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OPTIMUM ETHANOL-HEXANOL FOAMS FOR FORMULATION OF DETERGENTS

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ABSTRACT

The accurate and speedy characterization of aqueous foams generated by the use of surfactants are critical for the formulation of soaps and detergents. This paper describes details of experimental studies directed towards measurement of foaming properties of mixture of ethanol, hexanol and sodium lauryl sulfate (SLS). The experimental results are of great relevance with respect to the formulation of detergents, which can be generated by use of alcohol-surfactant mixtures. The foams were generated by the injection of air at a constant flow rate of 5 mL/sec into aqueous mixtures of ethanol, hexanol with sodium lauryl sulfate. The foams were then characterized using a Dynamic Foam Analyzer (Kruss GmbH, Germany). The foamability, stability parameters like foam capacity, RMI 30, were determined and their variation with changes in ethanol, hexanol and SLS amounts added to solution were noted. Model equations for foam capacity and RMI 30 were developed by using the Central Composite Experimental Design (CCD) approach of Response Surface Methodology (RSM), statistically analyzed and validated. The responses obtained in the study were foam capacity (FC) and RMI 30 in mL. Numerical optimization for two different strategies for the responses, were subsequently performed by using Design-Expert Software version 9.0.4.1. The optimum responses FC and RMI 30 were found to be in reasonable agreement with the experimental results.

Key words: Foam capacity, Detergents, Foam stability, Ethanol, Hexanol, Sodium lauryl sulfate.

INTRODUCTION

The foaming capability of the foam[1-3] constituents determines its application in various industrial processes ranging from oil recovery to the manufacture of pharmaceutical products[4]. Aqueous foams produced by the use of surfactants[5,6] are used in many industrial applications including detergents, cosmetics formulation, etc. The current relevance of evaluations of aqueous foam properties in formulations of final product are emphasized in various industries worldwide, starting from soap to shampoo manufacture. Thus, the

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evaluation of various foam properties are important factors for judging industrial products like detergents from the customer standpoint. This paper evaluates the optimum foaming power of ethanol-hexanol mixtures with surfactant sodium lauryl sulfate, to be used for detergent formulations. The alcohol mixture of ethanol-hexanol is selected keeping in mind, the penetration power of the detergent towards fabric as well as the foam stability aspect-critical for detergent formulations.

EXPERIMENTAL

Materials

Ethanol and hexanol with purity of 99.9%, were procured from Varun Industries, India. Powdered surfactant sodium lauryl sulfate (SLS) of purity 99% was first weighed, using a digital weighing balance and then mixed with aqueous mixture of ethanol-hexanol solution. Distilled water was used preparing for all solutions,. The total volume of solutions prepared was 100 mL.

Foam characterization

Aqueous foams of ethanol-hexanol-SLS were characterized by using a Dynamic Foam Analyzer DFA 100 (Kruss GmbH, Germany). All the experiments reported here, were conducted in laboratory at room temperature of 303 ± 2 K. Foams were generated in a glass column of 250 mm length with inside diameter 40 mm, by a stream of air that was introduced into the aqueous solutions through a porous glass filter (pore size: 16-40 μm) with a constant flow rate of 5 mL/s. The air was passed for 12 s from the start of each run to produce the foam. The Foam Analysis Software version 1.4.2.3 (Kruss GmbH, Germany) was used for determination of foam properties. Each foaming experiment was run for a total of 15 min (900 s) and each run was repeated three times for better accuracy. The foam capacity (at the end of air injection)[7] was considered as the ratio of the foam volume to the air volume entered. RMI 30 was considered to be the foam volume after 30 s collapse.

