Journal of New Developments in Chemistry

Open Access - Peer-Reviewed - ISSN 2377-2549

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Research Article

Statistical Taguchi Optimization for Preparation and Adhesion Evaluation of Epoxy Insulator to the Surface of Double Base Propellant Grain

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Published citation: Saeed Babaee, Zahra Monjezi, Milad Saadat Tagharoodi (2018) **Statistical Taguchi Optimization for Preparation and Adhesion Evaluation of Epoxy Insulator to the Surface of Double Base Propellant Grain**. *Journal of New Developments in Chemistry* - 2(1):41-50. https://doi.org/10.14302/issn.2377-2549.jndc-18-2479

Article DOI: 10.14302/issn.2377-2549.jndc-18-2479

Abstract

In this work, Taguchi design (orthogonal array, OA_9) was used for the adhesion investigation of an epoxy insulator to a double base (DB) propellant grain. In this manner three epoxy resins based on diglycidylether bisphenol A (DGEBA) and three polyamine curing agents with an active diluent based on DGEBA were used. Therefore, the effects of resin type, curing agent type with its amount and diluent quantity as main factors were investigated on the single lap shear strength (adhesion strength) and then the results were quantitatively evaluated by the analysis of variance (ANOVA). The data given of ANOVA predicted that the best adhesion strength of 15.584 ± 1.606 MPa was obtained for the optimum conditions of MANA POX-102 as epoxy resin, H-37 as curing agent with 57 phr, ERYSYS GE-30 as diluent with 5 phr. In comparison, practical result of adhesion strength obtained for the optimum conditions was 15.4 ± 0.2 MPa. Also the Pull-off test results on the surface of the DB propellant showed that the maximum adhesion strength (related to the optimal conditions) is 2.64 ± 0.2 MPa.

Keywords:
Epoxy; Insulator; Propellant; Taguchi; Adhesion Strength; ANOVA

Article Information	
Academic Editor	Zhe-Sheng Chenz, Professor, Department of Pharmaceutical Sciences, College of Pharmacy and Allied Health Professions, St. John's University, United states
Received Date	11 November 2018
Accepted Date	7 December 2018
Published Date	8 December 2018
Online	8 December 2018
Publication	Volume 2; Issue 1; Pages 41–50
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Introduction

Insulation and insulators for rocket motors containing double base (DB) propellants were developed in recent decades [1]. One way to provide thermal protection of a DB propellant motor is applying a polymeric material between the inner surface of the case and the cast propellant. One of the important characteristics of the insulating material is proper mechanical properties such as tensile strength, elongation and adhesion. This prevents deformation of the DB grain along with separation of the insulator from the grain, liner and motor during various phases of manufacturing, storage and handling [2].

The matrix of DB propellant insulators usually consists of organic materials such as epoxy resins, plastics and phenolic compounds. Epoxy resins provide excellent strength and adhesion to a wide range of materials such as solid propellants. This advantage makes them indispensable in adhesive applications where high strength is a prerequisite [2], [3].

Epoxy resins belong to a family of molecules or oligomers having epoxide groups (oxiranes). The greatest commercial epoxy resin is formed by the reaction of bisphenol A and epichlorohydrin. Therefore, this resin is well known as the diglycidyl ether bisphenol A or DGEBA [4]. Some advantages of epoxy resins are high chemical resistance, flexibility, processability, curing at ambient temperature and compatibility with DB propellants. Also, their mechanical properties such as tensile strength and adhesion strength are good [5], [6], [7]. The three elements of an epoxy resin are the base resin, the hardener and the diluent. The base resin determines many of the typical properties of the final polymer, such as operational temperature, while the hardener defines the curing properties such as reaction time. Diluents are added to modify the specific physical and mechanical properties [8].

A group of chemical compounds with active hydrogen that reacts with these epoxides is aliphatic polyamines which are known as the curing agent (or hardener). Hardeners modify the properties of epoxy polymers and provide appropriate flexibility, tensile strength, toughness for them. Traditional aliphatic polyamines such as diethylenetriamine (DETA), triethylenetetramine (TETA) and also aliphatic amines have been extensively used as hardeners for the curing of epoxy resins in ambient temperatures [9].

