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The Development of High Conversion PVC Suspension Resins – A Continual Improvement Case Study

By

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INTRODUCTION

In September 1978 a three-man team of BF Goodrich Chemical Company scientists was designated as the PVC High Conversion Suspension Resin Task Force. The Task Force assignment was to develop polymerization technology which would increase the conversion of both flexible and rigid grade resins without the loss of critical resin properties. This outcome was needed by June 1979 in order to help meet expected market demand. To some extent this objective was like that of the TV series, "Mission Impossible" because with the existing technology, conversion for suspension resins was in a state of statistical control with an average of 68%. The PVC industry had accepted that going to higher conversion decreased porosity and increased powder-mix-time, both of which were undesirable.

The purpose of this paper is to describe how the efforts of the Task Force increased the percent conversion of PVC resins to the mid-80's by April 1979 - two months ahead of the deadline - while improving the processing properties. The goal was attained because a process of continual improvement was used based on the following steps:

1. retrieving and analyzing historical lab data.
2. augmenting historical data using sequential experimental strategies.
3. modeling processes using statistical methods.
4. optimizing conflicting property objectives.
5. scaling-up the technology from one reactor configuration to another.

These five diagnostic steps to be described required a combination of subjective and statistical knowledge in order to achieve the new paradigm. This is the synergy of engineering and statistical thinking recommended by Shewhart in 1939.[1] What will not be presented is the guidance and direction provided to the Task Force by top management. The reader is advised to study Juran's discussion of "breakthrough in knowledge" in order to understand this function.[2]

STEP 1. RETRIEVING AND ANALYZING HISTORICAL LAB DATA

Before initiating a search for information and data relative to this project, it was deemed essential to develop a theory in response to the following question, "Why had suspension grade PVC resin not been polymerized beyond 68% conversion?" Although not always recognized, or even if recognized not adhered to, the development of knowledge in general must be guided by theory. Toward this end, scanning election micrographs of PVC sections showed that as conversion increased beyond 68% the resin morphology became more and more closed. As a result, at higher conversions, the porosity, a key resin property, was lower than the customer's minimum acceptable requirements.

Thus, the Task Force began their assignment by brainstorming the variables which potentially could cause the resin to become non-porous as conversion increased. The output from these sessions is shown in a Cause and Effect Diagram format in Figure 1.

Using a system of computerized data retrieval developed by the Statistics and Computer Group at the BF Goodrich Technical Center, a portion of the information identified as potentially important was assembled. (see Table 1.) Specifically, available for analysis were data on 268 1100-gallon scale polymerizations, a tremendous quantity of data. But was this quality data in the statistical sense?

After many attempts to analyze the effect of these ten variables on porosity using multiple correlation, the results indicated that

- few meaningful relationships were identified,
- few relationships were statistically significant ($|t| < 2$),
- the ratio, scatter about the correlation divided by the experimental error, was large (greater than 1.7).

Thus, with these data it was not possible to develop process models which could guide the Task Force in their search for technology that would show how conversion can be increased without a loss in porosity.

Part of the problem was caused by the fact that many of the controllable variables were correlated or confounded with each other. In addition, the controlled variable ranges in some cases were extremely narrow. Both situations were not surprising in as much as the 298 runs used in the analysis were gleaned from several development efforts rather than a single, well planned set of trials. However, it is always valuable to examine the existing information to avoid the possibility of "re-inventing the wheel". Therefore, augmenting the existing data with the appropriate number of key trials was the next step recommended.

STEP 2. AUGMENTING HISTORICAL DATA

Many experimenters (scientists and engineers) and applied statisticians next will proceed to start from the beginning and plan a series of trials to run based on statistical principles. Unfortunately such an approach typically ignores valuable historical information and, consequently, is not very efficient. An alternative to such a strategy is to augment existing data in order to

(a.) minimize the effects of "uncontrollables",
(b.) extract the most information for the least cost,
(c.) quantify important effects and interactions,
(d.) screen many variables efficiently,
(e.) minimize the number of low information trials.