RESULTS AND DISCUSSION

Experimental design and statistical analysis

In this work, a three-variable (three levels of each variable) Central Composite Design (CCD) and a Response Surface Methodology (RSM)[8,9] were used for the design of experiments for ethanol-hexanol-SLS systems, with the help of Design-Expert Software version 9.0.4.1, Stat-Ease Inc., MN, USA[10]. RSM is an important technique for investigating the impact of various independent variables on system response[11,12]. Table 1 shows the design matrix of the experimental design for the foaming characteristics. The independent variables considered were ethanol volume, SLS weight and hexanol volumes. The interactions of ethanol, hexanol volumes and SLS weights on the foaming characteristics were studied in 27 runs for ethanol-hexanol-SLS systems. The response functions y (dependent variables) for ethanol-hexanol-SLS systems were foam capacity (FC), RMI 30 (mL). The coded values of independent variables for ethanol volume (mL), SLS weights (g) and hexanol volume (mL) were denoted by X_1, X_2, X_3 respectively, as shown in Table 1. Regression analysis and analysis of variance (ANOVA) were conducted for fitting the models and to understand the statistical significance of the model terms. The adequacy of the models were investigated using model analysis and R^2 (coefficient of determination) analysis[13-16].

Table 1: Effect of process variables on FC and RMI 30 of ethanol-hexanol-SLS foam (FC indicated foam capacity at the end of air injection, RMI 30 indicated the foam volume after 30 s collapse (mL))

Experiment No.	Process variables[a]			Response	
	mL of ethanol (X_1)	g of SLS (X_2)	mL of hexanol (X_3)	Foam capacity	RMI 30 (mL)
1	5 (-1)	0.002 (-1)	0.1 (-1)	1.4	82.2
2	5 (-1)	0.004 (0)	0.1 (-1)	1.4	80.6
3	5 (-1)	0.006 (+1)	0.1 (-1)	1.5	83.1
4	5 (-1)	0.002 (-1)	0.2 (0)	1.4	81.4
5	5 (-1)	0.004 (0)	0.2 (0)	1.5	84.4
6	5 (-1)	0.006 (+1)	0.2 (0)	1.5	85.2
7	5 (-1)	0.002 (-1)	0.3 (+1)	1.4	81.1
8	5 (-1)	0.004 (0)	0.3 (+1)	1.5	87.1
9	5 (-1)	0.006 (+1)	0.3 (+1)	1.5	87.3
10	10 (0)	0.002 (-1)	0.1 (-1)	1.3	65

Cont…

Experiment No.	Process variables[a]			Response	
	mL of ethanol (X_1)	g of SLS (X_2)	mL of hexanol (X_3)	Foam capacity	RMI 30 (mL)
11	10 (0)	0.004 (0)	0.1 (-1)	1.4	84.8
12	10 (0)	0.006 (+1)	0.1 (-1)	1.5	87.7
13	10 (0)	0.002 (-1)	0.2 (0)	1.4	75.2
14	10 (0)	0.004 (0)	0.2 (0)	1.4	82.9
15	10 (0)	0.006 (+1)	0.2 (0)	1.5	86.5
16	10 (0)	0.002 (-1)	0.3 (+1)	1.3	79.4
17	10 (0)	0.004 (0)	0.3 (+1)	1.4	84.1
18	10 (0)	0.006 (+1)	0.3 (+1)	1.5	89.8
19	15 (+1)	0.002 (-1)	0.1 (-1)	1.2	32.7
20	15 (+1)	0.004 (0)	0.1 (-1)	1.3	52.9
21	15 (+1)	0.006 (+1)	0.1 (-1)	1.5	76
22	15 (+1)	0.002 (-1)	0.2 (0)	1.2	35.4
23	15 (+1)	0.004 (0)	0.2 (0)	1.4	51.8
24	15 (+1)	0.006 (+1)	0.2 (0)	1.5	76.1
25	15 (+1)	0.002 (-1)	0.3 (+1)	1.3	44.2
26	15 (+1)	0.004 (0)	0.3 (+1)	1.3	50.1
27	15 (+1)	0.006 (+1)	0.3 (+1)	1.4	66.8

[a]Data in parenthesis were the coded values of the process variables

ANOVA analysis

The experimental results for FC, and RMI 30 with the combination of independent variables are shown in Table 1. The analysis of variance (ANOVA) showed that the models are significant for all the responses (Tables 2-3). The p values of the overall models were both lesser than 0.05 (selected confidence interval) and thus, the models were all significant. As per ANOVA analysis, the individual model terms for all cases were considered significant, if p value < 0.05.