DGEBA epoxy resins have high viscosities that lead to numerous difficulties on their processes and the applications. Therefore, it is necessary to use a diluent. There are many various diluents, depending on their reactive or non-reactive nature. When these epoxy resins mix with a reactive diluent, due to the diluent epoxy groups, some important properties of the original system (such as tensile strength, glass transition temperature, and lifetime) are changed. The extent to which properties are altered is a function of both diluent type and its concentration [10], [11].

One of the most widespread approaches for optimizing the design factors is the (Genichi) Taguchi method [12]. It can identify and organize system interactions within experimental data for an analysis leading to an optimal design [13], [14]. For example, Chien and Tsai employed the Taguchi method to design a two-level screening experiment to determine the essential factors for an environmental stress screening usage [15]. Moreover, it is proven that this method can be capable of solving a variety of problems including continuous, discrete and qualitative design variables [16], [17].

Taguchi method classifies the factors as controllable factors and noise factors. Noise factors are variables that influence the response of a process but can not be controlled economically. They are not maintained at particular levels during the process period for the expected performance in optimum conditions with the least variations [18]. These factors usually are the first sources of variation and due to their control difficulties can not be considered [19], [20].

In this research, after preparation of an epoxy insulator at ambient temperature the adhesion of it on a DB propellant surface was investigated by the Taguchi method. It is used for design of the main effects (average effects of factors and interactions) of four control factors of the forecasting model. These four factors were epoxy resin type, curing agent type, curing agent amount and diluent quantity. To match the objectives of this study, a design affected minimally by noise is considered.

Materials and Methods

Chemicals

Epoxy resins based on bisphenol A: Epon 828 (Epoxy Equivalent Weight, EEW: 185-192 $g.eq^{-1}$, viscosity: 11000-15000 cP), MANA POX 95 (EEW: 190 $g.eq^{-1}$, viscosity: 10000-14000 cP) and MANA POX 102 modified with a reactive diluent based on bisphenol A (EEW: 205 $g.eq^{-1}$, viscosity: 500-900 cP). Polyamine curing agents H-30 (Active Hydrogen Equivalent Weight, AHEW: 106 $g.eq^{-1}$, viscosity: 2000-4000 cP), H-35 (AHEW: 110 $g.eq^{-1}$, viscosity: 2000-4000 cP) and H-37 (AHEW: 110 $g.eq^{-1}$, viscosity: 1500-3000 cP) were obtained from Mana polymer Co. (Iran). Reactive diluent by name ERYSYS GE-30, based on bisphenol A with three functional groups (EEW: 135-150 $g.eq^{-1}$, viscosity: 100-200 cP) was prepared from C.V.C Co. (USA).

Equipment

Vacuum oven (J.P. Selecta, model: TV-4001490). Universal testing machine (UTM) or tensile-compressive apparatus (Hiva, model: HIWA-2126, under the standard ASTM-D1002) with the accuracy of measurement 0.1 MPa (25˚C) was used for measurements of single lap shear strength of epoxy insulators. Pull-off device (Defelsko, model: Posi-ATA, under the standard ASTM-D4541) was used to evaluate the adhesion strength of epoxy insulator to the surface of a double base propellant. All data handling of Taguchi method was performed by using Qualitech-4 software.

Procedure

10 g of the epoxy resin was weighed and after adding of a certain amount of diluent, the resulting mixture was stirred for 5 minutes. Then, the mixture was placed for 25 minutes in a vacuum oven. Afterward certain amount of the curing agent was added to the above mixture and was stirred for 5 minutes again. The prepared blend was put between two pieces of sandblasted steel (10×2.5 cm^2) for complete curing at ambient temperature (25˚C). After 7 days the cured samples (insulators) were subjected to single lap shear testing by using a constant crosshead speed of 2 mm/min and the gauge length of 45 mm. Also, tensile strength of adhesive (adhesion strength) of the optimum insulator (a blend prepared under the optimal conditions) was determined by Pull-off test after complete adhesion of it (7 days) to the surface of a double base propellant.

Results and Discussion

One of the most important tests for determination of the mechanical properties and joint behavior of the polymeric adhesives is a single-lap-shear test. Single lap shear testing is a method for evaluation of adhesion by pulling bonded layers along the plane of adhesion. In this test, the apparent shear strengths

of adhesives or the ability of an adhesive to tolerate shear forces in the joint surfaces under specific conditions of preparation and test characterized [21], [22].

