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Optimization of chromium extraction from aqueous solutions by emulsion liquid membrane

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ABSTRACT

Taguchi method has been realized efficient for output improvement in research and development stage; so that high-quality items can be quickly produced at lower costs. Optimization of emulsion liquid membrane (ELM) for chromium extraction is achieved by Taguchi method. One of the main objectives of this work deals with the extraction of Cr(VI) using tri-octyl phosphine oxide (TOPO) as a carrier. Six parameters, namely carrier, surfactant, and internal phase concentration, feed phase pH, stirring speed and emulsion/external feed phase ratio, were chosen as the control factors while the initial concentration of chromium was chosen as the external noise factor. The results have been analyzed using analysis of variance (ANOVA) and signal-to-noise (S/N) ratio. From our results, combinations of all factors studied have a potential interaction. It can be noticed that the optimum external to emulsion ratio is the highest level. Indeed, this trend leads to treating large volumes of more dilute Cr(VI) solutions. This reflects the importance of ELM in which our system deviates from equilibrium that means a high mass transfer occurred. Maximum contribution for Cr(VI) extraction is obtained as 89.498% for TOPO concentration followed by Span 80 (4.393%), and the other parameters have a lower significance on ELM process (ranging from 1.750% to 1.299%).

Keywords: Emulsion liquid membrane; Chromium; Optimization; TOPO; Taguchi method

1. Introduction

Chromium compounds are applied in many industrial processes, e.g., in metal electroplating, glass coloring, textile dyeing, preservation of wood, and leather processing as a tanning agent. However, such processes generate liquid and solid wastes containing large quantities of Cr(VI) and Cr(III) compounds. Effluents and solid wastes containing compounds of chromium create an environmental hazard and risk for the humane health when they are discharged without purification into the landfill or rivers. Therefore, recovery of chromium (III) from solid and liquid industrial wastes has an ecological aspect [1].

Because of Cr(VI) extreme toxicity, a number of studies have been devoted to its removal by solvent extraction using tricaprylmethylammonium chloride [1–4] and tri-*n*-butyl phosphate [5].

Emulsion liquid membranes (ELM) is known to be one of the most effective methods for separation and concentration when the material being extracted is present in very low concentration. As a result, ELM has been considered a promising alternative technology for diverse separation processes including removal and recovery of various heavy metals such as copper, zinc, nickel, cadmium and chromium [6]. ELM combines a single step extraction and stripping processes that are generally carried out in two separate steps in conventional solvent extraction processes. ELM is created by forming a stable emulsion, such as a water-in-oil emulsion, between two immiscible phases, followed by dispersion of the emulsion into a third, continuous phase by agitation. The membrane phase is the oil phase that separates the encapsulated, internal phase droplets in the emulsion from the external feed phase [7].

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Recently, ELM has gained greater attention for the removal of chromium from aqueous effluents using tri-octyl phosphine oxide (TOPO) [8], tri-*n*-butyl phosphate (TBP) [7,9], tri-*n*-octyl methylammonium chloride [10–12], cyanex 923 [13–15], Amberlite LA-2 [16] and 2-ethylhexyl phosphonic acid mono-2-ethylhexyl ester (PC-88A) [17].

The conventional studies during the development of a process involve a variation of one factor at a time, keeping all other factors constant. The different range of values used as an optimum for the other parameters cannot be fully explained, and furthermore, the optimum level of these factors might also be influenced by the level of the other important factors. But, the experiments conducted using the factorial designs enable factors to vary simultaneously. This helps in quantifying the interactive effects of the test variables. Moreover, experimental designs can be changed easily until a suitable target is found.

The Taguchi method established by Genichi Taguchi has been generally adopted to optimize the design variables because it can significantly reduce the overall testing costs and time. The method uses a specifically designed orthogonal array consisting of controllable parameters and their variation levels to optimize experimental conditions [18]. Taguchi method has been realized efficient for output improvement in research and development stage so that high-quality items can be quickly produced at lower costs [19]. It has been recently used for chromium removal in a rotating packed bed contractor [20], optimizing Fenton process for the removal of amoxicillin from the aqueous phase [21], adsorption of Cr(VI) from aqueous solutions by NiO nanoparticles [22] and Ni(II) removal from wastewater by calcined oyster shell powders [23]. Few studies have been done on the application of Taguchi methodology for the separation of gold ions [24], amino acids [25], and arsenic [26] by ELM.

