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Extraction of Naphthalene using Methanol in Batch and Dynamic Mode, Optimization Applying Response Surface Methodology

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ABSTRACT

A laboratory study on liquid-liquid extraction (LLE) system was carried out in batch and dynamic modes to extract naphthalene from fuel-oil, using methanol as extractant.
This study investigates the optimization of naphthalene extraction using methanol in both batch and dynamic LLE modes.
In batch mode, a Central Composite Design (CCD) was applied to optimize extraction parameters: extraction ratio (R), contact time (t), temperature (T), and stirring speed (SS). In dynamic mode, a Box-Behnken Design (BBD) was used to evaluate the influence of feed input, extraction ratio, and successive extractions.
Batch extraction yielded an optimum naphthalene removal of 80.62% at R = 1.5, t = 5 min, T = 5°C, and SS = 100 rpm. Successive extractions led to complete removal. Dynamic extraction achieved total elimination at 30 mL/h, R = 1.5, and three successive extractions.
This study demonstrates the feasibility of using methanol for efficient naphthalene extraction. Future research should focus on solvent regeneration and industrial-scale applications.

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INTRODUCTION

Naphthalene is a hazardous polycyclic aromatic hydrocarbon (PAH) widely present in industrial emissions, particularly from the steel and coking industries. Exposure to naphthalene is associated with severe environmental and health risks, including air and water contamination, respiratory issues, and potential carcinogenic effects (Zahid et al., 2024). In Annaba, Algeria, steel plants release approximately 14.5 tons of fuel oil contaminated with naphthalene daily, highlighting the urgency of efficient removal methods.

Various techniques have been explored for PAH removal, including adsorption on carbon-based materials (Anbia et *al*., 2009), oxidation (Fazlali et *al*., 2021), and bioremediation (Abdelzaher & Awad, 2022). However, LLE remains a promising approach due to its efficiency in separating organic pollutants. Recent studies have investigated optimized solvent combinations to improve extraction yields (Bendebane et *al*., 2010, 2013, 2014, and 2016). Despite these advances, challenges remain in solvent regeneration and scalability.

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This study aims to develop an optimized methanol-based LLE process for naphthalene removal, evaluating its performance in both batch and dynamic extraction modes.

A key challenge in previous studies was the regeneration of the extractant. However, a new study was carried out using methanol as extractant. Methanol is an advantageous solvent, because its regeneration was easier, and pure naphthalene can be recovered through simple crystallization after decantation of the oil phase. It has been observed that the use of methanol alone resulted in a low yield due to rapid saturation of naphthalene (6%) in methanol. Consequently, a successive dynamic extraction method (Hsieh, et *al.*, 2015; Kula, et *al*., 1982; Lakade, et *al*., 2016; Rieger, et *al.*, 1996; Biery, et *al*., 1963) was developed to improve extraction efficiency.

The aim of this paper is not only to eliminate naphthalene-using methanol as an extractant in both static and dynamic modes but also to regenerate all produces used.

In static mode, we applied a composite centered design (CCD), varying four factors: temperature, time contact, ratio R (volume of the fuel phase on the methanol) and speed stirring SS. This design is the most commonly used response surface designed experiment (Box, et *al*., 1951; Box, et *al*., 1957). Central composite designs are a factorial or fractional factorial design with center points, augmented with a group of axial points (also called star points) that let you estimate curvature.

In second part of this work (dynamic mode), we examine the extraction of naphthalene from organic phase (fuel oil) using a methanol as an extractant. The process was carried out by varying the feed input, the volume ratio O/A (R) and number of successive extraction N according to response surface methodology (RSM) type Box-Behken Design. This design described by George E.P. Box and Donald Behnken. The design included three levels per factor. It should be sufficient to fit a quadratic model, that is, one containing squared terms and products of two factors. Nowadays, it is the most popular second-order designs (Kula, et *al*., 1982; Tinsson, 2010; Goupy, 1999; Box, et *al*., 2007).

BATCH EXTRACTION OF NAPHTHALENE

In the first phase of this study, the extraction of naphthalene from fuel oil was investigated using batch mode. A fuel oil solution containing 7.75% naphthalene was mixed with methanol in a fully stirred reactor to ensure optimal phase interaction. Four key factors—stirring speed, temperature, volume ratio (O/A), and contact time—were systematically varied following the Central Composite Design (CCD) methodology (Table 1). This approach enables a comprehensive evaluation of the influence of these variables on extraction efficiency.

The parameter ranges were selected based on previous studies that demonstrated their critical role in optimizing extraction efficiency (Collin et *al*., 2000; Anbia et *al*., 2009; Fazlali et *al*., 2021; Matar et *al*., 2001; Bendebane et *al*., 2010). By exploring a range of minimum and maximum values for each factor, this study aimed to identify the optimal conditions necessary to achieve high naphthalene removal while ensuring process feasibility and solvent economy.

The yield of extraction was then calculated using Eq. (1).

Table 1. Factors and domain studied

Factors	Units	Levels				
		Min(-1)	Middel (0)	max (+1)	- α	+ α
Temperature (T)	°C	15	25	35	5	45
Ratio (R)	/	1	1.5	2	0.5	2.5
Time (t)	Min	10	15	20	5	25
Stirring Speed (SS)	rpm g/ml	200	300	400	100	500

$$Y_{exp.}\ \% = [1 - \frac{[Naph.]_f}{[Naph.]_0}] \times 100 \quad (1)$$

Where $[Naph.]_f$ and $[Naph.]_0$ represent the concentrations of naphthalene in fuel oil solution at equilibrium and initial times, respectively.

In order to compensate the variation of the temperature from one experiment to another during the use of the refractometer, all the refractive indices are reduced to 20 °C using equation 2 (Bendebane, et *al*., 2010).

$$N_{20} = N_t - 0.0004 \times (20 - t) \quad \text{(Bendebane, et } al\text{., 2010)} \quad (2)$$

Due to the weak solubility of methanol in organic phase, the effect of methanol in the refractive index of fuel oil was affected. So an experimental correcting refractive index N_b 0% of naphthalene was measured for each experience in the same operator conditions. Then the percentage of naphthalene was determined from Eq.(3).

$$[naph.]_f = \frac{(N_m - N_b)}{0,0014} \quad \text{(Bendebane, et } al\text{., 2010)} \quad (3)$$

Where N_m and N_b are the refractive index measured of the fuel oil solution and a refractive index correcting experimental 0% of naphthalene, respectively.

The extraction process was also monitored using infrared spectroscopy, where the primary IR band at 780 cm^{-1} is characteristic of naphthalene. This band becomes smaller with the decrease in the concentration of the naphthalene (Fig. 2). This technique, which is much more qualitative than quantitative, serves mainly to reinforce the results of refractometry.

