Lubrizol

TECHNICAL DATA SHEET

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Optimizing Surfactant Systems Thickened with Carbopol®* ETD 2020 Polymer Using a Statistical Design

Introduction

An experimental design has been developed by Lubrizol Advanced Materials scientists to predict the viscosity and yield value of surfactant combinations thickened with Carbopol® ETD 2020 polymer. The information generated from the experimental design is available as a service to Lubrizol Advanced Materials, Inc. customers to facilitate the formulation of surfactant systems.

Use of the design will save time by providing formulating guidance and will help to predict the viscosity and yield value of finished products. The design is also beneficial because surfactant systems will be more effectively optimized, resulting in cost savings.

Background

Carbopol® polymers are well known for their ability to thicken, stabilize and provide suspending properties in a wide variety of personal care products. Carbopol® polymers are increasingly being used in surfactant systems such as bath gels, cleansing products and multifunctional shampoos, particularly with the recent introduction of Carbopol® ETD 2020. This polymer enhances the aesthetic properties and performance of surfactant systems by providing thickening, suspension stabilization and improved foam stability.

Optimizing surfactant systems can be a difficult and time-consuming process. Past literature has demonstrated the ability of Carbopol® polymers to thicken many single surfactants. However, when a formulator begins to actually formulate a shampoo, several questions may arise. What happens when several surfactants are mixed together? How much Carbopol® ETD 2020 polymer should be used to achieve a particular viscosity? What interactions will occur between surfactants? What are the optimum levels of surfactants to achieve the desired properties of the finished product?

Due to the difficulty of evaluating the interactions of all surfactants in combination with Carbopol® ETD 2020 polymer, a systematic approach was needed. A statistical design was developed to optimize and predict surfactant combinations with Carbopol® ETD 2020 polymer in order to minimize the number of experiments necessary to evaluate all interactions. Nine different surfactants—anionic, amphoteric and nonionic—were evaluated in various combinations with Carbopol® ETD 2020 polymer. Propylene glycol and ethanol were also included in the design to determine their effect on the viscosity and yield value of surfactant systems.

Carbopol® ETD 2020 Polymer

Carbopol® ETD 2020 polymer is an "easy-to-disperse" crosslinked polyacrylic acid copolymer used to thicken various surfactant systems. The CTFA name for this polymer is Acrylates/C10-30 Alkyl Acrylate Crosspolymer. Carbopol® ETD 2020 polymer is very similar to Carbopol® 1342 and 1382 polymers in thickening surfactant systems and providing yield value. Carbopol® ETD 2020 polymer has the advantages of being polymerized in a toxicologically-preferred cosolvent system, being easier to disperse and having a higher viscosity profile than its predecessors. Carbopol® ETD 2020 polymer provides the following benefits:

- Permanent suspension of insoluble ingredients - beads, mica, abrasives, actives
- Viscosity modification - thickens surfactants that do not respond to salt

- Foam stabilization - longer lasting foam
- Stability improvement - no separation or change in viscosity at long term high temperature and freeze/thaw stabilities
- Imparts slip - modifies feel
- Stabilization of silicones or oils - formulates 2-in-1 shampoos
- Rheology modification - modifies appearance, flow and texture

The Experimental Design

This design is a "response surface model" type. A response surface model (RSM) examines the linear effects of design variables as well as quadratic effects. In addition, this type of model examines synergistic effects between all design variables. The design was analyzed using the computer program RS/1®. Thirteen variables (ingredients) were used in the design, ten of which were surfactants.

The Experiments

Because of the number of variables, the RS/1 program required that 109 surfactant combinations (experiments) be made. (See Appendix I for examples.) This was the minimum number necessary to look at all possible interactions between experimental variables (ingredients). Once made, each experiment was measured for viscosity and yield value using the Brookfield method. These responses were entered into the program and interpreted. This data was then used to produce graphical predictions for viscosity and yield value for various surfactant combinations.

Experimental Variables, Constraints and Responses

In this type of design, certain factors must be constrained, while others vary.