Finally, it is noticeable that despite the vast research being conducted on the extraction of chromium by ELM; almost none of them has employed the Taguchi design method to investigate the optimum parameters.

The main objective of this study is to find out the applicability of Taguchi method to obtain the optimum operating conditions and the interactive effects of process parameters for chromium extraction by ELM. The operating parameters such as pH, internal phase, and surfactant concentration, mixing time, carrier concentration and external/emulsion ratio are chosen for the purpose.

2. Experimental

2.1. Reagents

The liquid membrane is composed of a surfactant, carrier and a diluent. The surfactant is Sorbitan monooleate, which is a product of Sigma-Aldrich Chemicals Co., UK and commercially known as Span 80. The mobile carrier is TOPO, which is purchased from Sigma-Aldrich Chemicals Co. Commercial kerosene (density 830 kg/m^3 and viscosity 7.3 mPa s at 20°C) was used as diluent. 1 M stock solution of TOPO in kerosene was prepared and used as stock solution. Sulfuric acid, sodium hydroxide and all other chemicals were used directly as received from the manufacturer. The external feed phases were 300 ppm solutions of Cr(VI) ion dissolved in distilled water. They were freshly prepared from 1,000 ppm Cr(VI) stock aged solutions.

2.2. ELM experiments

The emulsion was prepared in a 500 ml beaker by dispersing 200 ml internal aqueous phase into 200 ml of organic phase. The organic phase was prepared by dissolving TOPO in kerosene and an appropriate amount of nonionic surfactant (Span 80) was added. The organic phase was homogenized for up to 2 min by ultraturax T25 homogenizer at 7,500 rpm. The emulsion was formed by adding internal aqueous phase (H_2SO_4 solution) dropwise into the organic phase, keeping the whole mixture homogenized for the next 5 min. The ratio of internal to organic phase (I/O) was kept at 1:1 for all the experiments. The surfactant concentration (Span 80) was ranged from 3% to 10% (v/v) to provide sufficient stable emulsion. 15 ml of the prepared emulsion was poured into another 250 ml beaker containing Cr(VI) feed phase of 300 ppm. The ratio of feed phase to emulsion was varied from 1 to 20. The pH of the feed phase was changed in the range 0.5–5. Sulfuric acid and sodium hydroxide solutions were used to adjust the pH of the feed phase to the desired value. The whole mixture was gently stirred by a magnetic stirrer, and an agitation speed of 500 rpm was found to be the best to generate fine globules of emulsion with lowest possible breakage. After a certain mixing time, separation of the emulsion was done using a cylindrical separating funnel. 10 ml samples feed phase were taken out, filtered and analyzed using ICP spectrophotometer (Perkin Elmer Optima 5300 DV).

2.3. Experimental design

To evaluate the effects of ELM parameters on extraction percentage of chromium and to identify the performance characteristics under the optimal extraction parameters, a specially designed experimental procedure is required. Classical experimental design methods are complex and difficult to use. Additionally, a large number of experiments have to be carried out when a number of parameters increases [19,27–29]. In this study, Taguchi technique was used to determine optimal extraction parameters for maximum extraction percentage. In the Taguchi technique, process parameters, which influence the products, are separated into two main groups: control factors and noise factors [30]. The control factors are used to select the best conditions for stability in the design of extraction process, whereas the noise factors denote all factors that cause variation. The most appropriate design is selected based on the best levels of control factors having maximum signal-to-noise (S/N) ratios. Taguchi method also considers the effects of noise factors, which are inconvenient to control. It is required to consider as an outer array to understand the effect of the noise factor. Moreover, the optimum condition will be insensitive to the noise factor [20].

