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STUDY OF THE INFLUENCE OF DEGREASING METALLIC SURFACES PROCESS PARAMETERS ON ELONGATION AT BREAK

BY

GIANNINA ACATRINEI (MERLAN)*, TIBERIU-MIHAI STURZU and MATEI MACOVEANU*****

Abstract. The paper offers some mathematical models which show the relationships between elongation at break and the parameters of degreasing process. Using a Central-Compound Routable Program of second degree, one found the mathematical model of elongation at break and the three parameters of degreasing process that were studied. Because there were found optimal points inside of the domain taken into analysis, the paper permits drawing of interesting conclusions on the management of metallic surfaces degreasing process.

Key words: degreasing process, elongation at break, central-compound routable program of second order, level curves, 3 D representation.

1. Introduction

To protect against rust and corrosion of metals, there are used surface-active agents either to foster self-professed agents action of rust and corrosion, or by having this effect themselves.

Metal objects are almost always covered with greasy stains, due to penetration of materials used for the machining or due to reaching the hands full of grease. The contamination must be removed.

They differ into saponified fats and unsaponifiable ones. The first are the animal and vegetable fats and they are true chemical ones, *i.e.* glycerides of fatty acids: stearic, palmitic, oleic etc. [1]. These fats are called saponified because they are decomposed under the action of alkali in order to form soaps, which are water soluble salts of fatty acids and glycerol [2]; other unsaponifiable fats, of mineral origin, by chemical point of view are not fats. They are mixtures of hydrocarbons - oil, oil, grease, mineral oils and waxes [1]. Fats of this group did not undergo chemical decomposition under the action of alkali and, therefore, are called unsaponifiable matter. Under these conditions they can form emulsions with alkali and thus may be removed relatively easily from the metal surface [2].

Whatever is the nature of the contaminants to be removed, you can always find an optimal degreasing agent, without knowing, until now, a panacea [3]. For the successful operation of surface corrosion, products must be removed prior films fat. Insufficient degreasing is one of the main causes of scrap metal from depositing protective coatings. Therefore, degreasing operation is necessary and obligatory processing in cutting metal coatings [4]. It is used to remove parts of the surface layer of fat thick mineral washing large pieces or complex configuration. Most frequently, washing in organic solvents is the first in a series of operations related to degrease metal surface.

Improvement of surface cleaning technology parts basically go towards application of new degreasing means, the degreased enhance and improve quality. The last fact becomes particularly important in industries of radio engineering, electronics and some other industries in which they operate influence thoroughness of cleaning parts.

2. Experimental

In the process for cleaning of the steel materials, there were used alkaline degreasing solutions. The alkaline solution used as a degreasing agent is a caustic soda solution. We worked with caustic soda in the form of white flakes, with a concentration in NaOH, of min .= 99%, Na_2CO_3, min .= 0.7%, NaCl, % max .= 0.09, $Fe_2 O_3$, % max .= 0.008, chlorates missing.

Metallic materials used were halter type specimens, all with the same size, made of cold rolled steel with low carbon content (SR EN ISO 10130), having a chemical composition of C, max.= 0.12%, P, max .= 0.045%, S, .max = 0.045%, Mn, max .= 0.60%. As fat material, we use a single type of oil.

Metal samples were prepared, which were initially weighed. Then we sank each one in oil and left them to drain until the next day. We were weighed again with fat oil specimens. Then we made degreasing with solvent or caustic soda solutions, varying work conditions. The samples were subjected to mechanical tests, determining, in order to calculate elongation at break, initial length, final length before breaking and elongation after fracture, A80%.

In order to study the influence of process parameters for degreasing metal surfaces on elongation at break, was achieved, first, a preliminary experiment. As a root level we choose the point: $z_1^o = C^o = 22.5\%$, $z_2^o = T^o = 80°C$, $z_3^o = t^o = 22.5$ min, where C is the concentration of NaOH solution, T is temperature of solution, [ºC], and t is the duration of degreassing process,

[min]. In order to determine the equation of search plan, we used a full factorial experiment 2^k, k being the number of independent variables.

The obtained plan search equation is:

$$Al = 6.29917 - 0.00067 \cdot C + 0.00417 \cdot T - 0.00667 \cdot t \quad (1)$$

where: Al is the elongation at break, [%], C, T and t being the concentration of NaOH solution, [%], the solution temperature, [°C], respectively degreasing process duration, [min].

