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CONTRIBUTIONS TO THE STUDY OF COSMETIC EMULSIONS USING ANALYTICAL – EXPERIMENTAL MATHEMATICAL MODELS

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ABSTRACT. The quality of a cosmetic cream is evaluated by classical methods in specific authorized laboratories by periodically measuring the physico-chemical and microbiological quality indicators of the basic emulsions. This paper aims to use systems theory to study cosmetic emulsions by establishing mathematical models that correlate the existing dependencies between different quality indicators. The main parameters that allow the quantification of the stability of the emulsions were monitored in time: the evaporation residue, the pH, the total number of germs, staphylococcus aureus, pseudomonas aeruginosa and the concentration of the active ingredients. Mathematical models were obtained with adequacy indicators that meet the requirements of a good approximation and that can be used as models for control and prediction of stability in the short and medium term, for the types of creams based on the recipes of the emulsions studied.

Keywords: *emulsion, quality indicator of cosmetic creams, mathematical model*

INTRODUCTION

There is little research in the literature on the use of systems theory and mathematical modeling techniques to characterize cosmetic emulsions. [1,2].

Cosmetic emulsions are heterogeneous dispersed mixtures consisting of two or more immiscible liquids dispersed in each other in the form of droplets, whose diameter is between 0.1 - 100 µm. Emulsions can be simple - U / A (oil in water) or A / U (water in oil), or multiple - U / A / U (U / A emulsion is dispersed in another oily phase) or A / U / A (A / U emulsion is dispersed in another aqueous phase) [3-6].

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The main property of a cosmetic emulsion that provides a high quality is the stability (chemical, physical and rheological) both in the manufacturing phase and in storage and use. In accordance with the legal rules of national and international law, stability is quantified by the values of specific quality indicators. These are: weight loss (evaporation residue), pH, viscosity, concentration of active ingredients, total number of germs, pseudomonas aeruginosa, staphylococus aureus, yeasts and molds [3-6].

In general, the mathematical model of a system (in this case the cosmetic emulsion) is a set of mathematical relations, equations and inequalities, which characterize and describe the interdependencies between output and input variables, and the limitations imposed for their validity and values [7,8,9].

Figure 1 shows the cosmetic emulsion as a system with input variables, output variables and perturbations [10].

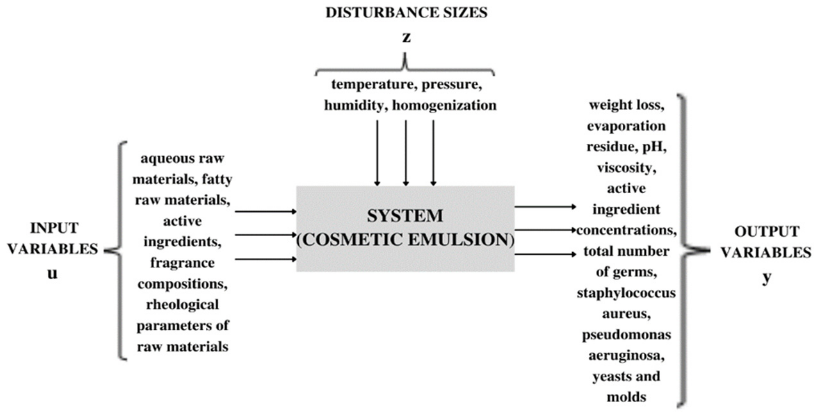


Figure 1. Representation of the cosmetic emulsion as a system

Cosmetic emulsions can present instability phenomena especially during storage, from physico-chemical, mechanical or visual changes of the component substances, to the appearance or presence of microorganisms and bacteria in the finished products. The experimental determinations were performed in a specialized laboratory over four years by analyzing four cosmetic emulsions.

The data obtained were used to generate 3D graphical representations and mathematical equations that are in fact analytical-experimental models. Based on these models, relevant conclusions were drawn on the existing dependencies between different quality indicators that have a major influence on the properties of the studied emulsions. From the point of view of the efficiency of the study, it should be noted its practical importance, which allows specifying the optimal values of physico-chemical parameters required both in manufacturing technology, storage and during use after opening bottles for sale, to ensure the best quality indicators.

The use of determined analytical-experimental mathematical models can replace the classical monitoring of the parameters characteristic of cosmetic emulsions, as well as allow predictions for the optimal values of quality indicators, which will ensure the physico-chemical and microbiological stability of creams.

In this paper, the analytical-experimental mathematical models that define the time dependencies between different physico-chemical and microbiological quality indicators using the database obtained for 4 years for four cosmetic emulsions were determined and verified.

RESULTS AND DISCUSSION

The results of the study performed for the 4 emulsions are presented in the form of sets of graphs in 3D format (figures 2 - 25), of some tables with mathematical expressions that represent the analytical-experimental models (tables 1, 3, 6, 8, 11,13), of some tables in which the values of the adequacy indicators are presented (tables 2, 4, 7, 9, 12, 14) and the tables with the calculated absolute errors (tables 5, 10, 15). The notations used are as follows: evaporation residue - RE (%), total number of germs - NTG (NTG / ml), pH, time - T (months) - T notation according to ISO 80000-1: 2009, error absolute - E (%).