Taguchi proposed to acquire the characteristic data by using orthogonal arrays and to analyze the performance measure from the data to decide the optimal process parameters. This method uses a special design of orthogonal arrays to study the entire parameter space with a small number of experiments only.

In Taguchi method, a loss function is used to calculate the deviation between the experimental values and the desired value. This loss function is further transformed into an S/N ratio. There are several S/N ratios available depending on a type of characteristics; lower is better (LB), nominal is better (NB) and higher is better (HB) [19]. For HB, the definitions of the loss function for extraction results Y_i of n repeated number is:

$$\frac{S}{N_B} = -10\log\left[\frac{1}{n}\right]\sum_{i=1}^{n} Y_i^{-2} \quad (1)$$

In this study, six parameters were used as control factors, and each parameter was designed to have five levels (Table 1). Initial Cr(VI) concentration of 300 ppm was considered as noise factor 1. According to the Taguchi quality design concept, an L25 orthogonal array table with 25 rows (corresponding to the number of experiments) was chosen for the experiments (Table 2). Each run was repeated three times. Minitab 16 software was used for analysis of the results and optimization of condition to arrange the control factors. To compare the effect of each factor on the extraction of chromium, the average S/N ratio of each level for each factor is calculated according to what level is used in which experiments. For example, level 1 for TOPO concentration factor is used in experiments 1–5, so the average S/N ratio for this level of the mentioned factor is equal to average S/N ratios of experiments 1–5.

3. Analysis of experimental results based on Taguchi methodology

3.1. Interactions of ELM controlling factors

Interactions between ELM variables affect process efficiency to a large degree. The interactions between the parameters are clearly presented in Fig. 1. When the slopes of two lines differ considerably, and they cross in the range tested, there is a potential interaction between the two parameters. From Fig. 1, it can be observed that the combinations of all factors studied have a potential interaction.

3.2. Effect of ELM parameters on chromium extraction

The observed extraction percentage and the results of S/N ratios for the L25 orthogonal array are given in Table 2. In general, the aim of any experiment is always to determine the highest possible S/N ratio that indicates the best database on the basis of minimum deviation and leads to the selection of optimum levels of each factor that are closest to the target [31]. Based on the experimental results shown in Table 2, an analysis of variance (ANOVA) is performed in order to estimate the predictive accuracy of the model. Also, by using Eq. (1), the S/N ratios for the five levels of each control factor are computed and tabulated in Table 2 to determine the relative significances of the different parameters. From Table 2, it can be seen that the optimal extraction performance (maximum S/N ratios value) was obtained at TOPO concentration 0.15 M (Level 3) with different levels of other factors.

Fig. 2 shows the main effects of ELM parameters on the mean values of chromium extraction.

By inspection of Fig. 2, we noticed that the higher absolute slope value of each definite factor of chart means a greater influence of that factor on the amount of chromium extraction. Considering this description, it is concluded that TOPO concentration has the most significant influence on the amount of chromium extraction. By increasing TOPO concentration from 0.05 to 0.2 M, the average S/N ratio increases from 34.5451 to 39.

According to Fig. 3, the increase of the TOPO and Span 80 concentrations lead, in general, to an increase in the chromium extraction percentage. This is due to the fact that increasing TOPO concentration gives more sites to Cr(VI) ions to form complex with TOPO molecules, but increasing Span 80 concentration increases the stability of emulsion and makes small globules when the emulsion is dispersed in the external aqueous phase.

The combination of these two factors may increase the possibilities of Cr(VI) ions to form complex with TOPO through a more stable emulsion, leading to an increase in chromium extraction. To compare the effect of each factor on the extraction of chromium, the average S/N ratio of each level for each factor is calculated according to what level is used in which experiments.

To make comparison clearer, the amounts of average S/N ratios are shown in a 3-D bar-type chart. The higher average S/N ratio for a level indicates that level is of more importance [19] and has the greater influence on chromium extraction. Regarding Fig. 3, it can be observed that fourth and fifth levels of TOPO concentration are sufficient to give higher average S/N ratios. Economically, level 4 is sufficient to give higher percentage extraction.