Having as an aim the statistical analysis of the proposed search plan, we made six determinations in the center of experimental domain. Based on the concordance dispersion, then we calculated the value of Fischer criterion, finding $F_c = 9.74694$. We compared this value with the tabulated value of Fisher criterion, for three degrees of freedom and a confidence level of 5% (95% probability), which is $F_t = 5.05$. Since $9.74694 < 5.05$, there was showed that the linear model is not appropriate. This improper linear model (search plan) may have several reasons That is why we started to test the response surface curvature [5]. Test response surface curvature ($10.43281 > 2.015$) showed that the curvature is significant, so we reach in the optimum aria. Linear model (search plan) was not adequate, so it was necessary to supplement the experimental program whith experiences in star (Table 4), for obtaining a Central Composed Routable Program of second order (CCRP2), which allows the deduction of an optimal point, whether it is in the analysed domain.

3. Results and Their Interpretation

We studied the influence of the NaOH solution concentration on elongation at break, for a constant degreasing process time and a constant temperature. Constant values were chosen as core values of the range of variation established. Experimental results are presented in Table 1. Using the method of least squares and a suitable Turbo-Pascal program, we obtained, under these conditions, the regressive equation:

$$Al = 6.46961 - 5.952 \cdot 10^{-3} \cdot C + 1.205 \cdot 10^{-4} \cdot C^2 \quad (2)$$

where: C is the concentration of NaOH solution, [%], and Al − the elongation at break, [%].

Fischer's test showed that the mathematical model is appropriate, the calculated Fischer's criterion having the value 0.569, being less than Fischer's tabled criterion for two degrees of freedom and a confidence level of 5%.

Graphical representations of experimental points and the curve which transpose graphically the equation are presented in Fig. 1.

The graphical analysis shows that, under the specified conditions (temperature 80°C and degrease duration of 22.5 min) one obtains a minimum for an elongation at break of approx. 6.4% at a concentration of 24.67% for NaOH solution.

We also studied elongation at break changes with temperature, the other two variables being maintained at the basic level. The experimental results are presented in Table 2.

The regressive equation which was obtained is:

$$\text{Al} = 7.4608 - 0.30033 \cdot \text{T} + 2.133 \cdot 10^{-4} \cdot \text{T}^2 \tag{3}$$

where: Al is the elongation at break, [%], and T − temperature, [°C].

Table 1

Influence of NaOH Solution Concentration on Elongation at Break

Temperature 80ºC, for 22.5 min.		
No.	NaOH solution concentration [%]	Elongation at break [%]
1.	10	6.42
2.	15	6.41
3.	20	6.40
4.	25	6.39
5.	30	6.40
6.	35	6.41

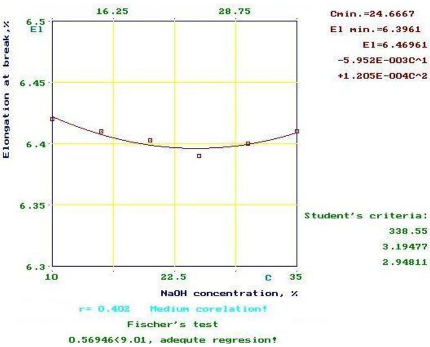

Fig. 1 − Influence of NaOH solution concentration on elongation at break.

In order to calculate Student's criteria, for verifying the significance of the regressive equation coefficents, repeated experiments have been performed, for a temperature of 80°C, the other variables being kept also constant. With these values it was calculated the dispersion of reproducibility, and then, Student's criterion value, which was compared with the tabulated value. All coefficients were found significant.

In order to verify the adequacy of the mathematical model is required Fischer test. For calculating the Fisher criterion value, there was calculated the dispersion of concordance ($s_0^2 = 4.187 \cdot 10^{-5}$). The calculated Fischer criterion is 0.15287 and Fischer tabulated criterion for a confidence level of 5% (probability of 95%) and 2 degrees of freedom is set to 19.25.

Since $0.15287 < 19.25$, Fischer test indicates an adequate regressive equation.

Graphical representations of experimental points and of the calculated curve, using the regressive equation are presented in Fig. 2.

Table 2
Influence of Temperature on Elongation at Break

NaOH solution concentration 22.5%, for 22.5 min.		
Nr. crt.	Temperature [ºC]	Elongation at break, [%]
1.	60	6.41
2.	70	6.38
3.	80	6.40
4.	90	6.46
5.	100	6.56

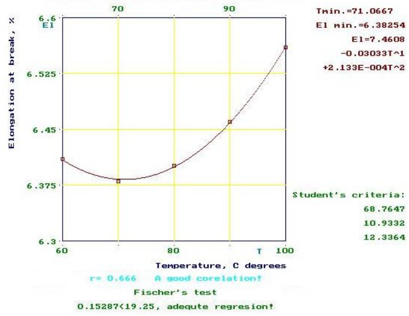

Fig. 2 − Influence of temperature on elongation at break.

