [20].

III. Results and Discussion

A. Experimental Designing from Diesel Fuel Dewaxing Tests

It was noted in general that the peaks representing paraffins are clearly separated one from another. Also, the increase in the number and the intensity of the peaks demonstrating the positive effect of the studied parameter was noted.

1) Effect of contact time

The effect of contact time on dewaxing was evaluated. Based on the literature, the other parameters values were fixed at: Solvent/Diesel fuel ratio of 10:1 and Acetone/Hexane ratio of 6:1. From the results obtained, an exponential increase of more than 10% of dewaxing between 90 min and 120 min of contact time was noted. Those two contacts time were selected as experimental zone of the parameter in the experimental design. The positive effect of the contact time on dewaxing is illustrated on the spectra of precipitated paraffins from the Diesel fuel of Fig. 2 below.

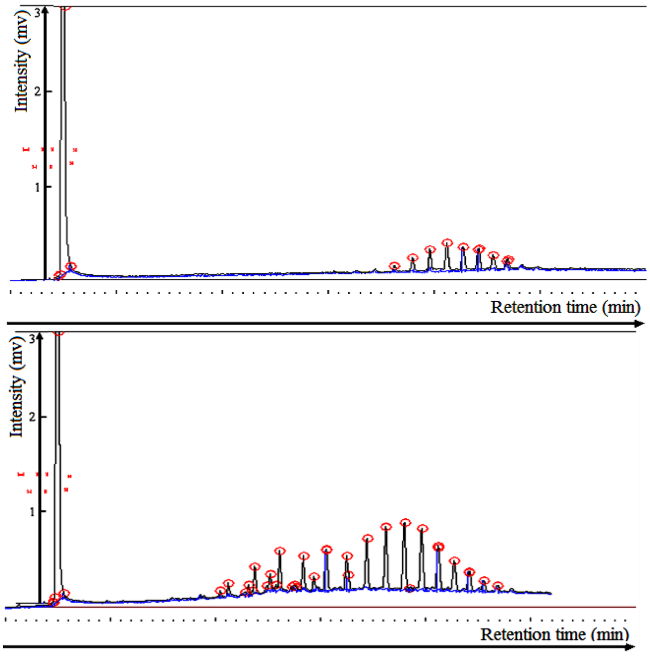


Fig. 2. Spectra of contact time effect for 15 min and 120 min on diesel fuel dewaxing (Solvent/Diesel fuel ratio = 10:1; Acetone/Hexane ratio = 6:1).

2) Effect of the Acetone/Hexane ratio (Solvent)

The effect of the Acetone/Hexane ratio named Solvent on dewaxing was evaluated. The Contact time of 105 min is the mean value of the experimental zone defined from the previous experiment while the Solvent/Diesel fuel ratio of 10:1 is based on the literature. From the results obtained, an exponential increase of 2% of dewaxing between 8:1 and 10:1 of Acetone/Hexane ratio appears. Those two Acetone/Hexane ratios were selected as experimental zone of the parameter in the experimental design. The positive effect of the Acetone/Hexane ratio on dewaxing is illustrated on the spectra of precipitated paraffins from the Diesel fuel of Fig. 3 below.