Table 1
Experimental parameters and levels

Independent variable	Levels					Observed parameter
	1	2	3	4	5	
X1 (carrier concentration, M)	0.05	0.10	0.15	0.20	0.25	Extraction, %
X2 (surfactant concentration, v%)	3	5	7	8	10	
X3 (internal phase concentration, M)	0.10	0.30	0.50	0.70	1.00	
X4 (external/emulsion ratio)	1	5	10	15	20	
X5 (stirring time, min)	1	5	10	20	30	
X6 (external phase, pH)	0.5	1	2	3.5	5	

Table 2
Experimental design orthogonal array, observed values and S/N ratios

Run	Input parameters						Observed Extraction, %	Calculated S/N ratios
	Carrier conc., M	Surfactant conc., v%	Internal phase conc., M	External/ emulsion ratio	Stirring time, min	External phase pH		
1	0.05	3	0.10	1	1	0.5	40.768	32.2064
2	0.05	5	0.30	5	5	1	49.998	33.9790
3	0.05	7	0.50	10	10	2	52.898	34.4688
4	0.05	8	0.70	15	20	3.5	61.058	35.7149
5	0.05	10	1.00	20	30	5	65.739	36.3564
6	0.10	3	0.30	10	20	5	72.135	37.1629
7	0.10	5	0.50	15	30	0.5	80.013	38.0652
8	0.10	7	0.70	20	1	1	81.279	38.1996
9	0.10	8	1.00	1	5	2	83.185	38.4009
10	0.10	10	0.10	5	10	3.5	90.352	39.1187
11	0.15	3	0.50	20	5	3.5	91.648	39.2424
12	0.15	5	0.70	1	10	5	100.000	40.0000
13	0.15	7	1.00	5	20	0.5	100.000	40.0000
14	0.15	8	0.10	10	30	1	100.000	40.0000
15	0.15	10	0.30	15	1	2	100.000	40.0000
16	0.20	3	0.70	5	30	2	99.609	39.9660
17	0.20	5	1.00	10	1	3.5	99.737	39.9771
18	0.20	7	0.10	15	5	5	99.840	39.9861
19	0.20	8	0.30	20	10	0.5	99.821	39.9845
20	0.20	10	0.50	1	20	1	99.902	39.9914
21	0.25	3	1.00	15	10	1	99.889	39.9903
22	0.25	5	0.10	20	20	2	99.803	39.9829
23	0.25	7	0.30	1	30	3.5	99.824	39.9847
24	0.25	8	0.50	5	1	5	99.863	39.9881
25	0.25	10	0.70	10	5	0.5	99.819	39.9842

By choosing the optimum concentration of TOPO (0.15 M), higher extraction efficiencies will be obtained. On the second level of importance, Span 80 concentration has a high influence on the extraction percentage. Therefore, by increasing Span 80 concentration in conjunction with TOPO concentration, the amount of extracted chromium in membrane phase will be noticeably increased. By further comparing Fig. 3 results, it can be observed that both internal phase concentration and stirring time factors (on the third level of importance) devote almost the same influences on the chromium extraction percentage. In addition, it can be seen that the external to emulsion phase ratio and acidity of external phase factors play a role on the fourth level of importance on the chromium extraction efficiency. Finally, it can be noticed that the external to emulsion phase ratio factor remains on the lowest level of importance compared with the other process factors. Indeed, this trend leads to treat large volumes of Cr(VI) solutions that reflect the importance of ELM in which our system (low concentration feed solutions) deviates from equilibrium that means a high mass transfer occurred. With regard to these results, by selecting the appropriate factors and levels, the consumed time, money and energy will be more economized. On the other hand, one of the most hazardous constituents of Cr(VI) will be removed at remarkable magnitudes.