Experimental Variables (Ingredients)

Abbreviation	CTFA Name	Trade Name	Actual Surfactant Active, %	Active Range in Experiment
TEALS	TEA Lauryl Sulfate	Standapol® T (Henkel)	40.0	0-10
ALES	Ammonium Laureth Sulfate	Standapol® EA3 (Henkel)	27.0	0-10
DLS	Disodium Laureth Sulfosuccinate	Mackanate™ EL (McIntyre)	32.3	0-10
P2000	Decyl Polyglucose	Plantaren® 2000	50.7	0-10
COCA-OX	Cocamido-propylamine Oxide	Mackamine™ CAO (McIntyre)	31.1	0-5
SCS	Sodium Cocyl Sarcosinate	Hamposyl® C-30 (Hampshire)	30.6	0-10
COCA-BET	Cocamido-propyl Betaine	Incronam® 30 (Croda)	29.9	0-5
COCA ACE	Cocampho-acetate	Miranol® Ultra (Rhone-Poulenc)	30.58	0-5
LDEA	Lauramide DEA	Ninol® 30LL (Stepan)	100.0	0-3
PG	Propylene Glycol	—	100.0	0-5
ETH	Ethanol	—	100.0	0-5
ETD 2020	Acrylates / C10-30 Alkyl Acrylate Crosspolymer	Carbopol® ETD 2020	100.0	0.5-1.0
SLS	Sodium Lauryl Sulfate	Standapol® WA-LC (Henkel)	31.0	0-12

[1]y (Brookfield Yield Value in dynes/cm^2) = $\dfrac{2r_1 (v_1 - v_2)}{100}$

Where: r_1 = slower spindle speed in rpm
v_1 and v_2 = apparent viscosities at r_1 and r_2 respectively

When: r_1 = 0.5 rpm and r_2 = 1.0 rpm
y (dynes/cm^2) = $\dfrac{\text{(Apparent viscosity @ 0.5 rpm – Apparent viscosity @ 1.0 rpm)}}{100}$

Responses

The responses were measured using the Brookfield method. Yield value is the determining factor in the ability to suspend.

Yield value[1]: 10 - 2,100 dynes/cm^2
Viscosity: 22 - 66,000 cP

Constraints

Surfactant actives: 5.0 - 25.0%
pH (adjusted with TEA): 6.0 - 6.5

Order of Addition

In order to minimize the effects of the order of addition on the outcome of the responses, a consistent order of addition was used:

1. Carbopol® ETD polymer was dispersed in deionized water.
2. Batch was partially neutralized with TEA (0.3% of Carbopol® polymer weight).
3. Ethanol and propylene glycol were added.
4. Primary anionic surfactants were added.
5. Plantaren® 2000 and amphoterics were added.
6. Amine oxide and DEA were added.
7. Final pH was adjusted to 6.0 - 6.5 with TEA.
8. Yield value and viscosity were measured after 2 days with Brookfield viscometer.

Graphical Predictions

The attached graphs demonstrate a small portion of the design's capabilities. The graphs in Figures 1 and 2 show the average main effects of each variable for viscosity and yield value. These graphs do not include interactions between variables.

The remaining 3-dimensional and contour plots (Figures 3 through 8) show trends and predictions of viscosity and yield value for surfactant combinations used in typical shampoo and bath gel formulations. Note that two ingredients vary while the others are held constant at the given usage levels. All ingredients are given in percent active. A key observation is how the viscosity and yield value change as the variable amounts are increased or decreased.