DYNAMIC EXTRACTION OF NAPHTHALENE

The dynamic study was conducted in a continuous system. The liquid to be treated passes through a continuously stirred reactor in countercurrent mode using a methanol. The concentration of naphthalene decreased from the oil phase to extractant phase. The two phases were then separated, and fresh fuel oil was recovered through decantation.

The figure 3 shows the schema for an experimental dynamic extraction.

RESULTS & DISCUSSION

Batch mode

Table 2 shows the different parameters studied by the CCD design, with their minimal, maximum, average limits and with the two lower and upper constraints (± α).

ANOVA

According to Table 3, the most significant factors influencing extraction efficiency in batch mode were identified an extraction ratio ($p = 0.000$) and the temperature ($p = 0.015$), the square ratio R^2 ($p = 0.008$), SS^2 the square stirring speed ($p = 0.046$) and a single significant interaction between time and stirring speed t×SS ($p = 0.036$). The other factors are not significant. Therefore the yield of the extraction does not vary too much according to these factors.

Polynomial regression

The polynomial models representing the relationship between the extraction yield of naphthalene and the factors studied (T, R t and SS) for the coded and uncoded units are given by equations 4 and 5:

Table 2. Extraction of yield in batch mode using CCD

StdOrder	T	R	t	SS	N_m	N_b	Naph.	Y_{Exp}
					Mes.	Ref.	(%)	%
25	25	1.50	15	300	1.4614	1.4569	3.21	58.53
21	25	1.50	5	300	1.4580	1.4562	1.29	83.41
1	15	1.00	10	200	1.4625	1.4583	3.00	61.29
14	35	1.00	20	400	1.4627	1.4559	4.86	37.33
23	25	1.50	15	100	1.4611	1.4554	4.07	47.47
5	15	1.00	20	200	1.4645	1.4575	5.00	35.48
19	25	0.50	15	300	1.4647	1.4574	5.21	32.72
8	35	2.00	20	200	1.4599	1.4545	3.86	50.23
11	15	2.00	10	400	1.4610	1.4550	4.29	44.70
15	15	2.00	20	400	1.4610	1.4575	2.50	67.74
2	35	1.00	10	200	1.4628	1.4565	4.50	41.94
4	35	2.00	10	200	1.4594	1.4546	3.43	55.76
31	25	1.50	15	300	1.4614	1.4569	3.21	58.53
3	15	2.00	10	200	1.4609	1.4575	2.43	68.66
12	35	2.00	10	400	1.4592	1.4550	3.00	61.29
27	25	1.50	15	300	1.4614	1.4569	3.21	58.53
26	25	1.50	15	300	1.4614	1.4569	3.21	58.53
13	15	1.00	20	400	1.4631	1.4574	4.07	47.47
17	5	1.50	15	300	1.4619	1.4578	2.93	62.21
16	35	2.00	20	400	1.4592	1.4553	2.79	64.06
7	15	2.00	20	200	1.4615	1.4575	2.86	63.13
30	25	1.50	15	300	1.4614	1.4569	3.21	58.53
10	35	1.00	10	400	1.4621	1.4559	4.43	42.86
9	15	1.00	10	400	1.4637	1.4580	4.07	47.47
28	25	1.50	15	300	1.4614	1.4569	3.21	58.53
22	25	1.50	25	300	1.4611	1.4556	3.93	49.31
29	25	1.50	15	300	1.4614	1.4569	3.21	58.53
24	25	1.50	15	500	1.4604	1.4553	3.64	53.00
18	45	1.50	15	300	1.4609	1.4561	3.43	55.76
6	35	1.00	20	200	1.4623	1.4556	4.79	38.25
20	25	2.50	15	300	1.4590	1.4546	3.14	59.45

$$Y_{exp}\% = 58.5300 - 2.3800 \times T + 7.3725 \times R - 3.6867 \times t + 0.3850 \times SS - 0.5008 \times T^2 - 3.7258 \times R^2 + 1.3429 \times t^2 - 2.6883 \times SS^2 + 1.1525 \times T \times R - 0.2300 \times T \times t + 2.5338 \times T \times SS + 3.1113 \times R \times t + 0.1150 \times R \times SS + 3.8025 \times t \times SS \quad (4)$$

$$Y_{exp}\% = 92.6767 - 1.0244 \times T + 34.3350 \times R - 6.3821 \times t - 0.01572 \times SS - 0.0050 \times T^2 - 14.9033 \times R^2 + 0.0537 \times t^2 - 2.6883E\text{-}04 \times SS^2 + 0.2305 \times T \times R - 0.0046 \times T \times t + 0.0025 \times T \times SS + 1.2445 \times R \times t + 0.0023 \times R \times SS + 0.0076 \times t \times S \quad (5)$$

Table 4 shows that the regression, the linear effect and the square effect are significant with a value of p respectively equal to 0.000; 0.000 and 0.025.

Table 3. Regression for yield in batch mode

Termes	Coefficients	Er-T Coeff	T	P
Constant	58,5300	2.510	23.322	0.000
T	-2.3800	1.355	-1.756	0.098
R	7.3725	1.242	5.440	0.000
T	-3.6867	1.242	-2.720	0.015
SS	0.3850	1.242	0.284	0.780
T^2	-0.5008	1.242	-0.403	0.692
R^2	-3.7258	1.242	-3.001	0.008
t^2	1.3429	1.242	1.082	0.295
SS^2	-2.6883	1.242	-2.165	0.046
T×R	1.1525	1.660	0.694	0.497
T×t	-0.2300	1.660	-0.193	0.892
T×SS	2.5338	1.660	1.526	0.146
R×t	3.1113	1.660	1.874	0.079
R×SS	0.1150	1.660	0.069	0.946
t×SS	3.8025	1.660	2.291	0.036

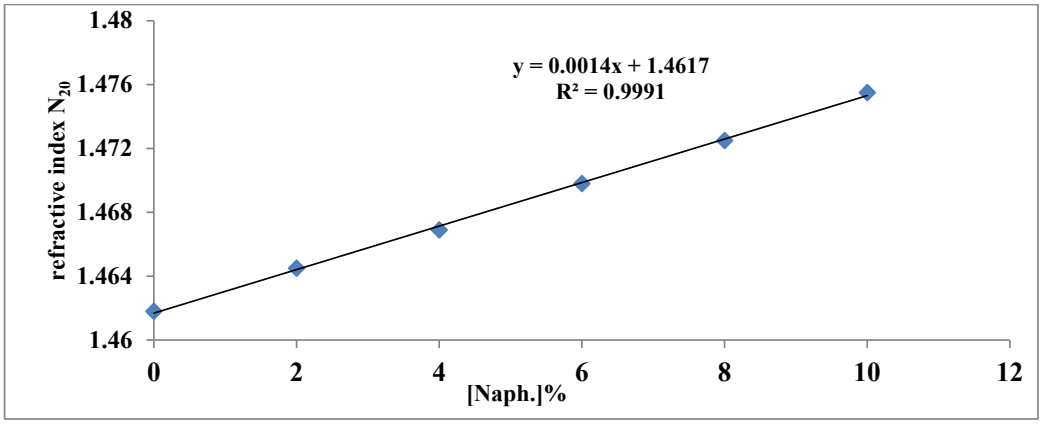

Fig. 1. Calibration curve fuel-naphthalene (Bendebane, et al., 2010).

