Scrub with Superior Exfoliation and Cleansing Properties Developed with Designed Experiments

Scrubs are personal care products that provide both cleaning and exfoliation. Exfoliation involves the removal of the oldest dead skin cells on the skin's outermost surface and has been used for ages to help maintain the skin. “In the past, the personal care industry developed products using what can be called the spray and pray method,” said Christopher Felski, Formulator for Alberto-Culver. “You'd try a little more of this and a little more of that. The problem was that even when you ended up with a great product, you didn’t know how or why you got there. Every new product would start from scratch.”

Alberto-Culver recently developed a new line of scrubs using the design of experiments (DOE) method in which all formulation factors are simultaneously varied to explore the entire design space. Statistical analysis of the results revealed the expected performance of any formulation, not just the ones that were specifically evaluated in the experiments. “Using DOE we have pushed the performance of our product well past the limits of what we thought was possible,” Felski said. “The new scrubs have an exfoliant concentration that considerably exceeds the 10% level that was the upper limit in the past.”

Ingredient	Effect on Foam	Effect on Viscosity
Foam Modifier	↑	↓
Surfactant	↑	↑
Exfoliant	↓	↑
Water/Other	↓	↓

Figure 1: The foaming scrub system

Challenge of formulating scrubs

The Alberto-Culver Company is a $1.4 billion manufacturer and marketer of beauty, personal care and household products that operates in more than 120 countries around the globe. Alberto-Culver's market-leading St. Ives line of scrubs uses foam modifiers to provide cleansing action and exfoliant to remove dead skin. The ideal scrub has a high level of foam and exfoliation and its viscosity falls within a certain range. Figure 1 shows the effect of each ingredient on the responses of foam and viscosity. Exfoliation is not a response because it is simply proportional to the amount of exfoliant used. Increasing the amount of exfoliant also increases the viscosity of the product which places practical limits on the amount of exfoliant that can be used.

Alberto-Culver previously used the same product development methods as the rest of the personal care products industry. Using these methods, the company created many industry-leading products. But formulators could never be sure that they had developed the best product that was possible with the current technology. They gained understanding of the effect of changing a single ingredient but not on the interactions between ingredients. Another weakness of the conventional approach was that there was no way of knowing the robustness of the product. Will a little more of one ingredient cause problems? Is there another formulation that will provide the same or better performance while being easier to manufacture because it is less sensitive to variation?

DOE drastically reduces the number of runs required to determine the optimal settings of each factor by varying the values of all factors in parallel. DOE determines not just the main effects of each factor but also the interactions between the factors, making it far superior to the traditional one-factor-at-a-time (OFAT) approach.

Mixture design experiments

"We selected Design-Expert® software from Stat-Ease, Inc. (Minneapolis, MN) because of its exceptional capabilities in the design and analysis of mixture design experiments," Felski added. "Mixture design reduces the number of experiments required to develop formulations of personal care products. The proportion of each ingredient in a mixture has to add up to 100%. This reduces the number of degrees of freedom, which in turn substantially reduces the number of runs required to achieve a given level of

statistical significance. The end result is that we can get products to market faster and at a lower cost than with conventional experiment designs."

Alberto-Culver marketers had a vision for a product that provides a higher level of exfoliation and cleansing than any existing product. In the past it had never been possible to increase the proportion of exfoliants beyond 10% because the viscosity of the product was too high. Felski set out to design an experiment that would enable him to break this barrier. The four components studied in the experiment are shown in figure 1. The ranges and other data provided in this article have been modified in order to protect Alberto-Culver's trade secrets.

A) Foam Modifier (0%-5%)
B) Surfactant (0%-10%)
C) Exfoliant (0%-10%)
D) Water/Other (80%-97.5%)