Figure 1
Main and Quadratic Effects
Adjusted Response Curves for Viscosity

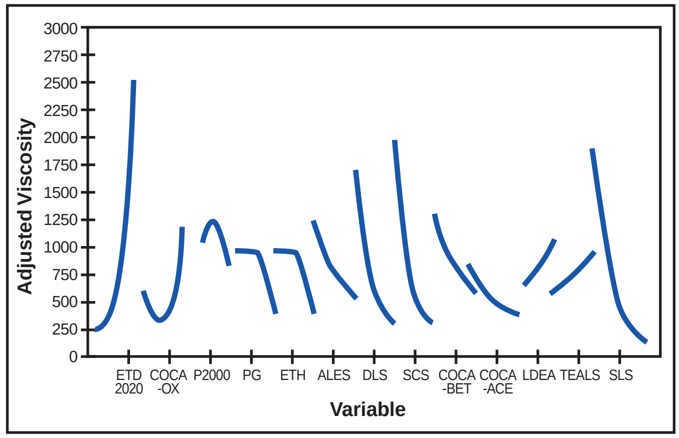


Figure 2
Main and Quadratic Effects
Adjusted Response Curves for Yield Value

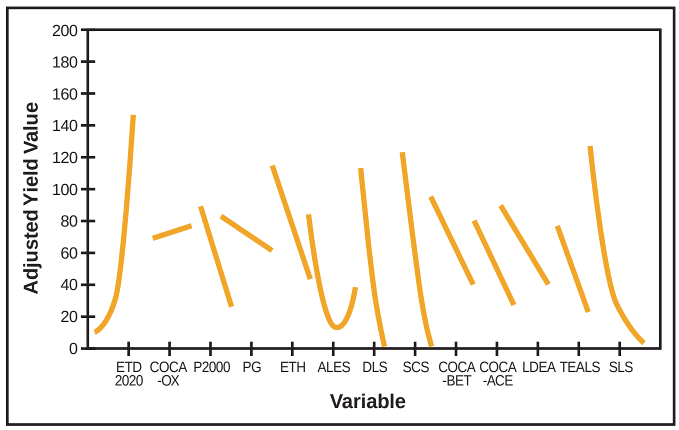


Figure 3
Experiment #9, Graphical Predictions for Viscosity
Variables: Carbopol® ETD 2020, Lauramide DEA
Constants: Sodium Lauryl Sulfate @ 10%

