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CHEMICAL SCIENCES

A Viscometric study of mixtures with Hydroxyl-terminated polybutadiene (HTPB) and short chain diols used in the formulations of solid composite propellants

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Abstract: Hydroxyl-terminated polybutadiene (HTPB) is widely used in the formulations of solid propellants used in rocket motors. Furthermore, in general, chain extenders and short chain diols, such as 1,4-butanediol and 1,2-propanediol, can also be used in propellant formulations to improve mechanical properties, especially tensile strength. However, the incorporation of these diols can result in a considerable increase in the viscosity of the mixture during the processing of propellants. Thus, the present study evaluated the compatibility of these diols with the HTPB prepolymer, through a viscometric study, with the aim to determine the order of addition that results in greater homogeneity for the mixture. It was concluded that 1,4-butanediol, due to its larger chain size, has better compatibility with HTPB resin than 1,2-propanediol. On top of that, it was found that when the resin is added first, it results in mixtures with greater compatibility.

Key words: Chain extenders, propellant, solid propulsion, viscometry.

INTRODUCTION

Solid composite propellants are considered as a material with solid particles, an oxidizer, such as ammonium perchlorate (AP), and a metallic fuel, normally aluminum powder, and with additives, such as plasticizer, antioxidant, and bonding agent, incorporated in an elastomeric matrix that acts as a binder providing a rubber-like elasticity to the grains (Shusser 2012). Polyurethane binder systems, formed by a hydroxyl-functional prepolymers, such as hydroxyl-terminated polybutadiene (HTPB) and diisocyanate curing agent are extensively used in composite solid propellants, due to convenient reaction conditions (Amtower 2006). In the aerospace area, they are used in the formulations of composite propellants and explosive materials as PBX type – Plastic explosive, acting as an agglomerant and providing a suitable mechanical behavior for the grain, especially tensile strength. The HTPB is also widely used as a starting material in polyurethane formulations applied to adhesives, paints and coatings (Cohen et al. 1974, Yang et al. 2002, Hailu et al. 2010, Amrollahi et al. 2011, Chen et al. 2011, DeLuca et al. 2013). HTPB can be classified as a prepolymer and has excellent physical-chemical properties, such as low glass transition temperature, tensile and tear resistance, low viscosity and good chemical resistance. The tensile mechanical characteristics of HTPB-based binder have been modified by increasing the hard segment content in the matrix, when short chain diol is used. Also, it is possible to increase the tensile strength of the polymeric matrix based on cured polyurethanes, obtained through the reaction between polyols and diisocyanates, by the increase of urethane groups and the use of rigid and symmetric molecules (Hepburn 1992, Wirpsza 1993, Oertel 1994). Due to the increase of quantity of urethane groups, the number of intermolecular interactions, as hydrogen bonds among polymer chains, increases as well. The amount of urethane groups can be increased by adding short chain diols and diisocyanates to the blend composition. These diols are classified as chain extenders, with 1,4-butanediol (BDO) being the most used (Wingborg 2002). Researches of polyurethanes based on HTPB, BDO and different diisocyanates have been carried out (Minoura et al. 1978, Siegmann et al. 1987, Ramesh et al. 1991, Sheikhy et al. 2013, Brzic et al. 2015). These studies concluded that the mixing mechanism and mixing dynamics are relevant in the mass rheology and in the final properties of the binder. Thermogravimetric Analysis, Differential Scanning Calorimetry, Dynamic Mechanical Thermal Analysis, Dynamic Mechanical Analysis, Scanning Electron Microscopy and Viscometry techniques are used to determining the miscibility of polymers (García et al. 1999, Zhang et al. 2002, Lewandowska 2005, Aroguz & Baysal 2006). The study of viscosity presents as advantages over these other methods its operational simplicity, low cost, and speed of analysis. Even with these instrumental advantages, there are no studies reported in the literature that evaluate viscometric studies of HTPB with chain extenders or additives used in solid composite propellant formulations. During the processing of propellants, considerable changes in viscosity and rheological characteristics of the mixes were observed after the addition of short chain diols as 1,4-butanediol and 1,2-propanediol. Thus, the compatibility of HTPB resin with 1,4-butanediol and 1,2-propanediol has to be evaluated. The resin is regarded as a polymeric solute in a short chain diol solvent. Hence, the viscometry technique was used in order to assess the compatibility of these components in a solution, using two methodologies presented in the literature (Bagchi et al. 1986, Mello et al. 2006). Therefore, a wide range of concentrations and diluted solutions of the HTPB polymer were evaluated.