Table 2: Analysis of variance (ANOVA) for FC model (Ethanol-hexanol-SLS)

Source	Sum of squares	df	Mean square	F Value	p-value Prob > F
Model	0.20	6	0.033	21.34	< 0.0001
X_1	0.00032	1	0.00032	0.20	0.6558
X_2	0.13	1	0.13	80.60	< 0.0001
X_3	0.00198	1	0.00198	1.28	0.2714
X_1X_2	0.013	1	0.013	8.60	0.0082
X_1X_3	0.00083	1	0.00083	0.54	0.4721
X_2X_3	0.00333	1	0.00333	2.15	0.1582
Residual	0.031	20	0.00155		
Cor total	0.23	26			

Table 3: Analysis of variance (ANOVA) for RMI 30 model (Ethanol-hexanol-SLS)

Source	Sum of squares	df	Mean square	F Value	p-value Prob > F
Model	7269.45	9	807.72	55.82	< 0.0001
X_1	3.40	1	3.40	0.24	0.6340
X_2	26.44	1	26.44	1.83	0.1942
X_3	26.88	1	26.88	1.86	0.1906
X_1X_2	763.21	1	763.21	52.75	< 0.0001
X_1X_3	8.50	1	8.50	0.59	0.4539
X_2X_3	63.94	1	63.94	4.42	0.0507
X_1^2	1000.18	1	1000.18	69.12	< 0.0001
X_2^2	0.098	1	0.098	0.00677	0.9354
X_3^2	0.16	1	0.16	0.011	0.9186
Residual	245.98	17	14.47		
Cor total	7515.43	26			

Neglecting the insignificant terms as per the ANOVA analysis and with the coded values of the independent variables, the following model equations (Eqs. 1 and 2) described the effect of significant process variables on the foam capacity (FC) and RMI 30 (mL) for ethanol-hexanol-SLS system, respectively.

$$FC = 1.57 + 0.083\ X_2 + 0.033\ X_1X_2 \qquad (R^2 = 0.87) \qquad \ldots(1)$$

$$RMI\ 30 = 99.382 + 7.975\ X_1X_2 - 12.911\ X_1^2 \qquad (R^2 = 0.97) \qquad \ldots(2)$$

As seen from the coefficients of the model equations, some of the model terms had a positive contribution and some negative contributions to the responses. The R^2 (coefficient of determination) for all the models developed were greater than 0.85, indicating goodness of the fit. The numerical optimization of the responses along with desirability values obtained by using Design Expert Software are depicted in Tables 4-5. The optimization strategies applied were:-

(i) FC was minimized and RMI 30 (mL) maximized (Table 4)

(ii) FC was maximized and RMI 30 (mL) minimized (Table 5)

Table 4: Results of numerical optimization (Desirability- 0.659)

Constraints	Goal	Lower limit	Upper limit	Importance	Optimized value	Experimental value
Ethanol (mL)	In range	5	15	3(+++)	9.075	
SLS (gms)	In range	0.002	0.006	3(+++)	0.002	
Hexanol (mL)	In range	0.1	0.3	3(+++)	0.1	
FC	Minimize	1.2	1.5	3(+++)	1.313	1.4
RMI30 (mL)	Maximize	32.7	89.8	3(+++)	72.545	77.4

Table 5: Results of numerical optimization (Desirability- 0.584)

Constraints	Goal	Lower limit	Upper limit	Importance	Optimized value	Experimental value
Ethanol (mL)	In range	5	15	3(+++)	15	
SLS (gms)	In range	0.002	0.006	3(+++)	0.005	

Cont…

Constraints	Goal	Lower limit	Upper limit	Importance	Optimized value	Experimental value
Hexanol (mL)	In range	0.1	0.3	3(+++)	0.1	
FC	Maximize	1.5	1.5	3(+++)	1.401	1.2
RMI30 (mL)	Minimize	32.7	89.8	3(+++)	60.781	55.8

CONCLUSION

On checking the optimum results for FC and RMI 30 for both the strategies, it was found that it was in reasonable agreement with the experimental results. All the models developed for the ethanol-hexanol-SLS system have shown good fit. However, even though the optimum foaming capability was determined, further tests will be required to be undertaken to check the cleansing power of the different ethanol-hexanol-SLS mixtures.

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