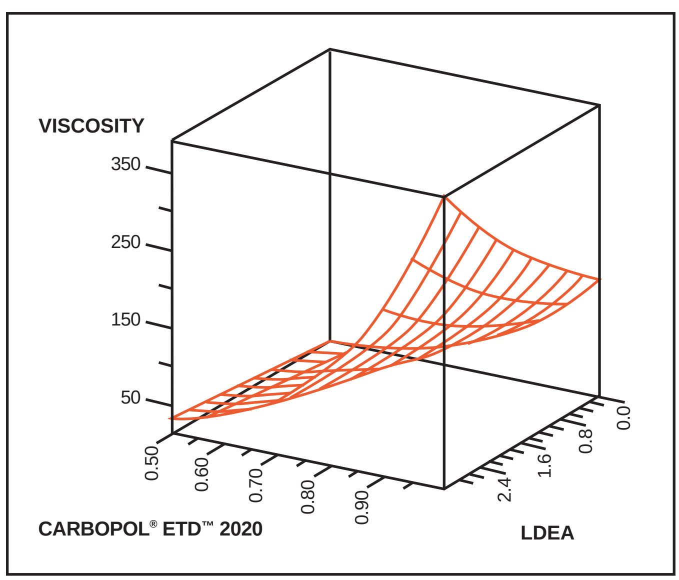


Figure 4

Experiment #9, Graphical Predictions for Yield Value

Variables: Carbopol® ETD 2020 polymer, Lauramide DEA

Constants: Sodium Lauryl Sulfate @ 10%

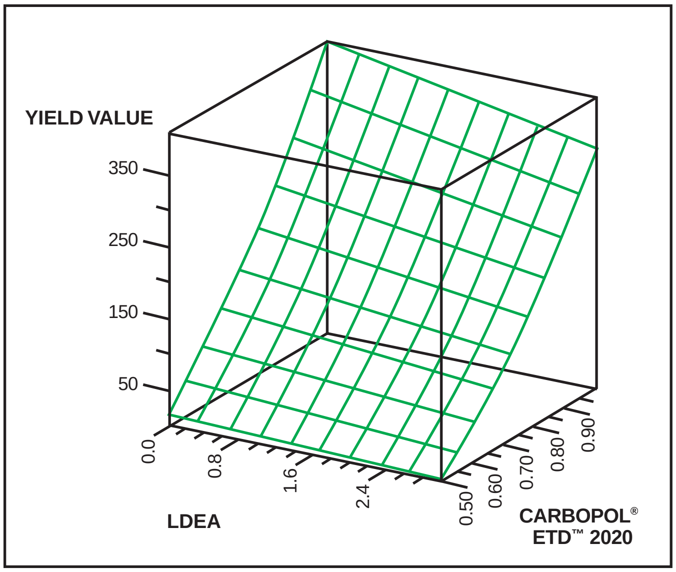


Figure 5

Experiment #10, Graphical Predictions for Viscosity

Variables: Sodium Lauryl Sulfate, Propylene Glycol

Constants: Carbopol® ETD 2020 polymer @ 1.0%, Lauramide DEA @ 2.5%, Miranol® Ultra @ 3.0%

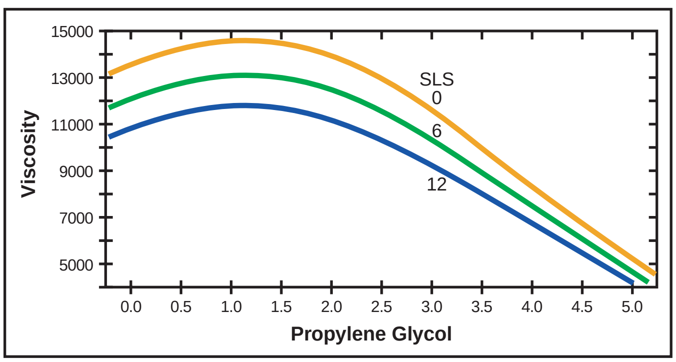
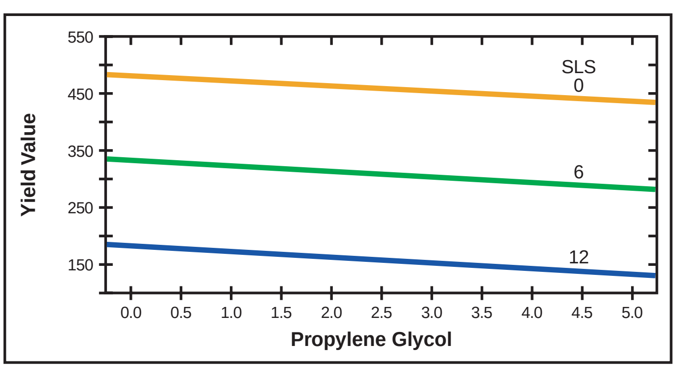


Figure 6

Experiment #10, Graphical Predictions for Yield Value

Variables: Sodium Lauryl Sulfate, Propylene Glycol

Constants: Carbopol® ETD 2020 polymer @ 1.0%, Lauramide DEA @ 2.5%, Miranol® Ultra @ 3.0%



For More Information

Using this experimental design, Lubrizol Advanced Materials, Inc. has the capability to generate graphical predictions for viscosity and yield value based on the ingredients of your individual formulation. We encourage you to contact our Technical Service Department at (800) 379-5389 or (216) 447-5000 and allow us to develop predictions for viscosity and yield value using the surfactants in your particular formula. (Please select surfactants from the experimental variables table on page 2.)