Optimization is a necessary step for stabilizing a multistage process and in this manner common procedures of optimization are sequential and simultaneous methods [23], [24]. In sequential methods (one each time optimization), the response surface is consecutively tracked until an optimum condition is obtained. These methods are suitable for few response surface designs and there are some difficulties with them such as slow convergence and too many experiments for a complex response surface with high dimensionality [25].

In simultaneous optimization methods such as mixture designs [26], [27] and factorial designs [28], [29], none of these problems exists. In these methods after an initial experiments plan, the experimental results are collected and afterward the optimum conditions would be determined with constructing a response surface or by an extrapolated graph. Mixture designs are perfect for experiments whose response is related to the proportions of ingredients in a mixture rather than their values, while factorial designs are used with the other variables. A clear defect of the factorial designs is that when the number of variables increases, the number of the required experimental trials increases geometrically. Therefore, in this case, the implementation of these trials is not feasible and fast. Of course this problem can be decreased by using fractional factorial experiments, such as Plackett-Burman schemes or orthogonal array designs (OAD) [26], [30], [31], [32], [33], [34]. In comparison to previous two-level designs, OAD has three-level designs and more precise information could result.

In designing experiments, Taguchi applied OAD, which represent the least fractional factorials and are used for the most experiment designs. The number of possible designs, N, in a full factorial design is as follows:

$$N = L^m \quad \textbf{(1)}$$

Where L is the number of levels for each factor and m = number of factors.

Thus, if the qualities of a given product depend on four factors (A, B, C, and D) and each factor is to be tested at three levels, a full factorial experiment would require 3^4 or 81 runs but may not provide appreciably more useful information. This array identified by the symbol L_9 (OA_9 or L-9) and is used to design experiments involving up to four three-level factors [18], [25].

In this research, four factors (epoxy resin type, curing agent type, curing agent amount and diluent quantity) at three levels were considered and Taguchi method was applied for the study of adhesion of an epoxy insulator to the DB propellant surface. The design of 9 experiments based on OA_9 (3^4) and also practical results of single lap shear strength were summarized in Table 1.

As shown in Table 1, the maximum and minimum single lap shear strength were resulted in experiments no. 9 and no. 4, respectively. It is emphasized that in rocket motor insulator, the highest adhesion strength is desirable. Figure 1 shows the average values for three levels of a factor and reveals how the single lap shear strength of epoxy insulator will change when the levels of the factor are changed.

Effect of the Resin Type

The effect of resin types (Epon 828, MANA POX 95 and MANA POX 102) on single lap shear strength of the epoxy insulator at three different levels was investigated. The analysis of Taguchi software on the preliminary data is shown in Figure 1(a). The results revealed that the effect of MANA POX 102 was relatively more than the two others, which can be due to its lower viscosity and higher EEW. Reduction of the viscosity will increase the permeability of the uncured resin to the pores of the sandblasted steel surfaces. Therefore, mechanical interactions of this resin with the steel surface were increased and so the adhesion strength would be added [35], [36]. Also the presence of reactive diluent (based on bisphenol A) in MANA POX 102 can also help to increase the resin crosslinking density and also concentration of hydroxyl groups. The hydroxyl groups of adhesive molecules attach to the specific sites on the steel by the intermolecular forces [4], [10]. Therefore, the adhesion strength of the cured insulator can be more elevated. Furthermore, higher EEW will increase the number of aromatic groups of bisphenol A in the chain length of the MANA POX 102 and therefore the strength (cohesive strength) of crosslinking networks in epoxy insulator was increased.

Table 1. Factors- levels arrangement of the experiments (OA_9 matrix) and practical results of the shear strength of adhesive (Adhesion strength).

Trial No.	Resin Type	Curing Agent type	Curing Agent Amount (phr[1])	Diluent Quantity (phr)	Adhesion strength[2] (MPa)
1	Epon 828	H-30	52	-	8.1 ± 0.5
2	Epon 828	H-35	57	5	12.3 ± 0.7
3	Epon 828	H-37	62	10	13.3 ± 0.2
4	MANA POX 95	H-35	62	-	7.5 ± 1.0
5	MANA POX 95	H-37	52	5	12.1 ± 0.0
6	MANA POX 95	H-30	57	10	9.7 ± 0.4
7	MANA POX 102	H-37	57	-	12.9 ± 0.7
8	MANA POX 102	H-30	62	5	8.8 ± 0.2
9	MANA POX 102	H-35	52	10	13.4 ± 0.1

[1] Per hundred resin.
[2] Average of two replicates run (n=2).