3.3. Analysis of variance (ANOVA)

Identification of the most effective parameters on the chromium extraction was performed using ANOVA technique. The results of variance analysis are given in Table 3. ANOVA was performed to see whether the process parameters are statistically significant. The optimal combination of the process parameters can be predicted using ANOVA analysis and performance characteristics. As seen in Table 3, TOPO concentration has a meaningful effect on the extraction of chromium by ELM. According to Taguchi method, the level with highest S/N ratio should be the optimum level for that particular control factor [20]. In accordance with that, the optimum levels of the control factors for the removal of chromium by ELM with

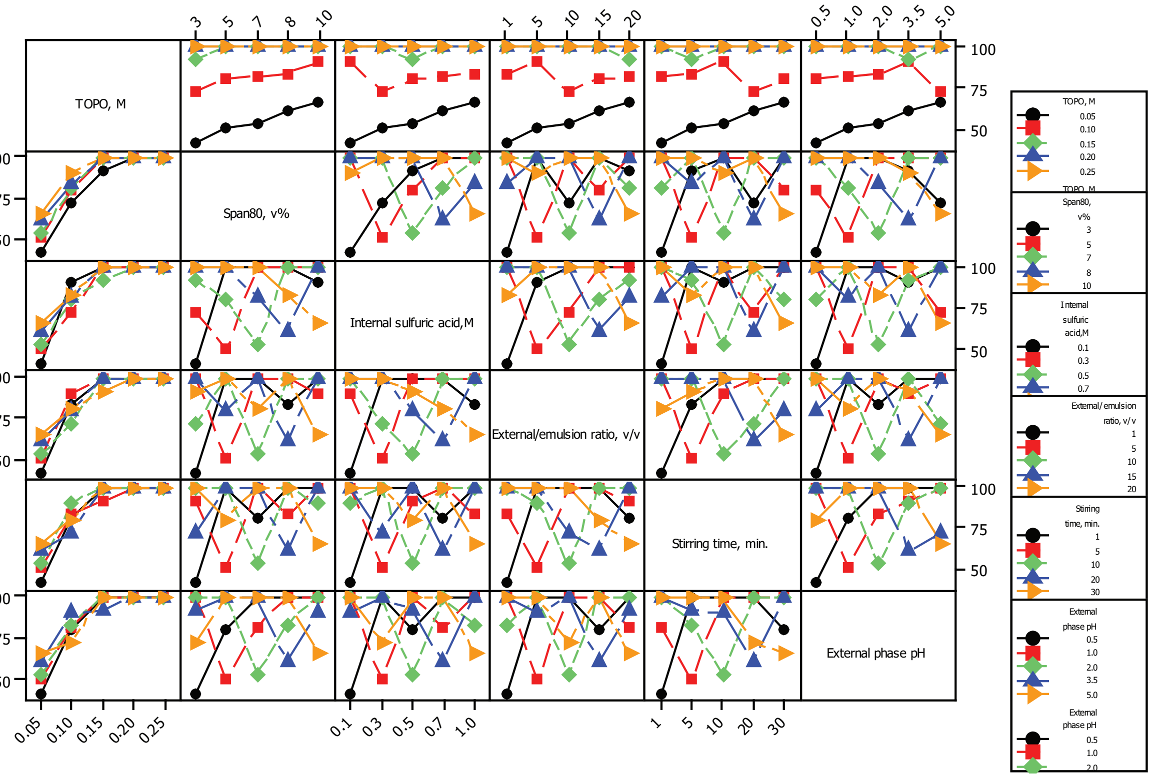


Fig. 1. Interactions between factors affecting chromium extraction by ELM.

