Application of Taguchi Method in Determining Optimum Level of Curing System of NBR/PVC Blend

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ABSTRACT: Taguchi method is applied in this study to determine the optimum level of curing system (sulfur, MBTS, and CBS) in a NBR/PVC blend. By considering physicomechanical properties of cured NBR/PVC blend, optimum level of curing system is determined. A fixed master batch formulation of NBR/PVC is used, and the effect of curing system is studied on the physicomechanical properties of NBR/PVC blend, such as tensile strength (TS) and elongation-at-break (EB) before and after aging and also hardness and abrasion. The L9 orthogonal array that includes nine rows and four columns is applied. In this matrix, rows show the experiments and three columns show the amount of three factors (sulfur, MBTS, and CBS) and one column is left arbitrarily as an empty column. In this array, the columns are mutually orthogonal. The optimum physicomechanical properties of cured NBR/PVC blend are at 2 phr sulfur, 2 phr MBTS, and 0.5 phr CBS of curing system. © 2006 Wiley Periodicals, Inc. J Appl Polym Sci 102: 5358–5362, 2006

Key words: Taguchi method; NBR/PVC blend; curing system; physicomechanical properties

INTRODUCTION

Rubber–plastic blends have become technologically important for use as thermoplastic elastomers (TPEs).[1] The plastic and rubber are blended to improve the physical, thermal, and mechanical properties of the product as well as the modification of processing characteristics.[2–4] Acrylonitrile butadiene rubber (NBR)/polyvinylchloride (PVC) is a miscible physical mixture which is important in commercials. NBR has good oil resistance and low gas permeability. Its use in automotive applications is remarkable. But its ageing resistance is limited because of the butadiene unsaturated backbone.

PVC improves ozone and mechanical resistance in blend with NBR.[5,6] The elastomeric component, NBR, can act as a permanent plasticizer in PVC appliances, such as in electrical wires and cable coatings, wrapping films for the food industry, conveyor belts, and domestic appliances and so on. The presence of PVC facilitates improving the ozone of NBR, which has given the possibility of using this blend in industries such as gaskets, wires and cables, and in the manufacture of soles, artificial leather, etc.[2–4] Crosslinking of NBR/PVC is accomplished by using chemical agents, heat, electron beam radiation, or a combination of them.[7]

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THEORETICAL CONCEPT

Taguchi method, also called the robust design method, pioneered by Dr. Genichi Taguchi, has had a great influence in improving engineering productivity.

By consciously considering the noise factors (environmental variation during the use of product and manufacturing variations and component deterioration) and also the cost of failure in the field, Taguchi method brings the costumer satisfaction.

Taguchi method focuses on improving the fundamental functions of products or the process which facilitate flexible engineering designs and concurrent engineering. In fact, it is the most powerful method which is accessible to reducing the cost of product and interval development and improving the quality. Complex systems with a lot of variables can be investigated through Taguchi method; this method can explain these items successfully, and optimize them at the best conditions.[8] It also provides a simple and systematic approach to optimizing design for performance in quality and cost. Two major tools are used in robust design[8,9]:

1. Signal-to-noise ratio (SN ratio), which measures the quality by emphasizing on variation, and
2. Orthogonal arrays, which accommodate many design factors simultaneously.

Taguchi method provides an orthogonal standard array to accommodate this requirement. Depending on

TABLE I
Formulation of Master Batch

Material	phr[a]	Producer
NBR/PVC (60/40)	100	Bayer Co., Germany
Carbon black N330	40	Ahvaz Factory, Iran
Zinc oxide	3	Gostar Jam, Iran
Stearic acid	1	Minko, Malaysia

[a] Parts per hundred of rubber.

the number of factors, interactions, and levels needed, upon to the user, we can choose the standard method or column-merging or idle-column or the other same methods.

In practice, the target mean value may change during the process development.

Two major concepts are used in robust design

1. SN ratio, which measures quality by emphasizing variation.
2. Orthogonal array, which accommodates different design factors simultaneously.

When the improvement of quality and measurement is considered, using SN ratio is useful, because it improves the quality through variability reduction. The SN ratio characteristics can be divided into three categories when the characteristic is continuous[8]:

1. Nominal (the best characteristic), which can be calculated in this way:

$$SN = -10\ \log \frac{\sum_{i=1}^{r}(Y_1 - M)^2}{r} \quad (1)$$

2. Smaller (the better characteristics), calculated from:

$$SN = -10\ \log \frac{\sum_{i=1}^{r} Y_1^2}{r} \quad (2)$$

and

3. Larger (the better characteristics), calculated from:

TABLE II
Sulfur Curing System

Material	phr	Producer
Sulfur	1–2	RPC,[a] Iran
MBTS[b]	1–2	Bayer Co., Germany
CBS[c] (vulkacit cz)	0.3–0.7	Bayer Co., Germany

[a] Razi Petrochemical Co.
[b] Dibenzothiazyl disulfide.
[c] *N*-Cyclohexyl-2-benzothiazole sulfonamide.