The graphic shows that, for a solution of NaOH, with a concentration of 22.5%, and a duration of 22.5 min, elongation at break has the minimum value of 6.38%, for a temperature of 71°C.

For studying the influence of the degreassing process duration on elongation at break, at constant temperature and constant concentration of NaOH solution, experiments were performed, whose results are summarized in Table 3. For calculating Student's criteria in order to verify the significance of the regressive equation, repeated experiments were performed, for 20 min of degreasing process, the other variables being kept also constant as they were mentioned above.

With these values, there was calculated the dispersion of reproducibility, which has the value $1.147 \cdot 10^{-5}$. Based on the reproducibility dispersion and the diagonal terms of covariance matrix, there were calculated Student criterion values, which were compared with the tabulated value.

All coefficients being significant, the regression equation has the form:

$$Al = 6.82354 - 0.03081 \cdot t + 5.348 \cdot 10^{-4} \cdot t^2 \tag{4}$$

where: t is the duration of degreasing process, [min]. Although the two coefficients are low, the Student's test indicates that coefficents are significant. Fischer test indicates - in this case - an appropriate mathematical model.

Fischer's criterion calculated value is 0.33287, a value lower than the Fischer criterion tabulated, which is 9.01. Graphical representations of experimental points and curve which describes the regressive equation are presented in Fig. 3.

Graphical analysis shows that under the mentioned conditions (temperature 80°C and NaOH solution concentration of 22.5%) one obtains a minimum elongation at break of 6.38%, at a time of degreasing process of 28.83 min.

Table 3
Influence of the Degrasing Process Duration on the Elongation at Break

NaOH solution concentration 22.5%, temperature 80ºC		
No. crt.	Duration [min]	Elongation at break, [%]
1.	10	6.57
2.	15	6.48
3.	20	6.42
4.	25	6.39
5.	30	6.38
6.	35	6.40

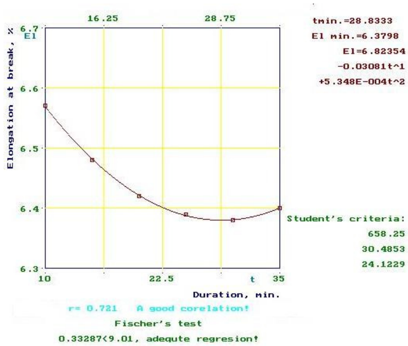

Fig. 3 – Influence of the degrasing process duration on the elongation at break.

In order to study the simultaneous influence of the three technological parameters on the elongation at break and to establish a generalized mathematical model of the process, we made an active experiment, developed on the basis of a Central Composite Rotatable Program of second order [5]. Experimental results for elongation of break and values of independent variables, codified (x_i) and real (z_i), are summarized in Table 4.

Table 4
Central Composed Routable Program Program

No. of det.	Rando-mization	Independent variables						Elongation at break [%]
		NaOH solution concentration [%]		Temperature [°C]		Time [min]		
		x_1	z_1 (C)	x_2	z_2 (T)	x_3	z_3 (t)	y (Al)
0.	1.	2.	3.	4.	5.	6.	7.	8.
1.	013	− 1	15	− 1	68	− 1	15	6.420
2.	003	+ 1	30	− 1	68	− 1	15	6.480
3.	015	− 1	15	+ 1	92	− 1	15	6.570
4.	018	+ 1	30	+ 1	92	− 1	15	6.600
5.	008	− 1	15	− 1	68	+ 1	30	6.400
6.	016	+ 1	30	− 1	68	+ 1	30	6.370
7.	019	− 1	15	+ 1	92	+ 1	30	6.500
8.	004	+ 1	30	+ 1	92	+ 1	30	6.400
9.	009	− 1.682	9.885	0	80.0	0	22.50	6.420
10.	005	+ 1.682	35.115	0	80.0	0	22.50	6.400
11.	007	0	22.50	− 1.682	59.816	0	22.50	6.410
12.	010	0	22.50	+ 1.682	100.184	0	22.50	6.550