Effect of Curing Agent Type

All three curing agents (H-30, H-35 and H-37) were based on polyamine compounds with differences in their AHEWs or viscosities. Figure 1(b) shows that the use of curing agent H-37 resulted in the highest single lap shear strength for the epoxy insulator system. It can be explained that the AHEW of H-37 is equal to H-35 and higher than H-30. Also its viscosity is lower than H-30 and H-35. By increasing the

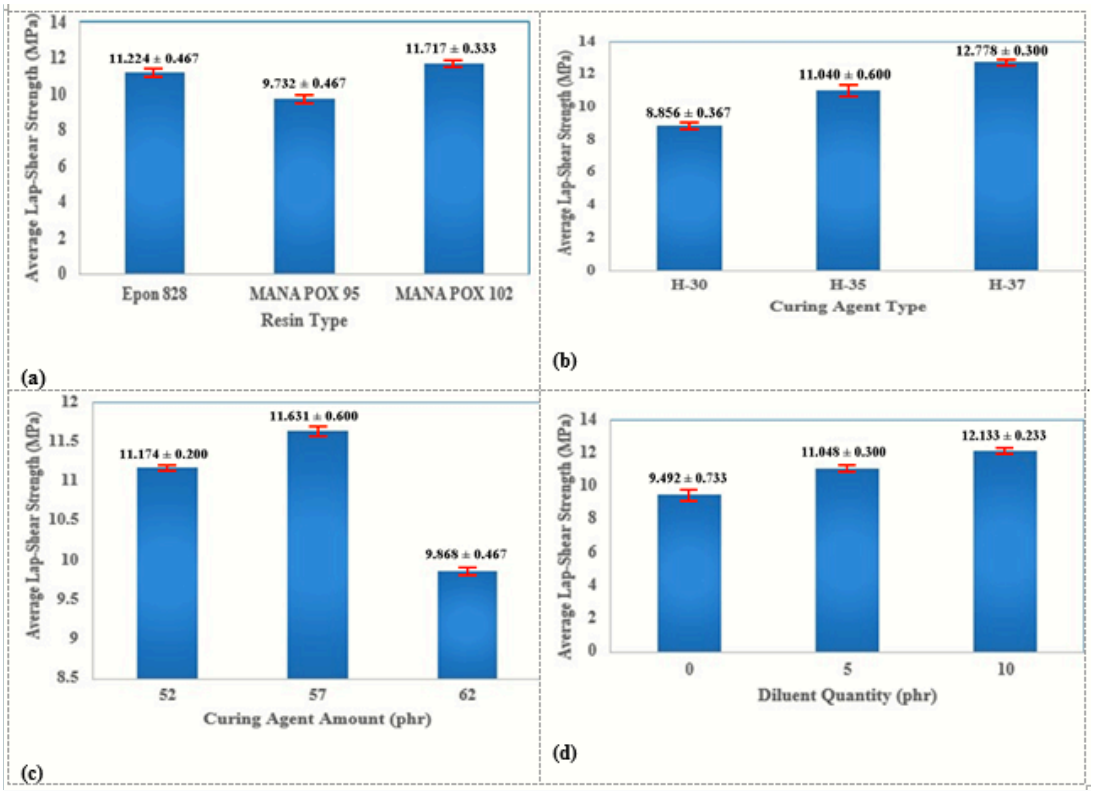

Figure 1. Effects of level variation for each parameter on the average shear strength of adhesive (n=2).
a: Resin type; b: curing agent type; c: Curing agent amount; and d: Diluent quantity.

AHEW of the curing agent, insulator network will be harder and its adhesion strength will be enhanced [4]. Furthermore, use of H-37 with lower viscosity can reduce the uncured blend viscosity more and therefore, in accordance with the mechanical interlocking theory, the adhesion strength was improved [4], [10].

Effect of the Curing Amount

Amount of curing agent is also another important factor for the single lap shear strength of the cured polymers. This test according to Figure 1(c) was performed at three levels of 52, 57 and 62 phr [4]. As it is seen addition of the curing agent to the epoxy resin up to 57 phr would improve the cross linking density of resin-hardener network and therefore the insulator strength can be elevated. At higher amounts, due to improper stoichiometry of resin-hardener and forming of the network defects, the insulator strength would be reduced.