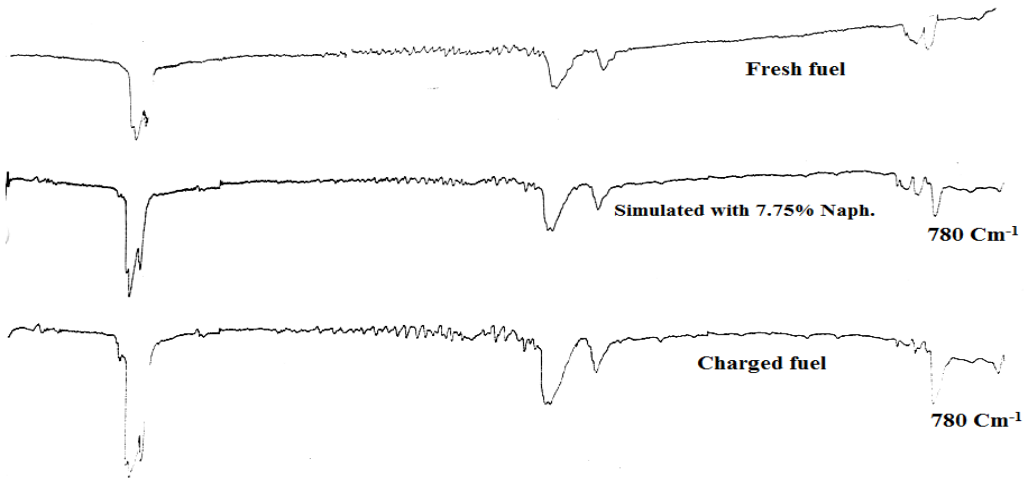

Fig.2. IR spectrum of fuels at different percentages of naphthalene.

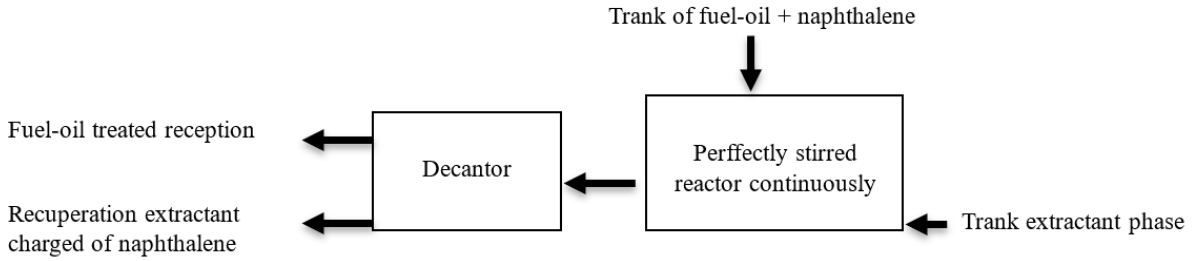

Fig. 3. Schema experimental for the dynamic liquid-liquid extraction of naphthalene.

Table 4. ANOVA for yield in batch mode

Source	DF	SeqSS	adjSS	AdjMS	F	P
Regression	14	2942.54	2942.54	210.18	4.77	0.002
Linear	4	1770.19	1770.19	442.55	10.04	0.000
Square	4	661.10	661.10	165.27	3.75	0.025
Interaction	6	511.25	511.25	85.21	1.93	0.137
Residual Error	16	705.41	705.41	44.09		
Lack-of-fit	10	705.41	705.41	70.54		
Pure Error	6	0.00	0.00	0.00		
Total	30	3647.95				

Response surfaces and contours

Naphthalene is widely found in industrial emissions, particularly from coking plants, petroleum refineries, and coal combustion. Exposure to naphthalene poses significant environmental and health risks, as it is classified as a possible human carcinogen by the U.S. Environmental Protection Agency (EPA) and the International Agency for Research on Cancer (IARC). Chronic exposure has been linked to respiratory issues, eye irritation, liver damage, and hematological disorders (Zahid et al., 2024). Additionally, naphthalene contamination in water sources poses threats to aquatic life due to its persistence and bioaccumulation potential (EPA, 2022).

Considering the environmental concerns associated with solvent use, future studies should explore greener extraction techniques, such as deep eutectic solvents (DES) and bio-based solvents, which have shown potential for selective PAH extraction with reduced toxicity (Koutsoukos et al., 2023).

To develop, improve and optimize processes mathematical and statistical techniques (Box, et *al*., 1978; Coursol, 1980) were collected in Response Surface Methodology (RSM) (Box, et *al*., 2007; Droesbeke, et *al*., 1997; Goupy, 1997; Lewis, et *al.*, 1998; Dagnelie, 2000) (Daudin, et *al*., 2002; Drapper, et *al*., 1996).This method was introduced by George E.P. Box and K.B. Wilson (Box, et *al*., 2007). It is applicable to many disciplines and all industries, because a maximum of information can be obtained with minimum of experiences (Droesbeke, et *al*., 1997; Goupy, 1997; Lewis, et *al.*, 1998; Dagnelie, 2000). Therefore, it is apparent that the response surface methodology not only gives valuable information on interactions between the factors but also leads to identification of feasible optimum values of the studied factors.

From the response and contour surfaces, it can be seen that the maximum extraction efficiency of naphthalene 60-80% is obtained at a minimum level for: a temperature (5-15 °C), a contact time (5min), a stirring speed between 100-200rpm and a range of the volume ratio A/O from minimum to medium (1-1.5). The low extraction yields are represented by the red contours and

Table 5. A table comparing our results with previous studies.