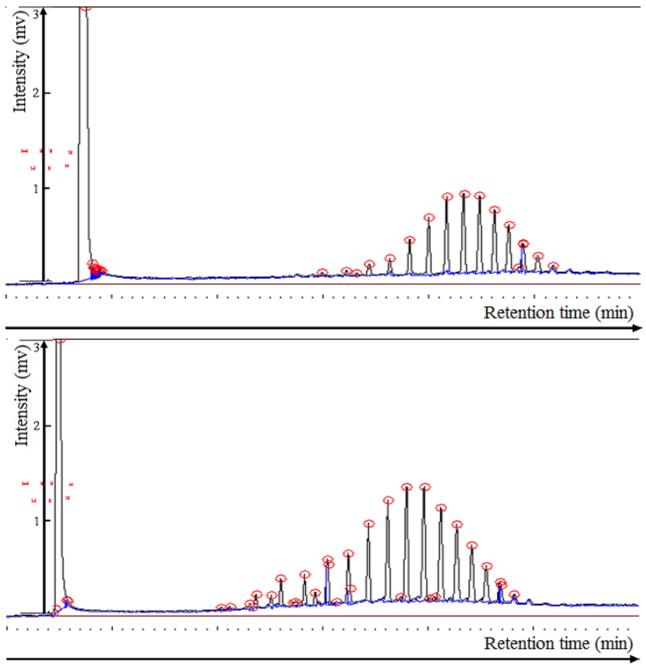


Fig. 3. Spectra of Acetone/Hexane ratio effect for 8:1 and 10:1 on diesel fuel dewaxing (Solvent/Diesel fuel ratio = 10:1; Contact time = 105 min).

3) Effect of Solvent/ Diesel fuel ratio

The effect of the Diesel fuel ratio on dewaxing was evaluated. The Contact time of 105 min and the Acetone/Hexane ratio of 9:1 are the mean value of the experimental zone defined from the previous experiments respectively. From the results obtained, an exponential increase of 3% of dewaxing between 4:1 and 6:1 of Solvent/Diesel fuel ratio was revealed. Those two Solvent/Diesel fuel ratios were selected as experimental zone of the parameter in the experimental design. The positive effect of the Solvent/Diesel fuel ratio on dewaxing is illustrated on the spectra of precipitated paraffins from the Diesel fuel of Fig. 4 below.

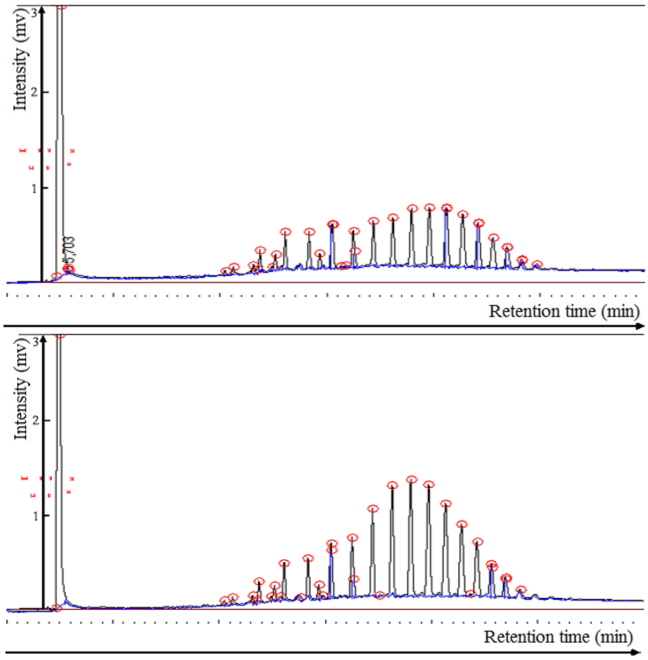


Fig. 4. Spectra of Solvent/Diesel fuel ratio effect for 2:1 and 6:1 on diesel fuel dewaxing (Acetone/Hexane ratio = 9:1; Contact time = 105 min).

An experimental domain was retained for each parameter test. These domains put together; minimal, center, and maximal values designated; coded values given; allowed the experimental design for oily sludge dewaxing to be set-up as shown on Table I below.

TABLE I: EXPERIMENTAL DESIGN FOR PARAFFINIC HYDROCARBONS DEWAXING

Factors	Minimal value	Center	Maximal value
Acetone/Hexane ratio (X_1)	8:1	9:1	10:1
Solvent/Hydrocarbon Ratio (X_2)	4:1	5:1	6:1
Contact time (Min) (X_3)	90	105	120
Coded value	-1	0	+1

B. Paraffinic Hydrocarbons Dewaxing

The oily sludge dewaxing was conducted as indicated earlier with sixteen (16) experimentations and the results are presented under the effects of parameters on dewaxing efficiency, the modelization of the oily sludge dewaxing and the identification/quantification of some precipitated paraffins.