I. a. Dependence of evaporation residue RE of time T and total number of germs NTG

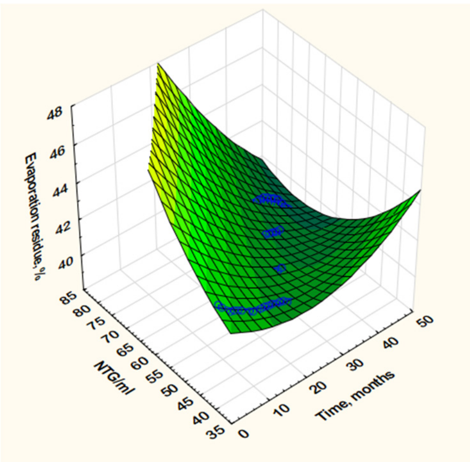


Figure 2 – EMULSION 1

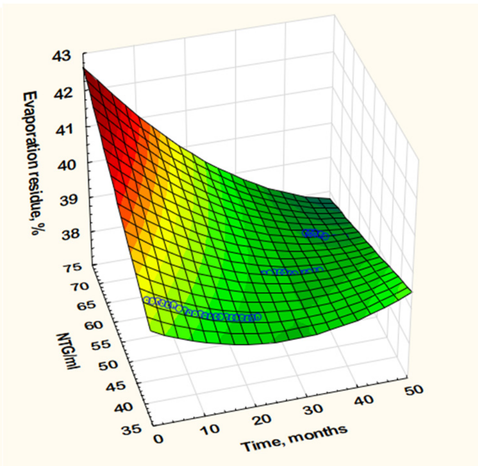


Figure 2 – EMULSION 1 **Figure 3** – EMULSION 2

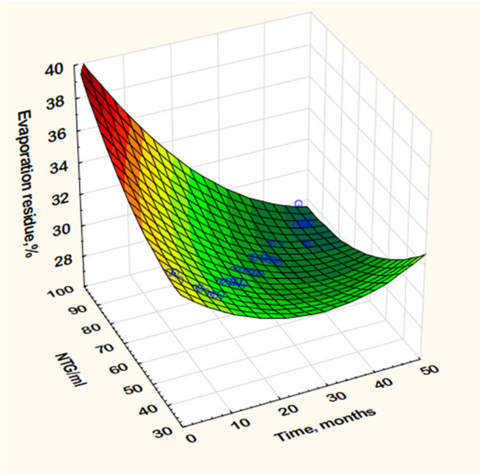


Figure 4 – EMULSION 3

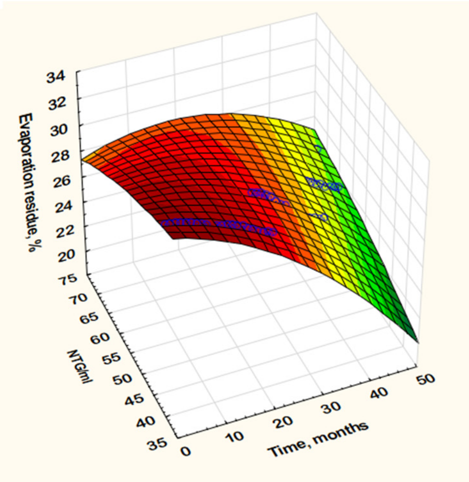


Figure 5 – EMULSION 4

Table 1. Equations of mathematical models

Emulsion 1	$RE = 44.3772 + 0.1506 \cdot T - 0.1773 \cdot NTG + 0.0032 \cdot T^2 - 0.008 \cdot T \cdot NTG + 0.004 \cdot NTG^2$
Emulsion 2	$RE = 37.2398 + 0.0096 \cdot T + 0.068 \cdot NTG + 0.0013 \cdot T^2 - 0.0023 \cdot T \cdot NTG + 5.2477E^{-5} \cdot NTG^2$
Emulsion 3	$RE = 35.0866 - 0.1323 \cdot T - 0.0689 \cdot NTG + 0.0041 \cdot T^2 - 0.0034 \cdot T \cdot NTG + 0.0013 \cdot NTG^2$
Emulsion 4	$RE = 34.7813 - 0.2796 \cdot T + 0.013 \cdot NTG - 0.0037 \cdot T^2 + 0.0055 \cdot T \cdot NTG - 0.0015 \cdot NTG^2$

Table 2. Adequacy indicators

Adequacy indicators	Emulsion 1	Emulsion 2	Emulsion 3	Emulsion 4
Dispersion, σ^2	0.04270	0.00354	0.03110	0.04107
Standard deviation, σ	0.20664	0.05950	0.17635	0.20266
Model accuracy indicator, R^2	0.96191	0.98318	0.99191	0.99419
Correlation coefficient, R	0.98077	0.99155	0.99594	0.99709