Study	Solvent	Extraction Efficiency (%)	Mode	Reference
This study	Methanol	100% (successive extraction)	Batch & Dynamic	Present work
Fazlali et *al.*	DMSO-Methanol	83.48%	Batch	Fazlali et *al.* (2021)
Zahid et *al.*	Biodegradation	78%	Dynamic	Zahid et *al.* (2024)

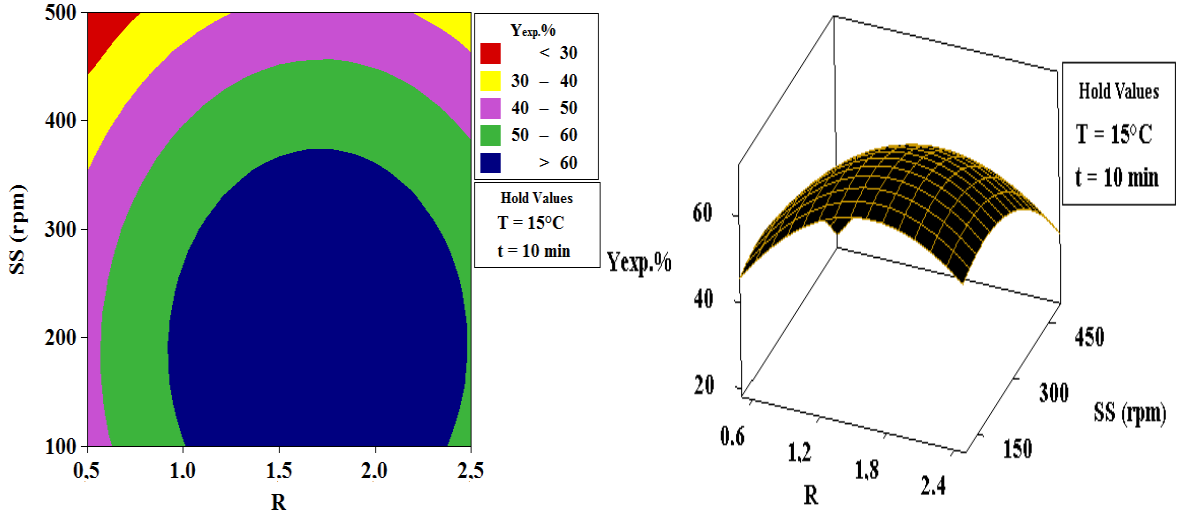

Fig. 4. Contour surface plot and its response surface for yexp. effects of SS and R at T and t min

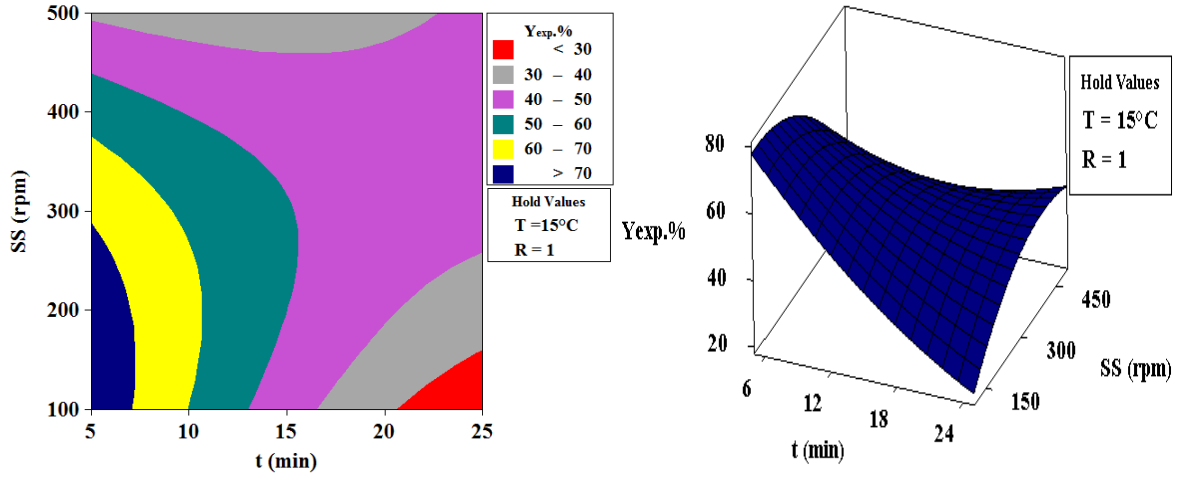

Fig. 5. Contour surface plot and its response surface for yexp. effects of SS and t at T and R min

also by the low of the response surface.

These findings are consistent with previous studies by Bendebane et *al.* (2010), which demonstrated that optimizing parameters such as temperature and volume ratio is crucial for improving naphthalene extraction efficiency.

Additionally, a study by Al Kaisy et *al.* (2014) applied response surface methodology to optimize naphthalene extraction using ionic liquids, further confirming the significance of these operational parameters.

Thus, the present results reinforce the notion that specific operating conditions, particularly

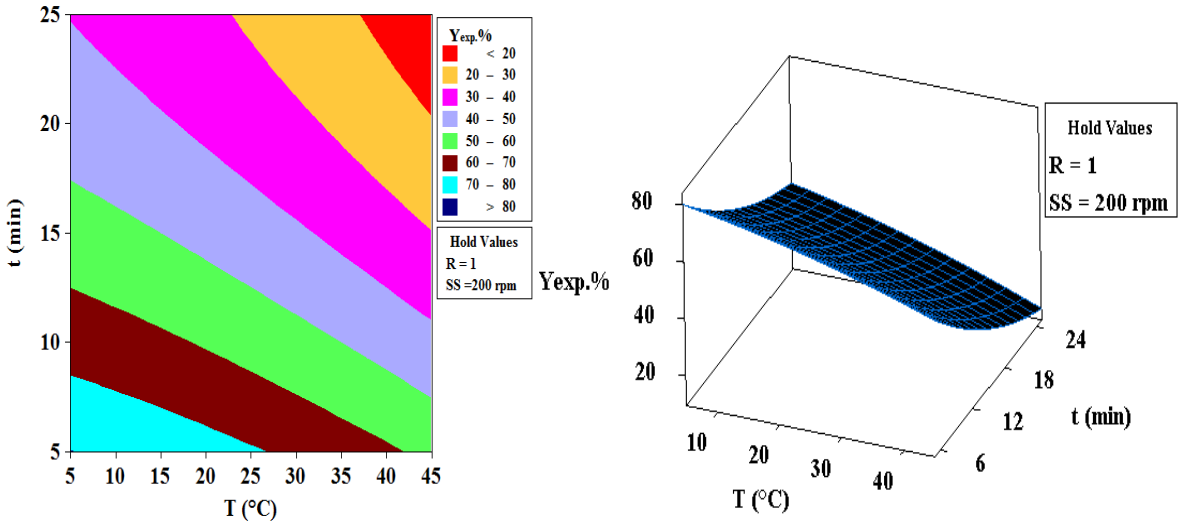

Fig. 6. Contour surface plot and its response surface for yexp. effects of t and T at R and SS min

