DETERMINATION OF THE RHEOLOGICAL PROPERTIES OF HYDROXYL TERMINATED POLYBUTADIENE (HTPB) MIXTURES WITH ENERGETIC MATERIALS AND PRESENTING EMPRICIAL MODELS

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Abstract Rheological Properties Such as Viscosity (η), Shear Stress (τ), and Torque (M) of the mixtures of (HTPB) with Octagon (HMX), Hexogen (RDX), and 2,6 Diamino-4-Phenyl-1, 3,5 Triazine (DAPTA) mixtures were measured. The experimental design was arranged for three factors at two levels (High and low levels). Temperature of the mixture (°C), Speed of the stirrer (rpm), Mixing Time (minutes) have been known to be effective in the quantity of Rheological properties. In this work, by using the appropriate equations the empirical model was presented for each property and mixture and the main effects on the responses were detected.

Keywords: Rheological Properties, HTPB/Energetic Materials Mixtures, Empirical Models, and Experimental Design.

چکیده *خواص رئولوژیکی* , نظیر ویسکوزیته, تنش برشی , و گشتاور مخلوط های پلی بوتادین خاتمه یافته با عامل هیدروکسیل با اکتاژن ,هگزوژن , و 2و6 دی آمینو – 4-فنیل – 1و3و5 تری آزین اندازه کیری شده است. طراحی آزمایش برای سه تا عامل در دو سطح (بالا و پایین) ترتیب یافته است.دمای مخلوط کردن, سرعت بهمزن , و زمان مخلوط کردن در میزان خواص *رئولوژیکی موثر شناخته شده اند.* در این فعالیت با استفاده از معادلات مناسب معادله تجربی برای هر خاصیت و مخلوط ارائه شده و تأثیرات اصلی در معادلات مشخص شده اند.

1. INTRODUCTION

Composite propellants are essentially composed of a binder, metallic fuel, and an oxidizer. Binder is used for mainting intimately and homogenousely the other ingredients of the propellants in the mixture. The binder must be chemically and physically stable during storage and operating conditions, and must be capable of bonding to insulating materials and the metallic parts of rocket. Among conventional binders for composite propellants Hydroxyl Terminated Polybutadiene (HTPB), has been regarded as the most suitable binder [1-17]. However, It is an inert binder. For increasing the energy, it is necessary to mix HTPB with an energetic material [18-25].

Preparing and using the propellant slurry is a critical step and an optimization must be done between the pot life of the slurry and casting time. So considering the rheological properties is very important.

The Rheological behavior of the propellant slurry has been studied in detail by Klager etal [26], Osgood [27], Killian [28], Rumbel [29], and Rajan etal [30] in order to understand its processiblity, pot life, and characteristics to obtain a free propellant grain.

Rheological behavior of Hydroxyl Terminated Polybutadiene (HTPB) based composite propellant with respect to solid loading, oxidizer particle size distribution and aluminum content was reported by Muthiah etal [31]. According to theire report by increasing the solid loading viscosity will be increased and by decreasing the solid particle size will be increased. In addition to these parameters the effects of mixing temperature and time and speed of the stirrer are important from the point of view of processability and final properties of propellants.

In all of the previous works only one parameter was considered as an independent variable. In this work the variations of rheological properties of HTPB mixtures with energetic materials (with respect to simultaneous variation of mixing temperature and time and speed of the stirrer) have been determined. Furthermore, an empirical model was presented for each case. So the present work thought to be as a complementary step to the previous works which have already been done by other researches in this field (Muthiah etal [32-33]) in the range of parameters involved.

2. EXPERIMENTAL PROCEDURES

The experimental design was arranged for three factors at two levels (High and low levels). The following parameters have been known to be effective in the quantity of rheological properties:

A: temperature of the mixture (°C)

B: Speed of the stirrer (rpm).

C: Mixing Time (minutes).

To simply the computations, factor levels are coded as +1 for the high level and –1 for the low-level .The coded parameters for A, B, and C are A^*, B^*, C^*respectively. The relationship between a factor's coded level, X_f^* and its actual value X_f, is given By [26]:

$$X_f = C_f + d_f X_f^* \quad (1)$$

Where C_f is the factor's average level, and d_f is the absolute difference between high and low values. Equation (1) is used to transform coded results back to their actual values.