I. b. Dependence of residue on evaporation RE of time T and pH

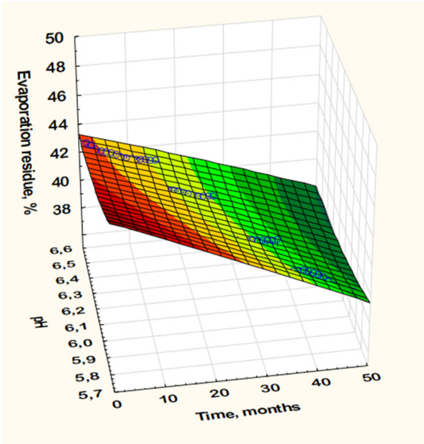


Figure 6 – EMULSION 1

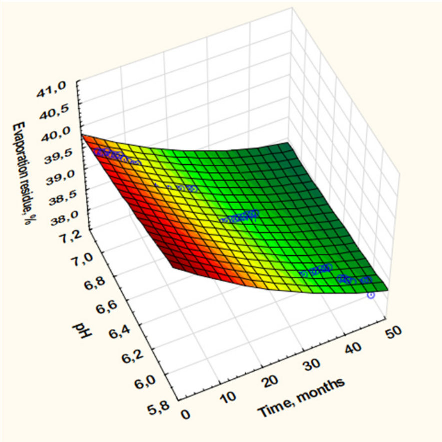


Figure 7 – EMULSION 2

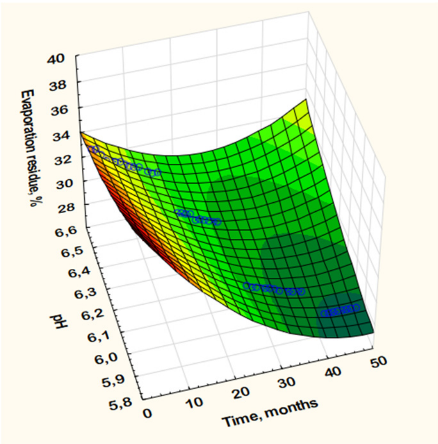


Figure 8 – EMULSION 3

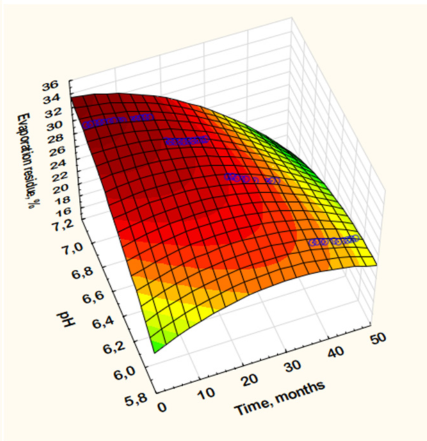


Figure 9 – EMULSION 4

Table 3. Equations of mathematical models

Emulsion 1	$RE = 176.1746 - 0.3555 \cdot T - 39.2725 \cdot pH - 0.0002 \cdot T^2 + 0.0391 \cdot T \cdot pH + 2.8993 \cdot pH^2$
Emulsion 2	$RE = 48.8903 - 0.1232 \cdot T - 1.9672 \cdot pH + 0.0005 \cdot T^2 + 0.0085 \cdot T \cdot pH + 0.0983 \cdot pH^2$
Emulsion 3	$RE = 235.6246 - 2.154 \cdot T - 57.3014 \cdot pH + 0.0051 \cdot T^2 + 0.285 \cdot T \cdot pH + 4.0552 \cdot pH^2$
Emulsion 4	$RE = -199.2458 + 2.087 \cdot T + 61.4851 \cdot pH - 0.0057 \cdot T^2 - 0.3003 \cdot T \cdot pH - 4.0488 \cdot pH^2$

Table 4. Adequacy indicators

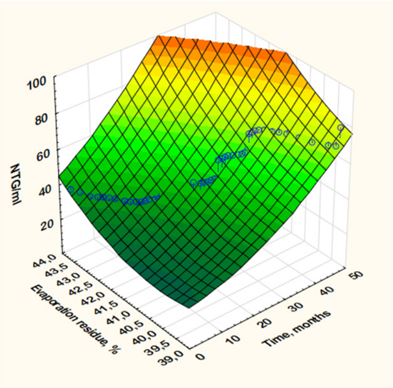
Adequacy indicators	Emulsion 1	Emulsion 2	Emulsion 3	Emulsion 4
Dispersion, σ^2	0.03909	0.00994	0.02411	0.04825
Standard deviation, σ	0.19771	0.09975	0.15527	0.21966
Model accuracy indicator, R^2	0.96513	0.95275	0.99372	0.99302
Correlation coefficient, R	0.98241	0.97609	0.99685	0.99658