The augmentation process is difficult with classical experimental design theory and orthogonal arrays as discussed by Box, Hunter, and Hunter[3] and Taguchi[4]. However, using computerized design programs such as EDO©[5], this assignment was easily accomplished by the Task Force.

The basis for this augmentation process was that given the historical design matrix,

$$\begin{array}{c} \text{variable} \\ 1 \; 2 \; . \; . \; . \; . \; . \; . \; . \; . \; . \; 10 \end{array}$$

$$X = \left[x_{ij} \qquad \begin{array}{l} i = 1, \dots 268 \\ j = 1, \dots 10 \end{array} \right]$$

268 rows x 10 columns

find a matrix with minimum number of rows

$$
\begin{array}{cc}
 & \text{variable} \\
 & 1 \quad 2 \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad . \quad 10 \\
X' = & \begin{bmatrix} x'_{ij} & \begin{array}{l} i = 1, \ldots, n \\ j = 1, \ldots, 10 \end{array} \end{bmatrix}
\end{array}
\quad \text{n rows x 10 columns}
$$

such that

$$
\frac{1}{K} \sum_{\substack{\text{all} \\ \text{admissible} \\ \underline{x}}} \underline{x}^T \left\{ \begin{bmatrix} X \\ X' \end{bmatrix}^T \begin{bmatrix} X \\ X' \end{bmatrix} \right\}^{-1} \underline{x} = 1.0
$$

where K, the number of admissible experiments (denoted by the 1 x 10 vector $\underline{x}$), was 3^{10} in this case study. Such designs are referred to as approximately D-optimal and are very effective for satisfying the criteria (a.) through (e.) above when used in conjunction with multiple correlation analysis. The Task Force found the number of new experiments to be 30 (i.e. n = 30).

STEP 3. PROCESS MODELING

After the new trials were run at the 1100-gallon development scale using the augmentation plan denoted by X', the data from the 30 trials were combined with that in Table 1 and re-analyzed. Using multiple correlation analysis, meaningful results were obtained using step-wise procedures. Figures 2 and 3 show the relationships for porosity and bulk density, two properties of critical importance to customers.

In order to verify the predictive accuracy of these models, six follow-up runs were made at conversions above 80% to determine if the resin porosity and powder mix time were acceptable with respect to customer specifications as expected. Once confirmation was realized, the next problem step, optimization, was addressed.

STEP 4. OPTIMIZING CONFLICTING OBJECTIVES

An examination of Figures 2 and 3 indicates that high temperature reduces Porosity but increases Apparent Bulk Density. Unfortunately these are conflicting objectives from the customers' perspective. Situations such as the one described here, therefore, require methodology to optimize conflicting objectives.

Toward this end it is essential to engage in a dialogue with customers in order to quantify their requirements. This can be accomplished by developing "Goodness" curves based on input from knowledgeable PVC users. For example Figure 4 illustrates such "Goodness" functions for Porosity and Apparent Bulk Density.

Using multiple property optimization software like MPO©[6], customer requirements expressed as "Goodness" functions and multiple correlation models can be combined with search logic to identify the best balance of polymerization recipe and process conditions to simultaneously optimize conflicting objectives like Porosity and Apparent Bulk Density.

Only after this step had been completed and results of predicted optimums verified in follow-up 1100-gallon development scale runs was the Task Force ready to address scale-up to production where recycle monomers were used, different agitation systems had been installed, and both spray and fluid bed dryers were used.

STEP 5. SCALE-UP

A scale-up strategy was needed to modify the models developed and confirmed at the 1100-gallon development scale and the associated optimum process for high conversion resin. To begin with, a D-Optimal experimental screening design was obtained using EDO© logic. This design contained the optimum from Step 4. as the first trial. These twelve experiments were run at the various production locations and measured results were compared with predictions from the models developed in Step 3.