Table 4
Continuation

No. of det.	Rando-mization	Independent variables						Elongation at break [%]
		NaOH solution concentration [%]		Temperature [ºC]		Time [min]		
		x_1	z_1 (C)	x_2	z_2 (T)	x_3	z_3 (t)	y (Al)
0.	1.	2.	3.	4.	5.	6.	7.	8.
13.	002	0	22.50	0	80.0	− 1.682	9.885	6.570
14.	012	0	22.50	0	80.0	+1.682	35.115	6.380
15.	014	0	22.50	0	80.0	0	22.50	6.400
16.	001	0	22.50	0	80.0	0	22.50	6.410
17.	020	0	22.50	0	80.0	0	22.50	6.390
18.	017	0	22.50	0	80.0	0	22.50	6.420
19.	006	0	22.50	0	80.0	0	22.50	6.380
20.	011	0	22.50	0	80.0	0	22.50	6.405

Regressive equation coefficients in coded variables were calculated using a Turbo-Pascal program and, using appropriate transformation relations, there were calculated and the regressive equation coefficients for real variables.

Using the diagonal terms of covariance matrix relations and the specialised literature, there were calculated Student's criteria for the regressive equation coefficients, and they were compared with the tabulated value.

Coefficients of terms x_1 and x_1^2 were found insignificant and removed at Student's test. Therefore, coded variable regressive equation has the form:

$$y = 6.40054+0.046653\cdot x_2-0.05268\cdot x_3-0.0125\cdot x_1\cdot x_2-0.0275\cdot x_1\cdot x_3- -0.0175\cdot x_2\cdot x_3 + 0.02993\cdot x_2^2 + 0.02816\cdot x_3^2 \quad (5)$$

Using this model and appropriate transformation relations [6], can be obtained a mathematical model for real variables. It is:

$$Al = 7.04639 - 0.02184\cdot T - 0.00299\cdot t - 0.00014\cdot C\cdot T - - 0.00049\cdot C\cdot t - 0.00019\cdot T\cdot t + 0.00021\cdot T^2 + 000050\cdot t^2 \quad (6)$$

To check the adequacy of the mathematical model, it is necessary to calculate the Fisher's criterion. We calculated the dispersion of reproducibility [7], which has been found the value $s_0^2 = 0.0002$. The coefficient of variation for central experiments is 0.22% and dispersion of concordance is set to $\sigma_{con}^2 = 0.00014$. Multiple correlation coefficient value is 0.99%, which shows a high correlation between independent variables chosen for study.

Calculated Fischer's criterion value is 0.69152, less than the tabulated Fischer's criterion for these conditions (4.95), indicating that the model is appropriate. Analyzing calculated Student's criteria, one can remark a considerable influence of temperature and the duration of degreasing process on the elongation at break.

For the coefficients b_1 and b_{11}, there were found insignificant values, which indicates a weak influence of the concentration of NaOH solution on elongation at break. NaOH solution concentration influences the elongation at break only in interaction with other parameters of the degreasing process, temperature and duration of the process. 3D and contour representations corresponding to one of representation for this model are shown in Figs. 4,...,7 taken from the monitor.

3 dimensional representations (3D) have been made using a proper Turbo-Pascal program by selecting an optimum angle which gives a perfect view of the response surface curvature. All 3D representations use the same angle of rotation. Contours from Fig. 7, which are corresponding to 3D representation from Fig. 6, were also created with a proper Turbo-Pascal program.

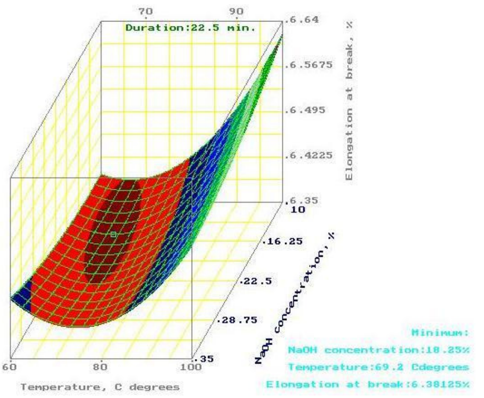

Fig. 4 – 3D representation temperature-concentration-elongation at break for a duration of degreasing process of 22.5 min.

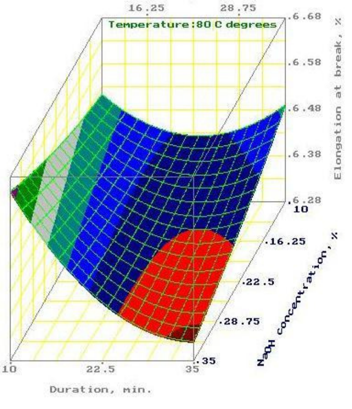

Fig. 5 – 3D representation concentration – degreasing process duration-elongation at break for a temperature of 80ºC.