Table 5. Absolute model errors

EMULSIONS	T, [months]	RE measured, [%]	RE calculated according to T and NTG, [%]	Error absolute of the model [%]	RE calculated according to T and pH, [%]	Error absolute of the model [%]
EMULSION 1	1	43.66	43.52	0.32	43.30	0.83
	48	39.18	39.67	1.24	39.29	0.28
EMULSION 2	1	39.92	39.96	0.10	39.87	0.12
	48	37.90	37.98	0.21	38.35	1.17
EMULSION 3	1	34.20	34.41	0.61	34.19	0.03
	48	27.83	27.97	0.50	27.78	0.18
EMULSION 4	1	32.83	32.84	0.03	32.74	0.27
	48	24.98	24.88	0.40	24.46	2.13

Based on the analysis of the 3D graphical representations (figures 2-9) and the equations that constitute the mathematical models obtained (tables 1,3) it is found that in a percentage of 75% the evaporation residue (RE) decreases during the 48 months.

II. a. Dependence of the total number of NTG germs of time T and the evaporation residue RE



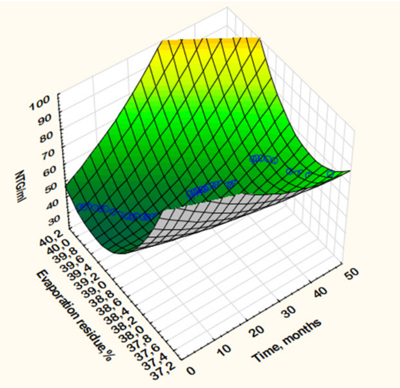
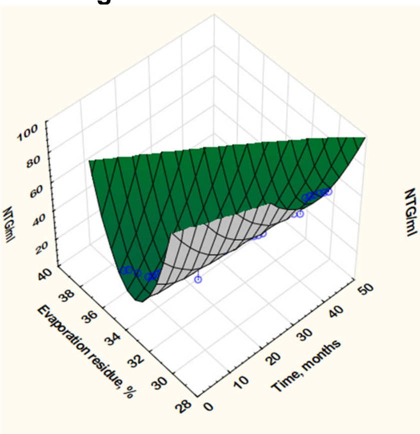


Figure 10 – EMULSION 1 **Figure 11** – EMULSION 2



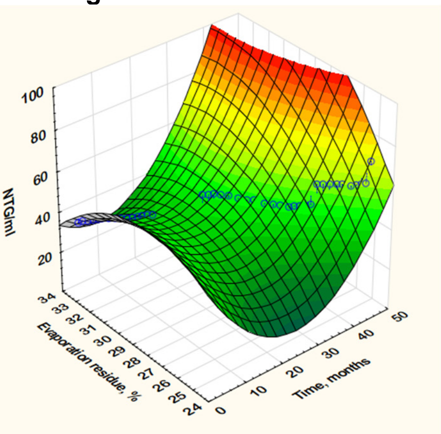


Figure 12 – EMULSION 3 **Figure 13** – EMULSION 4

Table 6. Equations of mathematical models

Emulsion 1	$NTG = 2621.5113-7.3024\cdot T -129.4987\cdot RE+0.0155\cdot T^2 +0.1949\cdot T \cdot RE+1.6122\cdot RE^2$
Emulsion 2	$NTG = 30338.1503-41.89\cdot T -1537.6807\cdot RE+0.0203\cdot T^2 +1.0702\cdot T \cdot RE+19.5066\cdot RE^2$
Emulsion 3	$NTG = 7410.9918-53.7877\cdot T -445.2677\cdot RE+0.1036\cdot T^2+1.6602\cdot T \cdot RE+6.7152\cdot RE^2$
Emulsion 4	$NTG = -507.7135-7.7856\cdot T +41.0303\cdot RE+0.0696\cdot T^2 +0.1879\cdot T \cdot RE-0.7384\cdot RE^2$

Table 7. Adequacy indicators

Adequacy indicators	Emulsion 1	Emulsion 2	Emulsion 3	Emulsion 4
Dispersion, σ^2	8.62415	6.84058	9.43264	5.06467
Standard deviation, σ	2.93669	2.61545	3.07126	2.25026
Model accuracy indicator, R^2	0.95115	0.90311	0.97126	0.92828
Correlation coefficient, R	0.97580	0.95032	0.98552	0.96347