Multiple correlation analysis was used to determine significant relationships for the differences (or Δ's) between the measured results and expected outcomes. In this way functional relationships, f_Δ were obtained such as the following for Porosity:

$$\text{Porosity (Prodn.)} - f_{porosity}\text{(development variables)} = f_{\Delta\text{-}porosity}\text{(development variables, production variables)}$$

Hence, the production scale predictor becomes $f_{porosity}$ (development variables) + $f_{\Delta\text{-}porosity}$ (development variables, production variables). A graphical representation of these scale-up correlations is shown in Figure 5.

Once again confirmation of these scale-up functions at each production facility enabled the Task Force to develop optimum recipes for producing consistent quality high conversion suspension PVC resins using the same approach outlined in Step 4.

CONCLUSION

Timing wise, Steps (1.) through (4.) were completed by the end of 1979. Scale-up, the last step, required about two weeks per production facility for experimentation, analysis of the data, and optimization. Thus it was possible to complete all phases of the development tasks by April 1979. As the ownership of the project transferred to marketing and sales, however, it was apparent that this would become the more time consuming aspect of the effort. It was no longer a scientific endeavor but a psychological one, trying to develop a cultural change that would accept PVC suspension resin polymerized to 85% conversion.

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Table 1. Historical Data (268 trials)

	Dispersant Type M	Dispersant Type A	Dispersant Type T	Dispersant Type P	RPM	No. Agit. Blades	Baffle Length (cm)	R_x Temp (°F)	Catalyst Type	Conversion (%)	Powder-Mix-Time-(sec)	Porosity (cc/g)
Variable Number	1	2	3	4	5	6	7	8	9	10	11	12
Max.	0	0	.001	.005	120	1	210	47	E	60	326	.144
Min.	.06	.07	.05	.025	190	2	239	60	S	90	992	.277