Effect of Diluent Quantity

This factor was considered at three levels of 0, 5 and 10 phr of the diluent (ERYSYS GE-30). According to Figure 1(d), the best insulator strength was obtained by using 5 phr of the diluent. This diluent contains three functional groups (bisphenol A) and 100-200 cP viscosity. By using it the viscosity of the uncured blend was decreased and therefore, according to the mechanical interlocking theory the adhesion strength on the steel surface was increased. Also due to its three functional groups, the crosslinking density and concentration of hydroxyl groups were increased. Thus with greater cohesive strength in resin-hardener network and adhesion strength at the interface of the sandblasted steels, the single lap shear strength of epoxy insulator was enhanced. In quantities more than 5 phr, with regard to specific properties of the diluent, the network behavior is similar to the diluent behavior and so properties of the final insulator would worsen.

Analysis of variance (ANOVA) was applied to a survey statistical or quantitative evaluation of effects of each factor on single lap shear strength for the insulator-steel system and the results are presented in Table 2. According to data given in Table 2 at 90 % confidence level, it is seen that the curing agent type with 46.065 percent has the highest influence on the single lap shear strength of the epoxy insulator. Influence of other parameters is in order to diluent quantity (20.157 %), resin type (11.604 %) and curing agent amount (8.744 %). By referring to the F-test data and the lowest number (6.534), the degrees of freedom (2 and 9) and related F- test Table [18] the critical F- value at 90 % confidence level is resulted 3.0065. Therefore, no factor became pooled. With respect to the results of Table 2, the main factors show that the optimum conditions proposed are sequentially as: curing agent type = H-37, diluent quantity = 5 phr, resin type = MANA POX 102, curing agent amount = 57 phr.

As a general rule, the optimum performance (here, for preparation of the epoxy insulator with the highest adhesion strength) could be calculated by Eq. (2):

Table 2. ANOVA results for adhesion strength of insulator-steel system by an OA_9 (3^4) matrix with single lap shear strength as the response (MPa).

Factor	Code	DOF	S	Variance	F-test	S′	P (%)
Resin type	R	2	12.826	6.413	8.344	11.289	11.604
curing agent type	H	2	46.352	23.176	30.157	44.815	46.065
Curing agent amount	C	2	10.044	5.022	6.534	8.507	8.744
Diluent quantity	D	2	21.147	10.573	13.758	19.61	20.157
Error		9	6.915	0.768	-	-	13.43

DOF: degrees of freedom, S: sum of squares, S′: Pure sum, P: Participation percentage
Note: The critical F value was at 90 % confidence level.

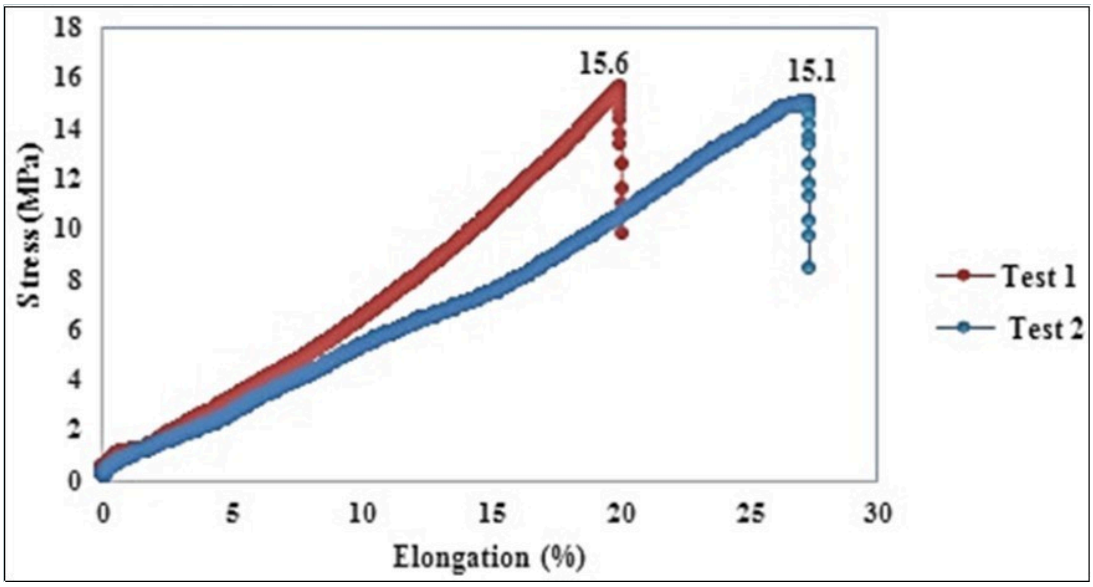

Figure 2. Stress-Elongation curves (n=2) for verification of the optimal conditions.