1) Effects of parameters

The effects of studied parameters on oily sludge dewaxing are represented on Pareto graphic for standardized extracted masse of paraffins in Fig. 5 below.

It is appearing that the Acetone/Hexane ratio is having a significant positive impact on the dewaxing efficiency the same as its quadratic effect, while the Solvent/Hydrocarbons ratio is significantly negative the same as its quadratic effect. It can be noted that the interactions between the two ratios and between the Solvent/Hydrocarbon ratio and Contact time have significant positive effects on oily sludge dewaxing.

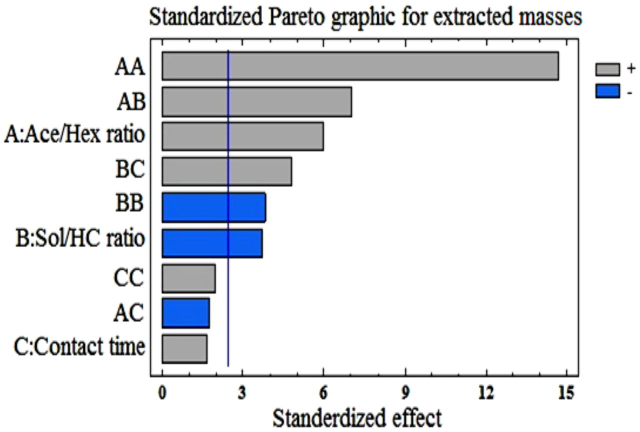


Fig. 5. Standardized Pareto graphic for extracted wax.

2) Paraffinic hydrocarbons dewaxing modeling

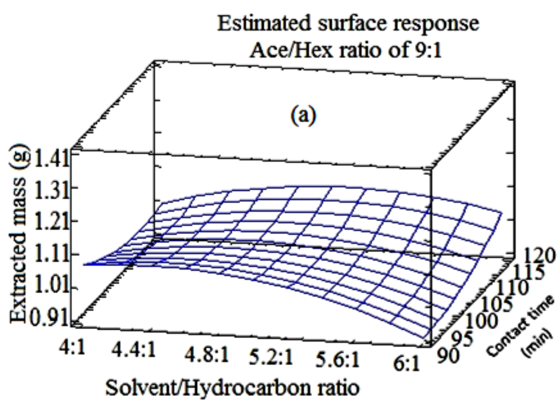
The analysis of the results from the experimental design generated an equation of second order as shown by the equation (1) below.

$$Y = 24.880 - 4.743X_1 - 0.333X_2 - 0.036X_3 + 0.254X_1^2 + 0.070X_1X_2 - 0.066X_2^2 + 0.003X_2X_3 \qquad (1)$$

where X_1 = Acetone/Hexane ratio, X_2 = Solvent/Hydrocarbon Ratio, X_3 = Contact time (Min).

The optimal condition of oily sludge dewaxing given by the model is: Acetone/Hexane ratio 10:1; Solvent/Hydrocarbon Ratio 5,8:1; and Contact time of 120 Min with an amount of extracted paraffins of 1.386 g over a sample of 3 g of paraffinic hydrocarbons extracted from oily sludge. In other words, heavy paraffinic hydrocarbons represent 46.2% of paraffinic hydrocarbons extracted from oily sludge.

The model also allows the estimated surface responses graphic representation as illustrated by Fig. 6 below.