MATERIALS AND METHODS

The materials used in this study was HTPB Liquiflex LIQ-P®, produced by Petroflex®, batch 700176, 1,4-butanediol, produced by Aldrich®, batch 50287, and 1,2-propanediol, produced by Reagen®, batch 881157.

Firstly, an evaluation of the mixtures with a wide range of concentration was performed taking into consideration a previous work (Bagchi et al. 1986). The methodology used was based on obtaining solutions of HTPB and 1,4-butanediol or 1,2-propanediol, with different levels of HTPB in mass basis (g of resin per g of chain extender as solvent). The following concentrations were used: HTPB:diol 0, 5, 10, 20, 40, 60, 80, 100, 200, 400, 600, 800, 1000:100 in mass basis. The experimental viscosity at each concentration was obtained using the Brookfield DV-II+ Pro Viscometer at 50°C, which was the process temperature used in the production of the propellants. The viscosity was measured once, as the viscometer is digital and the error of +/- 1% is intrinsic to the equipment. To determine viscosity, both orders of addition were considered, when the diol was added first and when the resin HTPB was added first. It should be noted that in the literature (Bagchi et al. 1986, Mello et al. 2006) this parameter was not considered.

Secondly, an evaluation of diluted mixtures was performed. Thence, five diluted resin solutions in BDO or 1,2-propanediol were prepared at 50°C, with concentrations up to 15.0 g.L^{-1}. The viscosities of pure solvents and solutions at this temperature were measured, using the viscometer mentioned previously. The relative (η_r), specific (η_{sp}), reduced (η_{red}) and inherent (η_{inh}) viscosities of all solutions were also determined mathematically from Equations 1, 2, 3 and 4, respectively, suggested in the literature (Lucas et al. 2001).

$$\eta_r = \frac{\eta}{\eta_0} \tag*{Eq.1}$$

$$\eta_{sp} = \eta_r - 1 \tag*{Eq.2}$$

$$\eta_{red} = \frac{\eta_{sp}}{C} \tag*{Eq.3}$$

$$\eta_{inh} = \frac{ln \quad \eta_r}{C} \tag*{Eq.4}$$

η_0 is the viscosity of pure solvent and C is the concentration in g.L^{-1}. Finally, the intrinsic viscosity of the solutions was determined through a procedure presented in the literature (Mello et al. 2006). From the reduced and inherent viscosity at different concentrations, at 50°C, it was possible to determine, by extrapolation, at zero concentration, the intrinsic viscosity values [η]. The least squares method was used for linear adjustment of the empirical equations for determining intrinsic viscosity, through Huggins and Kraemer equations, presented respectively in Equations 5 and 6.

$$\eta_{red} = \frac{\eta_{sp}}{C} = [\eta] + K'[\eta]^2 C \tag*{Eq.5}$$

$$\eta_{inh} = \frac{\ln \eta_r}{C} = [\eta] + K''[\eta]^2 C \tag*{Eq.6}$$

K' and K'' are the constant of Huggins and Kraemer, respectively.

RESULTS AND DISCUSSION

The viscosity results for 1,4-butanediol solutions and 1,2-propanediol with HTPB, in both orders of component addition, at 50°C are shown in Table I, Figures 1 and 2. They suggest that the order of addition of the resin and diols influences the viscosity of the mixture.

As previously mentioned, it was observed that the order of addition for mixtures of 1,4-butanediol and HTPB influences the viscosity of the mixtures when there is a resin concentration greater than 38 % by mass of resin. When adding HTPB resin first, the viscosity was increased, in relation to the pure resin, for concentrations above 55 % in mass basis. This phenomenon suggests that 1,4-butanediol is considered a good solvent for HTPB resin (Lucas et al. 2001), when it is used in this order of addition and from this concentration, because there was an increase in viscosity in the final mixture in comparison to the pure resin. On the other hand, the inverse order of addition results in concentrated solutions of resin, which have lower viscosity, in comparison to the results obtained previously. Ergo, based on the data, it is preferable to add HTPB first to obtain a higher viscosity of the mixture.