TABLE III
Specification of NBR/PVC Blend

Grade	Ratio NBR/PVC	Mooney viscosity (ML 1 + 4, 100°C)	Density (g/cm^3)	Shape
866/20	60/40	20 ± 5	1.04	Sheet

$$SN = -10\ \log \frac{\sum_{i=1}^{r}\left(\frac{1}{Y_1^2}\right)}{r} \quad (3)$$

where M is the average of observed data, r is the number of observations, and Y is the observed data; with considering the above SN ratio transformation, for any type of the characteristic, the higher SN ratio shows the better result.

One of the methods for analyzing data in process of optimization is the use of ANOVA (analysis of variance).[8]

It is a quick and easy method to analyze results of parameter design. The ANOVA is performed, which is suitable for engineers and industrial practitioners because it can be done even with a little information. In data analysis, signal-to-noise (SN) ratio is used to allow the control of the response as well as to reducing the variability of response. The use of ANOVA is to calculate the statistical confidence associated with the conclusions drawn.

Since TS and hardness should be maximum, *larger*, the better type of SN ratio has been used. Also since %EB and %abrasion should be minimum, *smaller*, the better type of SN ratio has been used.

EXPERIMENTAL

Material

The fixed master batch formulation is used in this study. The producer and the amount of the fixed master batch are shown in Table I. The curing system, the amount of them, and the producer are shown in Table II. Table III shows specification of NBR/PVC blend.

TABLE IV
L9 Orthogonal Array of Taguchi Method

Experiment no.	A	B	C	E
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

[a] Empty column is denoted by e.

TABLE V
Mean Value for Physicomechanical Properties of NBR/PVC

Experiment no.	TS before aging (MPa)	TS after aging (MPa)	%EB before aging	%EB after aging	%Abrasion	Hardness before aging (shore A)	Hardness after aging (shore A)
1	19.1	20.2	328	207	7.9	64	82
2	19.8	20.6	317	206	7.4	68	84
3	20.1	21.1	285	192	7.2	70	86
4	19.6	20.6	278	185	7.0	73	86
5	19.8	20.8	270	190	5.6	75	87
6	20.2	21.3	277	186	6.1	77	87
7	20.4	21.5	259	178	5.8	79	88
8	20.4	21.5	258	174	6.3	79	88
9	20.5	21.8	221	170	5.7	81	89

Compounding and cure characteristics of NBR/PVC

Depending on considering four factors and three levels in this study, L9 orthogonal array of Taguchi method is selected. The L9 orthogonal array is shown in Table IV. Table IV consists of nine experiments corresponding to the nine rows and four columns, with three of them corresponding to sulfur, MBTS, and CBS and last one is considered as an empty column. In this case, for any pair of columns, the combinations of all factor levels occur and the frequency of occurring is the same. The point is that, using this design reduces 81 × (34) configurations into nine experimental evaluations.

Mixing is carried out on a laboratory-size two-roll mill based on ASTM D3182. Cure characteristic of mixes, for example, scorch time, t_2 and cure time, t_{90} is determined by using a Hiwa Rheometer model 100 based on ASTM D2084. Various rubber compounds were compression molded at 160°C under pressure (120 kg/cm^2) in an electrical heated press so as to prepare sheets of 2 ± 0.05 mm thickness. Dumbbell test pieces are cut from the sheets.

Measurements

Tensile strength (TS) and elongation-at-break(EB) is measured on dumbbell specimens based on ASTM D412.

Tensile tests are carried out based on a Hiwa 200 tensometer at 500 mm/min cross-head speed. The hardness test is done by using shore type A Durometer based on ASTM D2240. The other physicomechanical test is abrasion (DIN5356) done by using Hiwa700. All tests are done at room temperature (25°C). The aging property was studied at 100°C for 48 h (in an oven), and then samples were kept at an ambient temperature for at least 16 h before testing based on ASTM D573. For all tests three samples are used.

RESULTS AND DISCUSSION

After completing the experimenter's log given in Table V, It was found that percent error of experiment is ±1 in all of the tests. The next step in data analysis is to estimate the optimum level of each control factor (SN ratio) and to perform ANOVA, as described in last test. SN ratio analysis is shown in Tables VI and VII for each curing system factor. The ANOVA of mean analysis is shown in Table VIII. In the table, Df is the number of degrees of freedom, SS is the sum of squares of source, V is the variance of source, F is the variance ratio, and $\%P$ is the percent contribution of source.

Tables VI and VII show that sulfur is the most important factor in vulcanization for NBR/PVC.