In Tables 1-3 the uncoded factor levels, coded factor levels, and responses for a 2^3 factorial design were listed for the mixtures of HTPB with HMX, RDX, and DAPTA respectively.

For example, the factor A has coded levels of +1 and –1 with an average factor level of 45, and d_A equal to 5, the actual factor levels are:

$$H_A = 45 + (1)\cdot(5) = 50$$
$$L_A = 45 + (-1)\cdot(5) = 40$$

In all mixtures the weight of the HTPB was 50g and the weight of the energetic material was 5g. Duplicate measurement was performed for each case.

The two levels three factorial designs and their corresponding responses at each case are given in the tables 1,2,and 3. The empirical model is described by the following equation [32]:

$$R = C_0 + C_a A + C_b B + C_c C + C_{ab} AB + C_{ac} AC + C_{bc} BC + C_{abc} ABC \quad (2)$$

WhereC_0, C_a, C_b, C_c, C_{ab}, C_{ac}, C_{bc}, C_{abc} were determined by the following equations:

$$C_0 = 1/n \sum R_i \quad (3)$$

$$C_a = 1/n \sum A_i^* R_i \quad (4)$$

$$C_b = 1/n \sum B_i^* R_i \quad (5)$$

$$C_c = 1/n \sum C_i^* R_i \quad (6)$$

$$C_{ab} = 1/n \sum A_i^* B_i^* R_i \quad (7)$$

$$C_{bc} = 1/n \sum B_i^* C_i^* R_i \quad (8)$$

$$C_{ac} = 1/n \sum A_i^* C_i^* R_i \quad (9)$$

$$C_{abc} = 1/n\sum A^{*}B^{*}C^{*}R_i \qquad (10)$$

3. SPECFICATION OF MATERIALS AND INSTRUMENTS

The following materials were used for preparing the mixtures:

1. HTPB having the microstructure:
Trans: 62%
Vinyl: 20%
Cis: 18%

It was prepared and characterized in our laboratory [1-5]. Polymerization was done by free radical method using hydrogen peroxide as an initiator. The yield of polymerization reaction (with respect to monomer) was 70%.

2. Granular RDX and HMX. These materials were prepared and characterized in our laboratory according the method presented in reference [18]. Analysis of the particles was:

Size	percent
50-100 micron	20
100-200 micron	60
200-250 micron	20

3. DAPTA from Merck. This material was used in the the purchased form.

The following Apparatuses have been used for preparing the mixtures and measuring the Rheological properties.

1-“RW-20” mixre from “Junkle and Kunkel Co.”

2 - “Rhomat 108” from Contraves . That is a coaxial Rheometer in which the inner cylinder is rotated at a prespecified speed and the inner

Table 1. Uncoded and coded parameters and the responses for the HTPB/HMX system

Run	A (°C)	B (rpm)	C (minutes)	A^*	B*	C^*	$R=R_\eta$ Viscosity (Pa.s)	$R=R_\tau$ Shear Stress (Pa)	$R=R_M$ Torque (mNm)
1	50	100	25	+1	+1	+1	5.68	101	5.22
2	50	100	15	+1	+1	-1	5.86	104	5.46
3	50	80	25	+1	-1	+1	5.75	102	5.44
4	50	80	15	+1	-1	-1	5.45	105.2	5.38
5	40	100	25	-1	+1	+1	6.96	122.4	6.38
6	40	100	15	-1	+1	-1	7.45	130	6.77
7	40	80	25	-1	-1	+1	7.40	128.4	6.71
8	40	80	15	-1	-1	-1	7.54	133.2	6.83

cylinder is stationary. A four blade metal stirrer was used for mixing.