Figure 7

Experiment #11, Graphical Predictions for Viscosity

Variables: Carbopol® ETD 2020 polymer, Sodium Lauryl Sulfate

Constants: Lauramide DEA @ 2.5%, Betaine @ 2.0%

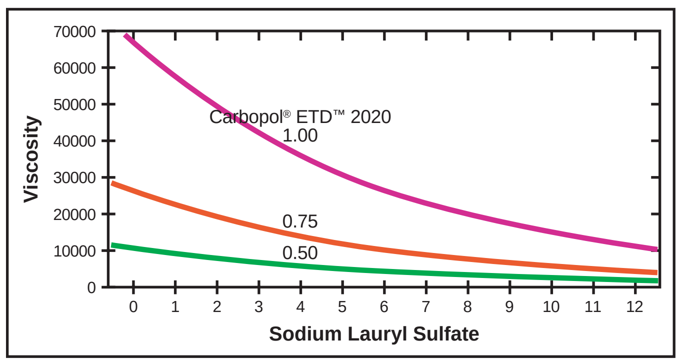
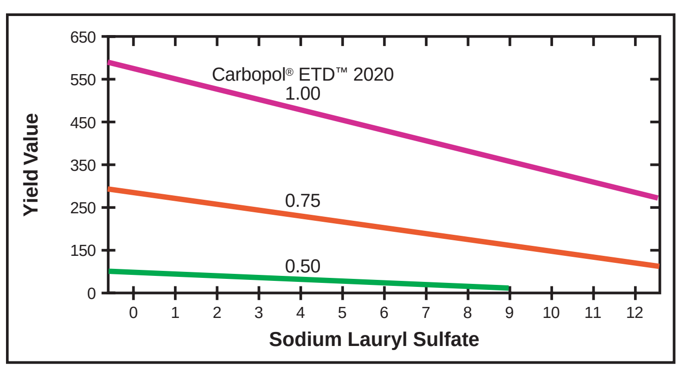