II.b. Dependence on the total number of NTG germs of time T and pH

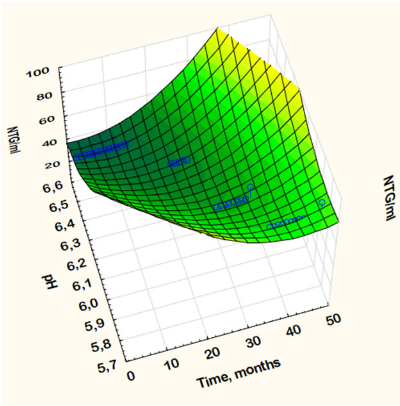


Figure 14 – EMULSION 1

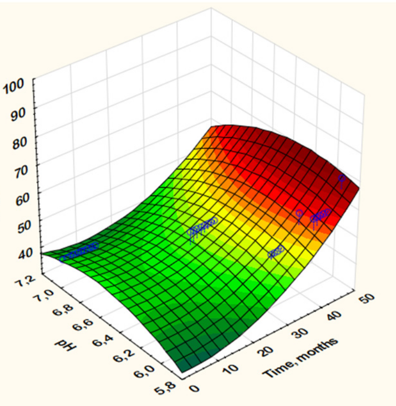


Figure 15 – EMULSION 2

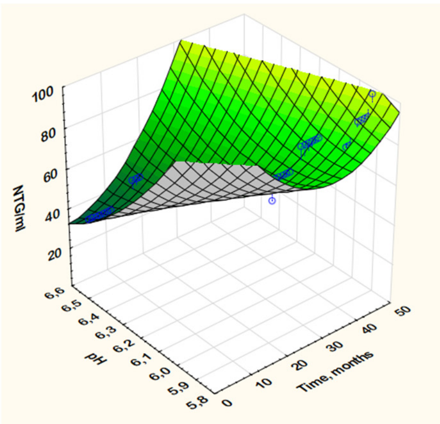


Figure 16 – EMULSION 3

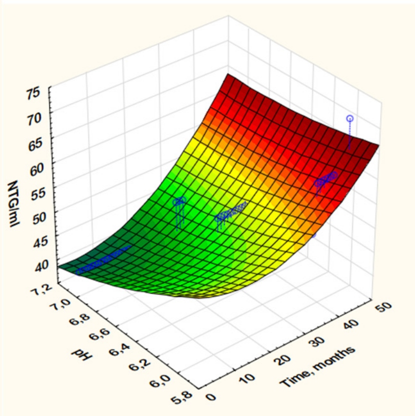


Figure 17 – EMULSION 4

Table 8. Equations of mathematical models

Emulsion 1	$NTG = 5570.2525 - 29.9381 \cdot T - 1645.7096 \cdot pH + 0.0524 \cdot T^2 + 4.4981 \cdot T \cdot pH + 122.3123 \cdot pH^2$
Emulsion 2	$NTG = -613.0804 + 1.231 \cdot T + 198.1065 \cdot pH + 0.0153 \cdot T^2 - 0.2238 \cdot T \cdot pH - 14.9611 \cdot pH^2$
Emulsion 3	$NTG = 4368.8048 - 32.6311 \cdot T - 1258.3122 \cdot pH + 0.0591\ T^2 + 4.9779 \cdot T \cdot pH + 91.108\ pH^2$
Emulsion 4	$NTG = 229.291 - 1.2878 \cdot T - 46.0972 \cdot pH + 0.012 \cdot T^2 + 0.1645 \cdot T \cdot pH + 2.7219 \cdot pH^2$

Table 9. Adequacy indicators

Adequacy indicators	Emulsion 1	Emulsion 2	Emulsion 3	Emulsion 4
Dispersion, σ^2	9.43602	6.50852	9.2884	6.63279
Standard deviation, σ	3.07181	2.55118	3.04770	2.57542
Model accuracy indicator, R^2	0.94769	0.90782	0.97170	0.90605
Correlation coefficient, R	0.97349	0.95279	0.98575	0.95187

Table 10. Absolute model errors

EMULSIONS	T, [months]	NTG /ml read	NTG/ml calculated according to T and RE	Error absolute of the model [%]	NTG/ml calculated according to T and pH	Error absolute of the model [%]
EMULSION 1	1	40	41.99	4.74	40.19	0.47
	48	80	74.33	7.63	75.69	5.69
EMULSION 2	1	40	40.63	1.55	40.25	0.62
	48	70	62.49	12.01	65.91	6.21
EMULSION 3	1	40	40.30	0.74	38.87	2.90
	48	100	94.82	5.46	95.85	4.33
EMULSION 4	1	40	41.91	4.56	39.86	0.35
	48	70	68.41	2.32	63.91	9.53

Analyzing 3D graphical representations (figures 10-17) and the equations of the obtained mathematical models (tables 6,8) it is observed that the total number of germs (NTG) increases in proportion of 85% starting from the end of the second year, but remaining in the optimal parameters and during the third.