Run	A (°C)	B (rpm)	C (minutes)	A^*	B*	C^*	$R=R_\eta$ Viscosity (Pa.s)	$R=R_\tau$ Shear Stress (Pa)	$R=R_M$ Torque (mNm)
1	50	100	25	+1	+1	+1	4.73	84.5	4.35
2	50	100	15	+1	+1	-1	4.82	87	4.55
3	50	80	25	+1	-1	+1	4.79	85	4.45
4	50	80	15	+1	-1	-1	4.99	87.7	4.48
5	40	100	25	-1	+1	+1	5.88	102	5.33
6	40	100	15	-1	+1	-1	6.21	109	5.64
7	40	80	25	-1	-1	+1	6.17	107	5.59
8	40	80	15	-1	-1	-1	6.28	111	5.69

Table 2. Uncoded and coded parameters and the responses for the HTPB/RDX system

4. RESULTS AND DISCUSSIONS

Using equations (3-10), parameters can be estimated .For example, for the viscosity of the HTPB/HMX mixture we have:

$$C_0 = 1/8(5.68 + 5.86 + 5.75 + 5.45 + 6.96 + 7.45 + 7.40 + 7.54)$$

$$C_0 = 6.510$$

$$C_a = 1/8(5.68 + 5.86 + 5.75 + 5.45 - 6.96 - 7.45 - 7.40 - 7.54)$$

$$C_a = -0.826$$

$$C_b = 1/8(5.68 + 5.86 - 5.75 - 5.45 + 6.96 - 7.45 + 7.40 - 7.54)$$

$$C_b = -0.036$$

$$C_c = 1/8(5.68 - 5.86 + 5.75 - 5.45 + 6.96 - 7.45 + 7.40 - 7.54)$$

$$C_c = -0.064$$

$$C_{ab} = 1/8(5.68 + 5.86 - 5.75 - 5.45 - 6.96 - 7.45 + 7.40 + 7.54)$$

$$C_{ab} = 0.109$$

$$C_{ac} = 1/8(5.68 - 5.86 + 5.75 - 5.45 - 6.96 + 7.45 - 7.40 + 7.54)$$

$$C_{ac} = 0.093$$

$$C_{bc} = 1/8(5.68 - 5.86 - 5.75 + 5.45 + 6.96 - 7.45 - 7.40 + 7.54)$$

$$C_{bc} = -0.091$$

$$C_{abc} = 1/8(5.68 - 5.86 - 5.75 + 5.45 - 6.96 + 7.45 + 7.40 - 7.54)$$

$$C_{abc} = -0.016$$

So the coded empirical model therefore is:

$$R_\eta = 6.510 - 0.826 \cdot A^* - 0.036 \cdot B^* - 0.064 \cdot C^* + 0.109 \cdot A^* \cdot B^* + 0.093 \cdot A^* \cdot C^* - 0.091 \cdot B^* \cdot C^* - 0.016 \cdot A^* \cdot B^* \cdot C^*$$

To check the result we substitute the coded factor levels for the first run into the coded empirical Model, giving:

$$R_\eta = 5.68$$

Which agrees with the measured response.
From the equation (1):

$$X_a = A = 45 + 5 \cdot A^* \qquad (11)$$

$X_b = B = 90 + 10 \cdot B^*$ (12) $X_c = C = 20 + 5 \cdot C^*$ (13)

Table 3. Uncoded and coded parameters and the responses for the HTPB/DAPTA system

Run	A (°C)	B (rpm)	C (minutes)	A^*	B*	C^*	R_η (Pa.s)	R_τ (Pa)	R_M (mNm)
1	50	100	25	+1	+1	+1	4.35	75.4	3.95
2	50	100	15	+1	+1	-1	4.52	79.4	4.09
3	50	80	25	+1	-1	+1	4.50	79	4.05
4	50	80	15	+1	-1	-1	4.54	79.9	4.11
5	40	100	25	-1	+1	+1	5.52	99	4.92
6	40	100	15	-1	+1	-1	5.78	105	5.05
7	40	80	25	-1	-1	+1	5.66	102	4.88
8	40	80	15	-1	-1	-1	5.91	109	5.12

From the equations (11-13), the coded factors are:

$$A^* = 0.2 \cdot A - 9 \quad (14)$$

$$B^* = 0.1 \cdot B - 9 \quad (15)$$

$$C^* = 0.2 \cdot C - 4 \quad (16)$$

Substituting the equations (14-16) into the coded empirical Model and simplifying gives the following result for the uncoded empirical Model:

$$R_\eta = 28.609 - 0.5510 \cdot A - 0.1229 \cdot B - 0.2756 \cdot C + 0.00346 \cdot A \cdot B + 0.001060 \cdot B \cdot C + 0.00948 \cdot A \cdot C - 0.000064 \cdot A \cdot B \cdot C$$

To check the results, we substitute the uncoded factor levels for the first run into the uncoded empirical Model giving:

$$R_\eta = 5.68$$

Which agrees with the measured response.

