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Improving Resistivity of Resin

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Abstract: Resistivity of resin plays a vital role for trouble free performance of electrostatic application of resin based paints. Low resistivity results in electrical shorting and high resistivity leads to poor deposition of paints due to insufficient charge pick up. Hence resin resistivity should be maintained within a specified range. The present study was undertaken in a reputed paints manufacturing enterprise. The concerned firm was not able to maintain the resistivity value within the desired specification. They were consistently getting low resistivity. It was decided to study on identifying significant factors for controlling resistivity through experimentation.

A 2^3 full factorial experiment was conducted with three factors phthalic anhydride%, water collection time, and solvent distillate collection time, each varying at two levels. The experiment could not be replicated due to time constraint. After analysing the data, using Bayesian method of posterior probability plot the main effect of water collection time and solvent distillate collection time were found to be significant at 5% level of significance. Considering expected values of resistivity at different levels of significant factors, optimum levels were arrived at so as to achieve the expected average resistivity value closed to the target value. Taking experimental error into account a 95% confidence interval was obtained which was lying within the desired specification. Confirmatory trials were made for few batches. Since the results of confirmatory trials were satisfactory the recommendations were accepted by plant management. As a result of implementation of the recommendations of the present study the concerned firm was able to get the value of resin resistivity within the specification.

1 Introduction

The present study was carried out in a renowned Indian paints company. Resin is an important constituent of paints. It is used as pigment binder. The role of resin in paints application is twofold:

1. to act as a binding agent of pigments and
2. to facilitate uniform disposition of paints.

Resistivity is playing an important role in paints application. For electrostatic spraying the resin based paint is applied in the form of atomized charged particles. The distribution of charged particles depends on the resistivity of resin. A low resistivity results in electrical shorting, whereas high resistivity leads to poor deposition of paint due to insufficient charge pick up. Hence, resin resistivity within an appropriate range is pre-requisite for trouble free electrostatic application of resin based paints.

2 Problem

The required specification for resin resistivity was (130-180) kohm whereas the concerned industry was achieving resistivity between 100-130 kohms in their regular production which was far below the lower limit of customer specification. Hence, a study was initiated by the R&D department with the objective to achieve resistivity of resin in the desired zone.

2.1 Process

The manufacturing process started with charging poly alcohol, polyacid solvent with agitator. The solution was then refluxed for half an hour and cooled. Refluxing was a process of recycling the solvent in given manner :

heating → vapourisation → condensation

Urea was then added at a fixed temperature and the solution was again refluxed. Then the solution was cooled and, subsequently, phthalic anhydride and xylene were added and stirred for 15 minutes. During this stage water was generated, which was removed in a phased manner over a predefined time interval. After completion of water removal, the solvent distillate was also removed. After filtering the end product was pumped to storage.

2.2 Objective of Study

The present study was taken up with the objective to have the average resin resistivity close to the nominal value of desired specification, i.e., 155 kohms.

2.3 Approach

Based on experience and technical knowledge, three process parameters (factors) were considered for the study., which are given below:

- Phthalic anhydride (%) (A) : For etherification and polymerisation.
- Water collection time (B) : For efficient water removal.
- Solvent distillate collection time (C) : For etherification.

It was decided to conduct an experiment with the help of statistical design considering the above mentioned factors, each at two levels. Factors and corresponding level considered for the study are given below.

Table 1: Factors and Levels

Factor	Levels	
	1	**2**
A	0.5	0.6
B	3.5 hr	4.5 hr
C	25 min	45 min

Because of lack of time only very few experiments could be performed, by considering a 2^3 full factorial design with only one execution. As a matter of fact, the small number of experiments cast the success into doubts, but nevertheless an attempt seemed to be worthwhile.

2.4 Data Collection

The 8 (=2^3) trials were carried out in the R&D department. Resistivity was measured as response. The following table gives the experiment layout along with response. Randomization was taken care of while carrying out the experiment.

Table 2: : Lay-out of experiment and response

Trial	Factors			
No.	**A**	**B**	**C**	**Response**
1	1	1	1	170.0
2	1	1	2	147.5
3	1	2	1	112.5
4	1	2	2	102.5
5	2	1	1	190.0
6	2	1	2	157.5
7	2	2	1	132.5
8	2	2	2	104.0

3 Analysis and Result

Since it is an experiment with no replications the data could not be analysed by usual ANOVA. However, Box and Meyer [1] have proposed a Bayesian procedure for an analysis of unreplicated data that produces posterior probabilities that an effect is active and which is rather efficient in the case a screening stage of an investigation.