III.a. pH dependence of time T and evaporation residue RE

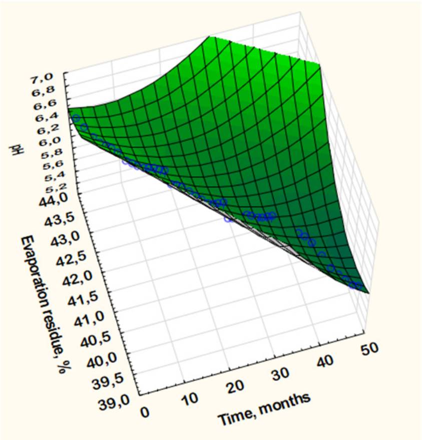


Figure 18 – EMULSION 1

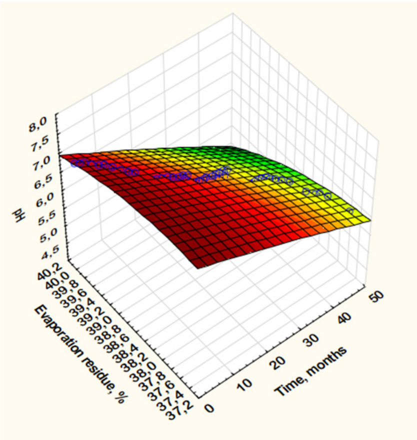


Figure 19 – EMULSION 2

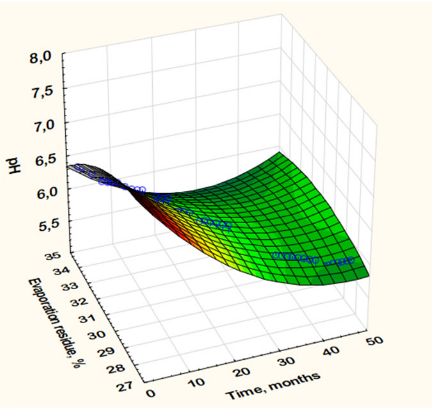


Figure 20– EMULSION 3

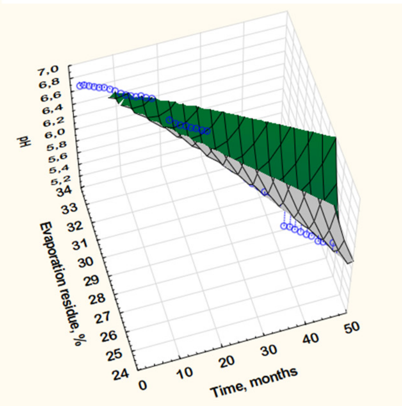


Figure 21 – EMULSION 4

Table 11. Equations of mathematical models

Emulsion 1	$pH = 347.7165 - 1.3413 \cdot T - 15.4457 \cdot RE + 0.0012 \cdot T^2 + 0.03 \cdot T \cdot RE + 0.1748 \cdot RE^2$
Emulsion 2	$pH = -152.9027 + 0.155 \cdot T + 8.5343 \cdot RE - 7.9467E\text{-}5 \cdot T^2 - 0.005 \cdot T \cdot RE - 0.1134 \cdot RE^2$
Emulsion 3	$pH = -0.977 - 0.1649 \cdot T + 0.6813 \cdot RE + 0.0008 \cdot T^2 + 0.0033 \cdot T \cdot RE - 0.0135 \cdot RE^2$
Emulsion 4	$pH = 318.0388 - 3.043 \cdot T - 18.8037 \cdot RE + 0.007 \cdot T^2 + 0.0918 \cdot T \cdot RE + 0.2842 \cdot RE^2$

Table 12. Adequacy indicators

Adequacy indicators	Emulsion 1	Emulsion 2	Emulsion 3	Emulsion 4
Dispersion, σ^2	0.00304	0.01055	0.00407	0.00904
Standard deviation, σ	0.05520	0.10270	0.06382	0.09506
Model accuracy indicator, R^2	0.96377	0.94496	0.93233	0.93956
Correlation coefficient, R	0.98172	0.97209	0.96557	0.96931