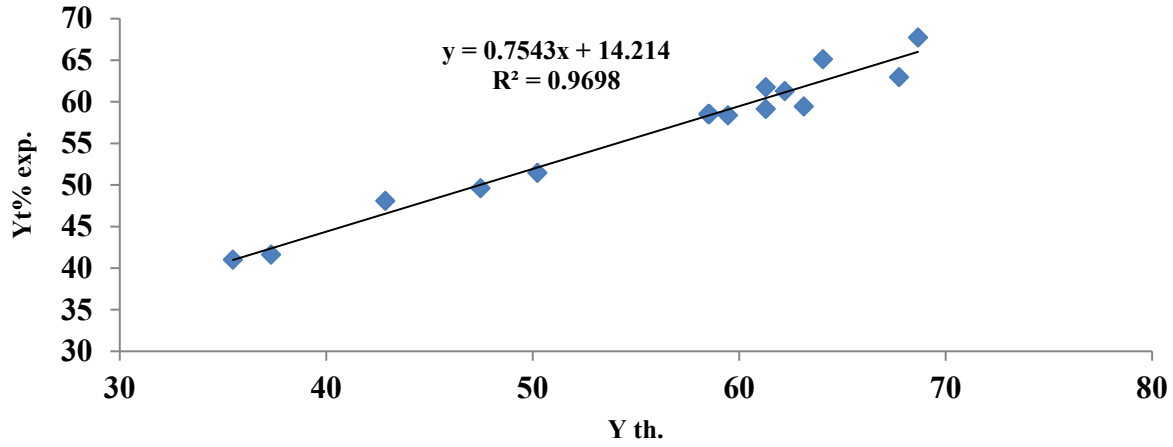

Fig. 7. Correlation between theoretical and experimental yield in batch mode.

low temperature, short contact time, moderate stirring speed, and an appropriate volume ratio, are essential for maximizing naphthalene extraction efficiency (Meziane, 2013).

Below is a table summarizing a comparison between this work and previous ones.

Search for an optimum

Exclusion of factors not significant

If we consider the most important terms of the linear regression are **T**, **t**, **R**, **SS** and only one terms of the interaction regression which is **t** × **SS**, the statistical study of the overall decline gives all interesting p-values less than or equal to **0.05**.

Excluding all non-significant terms one by one until the simplest possible model. The regressions are represented by polynomial equations (6) and (7) corresponding to the coded and uncoded parameters respectively.

$$Y_{exp}\% = 59.3073 - 2.3800 \times T + 7.3725 \times R - 3.6867 \times t + 0.3850 \times SS - 3.8068 \times R^2 - 2.7693 \times SS^2 + 3.8025 \times t \times SS \quad (6)$$

$$\boldsymbol{Y_{exp}\%} = 28.0824 - 0.238000 \times \boldsymbol{T} + 60.4266 \times \boldsymbol{R} - 3.01883 \times \boldsymbol{t} - 0.0559332 \times \boldsymbol{SS} - 15.2272 \times R^2 - 2.76930E\text{-}04 \times SS^2 + 0.00760500 \times \boldsymbol{t} \times \boldsymbol{SS} \quad \textbf{(7)}$$

Regression line representing the experimental extraction Y_{exp} as a function of Y_{th}

According to this regression line representing the experimental extraction yields of naphthalene as a function of the theoretical yields, it is observed that the points are well distributed around the straight line with a correlation constant of **0.969**.

Research of optimum

Several optimizations have been performed; the best is shown in Table.

Successive extraction under the same optimal conditions gave total (100%) removal of the naphthalene.

Dynamic mode

Using a Box-Behnken design with three factors (Daudin, et *al*., 2002; Drapper, et *al*., 1996), the input, the volume ratio O/A (R)and the number of the successive extraction N, the tests were performed. In fact, all manipulations are performed following the experimental design matrix (table 7).

The extraction of naphthalene followed by refractometry and the extraction efficiency is calculated via Equation 1.

Statistical analysis (ANOVA): p-values

Depending on the values of p (table 8), the effect of linear ratio R and the number of the

Table 6. The criteria of optimization in batch mode

Lower	Taget	Factors				$Y_{Theo.}$ %	$Y_{Exp.}$ %
		T (°C)	R	t (min)	SS (rpm)		
60	87	15	1.5	5	100	81.45	78.62

Table 7. Experiment Results by experimental matrix Box-Behnken

N°	Input	R	N	$[naph.]_f$	$Y_{exp.}$ %	$Y_{théor.}$ %
1	60	1.0	2	2.86	63.13	63.13
2	60	1.5	3	0.21	97.24	95.97
3	30	0.5	2	3.36	56.68	54.38
4	60	0.5	1	5.36	30.88	32.15
5	60	1.0	2	2.86	63.13	63.13
6	30	1.0	1	3.29	57.60	58.64
7	60	0.5	3	3.14	59.45	58.87
8	90	1.0	1	3.14	59.45	56.57
9	30	1.0	3	1.00	87.10	89.98
10	60	1.5	1	2.93	62.21	62.79
11	90	0.5	2	3.21	58.53	60.14
12	90	1.5	2	1.36	82.49	84.79
13	60	1.0	2	2.86	63.13	63.13
14	30	1.5	2	0.07	99.08	97.47
15	90	1.0	3	1.07	86.18	85.14

Table 8. Regression of linear factors, squares and their interactions

Termes	Coefficients	Er-T coef	T	P
Constant	63.1300	1.6007	39.438	0.000
Input	-1.7262	0.9802	-1.761	0.139
R	16.9350	0.9802	17.276	0.000
N	14.9788	0.9802	15.281	0.000
$(Input)^2$	10.6012	1.4429	7.347	0.001
$(R)^2$	0.4637	1.4429	0.321	0.761
$(N)^2$	-1.1488	1.4429	-0.796	0.462
Input × R	-4.6100	1.3863	-3.325	0.021
Input × N	-0.6925	1.3863	-0.500	0.639
R ×N	1.6150	1.3863	1.165	0.297

Table 9. Analysis of variance for yield extraction efficiency.

Source	DF	SeqSS	AdjSS	AdjMS	F	P
Regression	9	4639.92	4639.92	1371.03	67.07	0.000
Linear	3	4113.10	4113.10	1371.03	178.36	0.000
Square	3	429.46	429.46	143.15	18.62	0.004
Interaction	3	97.36	97.36	32.45	4.22	0.078
Residual Error	5	38.44	38.44	7.69		
Lack-of-fit	3	38.44	38.44	12.81	*	*
Pure Error	2	0.00	0.00	0.00		
Total	14	4678.35				

successive extraction N are highly significant, which the values of p is equal zero. For the factors square and interaction, it has been found only one signification with a p = 0.001 for square input and p = 0.021 for interaction input-R. However, the other terms do not have a significant effect on the studied phenomenon.

Effects of factors

From table 9: we found highly significant values with a regression model, linear and square terms. We also observed that there are no significant interactions.