Using the equations (3-10) and (14-16) the coded and uncoded empirical Model for the mixtures of the HTPB with HMX, RDX, and DAPTA was derived and the results were given in the tables 4,5,and 6.

By substituting the coded factor levels in the coded empirical equations and the uncoded factor levels in the uncoded empirical equations the results for all runs were checked and they well agreed with the measured responses. This indicates that the selected empirical model is very effective and can be used for modeling the experimental results.

5. CONCLUSION

From the Results presented in tables 1-3; it can be concluded that by raising the temperature, the speed of stirrer and mixing time; the viscosity, shear stress, and Torque are reduced. However, by comparing the results, it will be found that the temperature has the most effect on the responses. This is a reasonable conclusion, because by raising the temperature the mobility of the polymer molecule increase and so the viscosity, shear stress, and Torque decreases. By raising the speed of stirrer and time of mixing, the rate of shear increases and therefor the viscosity, shear stress, and Torque reduced. However, the temperature influences are more than the other factors and so it has the most effect on the responses. The results (viscosity) obtained by this models are relatively agreed with the results obtained by Muthiah etal [32-33] in the range of parameters involved.

Referring to the tables 4-6 it can be seen that the responses were exactly fitted in the empirical models. So because of the simplicity of the method employed in this work (from point of view of designing the experiments[34] and presenting empirical models) the results of this research activity can be used as a preliminary guide to experimental designof the processing parameters of the composite propellants.

Table 4. Coded and uncoded empirical equations for HTPB/HMX system.

Coded	**Uncoded**
$R_\eta = 6.510 - 0.826 \cdot A^* - 0.036 \cdot B^*$ $- 0.064 \cdot C^* + 0.109 \cdot A^* \cdot B^* + 0.093 \cdot A^* \cdot C^*$ $- 0.091 \cdot B^* \cdot C^* - 0.016 \cdot A^* \cdot B^* \cdot C^*$	$R_\eta = 28.609 - 0.5510 \cdot A - 0.1229 \cdot B$ $- 0.2756 \cdot C + 0.00346 \cdot A \cdot B + 0.001060 \cdot B \cdot C$ $+ 0.00948 \cdot A \cdot C - 0.000064 \cdot A \cdot B \cdot C$
$R_\tau = 115.775 - 12.725 \cdot A^* - 1.625 \cdot B^*$ $- 2.325 \cdot C^* + 0.875 \cdot A^* \cdot B^* + 0.900 \cdot A^* \cdot C^*$ $- 0.325 \cdot B^* \cdot C^* - 0.375 \cdot A^* \cdot B^* \cdot C^*$	$R_\tau = 224.3 - 2.14 \cdot A + 0.53 \cdot B$ $+ 4.575 \cdot C - 0.0125 \cdot A \cdot B - 0.074 \cdot B \cdot C$ $- 0.099 \cdot A \cdot C + 0.0015 \cdot A \cdot B \cdot C$
$R_M = 6.024 - 0.649 \cdot A^* - 0.066 \cdot B^*$ $- 0.086 \cdot C^* + 0.31 \cdot A^* \cdot B^* + 0.041 \cdot A^* \cdot C^*$ $- 0.071 \cdot B^* \cdot C^* - 0.004 \cdot A^* \cdot B^* \cdot C^*$	$R_M = 15.53 - 0.2472 \cdot A - 0.0205 \cdot B$ $- 0.028 \cdot C + 0.00094 \cdot A \cdot B - 0.0007 \cdot B \cdot C$ $+ 0.00308 \cdot A \cdot C - 0.000016 \cdot A \cdot B \cdot C$

Table 5. Coded and uncoded empirical equations for HTPB/RDX system.