III.b. pH dependence of time T and total number of germs NTG

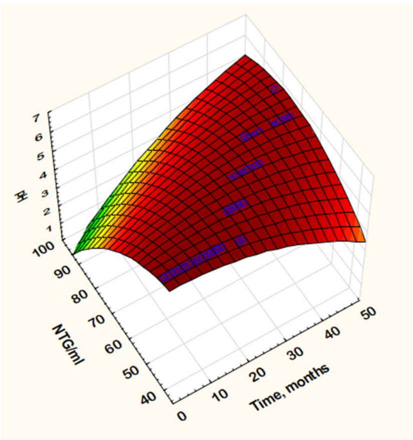


Figure 22 – EMULSION 1

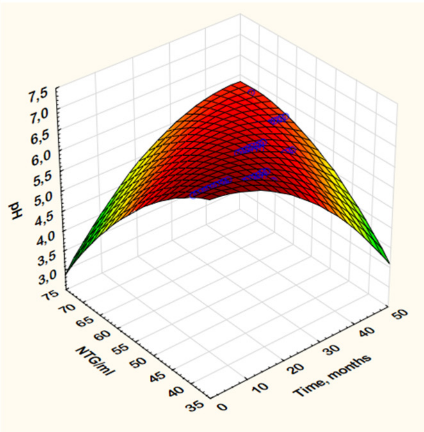


Figure 23 – EMULSION 2

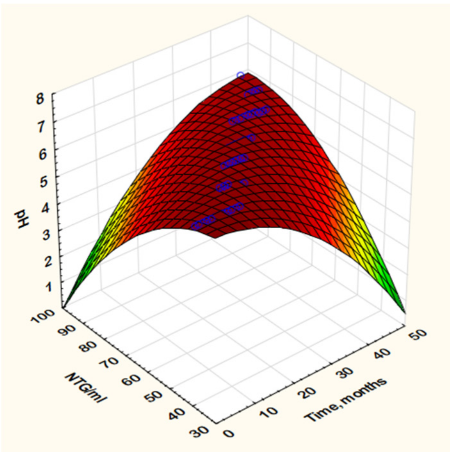


Figure 24 – EMULSION 3

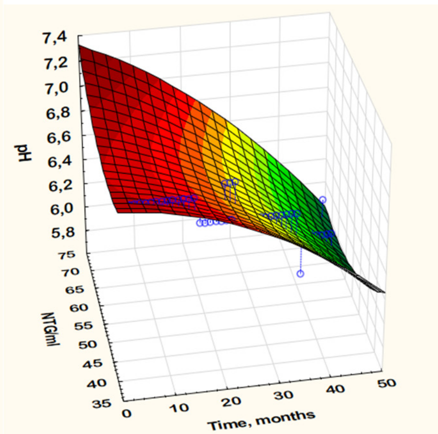


Figure 25 – EMULSION 4

Table 13. Equations of mathematical models

Emulsion 1	$pH = 6.2524 - 0.0983 \cdot T + 0.0586 \cdot NTG - 0.0014 \cdot T^2 + 0.0029 \cdot T \cdot NTG - 0.0013 \cdot NTG^2$
Emulsion 2	$pH = 6.8415 - 0.1246 \cdot T + 0.0687 \cdot NTG - 0.0013 \cdot T^2 + 0.0033 \cdot T \cdot NTG - 0.0016 \cdot NTG^2$
Emulsion 3	$pH = 6.1796 - 0.1109 \cdot T + 0.0534 \cdot NTG - 0.0024 \cdot T^2 + 0.0035 \cdot T \cdot NTG - 0.0012 \cdot NTG^2$
Emulsion 4	$pH = 8.1387 + 0.0098 \cdot T - 0.0465 \cdot NTG - 0.0003 \cdot T^2 - 0.0003 \cdot T \cdot NTG + 0.0005 \cdot NTG^2$

Table 14. Adequacy indicators

Adequacy indicators	Emulsion 1	Emulsion 2	Emulsion 3	Emulsion 4
Dispersion, σ^2	0.00354	0.98790	0.00475	0.94983
Standard deviation, σ	0.05953	0.09939	0.06886	0.09745
Model accuracy indicator, R^2	0.95787	0.94844	0.92124	0.93648
Correlation coefficient, R	0.97870	0.97388	0.95981	0.96772

Table 15. Absolute model errors

EMULSIONS	T, [months]	pH read	pH calculated according to T and RE	Error absolute of the model [%]	pH calculated according to T and NTG	Error absolute of the model [%]
EMULSION 1	1	6.50	6.53	0.46	6.53	0.46
	48	5.80	5.69	1.93	5.81	0.17
EMULSION 2	1	7.00	7.03	0.43	7.04	0.57
	48	5.90	5.82	1.37	5.92	0.34
EMULSION 3	1	6.50	6.48	0.31	6.42	1.24
	48	5.90	5.86	0.68	5.47	7.86
EMULSION 4	1	7.00	7.01	0.14	7.08	1.13
	48	6.00	5.80	3.45	6.10	1.64

The 3D graphical representations of pH (figures 18-25) and the equations that constitute the mathematical models obtained (tables 11,13) it is observed a 90% decrease in its values by the end of the study.

Regarding the quality of the mathematical models obtained for the 4 emulsions, it can be seen that they have indicators of adequacy that fall within the requirements of a good approximation (tables 2,4,7,9,12,14). Thus the correlation coefficient R is higher than 0.95, and the absolute errors fall in the range of 0.03% and 12.01% (tables 5,10,15). This situation leads to the conclusion that the mathematical models obtained are true and accurately reflect the behavior of the real model (cosmetic emulsion). Based on the equations of the obtained analytical-experimental mathematical models, presented in tables 1,3,6,8,11 and 13, the variations of the specific quality indicators can be correlated with each other and depending on the time.

This method is comparable to the classical methods of studying cosmetics based on direct experimental determinations with the appropriate equipment. However, these classical methods do not offer the possibility to evaluate qualitatively and quantitatively the structural changes of the emulsions highlighted by the shape of the graphical representations of the determined mathematical models.