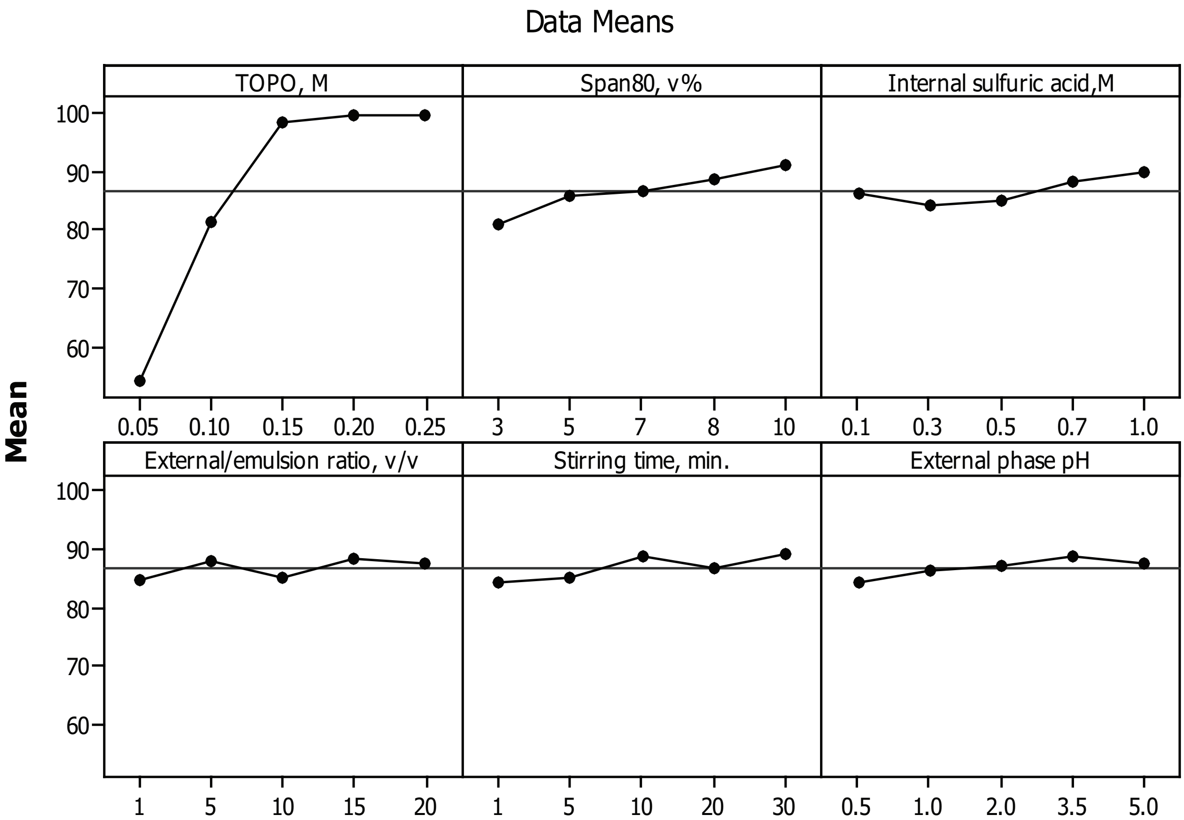


Fig. 2. Main effects plot of chromium extraction by ELM.

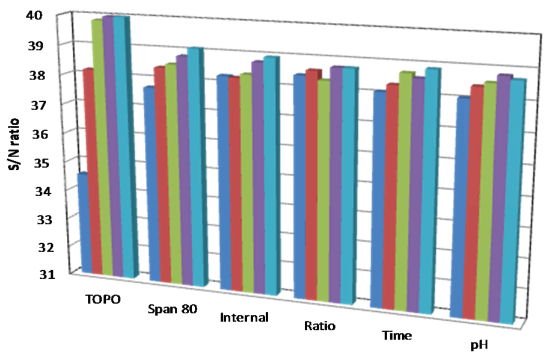
TOPO are tabulated in Table 3. From Table 3, it can be seen that the optimal extraction percentage for Cr(VI) (maximum S/N ratios) was obtained at the highest (Level 5) concentration of TOPO, Span 80, internal phase, feed/emulsion ratio and stirring time. But with the acidity of the external phase parameter, a medium value (level 4) is setting.

The degrees of the influences of parameters on the extraction percentage are given in Fig. 4. From the analysis of extraction data (Fig. 4), it is observed that TOPO is the most significant parameter affecting Cr(VI) extraction by ELM process (89.498% contribution) followed by Span 80 (4.393% contribution), and the four other parameters have a lower significant on ELM process (ranging from 1.750% to 1.299% contribution).