Mathematic model

The regressions are represented by polynomial equation 8 and 9 corresponding to the coded and uncoded parameters respectively.

$$Y_{dymanic}\% = 63.1300 - 1.7262\times Input + 16.9350\times R + 14.9788\times N + 10.6012\times Input^2 + 0.4637\times R^2 - 1.1488\times N^2 - 4.6100\times Input\times R - 0.6925\times Input\times N + 1.6150\times R\times N \quad (8)$$

$$Y_{dynamic}\% = 27.6700 - 1.11754\times Input + 42.1400\times R + 17.7287\times N + 0.0117792\times Input^2 + 1.85500\times R^2 - 1.14875\times N^2 - 0.307333\times Input\times R - 0.0230833\times Input\times N + 3.23000\times R\times N \quad (9)$$

Response surfaces and contours

The extractions of naphthalene in dynamic mode using methanol are tested. The three factors

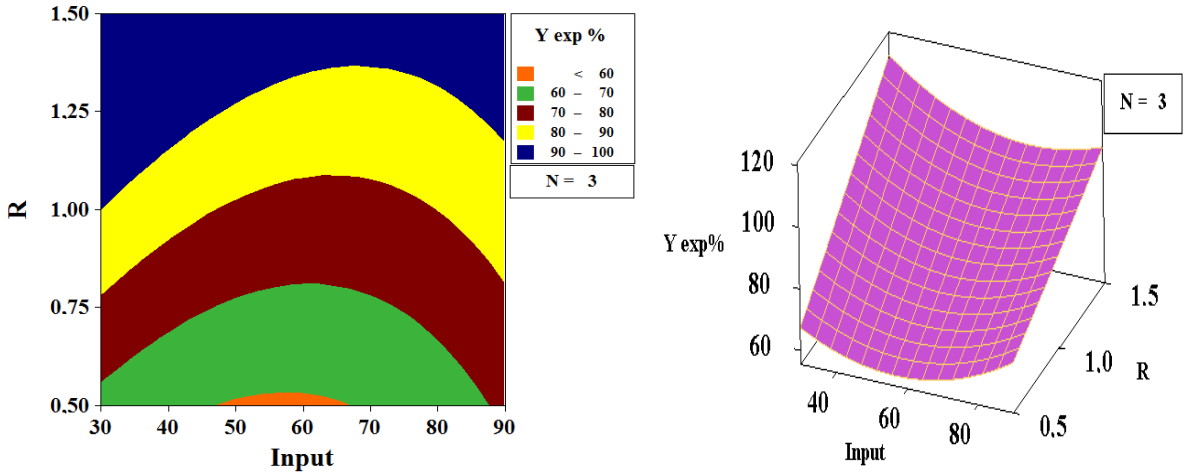

Fig. 8. Contour surface plot and its response surface for yield dynamic: effects of Input and R at N max

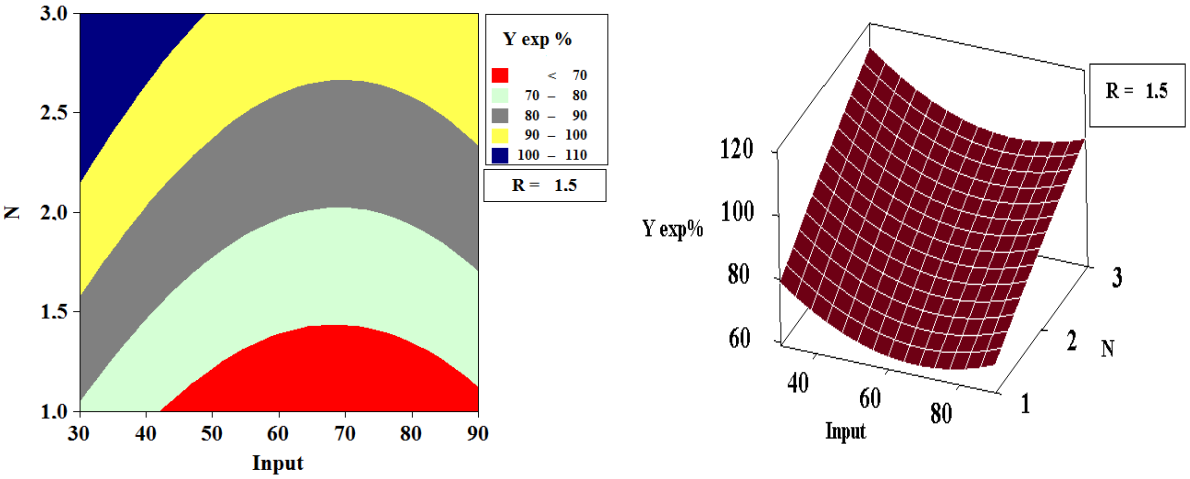

Fig. 9. Contour surface and response surface of yield dynamic according to Input and N at R min

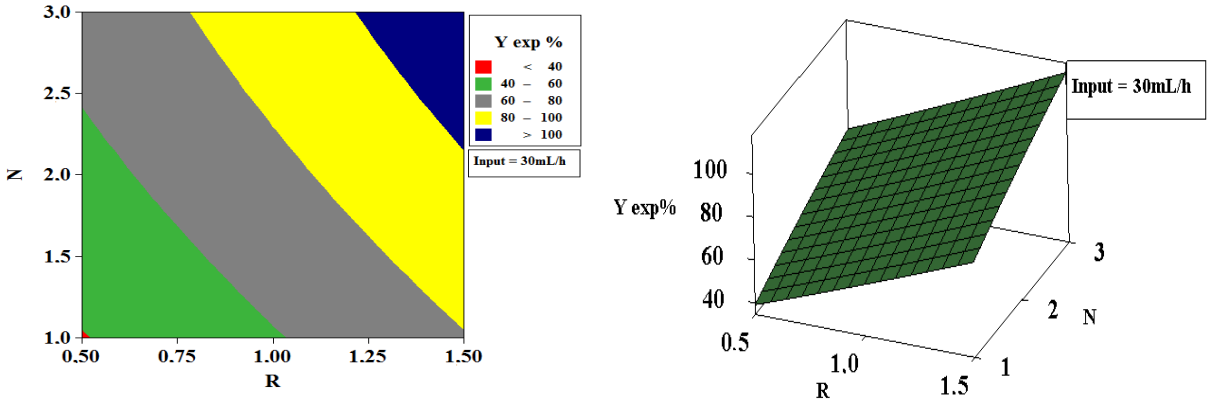

Fig. 10. Contour surface and response surface of yield dynamic according to R and N at Input min.