Figure 1
Potential Factors Causing Loss of PVC Resin Porosity

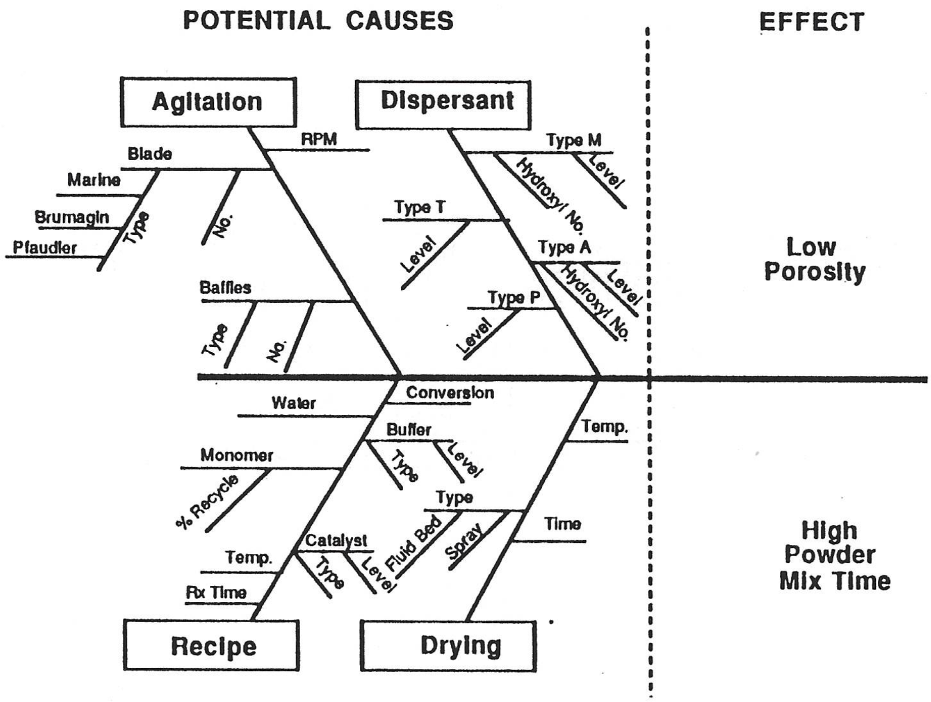


Figure 2

Porosity

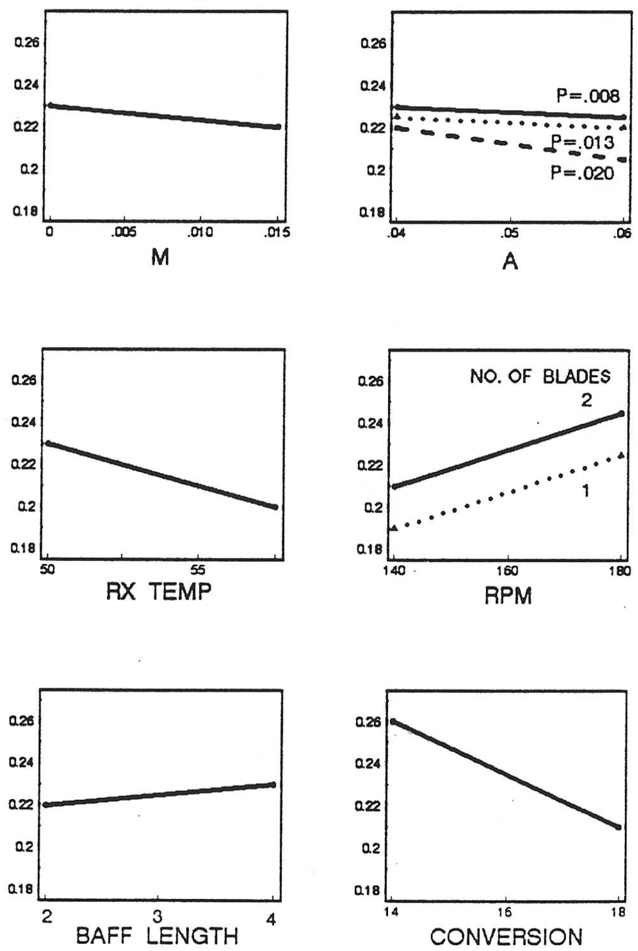


Figure 3

Apparent Bulk Density

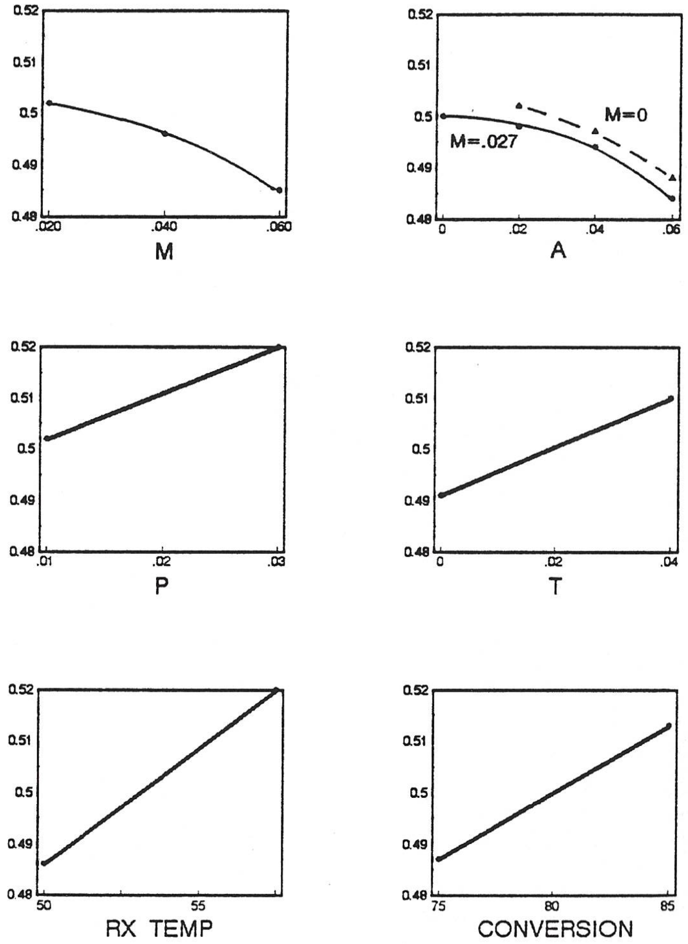