Coded	**Uncoded**
$R_\eta = 5.484 - 0.651 \cdot A^* - 0.074 \cdot B^*$ $- 0.091 \cdot C^* + 0.0163 \cdot A^* \cdot B^* + 0.019 \cdot A^* \cdot C^*$ $- 0.014 \cdot B^* \cdot C^* + 0.042 \cdot A^* \cdot B^* \cdot C^*$	$R_\eta = 0.2653 + 0.12766 \cdot A + 0.13473 \cdot B$ $+ 0.6532 \cdot C - 0.003034 \cdot A \cdot B - 0.00784 \cdot B \cdot C$ $- 0.01436 \cdot A \cdot C + 0.000168 \cdot A \cdot B \cdot C$
$R_\tau = 96.650 - 10.600 \cdot A^* - 1.025 \cdot B^*$ $- 2.025 \cdot C^* + 0.725 \cdot A^* \cdot B^* + 0.725 \cdot A^* \cdot C^*$ $- 0.350 \cdot B^* \cdot C^* + 0.400 \cdot A^* \cdot B^* \cdot C^*$	$R_\tau = 152 - 1.125 \cdot A + 0.825 \cdot B$ $+ 5.4 \cdot C - 0.0175 \cdot A \cdot B - 0.079 \cdot B \cdot C$ $- 0.115 \cdot A \cdot C + 0.0016 \cdot A \cdot B \cdot C$

$R_M = 5.009 - 0.551 \cdot A^* - 0.044 \cdot B^*$ $- 0.082 \cdot C^* + 0.036 \cdot A^* \cdot B^* + 0.024 \cdot A^* \cdot C^*$ $- 0.049 \cdot B^* \cdot C^* + 0.006 \cdot A^* \cdot B^* \cdot C^*$	$R_M = 10.744 - 0.151 \cdot A + 0.0044 \cdot B$ $+ 0.1258 \cdot C + 0.00024 \cdot A \cdot B - 0.00206 \cdot B \cdot C$ $- 0.0012 \cdot A \cdot C + 0.000024 \cdot A \cdot B \cdot C$

Table 6. Coded and uncoded empirical equations for HTPB/DAPTA system.

Coded	Uncoded
$R_\eta = 5.098 - 0.620 \cdot A^* - 0.055 \cdot B^*$ $- 0.090 \cdot C^* + 0.0125 \cdot A^* \cdot B^* + 0.0375 \cdot A^* \cdot C^*$ $- 0.0175 \cdot B^* \cdot C^* - 0.015 \cdot A^* \cdot B^* \cdot C^*$	$R_\eta = 18.1255 - 0.2845 \cdot A - 0.06375 \cdot B$ $- 0.297 \cdot C + 0.00145 \cdot A \cdot B + 0.00235 \cdot B \cdot C$ $+ 0.0069 \cdot A \cdot C - 0.00006 \cdot A \cdot B \cdot C$
$R_\tau = 91.088 - 12.663 \cdot A^* - 1.388 \cdot B^*$ $- 2.238 \cdot C^* + 0.363 \cdot A^* \cdot B^* + 1.013 \cdot A^* \cdot C^*$ $- 0.263 \cdot B^* \cdot C^* - 0.513 \cdot A^* \cdot B^* \cdot C^*$	$R_\tau = 449.3732 - 7.69576 \cdot A - 2.20998 \cdot B$ $- 10.12116 \cdot C + 0.048364 \cdot A \cdot B + 0.087224 \cdot B \cdot C$ $+ 0.225488 \cdot A \cdot C - 0.0020552 \cdot A \cdot B \cdot C$
$R_M = 4.521 - 0.471 \cdot A^* - 0.019 \cdot B^*$ $- 0.071 \cdot C^* - 0.011 \cdot A^* \cdot B^* + 0.021 \cdot A^* \cdot C^*$ $+ 0.004 \cdot B^* \cdot C^* - 0.024 \cdot A^* \cdot B^* \cdot C^*$	$R_M = 17 - 0.264 \cdot A - 0.0800 \cdot B$ $- 0.448 \cdot C + 0.0017 \cdot A \cdot B + 0.0044 \cdot B \cdot C$ $+ 0.00948 \cdot A \cdot C - 0.000096 \cdot A \cdot B \cdot C$

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