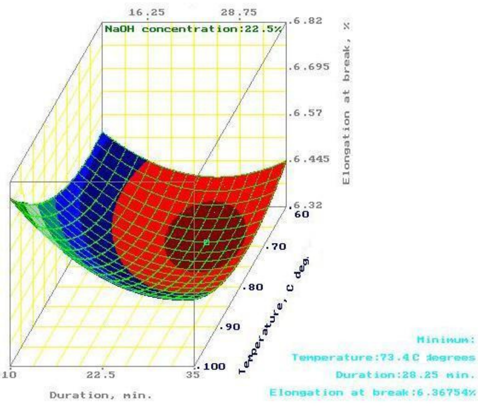

Fig. 6 − 3D representation temperature-duration of degreasing process elongation at break for the NaOH solution concentration of 22.5%.

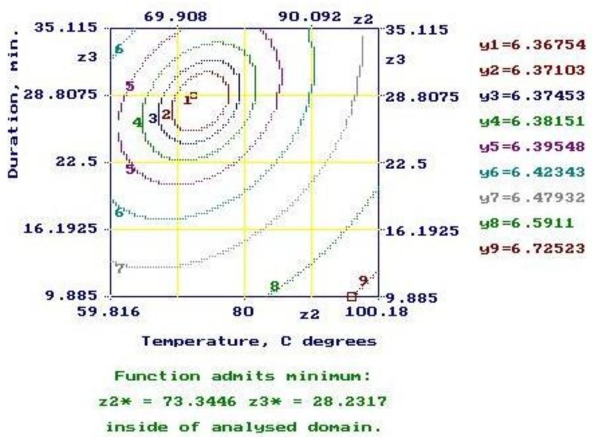

Fig. 7 – Level contours temperature-duration of degreasing process for NaOH solution concentration of 22.5%.

4. Conclusions

Based on experimental findings and analysis of 3D representations and corresponding level contours, the following conclusions can be drawn:

a) there was established the equation of a search plan, which has, however, a pronounced curvature response, that is why we started to study the influence of each parameter of the degreasing process and the concomitent influence of them, using a Central Composed Routable Program of second order (CCRP2);

b) the analysis of the influence of each parameter on elongation at break showed a less influence of NaOH solution concentration, but a great enough influence of temperature and duration of degreasing process;

c) based on a Central Composed Routable Program of second order, was obtained a mathematical model of the influence of three parameters on the elongation at break, both in real variables and in coded variables;

d) all mathematical models presented were statistically analyzed, and, following Fischer's tests, proved to be adequate;

e) based on 3D representation temperature-concentration-elongation at break for a duration of degreasing process of 22.5 min, we found a minimum elongation at break of 6.38125%, for 18.25% NaOH concentration and a temperature of 69.2ºC;

f) based on 3D representation time-temperature-elongation at break, for the NaOH solution concentration of 22.5% NaOH, we found a minimum elongation at break of 6.36754%, for a temperature of approx. 73.4ºC and a duration of degreasing process of about 28.25 min;

g) based on the experimental results analysis is recommended the management of metal surfaces degreasing processes of the kind discussed at the values obtained at point f), values that allow minimum deformations of defatted metal surfaces.

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**SC SAGGA CONSULTING SRL Iaşi*
e-mail: giaacatrinei@yahoo.com

***Scholar Group „Radu Cernătescu“ Iaşi*
e-mail: tibisturzu@yahoo.com

**** "Gheorghe Asachi" Technical University of Iaşi,*
Faculty of Chemical Engineering
and Environment Protection
e-mail: mmac@ch.tuiasi.ro

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STUDIUL INFLUENȚEI PARAMETRILOR PROCESULUI DE DEGRESARE A SUPRAFEȚELOR METALICE ASUPRA ALUNGIRII LA RUPERE

(Rezumat)

Lucrarea prezintă trei modele matematice care arată dependența dintre alungirea la rupere şi fiecare dintre parametrii analizați ai procesului de degresare. Utilizând un program central compus rotitor de ordinal al II-lea, s-a găsit un model matematic care corelează alungirea la rupere cu toți cei trei parametri ai procesului de degresare care au fost studiați. Deoarece au fost găsite puncte de optim în interiorul domeniului analizat, lucrarea permite tragerea unor concluzii interesante privind conducerea proceselor de degresare a suprafețelor metalice.