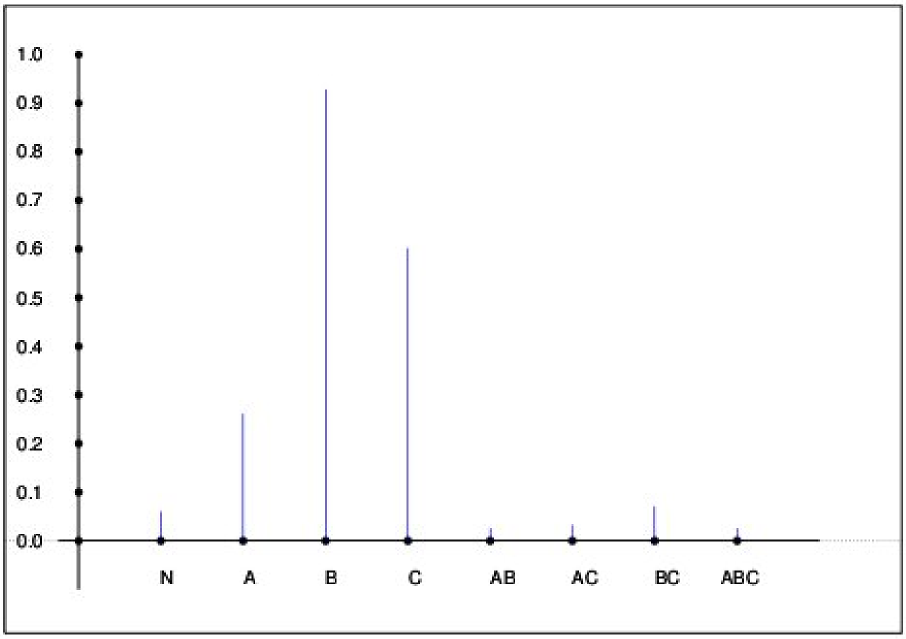
In 1989 Stephenson, Fredrick and Moore [3] developed a Fortran based computer program for calculation and plotting the posterior probabilities using the method proposed by Box and Meyer. The data was analysed by using this computer program.

Table 3: Output of Stephenson, Fredrick and Moore's program

Prior probability selected for an active effect is: .20
Multiplier of SIGMA selected for an active effect is: 10.00

ALPHA	K	EFFECT	ESTIMATED EFFECT	PROBABILITY EFFECT IS ACTIVE
.2000	10.00	NONE		.06040
		1	12.87500	.26177
		2	-53.37500	.92822
		3	-23.37500	.60141
		4	-2.12500	.02609
		5	4.12500	.03265
		6	-7.12500	.07047
		7	-2.12500	.02609

Figure 1: Graphical representation of the probabilities that factors are active



From the above analysis shown in Figure 1 it is clear that none of the interactions of the factors must be considered and, moreover, that factor A has a comparatively small effect. Hence, for the subsequent analysis only the factors B and C were considered as active. Next, an analysis of variance was performed by taking the other effects as error and the effect of B and C were tested against it.

Table 4: ANOVA Table

source of variation	degree of freedom	sum of square	mean square error	F	$F_{0.05,1,5}$
A	1	331.531	331.531		
B	1	5697.781	5697.781	58.72	6.61
C	1	1092.781	1092.781	11.262	
A,B	1	9.031	9.031		
B,C	1	101.531	101.531		
C,A	1	34.031	34.031		
A,B,C	1	9.031	9.031		
ERROR*	5	485.155	97.031		

ERROR was obtained by pooling SS due to A, AB, BC, AC, ABC which were found to be insignificant from posterior probability analysis. Factors B and C were found to be significant at 5 % level.

Next, the average response table was computed to determine appropriate levels for the significant factors.

Table 5: Average response table

Factor	**Average response at**	
	Level - 1	**Level - 2**
B	166.25	112.875
C	151.25	127.875

The following table was computed showing the expected average response at 4 combinations of the significant factors.

Table 6: Expected average response table

Factor	**C**		
	Level	**1**	**2**
B	**1**	177.94	154.56(*)
	2	124.56	101.19

That combination of factor levels was selected with expected average response closest to the desired target value given by the center of the specification interval $\frac{130+180}{2} = 155$.

Hence, the selected factor level combinations was B1 and C2, i.e.:

water collection time:	3.5 hr
solvent distillate collection time:	45 min

with expected average response (resistivity) 154.56, being rather close to the desired value, with a 95% confidence interval giben by (154.56 ± 19.30), i.e., (135.26, 173.86), which lies completely within the specification interval.

4 Conclusions

The following conclusions were drawn from the experiments:

- Water collection time and solvent distillate collection time were found to be significant for resistivity.
- Lower water collection time resulted in higher resistivity and lower solvent distillate collection time showed the same effect.
- The two levels for phthalic anhydride % were found not to be significant with respect to resistivity.
- No interaction was found to be significant.

The combination selected (i.e. B1 and C2) was tried and two batches were produced. As expected, resistivity was observed to lie within the bounds of the confidence interval.

Thus, it was recommended to use a water collection time of 3.5 hr and a solvent distillate collection time of 45 min for obtaining an average resistivity within specification. This recommendation was discussed with plant managers and they accepted it. After successful implementation of the recommendation the problem of low resistivity was substantially reduced and henceforth the recommendation was implemented as standard operating practice.

References

[1] Box and Meyer, (1986): An analysis of unreplicated fractional factorial. *Tecnometrics* 28, 11-18.

[2] Montgomery, D. C. (): *Design and Analysis of Experiments.*

[3] Stephenson W. R., Hulting F. L. and Karen Moore, (1989): Posterior probabilities for identifying active effects in unreplicated experiments. *JQT* 21, 202-215.

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