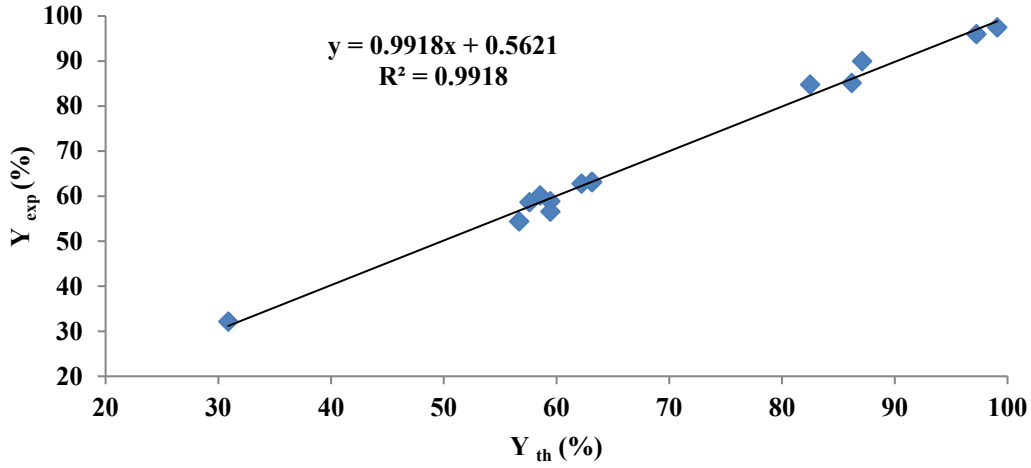

Fig. 11. Correlation between theoretical and experimental yield.

Table 10. The criteria of optimization

Parameters	Lower	Target	Upper	FACTORS			$Y_{dyn.\ theo}$ %	Desirability
				Input (mL/h)	R	N		
	60	100	100	30	1.5	3	100	1

(Input, ratio R and number of successive extraction N) are varying simultaneously in the study area chose applying Box-Behnken design (Drapper, et *al*., 1996). It has been found that a total extraction yield was obtained after 3 successive extractions with R = 1.5 and an Input = 30ml/h.

These results are consistent with previous studies particularly that of Bendebane et al. (2016), who used an optimized solvent combination to extract naphthalene from fuel in dynamic mode, achieving an extraction yield of 79%.

Furthermore, Fazlali et al. (2021) optimized the extraction of naphthalene from cracked fuel using a crystallization method followed by solvent extraction, achieving a purity of 99.42% and an extraction yield of 97.36%.

These studies demonstrate the efficiency of optimized extraction methods for removing naphthalene from petroleum matrices, highlighting the importance of parameters such as volume ratio, feed input, and the number of successive extractions.

A comparison between batch and dynamic models highlights key differences in extraction efficiency, process feasibility, and scalability.

In the batch model, the extraction process is carried out in a single-stage reactor with controlled stirring. This method allows precise control over parameters such as temperature, stirring speed, and contact time. The batch mode yielded an optimal naphthalene removal of 80.62% under specific conditions (R = 1.5, t = 5 min, T = 5°C, and SS = 100 rpm). However, to achieve complete removal, multiple successive extractions were necessary, which increases the process duration and solvent consumption.

On the other hand, the dynamic model enables continuous extraction, significantly improving process efficiency. With a feed input of 30 mL/h, R = 1.5, and three successive extractions, total naphthalene elimination was achieved. The continuous operation enhances mass transfer, reduces solvent usage per extraction cycle, and makes the process more suitable for industrial applications. Additionally, the dynamic mode offers better scalability since it can be integrated

into large-scale separation systems with minimal downtime.

In conclusion, while the batch model allows precise control of operational variables and is well-suited for small-scale laboratory studies, the dynamic model is more efficient for large-scale applications. The choice between these two methods depends on the specific requirements of the extraction process, including desired purity, process duration, and economic feasibility.

COMPARISON BETWEEN Y_{exp} AND Y_{th} in dynamic mode

To check the model, a comparison between theoretically and experimentally values was examined. Indeed, from the figure 13 we ought to say that the points are fairly distributed around the regression line with a correlation constant of 0.991. This proves that the mathematical model represents well the extraction phenomenon in this context.

Optimization

Exclusion of terms

The result of the exclusion is shown in equations 10 and 11.

$$\boldsymbol{Y\,\% = 62.739 - 1.726 \times Input + 16.935 \times R + 14.979 \times N + 10.650 \times Input^2 - 4.610 \times Input \times R} \quad \textbf{(10)}$$

$$\boldsymbol{Y\,\% = 54.8392 - 1.17023 \times Input + 52.3100 \times R + 14.9788 \times N + 0.0118335 \times Input^2 - 0.307333 \times Input \times R} \quad \textbf{(11)}$$

Research of optimum

Several optimizations have been carried out in order to maximize the extraction efficiency in dynamic mode. The best optimization results are summarized in Table 10.

For the purpose of optimizing the dynamic extraction yield, the functionality of minitab was used by fixing the lower and target values we obtain a global solution for the three factors: A Input of 30 mL/h, a ratio of 1.5 and N = 3 for a theatrically response of 100% and desirability equal 1.

Although batch mode proves valuable for exploratory studies thanks to its precise control of experimental parameters, its effectiveness remains limited without multiple extraction cycles. In contrast, the continuous mode stands out for its superior overall efficiency and better suitability for industrial applications, achieving complete extraction in fewer steps and demonstrating strong potential for scale-up.

In summary, for large-scale operations where substantial volumes need to be treated, the continuous mode offers clear advantages. However, batch mode retains its importance for laboratory trials and small-scale production, where flexibility and control are essential.

CONCLUSION

This study examined the extraction of naphthalene using methanol in both batch and continuous modes. In batch mode, an initial extraction yield of 78.6% was achieved under optimal conditions (15°C, R = 1.5, 5 minutes, 100 rpm), with successive extractions allowing complete removal of naphthalene from fuel oil. In continuous mode, the extraction efficiency was optimized through three key parameters: input flow rate, ratio R, and the number of successive extractions. Under optimal conditions (30 ml/h, R = 1.5, N = 3), a theoretical 100% extraction yield was obtained and validated experimentally.

Methanol regeneration by distillation was highly effective, achieving over 90% recovery, while naphthalene was recovered at 88.8% purity, verified by melting point analysis. Although the method shows great promise, challenges remain for industrial scaling, including process

optimization, solvent reuse, and compliance with environmental regulations.

The findings highlight the potential of this process for large-scale naphthalene recovery, offering economic and environmental benefits by conserving fuel and recycling solvents. Future research should target industrial-scale optimization, alternative green solvents, and enhanced purification methods to maximize recovery and sustainability.

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The present research did not receive any financial support.

CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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REFERENCES

Anbia, M., & Moradi, S. E. (2009). Removal of naphthalene from petrochemical wastewater streams using carbon nanoporous adsorbent. *Applied Surface Science, 255*(9), 5041–5047. https://doi.org/10.1016/j.apsusc.2008.12.065

Abdelzaher, M. A., & Awad, M. M. (2022). Sustainable development goals for the circular economy and the water-food nexus: Full implementation of new drip irrigation technologies in Upper Egypt. *Sustainability, 14*(21), 13883. https://doi.org/10.3390/su142113883

Bendebane, F., Bouziane, L., & Ismail, F. (2010). Extraction of naphthalene: Optimization and application to an industrial rejected fuel oil. *Journal of Industrial and Engineering Chemistry, 16*(2), 314–320. https://doi.org/10.1016/j.jiec.2010.01.033

Bendebane, F., Bouziane, L., & Ismail, F. (2013). Liquid-liquid extraction of naphthalene: Application of a mixture design and optimization. *Energy Procedia, 36*, 1241–1248. https://doi.org/10.1016/j.egypro.2013.07.140

Bendebane, F., Ismail, F., & Bouziane, L. (2014). Extraction of naphthalene from an organic phase with the mixture DMSO-methanol-phenol in batch mode: Application of a composite centered design (CCD). *Energy Procedia, 50*, 642–651. https://doi.org/10.1016/j.egypro.2014.06.079

Bendebane, F., Mohammedi, D., Boukhari, A., & Ismail, F. (2016a). Modeling and optimization of naphthalene extraction from fuel oil: Application of a mixture design. *Chem Xpress, 9*(2), 148–155.

Bendebane, F., Halaimia, F., Bahloul, L., Bouziane, L., & Ismail, F. (2016b). [Title missing]. *Pollution Research, 35*(4), 23–29.

Biery, J. C., & Boylan, D. R. (1963). Dynamic simulation of liquid-liquid extraction column. *Industrial & Engineering Chemistry Fundamentals, 2*(1), 44–50. https://doi.org/10.1021/i160005a009

Box, G. E. P., & Wilson, K. B. J. (1951). [Title missing]. *Journal of the Royal Statistical Society: Series B (Statistical Methodology), 13*, 1–45.

Box, G. E. P., & Hunter, J. S. (1957). [Title missing]. *Annals of Mathematical Statistics, 28*(1), 195–241.

Box, G. E. P., & Draper, N. R. (2007). *Response surfaces, mixtures, and ridge analyses* (Vol. 649). John Wiley & Sons.

Box, G. E. P., Hunter, W., & Hunter, J. R. (1978). [Titre manquant]. *Journal of the Royal Statistical Society: Series A (General), 141*(2). *(À compléter si tu as le titre)*

Coursol, J. (1980). *Technique statistique des modèles linéaires*. CMPA, Nice-Paris.

Collin, G., Höke, H., & Greim, H. (2000). Naphthalene and hydronaphthalenes. In *Ullmann's encyclopedia of industrial chemistry*. Wiley-VCH. https://doi.org/10.1002/14356007.a17_001.pub2

Dagnelie, P. (2000). La planification des expériences : choix des traitements et dispositif expérimental. *Journal de la Société Française de Statistique, 141*(1–2), 5–29.

Daudin, J. J., & Duby, C. (2002). *Techniques mathématiques pour l'industrie agroalimentaire*. *(Maison d'édition à préciser si possible)*

Draper, N. R., Lin, D. K. J., Ghosh, S., & Rao, C. R. (1996). *Response surface designs* (Vol. 13). Elsevier Science.

Droesbeke, J.-J., Saporta, G., & Fine, J. (1997). *Plans d'expériences : applications à l'entreprise*. Éditions Technip.

EPA. (2022). *Naphthalene: Health and environmental effects*. United States Environmental Protection Agency. *(Lien à ajouter si disponible)*

Fazlali, A., Ghalehkhondabi, V., & Asadi, Z. (2021). Optimization of naphthalene extraction from cracked fuel oil (CFO) in Shazand petrochemical complex. *Petroleum Research, 31*(116), 11–13. https://doi.org/10.22078/pr.2020.4127.2873

Goupy, J. (1999). Plan d'expériences pour surface de réponse. Dunod. ISBN 2-10-005765-0.

Goupy, J. (1997). Plans d'expériences (Vol. P230). Dunod.

Hsieh, K.-T., Liu, P.-H., & Urban, P. L. (2015). Automated on-line liquid–liquid extraction system for temporal mass spectrometric analysis of dynamic samples. Analytical Chimica Acta, 894, 35–43. https://doi.org/10.1016/j.aca.2015.08.045

Al Kaisy, G. M. J., Mutalib, M. I. A., Bustam, M. A., & Razali, R. B. (2014). Optimization of naphthalene extraction from its mixture with base oil and dibenzothiophene by BMIMDMP ionic liquid using response surface methodology (RSM). Applied Mechanics and Materials, 625, 50–56. https://doi.org/10.4028/www.scientific.net/AMM.625.50

Kula, M.-R., Kroner, K. H., & Hustedt, H. (1982). Purification of enzymes by liquid-liquid extraction. In Reaction Engineering (pp. 73–118). Springer. https://doi.org/10.1007/3-540-11699-0_11

Koutsoukos, S., Papadopoulou, M., Nikolaou, A., & Kalogianni, E. (2023). Application of deep eutectic solvents for the extraction of polycyclic aromatic hydrocarbons (PAHs). Applied Sciences, 13(6), 3502. https://doi.org/10.3390/app13063502

Lakade, S. S., Borrull, F., Furton, K. G., Kabir, A., Marcé, R. M., & Fontanals, N. J. (2016). Dynamic fabric phase sorptive extraction for a group of pharmaceuticals and personal care products from environmental water. Journal of Chromatography A, 1456, 19–26. https://doi.org/10.1016/j.chroma.2016.05.097

Lewis, G. A., Mathieu, D., & Phan-Tan-Luu, R. (1998). Pharmaceutical experimental design. CRC Press.

Matar, S., & Hatch, L. F. (2001). Chemistry of petrochemical processes (2nd ed., Chapter 2). Gulf Professional Publishing.

Meziane, S. (2013). Optimization of oil extraction from olive pomace using response surface methodology. Food Science and Technology International, 19(4), 315–322. https://doi.org/10.1177/1082013212452476

Rieger, R., Weiss, C., Wigley, G., Bart, H.-J., & Marr, R. (1996). Investigating the process of liquid-liquid extraction by means of computational fluid dynamics. Computers & Chemical Engineering, 20(12), 1467–1475. https://doi.org/10.1016/0098-1354(95)00232-4

Tinsson, W. (2010). Plans d'expérience : constructions et analyses statistiques (Vol. 67). Springer Science & Business Media.

Aziz, Z. S., Jazza, S. H., Dageem, H. N., Banoon, S. R., Balboul, B. A., & Abdelzaher, M. A. (2024). Bacterial biodegradation of oil-contaminated soil for pollutant abatement contributing to achieve sustainable development goals: A comprehensive review. Results in Engineering, 22, 102083. https://doi.org/10.1016/j.rineng.2024.102083