FIGURE 4

Goodness Functions for Porosity and Apparent Bulk Density

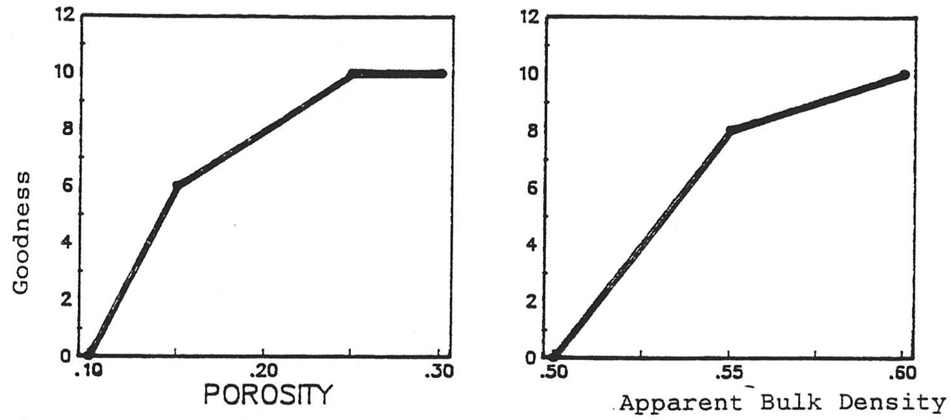
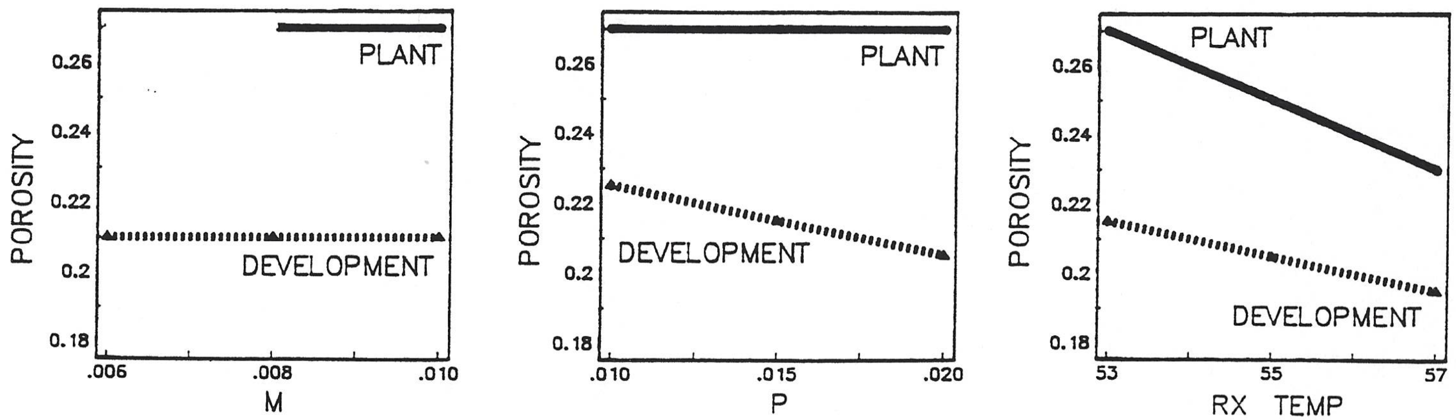


FIGURE 5

Scale-Up Correlation



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