$$Y_{opt} = T/N + (R_3 - T/N) + (H_3 - T/N) + (C_2 - T/N) + (D_3 - T/N) \quad \textbf{(2)}$$

Where Y_{opt} (single lap shear strength at the optimum conditions) is equal to the T/N (ratio of the grand total of all results to the total number of all experiments) plus the contributions of R_3 (resin type at level 3 (MANA POX 102)), H_3 (curing agent type at level 3 (H-37)), C_2 (curing agent amount at level 2 (57 phr)) and D_3 (diluent quantity at level 3 (10 phr)). The procedure for computing the confidence interval (CI) of the optimum performance is explained following by Eq. (3).

$$CI = \pm\sqrt{((F_\alpha (f_1, f_2) V_e)/n_e)} \quad \textbf{(3)}$$

Where F_α (f_1, f_2) is the critical value for F at degrees of freedom (DOF) f_1 and f_2 at the significance confidence level (In this work α= 90%). f_1 is DOF of the mean (which always equals 1), f_2= DOF of the error term, V_e is the variance of error term (from ANOVA), n_e is defined as effective number of replications, and expressed by n_e = number of trials/(f_1 + DOF of all factors applied in the estimation of optimum results) [14]. Statistical calculations for predicting the result and CI at optimum conditions revealed that the single lap shear strength of the epoxy insulator will be 15.584 ± 1.606 MPa.

Due to validation of the optimal conditions obtained by Taguchi method, a single lap shear testing was applied for an insulator mixture of the resulted ingredients. According to Figure 2 the average adhesion strength of two replicate runs is 15.4 ± 0.2 MPa. This result is in the range of formerly confidence interval and so it is acceptable for this work.

Finally, in order to examine the applicability of the proposed method, adhesion strength of the blends of trials no. 3, 7 and 9 (due to their highest strength of adhesive) along with a blend prepared under the optimal conditions were determined. This work was performed by using pull-off test with a 500 µm thickness of the cured blends on the surface of the DB propellant (with three replicate runs) and the results are given in Table 3.

The data given in Table 3 shows that the highest adhesion strength is obtained for the blend prepared under optimum conditions. So the Taguchi method was suitable and could be successfully applied to predict the optimal construction of the epoxy insulator with the best adhesion to the DB propellant.

Table 3. Comparison of Pull-off test results of the selected blends for a DB propellant.

Blend- Trial number	Adhesion strength [1] (MPa)
3	1.82 ± 0.4
7	1.91 ± 0.3
9	2.07 ± 0.3
Optimum conditions	2.64 ± 0.2

[1] Average of three replicate runs (n=3).

Conclusions

In this study, an epoxy insulator with the best adhesion strength was prepared for a DB propellant grain at ambient temperature. Taguchi robust design method using Qualitech-4 software was applied to optimize experimental conditions of the preparation process. In this manner the resin type, curing agent type with its amount and diluent quantity were chosen as the main parameters. Under the optimum conditions, the participation of each parameter on the yield of the process is: curing agent type (H-37, 46.065%), diluent quantity (ERYSYS GE-30, 20.157%), resin type (MANA POX 102, 11.604 %) and curing agent amount (8.744 %). All of the mentioned parameters were important and any of them were not pooled. According to Taguchi method and mechanical test results, the optimal composition of epoxy insulator was obtained as MANA POX 102, 57 phr H-37 and 5 phr ERYSYS GE-30. The results obtained from predicted data were analyzed by the Taguchi design (adhesion strength = 15.584 ± 1.606 MPa) and those of practical samples (average adhesion strength = 15.4 ± 0.2 MPa) were in satisfactory agreement. Finally, the results of pull-off tests revealed that Taguchi method could be successfully applied to predict the optimal insulator with the highest adhesion to the DB propellant.

Competing Interests

The authors have declared that no competing interests exist.

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