Since a relatively low degree of vulcanization is obtained by using uncombined accelerator (MBTS

TABLE VI
Response Table for Mean Analysis

Factor	Level (phr)	TS before aging (MPa)	TS after aging (MPa)	%EB before aging	%EB after aging	%Abrasion	Hardness before aging (shore A)	Hardness after aging (shore A)
S	1	19.7	20.6	310	201	7.4	67	84
	1.5	19.9	20.9	275	186	6.3	75	87
	2	20.4	21.6	260	174	5.9	79	88
MBTS	1	19.7	20.7	288	190	6.9	72	85
	1.5	20.0	21.0	282	190	6.5	74	86
	2	20.3	21.4	261	183	6.4	76	87
CBS	0.3	19.7	21.0	288	189	6.8	73	85
	0.5	19.9	21.0	272	187	6.7	74	86
	0.7	20.1	21.1	271	187	6.2	75	87

TABLE VII
Response Table for SN Ratio Analysis

Factor	Level	TS before aging (MPa)	TS after aging (MPa)	%EB before aging	%EB after aging	%Abrasion	Hardness before aging (shore A)	Hardness after aging (shore A)
S	1	25.86	26.27	49.84	46.22	17.53	36.55	38.48
	1.5	25.98	26.40	48.79	45.44	15.95	37.50	38.76
	2	26.21	26.67	47.84	44.81	15.46	38.02	38.92
MBTS	1	25.87	26.32	49.25	45.52	16.82	37.05	38.61
	1.5	26.02	26.43	49.03	45.50	16.25	37.27	38.72
	2	26.14	26.61	48.38	45.24	16.11	37.57	38.82
CBS	0.3	25.95	26.42	49.22	45.46	16.68	37.19	38.62
	0.5	26.00	26.44	48.78	45.45	16.59	37.32	38.72
	0.7	26.07	26.48	48.68	45.43	15.92	37.43	38.81

and CBS), interaction will not be significant.[7] On the basis of pervious experiment, the incorporation of sulfur resulted in a moderate increase in the TS. Similar trends are recorded for EB. This is indicated by increase in hardness and the decrease in %abrasion with sulfur loading, presented in Tables VI and VII.[10] All of the physicomechanical properties improve with increasing the amount of sulfur, MBTS, or CBS alone. This is due to the increasing crosslinking with increasing the amount of curing factor in vulcanization.[5] Using more accelerators in curing system cause better use of sulfur in vulcanization. When the amount of sulfur is less, MBTS and CBS can not produce high crosslinking density.[5] For equal crosslinking density, MBTS must use more than CBS in fix amount of sulfur. From the results obtained, it was found that the best composition to get high TS, low %EB and high hardness is at 2 Phr sulfur. 2 Phr MBTS and 0.5 Phr CBS. For example, composition no. 9 of Table IV is best for sulfur-curing system. As sulfur and then MBTS have strong effect on TS, %EB and chemical resistance. From Table VIII, it is clear that %*P* for sulfur is salient and %*P* for error is less than 15 for all tests.

Confirmation experiment to validate the optimal conditions found is performed and it confirms the results.

TABLE VIII
Response Table for ANOVA Analysis

Test	Factor	SS	Df	*V*	*F*	%*P*
TS before aging (MPa)	S	0.98	2	0.9	12.52	55.3
	MBTS	0.54	2	0.27	38.57	30.2
	CBS	0.09	2	0.045	6.43	4.3
	error	0.15	20	0.007	–	10.2
TS after aging (MPa)	S	1.34	2	0.67	67	56.9
	MBTS	0.75	2	0.35	35	31.5
	CBS	0.02	2	0.01	1	0.8
	error	0.2	20	0.01	–	10.8
%EB before aging	S	1298	2	1975	81.12	63.6
	MBTS	98	2	598059	26.4	18.8
	CBS	68	2	267069	11.81	8.0
	error	324.2	20	22067	–	9.6
%EB after aging	S	3.78	2	649	4.03	80.8
	MBTS	0.96	2	49	3.02	3.7
	CBS	0.57	2	34	2.10	2.0
	error	0.39	20	16.21	–	13.5
%Abrasion	S	3.78	2	1.89	94.50	65.5
	MBTS	0.94	2	0.48	24.00	16.1
	CBS	0.57	2	0.28	28.50	9.3
	error	0.39	20	0.02	–	9.1
Hardness before aging (shore A)	S	224	2	112	205.88	85.1
	MBTS	24.18	2	12.09	22.22	8.8
	CBS	2.98	2	1.49	2.74	0.7
	error	10.88	20	0.54	–	5.4
Hardness before aging (shore A)	S	30.38	2	15.19	3797.5	77.9
	MBTS	6	2	3	750.0	15.4
	CBS	2.54	2	1.27	317.5	6.5
	error	0.08	20	0.004	–	0.2

CONCLUSIONS

1. Taguchi method is used for improving productivity during an NBR/PVC curing so that high-quality products can be produced at low cost. The orthogonal array technique is described for experimental design as it reduces the number of experiments required to investigate a set of parameters, and to minimize time and expenses while performing experiments we use a full-scale plant. Through experimental investigations, the effect of parameters are distinguished and also the effect of design parameters for performance, quality, and expenses.
2. Sulfur is more effective than MBTS and CBS in NBR/PVC curing.
3. Error is less than 15% in any test; therefore, the results are acceptable for this system.

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