Figure 8

Experiment #11, Graphical Predictions for Yield Value

Variables: Carbopol® ETD 2020 polymer, Sodium Lauryl Sulfate

Constants: Lauramide DEA @ 2.5%, Betaine @ 2.0%



Appendix I

Examples of Experiments Generated by RS-1 Computer Design

	Variables												
Experi-ment #	TEALS	ALES	DLS	P2000	Coca Oxide	SCS	Ampho Type	Ampho Level	Laura-mide DEA	PG	ETH	Carbopol® ETD 2020 Polymer	Total Surfactant Actives
1	0.0	10.0	0.0	0.0	0.0	10.0	COCA_BET	2.5	2.5	5.0	2.5	1.00	25.0
2	0.0	10.0	0.0	0.0	5.0	0.0	COCA_BET	5.0	5.0	2.5	5.0	0.50	25.0
3	0.0	0.0	0.0	10.0	0.0	5.0	COCA_BET	5.0	5.0	0.0	0.0	1.00	25.0
4	10.0	5.0	0.0	10.0	0.0	0.0	MIRA_ULT	0.0	0.0	2.5	0.0	0.75	25.0
5	0.0	0.0	5.0	0.0	0.0	0.0	MIRA_ULT	5.0	0.0	2.5	2.5	0.75	10.0
6	10.0	5.0	0.0	0.0	2.5	0.0	COCA_BET	5.0	0.0	2.5	0.0	1.00	22.5
7	0.0	10.0	10.0	0.0	0.0	0.0	COCA_BET	0.0	0.0	0.0	0.0	0.75	20.0
8	0.0	5.0	0.0	5.0	0.0	10.0	COCA_BET	5.0	0.0	0.0	2.5	1.00	25.0
9	5.0	5.0	0.0	0.0	0.0	5.0	COCA_BET	5.0	0.0	5.0	2.5	1.00	20.0
10	10.0	0.0	0.0	5.0	5.0	0.0	COCA_BET	2.5	2.5	0.0	2.5	0.50	25.0
11	0.0	0.0	10.0	5.0	2.5	5.0	COCA_BET	0.0	0.0	5.0	0.0	0.75	22.5
12	10.0	0.0	0.0	0.0	0.0	10.0	COCA_BET	2.5	2.5	5.0	0.0	0.50	25.0
13	5.0	10.0	0.0	0.0	0.0	0.0	MIRA_ULT	2.5	0.0	5.0	0.0	0.50	17.5
14	0.0	0.0	0.0	0.0	0.0	10.0	MIRA_ULT	0.0	5.0	5.0	2.5	1.00	15.0
15	0.0	5.0	0.0	10.0	0.0	0.0	COCA_BET	0.0	5.0	2.5	0.0	0.75	20.0
16	0.0	5.0	5.0	10.0	2.5	0.0	COCA_BET	0.0	2.5	0.0	0.0	0.50	25.0
17	10.0	0.0	10.0	0.0	0.0	0.0	MIRA_ULT	0.0	5.0	2.5	5.0	0.50	25.0
18	5.0	10.0	0.0.	0.0	5.0	0.0	MIRA_ULT	0.0.	0.0	0.0	0.0	1.00	20.0
19	0.0	5.0	5.0	0.0	5.0	0.0	MIRA_ULT	5.0	5.0	5.0	0.0	0.75	25.0
20	0.0	10.0	5.0	0.0	5.0	0.0	COCA_BET	0.0	2.5	0.0	2.5	1.00	22.5
21	0.0	0.0	0.0	5.0	2.5	0.0	MIRA_ULT	0.0	0.0	0.0	5.0	0.50	7.5
22	5.0	10.0	0.0	0.0	0.0	5.0	MIRA_ULT	0.0	0.0	0.0	0.0	0.50	20.0
23	5.0	10.0	0.0	5.0	5.0	0.0	COCA_BET	0.0	0.0	5.0	2.5	0.75	25.0
24	0.0	5.0	0.0	10.0	2.5	0.0	COCA_BET	5.0	2.5	2.5	5.0	1.00	25.0
25	5.0	5.0	10.0	0.0	0.0	0.0	MIRA_ULT	5.0	0.0	2.5	5.0	0.75	25.0
26	0.0	0.0	0.0	5.0	0.0	10.0	COCA_BET	5.0	5.0	5.0	0.0	0.50	25.0
27	10.0	0.0	0.0	0.0	0.0	0.0	MIRA_ULT	2.5	0.0	0.0	2.5	0.50	12.5
28	0.0	5.0	0.0	0.0	0.0	0.0	MIRA_ULT	0.0	0.0	0.0	0.0	1.00	5.0
29	5.0	0.0	0.0	0.0	0.0	10.0	COCA_BET	2.5	0.0	0.0	5.0	0.50	17.5
30	0.0	5.0	10.0	0.0	0.0	0.0	COCA_BET	0.0	0.0	0.0	5.0	0.75	15.0
31	0.0	5.0	0.0	0.0	2.5	10.0	COCA_BET	2.5	5.0	0.0	0.0	0.75	25.0
32	0.0	0.0	5.0	0.0	2.5	10.0	COCA_BET	0.0	5.0	5.0	2.5	1.00	22.5
33	5.0	5.0	0.0	0.0	5.0	0.0	COCA_BET	2.5	5.0	5.0	0.0	1.00	22.5
34	5.0	0.0	0.0	0.0	5.0	0.0	COCA_BET	5.0	0.0	2.5	5.0	0.50	15.0
35	0.0	10.0	0.0	0.0	0.0	0.0	MIRA_ULT	5.0	5.0	0.0	5.0	0.50	20.0
36	0.0	0.0	0.0	5.0	2.5	5.0	MIRA_ULT	5.0	5.0	2.5	5.0	1.00	22.5
37	5.0	0.0	10.0	0.0	5.0	5.0	COCA_BET	0.0	0.0	5.0	5.0	0.75	25.0

KEY: TEALS=TEA Lauryl Sulfate; ALES=Ammonium Laureth Sulfate; DLS=Disodium Laureth Sulfosuccinate; P2000=Plantaren 2000; Coca Oxide=Cocamidopropylamine Oxide; SCS=Sodium Cocyl Sarcosinate; Ampho Type=Amphoteric Type (MIRA_ULT=Miranol Ultra, COCA_BET=Cocamidopropyl Betaine); PG=Propylene Glycol; ETH=Ethanol; ETD2020=Carbopol ETD 2020