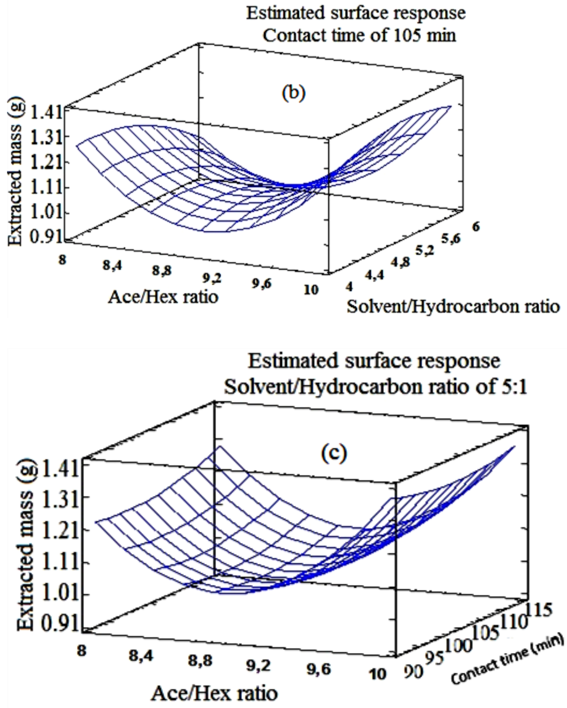


Fig. 6. Estimated response surfaces for extracted masses of paraffins from oily sludge.

C. Identification and quantification of precipitated wax

The gas chromatographic analysis that was realized on each wax extract after being cleaned-up allow the identification and quantification of hydrocarbons having from 12 to 26 carbons.

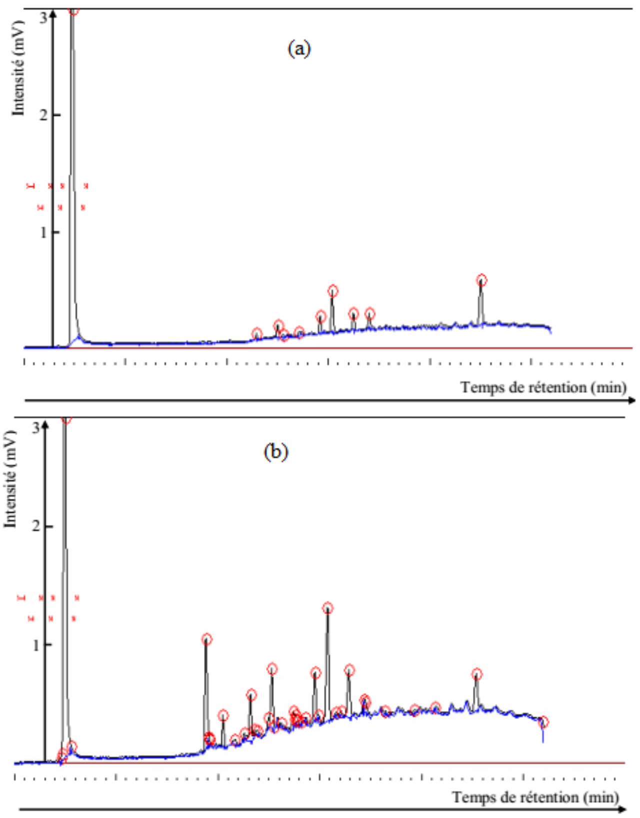


Fig. 7. Spectra of gas chromatographic analysis of wax extracts. (a) Acetone/Hexane ratio 8:1; Solvent/Hydrocarbon Ratio 4:1; and Contact time of 120 Min; (b) Acetone/Hexane ratio 10:1; Solvent/Hydrocarbon Ratio 6:1; and Contact time of 120 Min.

As illustrated by the above Fig. 7, the spectra look similar but presents different compounds along with different quantities.

It has been noted that the amount of extracted wax increases slightly when the Acetone/Hexane ratio is increased till a limit, while other parameters are kept constant. The same observation is made for Solvent/Hydrocarbon ratio. The contrary effect is reveled with contact time showing that for the other parameters kept constant, the amount of extracted was tends to decrease when the contact time increases. These observations are in perfect conformity with the Pareto diagram in Fig. 5 that illustrates the effects and interactions of parameters on the extraction efficiency.

2,6-Dimethylundecane (C13) and Tetracosane (C24) are firstly the only compounds that are found in all extracts and secondly the most abundant compounds with concentrations ranged from 49 to 225 ppm and from 22 to 105 ppm respectively for 2,6-Dimethylundecane (C13) and Tetracosane (C24). Nonadecane and Pristane are absent in extract n°1 (8:1; 4:1; 90) and n°4 (10:1; 6:1; 90) respectively. Phytane is completely absent from all extracts. The extraction condition that gave the highest amount of wax is the extraction n°10 (9:1; 4:1; 105).