The disadvantage of the proposed method is that the determined mathematical models are valid only for well-defined case studies, respectively for concrete limitations of the variations of the parameters taken into account when calculating the quality indicators. In order to ensure a high physico-chemical and microbiological stability of the studied emulsions, ingredients were chosen to ensure a physico-chemical interaction between the components as low as possible, especially after preparation.

CONCLUSIONS

The results obtained in the research show that for the case study presented, the experimental-computational mathematical models describe the physico-chemical processes that take place in cosmetic emulsions, which can lead to changes in the values of some parameters and quality indicators specified by law the EU.

Taking into account the experimental models obtained by the multi-correlation method, it can be seen that preserving emulsions with a well-defined amount of 0.8% methyl paraben improves their quality and stability over time (NTG, RE and pH have changed in the limits imposed by the standards in force, even for emulsions in which the aqueous phase is above 60% found in figures 3,4,5,7,8,9) [11]. The organoleptic properties of the four emulsions were also monitored throughout the 4-year study. It was observed that for almost three years they did not change, so they kept their homogeneous shape, without separation phases, without foreign smell and without color change.

Staphylococcus aureus and Pseudomonas aeruginosa, the other microbiological parameters, remained absent throughout the testing period (as required by legal standards for cosmetics). The final conclusion of the study is that the 4 emulsions cosmetics can have a safe shelf life of up to 3 years, if kept in optimal storage conditions. It is important to note that these cosmetic emulsions (creams) have were packed in polypropylene boxes. Their lids are opened every time they are subjected to quality control analyzes, and when they reach consumers they open daily. As such, in order to maintain a superior quality of cosmetic creams, it is recommended to use airless bottles; the packaging of these vials is airtight and there is no risk of contamination during quality control or during use by consumers.

Given the accelerated development of the use of artificial intelligence, we believe that future technologies for obtaining cosmetic emulsions will be developed very soon only on the basis of computational mathematical models.

EXPERIMENTAL

Preparation of the emulsions

The preparation of the emulsions was carried out in accordance with the specific technology for obtaining them [12,13]. Both the aqueous phase (1) and the oily phase (2) are heated at 80°C and then, the aqueous phase was added over the oily phase and mixed for 8 minutes with a Lab High-shear Homogenizer, at 10000 rpm. After homogenization, the emulsions were cooled to 40°C under continuos stirring (5000 rpm) after which the active ingredients and the perfume were added. The composition of the prepared and analysed emulsions is presented in table 16.

Table 16. The composition of the analysed samples

Ingredients	Composition, wt.%			
	E1	**E2**	**E3**	**E4**
Aqueous phase (1)				
Glycerol	10	5	5	7
Methyl 4-hydroxybenzoate (methylparaben)	0.8	0.8	0.8	0.8
Distilled water	57.29	60.49	63.8	67.39
Sodium lauryl sulfate (anionic emulsifier)	0.5	0.5	0.5	0.5
Oily phase (2)				
Paraffin oil	25	8	6	14
Vaseline	4	20	15	3
Cocoa butter	-	-	0.5	-
Cetaceum	-	-	-	2
Cetostearyl alcohol	2	5	-	2
Cetyl alcohol	-	-	8	-
Stearic acid	-	-	-	2
Active ingredients (3)				
Spirulina	0.1	-	-	-
Honey	-	-	0.2	1
Vitamin E	0.01	0.01	-	0.01
Vitamin A	-	-	-	0.01
Perfume (4)	0.3	0.2	0.2	0.3

After preparation, the emulsions were packed in polypropylene boxes with a lid for sealing. The samples were stored in a room where temperature (15÷25ºC) and air humidity (55÷65%) were monitored.

Determination of pH value and the evaporation residue

The pH value was determined with InoLab pH-meter. The evaporation residue (RE) was determined with thermobalance PCE-MA 50X.

Determination of physico-chemical and microbiological parameters

These parameters were determined using the specific methods of analysis recommended by the current standards [14,15].

Experimental-computational mathematical modeling methodology

As a modeling strategy, the technique of obtaining analytical-experimental models or the so-called computational modeling was chosen. As such, the following steps were taken [7,16-19]:

• Data collection, analysis and interpretation (the database was obtained using experimental determinations performed in the laboratory using physical-chemical and microbiological analysis methods, and the results were presented in tabular form).

• The elaboration of the analytical-experimental mathematical models was performed by processing the database obtained in the laboratory, with the computer program STATISTICA 14.0 (nonlinear regression method), directly obtaining the corresponding graphical representations, the corresponding mathematical equations and the adequacy indicators.

• The testing of the analytical-experimental mathematical models was performed based on the determined adequacy indicators: the adequacy dispersion (σ^2), the standard deviation (σ), the model accuracy indicator (R^2) and the multiple correlation coefficient (R).

• Verification of the authenticity of mathematical models was performed by the classical method of calculating the absolute error according to formula (1):

$$E = 100(y_c - y) / y_c, \% \qquad (1)$$

where:

y_c - the value of the output variable calculated based on the mathematical model,

y - the value of the measured / readable output variable on the graph.

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