4. Conclusions

In this study, Taguchi method is proposed to optimize the extraction of chromium by ELM. The extraction of Cr(VI) ions by an emulsion formed with Span 80 as emulsifier has been investigated. For this purpose, six operating parameters at five levels are chosen. Hence, the orthogonal array of L25 has been selected, and computations are carried out for all the 25 trial runs. The S/N ratio and the ANOVA are employed to optimize the operating parameters. Computations are carried out using a Minitab 16 program. Based on the results, the following conclusions have been arrived at:

- Using the Taguchi optimization analysis, it is found that TOPO and Span 80 concentration are the most influencing parameters for achieving the maximum extraction percentage whereas the internal acid concentration and the acidity of external phase are of less important as process parameters and stirring time and emulsion/external phase ratio as an operational parameter.
- The optimum combinations of factors and levels was introduced by Taguchi method in the order of 0.25 M TOPO, 10 v% Span 80, 1 M $[H_2SO_4]_{int}$, 15 external/emulsion phase ratio, 30 min stirring time and pH 3.5 for external phase. With this combination, almost complete extraction of chromium was achieved.



	TOPO	Span 80	Internal	Ratio	Time	pH
Level 1	34.5451	37.7136	38.25882	38.4365	38.07424	38.04806
Level 2	38.18946	38.40084	38.22222	38.61036	38.31852	38.43206
Level 3	39.84848	38.52784	38.35118	38.3186	38.71246	38.56372
Level 4	39.98102	38.81768	38.77294	38.7513	38.57042	38.80756
Level 5	39.98604	39.09014	38.94494	38.75316	38.87446	38.6987

Fig. 3. Average S/N ratio for different levels of extraction parameters.

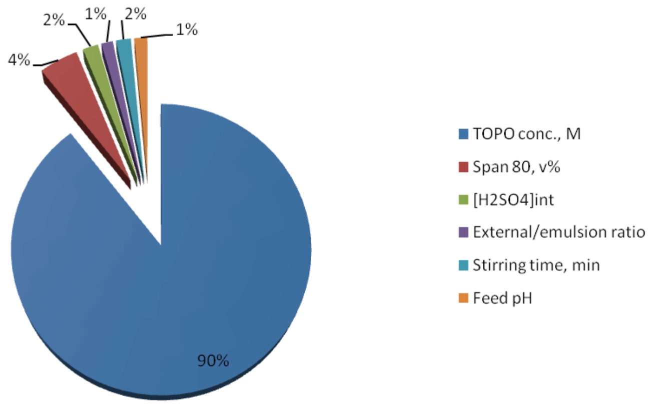


Fig. 4. Pie chart fot the contribution percentage of extraction parameters.

Table 3
Analysis of chromium extraction results

Factor	S/N ratio by factor level					Delta	Rank	DF	Seq. SS	Adj. MS	Contribution, %
	1	2	3	4	5						
TOPO conc., M	34.5451	38.18946	39.8484	39.9810	39.9860[a]	5.4409	**1**	4	109.78	27.446	89.498
Span 80, v%	37.7136	38.4008	38.5278	38.8176	39.0901[a]	1.3765	**2**	4	5.389	1.3472	4.393
$[H_2SO_4]_{int}$	38.2588	38.2222	38.3511	38.7729	38.9449[a]	0.7227	**5**	4	2.147	0.5368	1.75
Feed/emulsion ratio	38.4365	38.6103	38.3186	38.7513	38.7531[a]	0.4327	**6**	4	1.594	0.3984	1.299
Stirring time, min	38.0742	38.3185	38.7124	38.5704	38.8744[a]	0.8002	**3**	4	2.02	0.505	1.647
Feed pH	38.0480	38.4320	38.5637	38.8075[a]	38.6987	0.7595	**4**	4	1.733	0.4331	1.445
Delta	3.8914	0.4209	1.5298	1.4106	1.2873						
Total								24	122.66		100

[a] Optimum conditions.

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