A total number of twenty (20) paraffinic hydrocarbons could be identified among which Linear paraffins (3), Paraffins with one ramification (12), Paraffins with two ramifications (2), Paraffin with more than three ramifications (1), and Cyclic paraffin (1). The number of carbons of those compounds were ranged from 13 to 26. Paraffins with 13 to 18 carbons are considered as liquid wax while those with 19 carbons and above are considered as hard wax.

All extracted wax could be shared in seven (6) deferent groups which are Linear paraffins, Paraffins with one ramification, Paraffins with two ramifications, Paraffin with more than three ramifications, Cyclic paraffin, and none identified wax. Their respective proportions in each extract are presented in the Table II below. It came out that paraffins with two ramifications where the most abundant with more than 45% in all extracts followed by linear paraffins.

IV. Conclusion

Solvent extraction conducted on hydrocarbon fraction of oily sludge for wax recovery following an experimental design allowed us to establish a model showing that the rate of wax precipitation is influenced by the three studied parameters. It was also shown that there are interactions existing among these parameters. The most extracted waxes are 2,6-Dimethylundecane (C13) and Tetracosane (C24) which were present in all extract with concentrations ranged from 49 to 225 ppm and from 22 to 105 ppm respectively. The highest amount of extracted wax was obtained at extraction condition of 9:1 for the Acetone/Hexane ratio (named Solvent), 4:1 Solvent/Hydrocarbon ratio and 105 min of contact time.

TABLE II. PROPORTIONS OF RESPECTIVE GROUPS OF EXTRACTED PARAFFINS IN EACH EXTRACT DEPENDING ON ACETONE/HEXANE RATIO, SOLVENT/HYDROCARBON RATIO AND CONTACT TIME

	N°1 (8:1-4:1-90)	*N°2 (10:1-4:1-90)*	*N°3 (8:1-6:1-90)*	*N°4 (10-6-90)*	*N°5 (8-4-120)*	*N°6 (10-4-120)*	*N°7 (8-6-120)*	*N°8 (10-6-120)*	*N°9 (8-5-105)*	*N°10 (9-4-105)*	*N°11 (9-5-90)*	*N°12 (10-5-105)*	*N°13 (9-6-105)*	*N°14 (9-5-120)*	*N°15 (9-5-105)*	*N°16 (9-5-105)*
TEP (ppm)	104	155	292	119	353	273	170	244	199	375	371	300	315	347	270	270
LP (%)	23.08	23.23	22.26	20.17	20.68	20.51	47.65	34.43	37.19	28.80	33.42	31.33	33.97	27.09	33.70	31.48
P1R (%)	2.88	10.32	1.37	0.84	6.52	4.76	0.00	0.00	3.52	10.40	1.35	3.00	1.59	0.00	1.48	6.67
P2R (%)	51.92	50.32	69.52	78.15	62.32	53.48	47.65	61.89	52.26	45.33	61.19	58.33	59.05	70.32	58.89	51.11
P3R (%)	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
P+3R (%)	13.46	14.19	4.79	0.00	9.35	5.13	2.94	3.28	6.53	12.80	3.50	7.33	4.76	2.02	5.93	7.04
CP (%)	0.00	0.00	1.03	0.00	0.00	0.37	1.76	0.41	0.50	0.53	0.54	0.00	0.63	0.58	0.00	0.74
NIW (%)	8.65	1.94	1.03	0.84	1.13	15.75	0.00	0.00	0.00	2.13	0.00	0.00	0.00	0.00	0.00	2.96

LP = Linear paraffins; P1R = Paraffins with one ramification; P2R = Paraffins with two ramifications; CP = Cyclic paraffin; P+3R = Paraffin with more than three ramifications; NIW = None identified wax; TEP = Total extracted paraffins.

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CONFLICT OF INTEREST

Authors declare that they do not have any conflict of interest.

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