Blend	A: Foam Modifier	B: Surfactant	C: Exfoliant	D: Water	R1: Viscosity (10 RPM) poise	R2: Foam Volume mL
1	2.5	10.0	5.0	82.5	200	700
2	5.0	0.0	10.0	85.0	180	375
3	0.0	10.0	10.0	80.0	570	175
4	0.0	10.0	0.0	90.0	290	275
5	0.0	5.0	5.0	90.0	340	275
6	0.0	5.0	0.0	95.0	215	350
7	0.0	0.0	10.0	90.0	490	75
8	5.0	10.0	10.0	75.0	260	750
9	2.5	0.0	0.0	97.5	95	412.5
10	5.0	10.0	0.0	85.0	60	900
11	5.0	0.0	10.0	85.0	195	225
12	5.0	5.0	0.0	90.0	50	900
13	0.0	0.0	5.0	95.0	300	150
14	2.5	0.0	0.0	97.5	75	400
15	5.0	10.0	10.0	75.0	315	675
16	2.5	5.0	10.0	82.5	390	312.5
17	5.0	5.0	5.0	85.0	105	675
18	2.5	10.0	5.0	82.5	230	637.5
19	0.0	10.0	10.0	80.0	590	200
20	5.0	0.0	3.33	91.67	50	425

Figure 2: Design run sheet and results

The two responses for this experiment were foam value and viscosity. Felski entered this information into Design-Expert software and the software set-up a 20-run experiment with 16 unique blends and 4 replicates that fully explored the design space. “The use of a mixture design experiment reduced the number of runs required to achieve the desired level of accuracy by 50% compared to a factorial design,” Felski said. Technicians ran the test runs as indicated by the software. Felski then entered in the results from each. The experimental design and results are shown in Figure 2.

Model shows excellent predictive capabilities

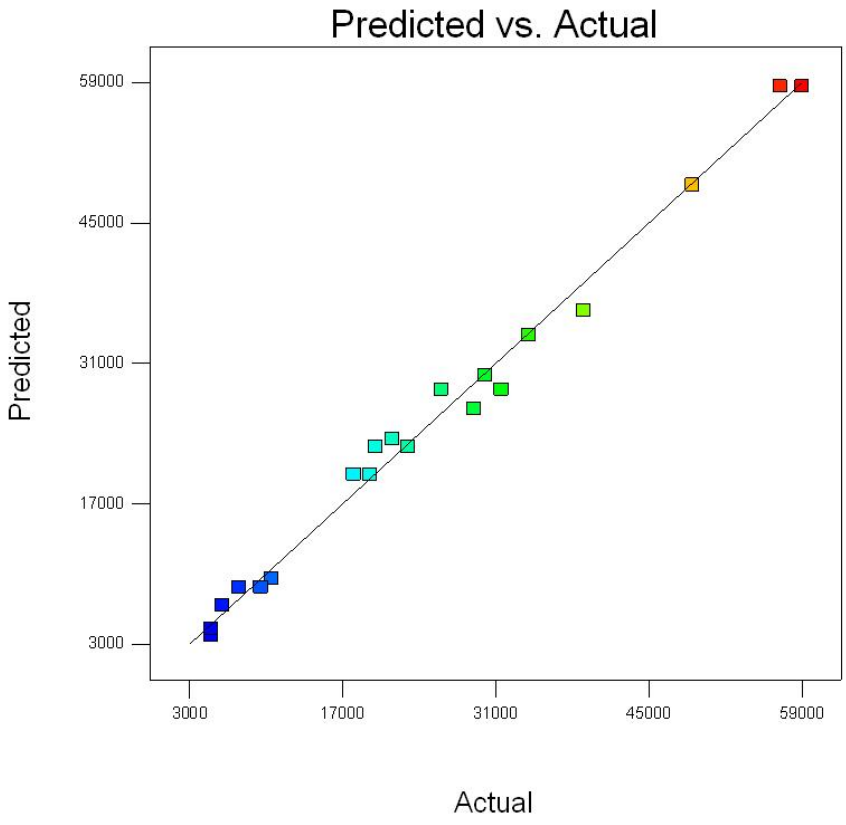

Figure 3: Model significance and predictions

Felski selected a quadratic model and checked its predictive ability on the viscosity response. Figure 3 shows the viscosity predicted by the model for each run graphed against the actual measured values. “The model demonstrated excellent predictive capabilities,” Felski said. The model has an F-value of 134.60 which implies only a 0.01% chance that the model

could be caused by noise. All of the factors and all of the two-factor interactions had a significant effect on the responses.

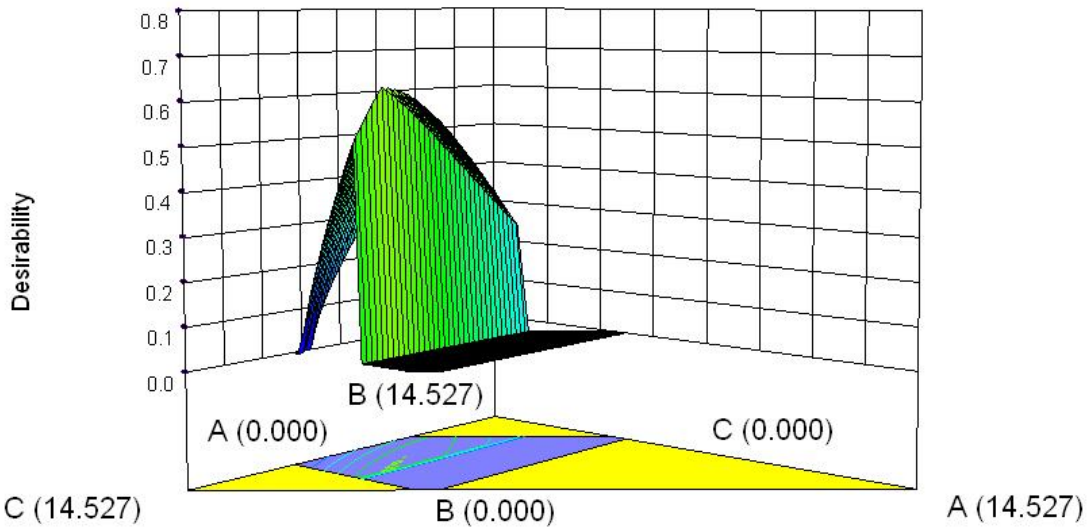

Figure 4: Numerical optimization shows most desirable region for formulation

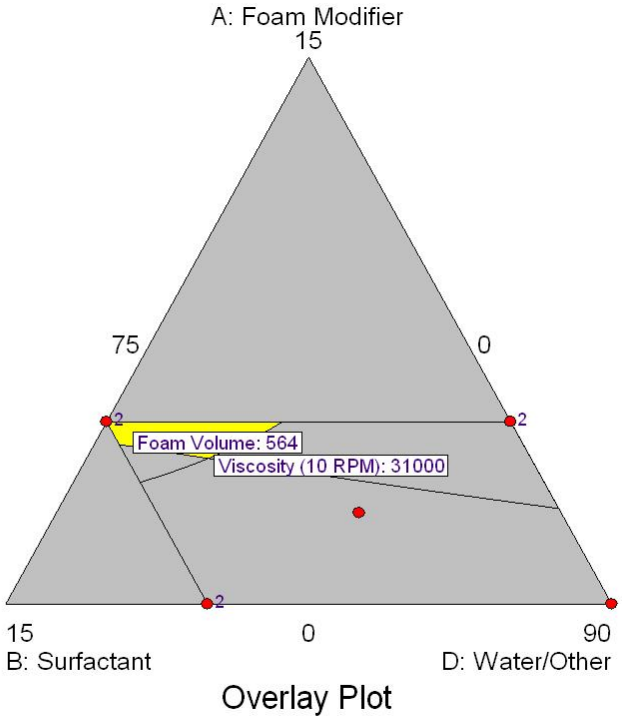

Figure 5: Graphical optimization shows the sweet spot

Confident in the predictive power of the model, Felski then used it to optimize the formulation. His objective function held viscosity within a range of 10,000 to 50,000 centipoise and maximized exfoliant and foam volume. Each of the three objectives was weighted equally in the desirability function. The optimization showed that it was possible to increase the amount of exfoliant (C) well beyond 10% while also increasing foaming (A) beyond the levels of current products and maintaining viscosity within the desirable range. Figures 4 and 5 show the numerical and graphical optimization. As mentioned earlier, the data has been modified to protect Alberto-Culver's confidential information.

Felski ran confirmatory experiments at each optimal value and the results closely matched the DOE predictions. He looked at the graphical optimization to determine how sensitive each optimal value was to changes in the proportions of each ingredient. He selected an optimum that was relatively inexpensive to manufacture and insensitive to variation. "The scaleup went very well because we had mapped out the formulation space and selected a very robust formulation with no steep cliffs," Felski said. 'Mixture DOE helped us push our product performance to a level that in the

past we thought was impossible. Market research shows that consumers give the new product a 25% higher rating than comparable existing products. The product will soon be introduced to the market."

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