For 1,2-propanediol, the results suggest that the order of addition for mixtures of 1,2-propanediol and HTPB also influences their viscosity at resin concentrations greater than 50 % by mass of resin. When the resin was added first, the viscosity increased, in comparison to the pure resin, for concentrations greater than 57 % in mass basis. This phenomenon suggests that 1,2-propanediol can be considered a good solvent for the resin (Lucas et al. 2001), when

Table I. **Viscosity data obtained from 1,4-butanediol or 1,2-propanediol and HTPB solutions.**

Mass content (HTPB:100g of diol)	C (% HTPB in mass)	Order of addition (First component)			
		Viscosity (cP)	Viscosity (cP)	Viscosity (cP)	Viscosity (cP)
		1,4-butanediol	HTPB	1,2-propanediol	HTPB
0.00	0.00	19.20	14.40	0.00	4.80
5.00	4.76	48.00	72.00	24.00	14.40
10.00	9.09	96.00	124.80	19.20	24.00
20.00	16.67	168.00	163.20	134.00	81.60
40.00	28.57	311.90	283.00	254.00	153.60
60.00	37.50	374.30	431.90	340.00	254.30
80.00	44.44	475.10	835.00	383.00	364.70
100.00	50.00	537.50	1075.00	446.30	662.70
200.00	66.67	1147.00	6200.00	508.00	4237.00
400.00	80.00	4000.00	5346.00	494.00	4180.00
600.00	85.71	3532.00	4720.00	606.40	3623.00
800.00	88.89	4000.00	5135.00	4300.00	3695.00
1000.00	90.91	2700.00	3900.00	4200.00	3474.00
∞	100.00	2000.00	1972.00	2000.00	1940.00

this order of addition is adopted at certain concentrations. However, when the inverse order of addition was used, it resulted in solutions whose viscosity did not vary until the concentrations reached about 87 % in mass basis. Whence, this suggests that 1,2-propanediol is not a good solvent under these conditions. Moreover, a considerable heterogeneity of the mixtures along with the visible presence of unsolvable resin was observed. Therefore, the most suitable order of addition for the application of 1,2-propanediol in the propellant formulations is adding the HTPB resin first.

Finally, for comparison purpose, Figure 3 shows the behavior of the Resin + 1,4-butanediol and Resin + 1,2-propanediol system, for the same addition order, starting from pure resin, the most indicated order of addition for both systems.

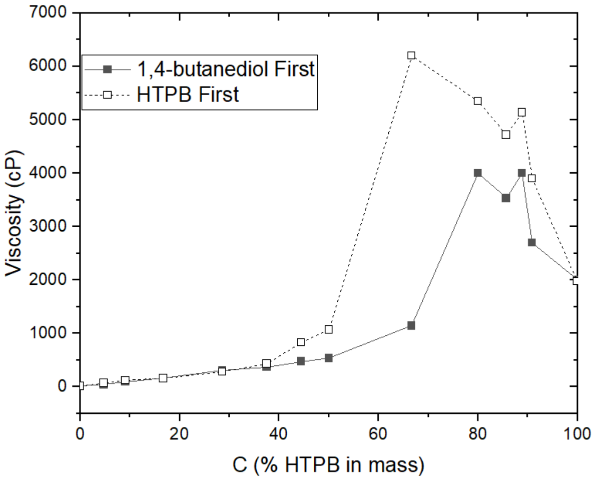

Figure 1. **Viscosity profile of 1,4-butanediol and HTPB resin solutions, depending on the resin concentration, for both order of addition.**

It was noticed that the solutions, which contain 1,4-butanediol, showed, in all evaluated ranges, higher viscosity than the solutions in which the 1,2-propanediol was used, suggesting that there is greater compatibility between the resin and the 1,4-butanediol. Consequently, it is possible to infer that, with a larger diol chain, the compatibility with the resin increases, because, in a simplified way, the resin is roughly a diol with a long unsaturated chain and two hydroxyls at the ends.

The analysis proposed in this work proves to be useful to evaluate the interaction between the studied diols and HTPB resin, to predict their rheological behavior and to determine their potential to be used in formulations of solid composite propellants. Thus, it is recommended that this analysis is adopted in future works when the addition of a new component to the formulation is being considered. Ergo, it is important to carry out this previous study involving resin viscosity and the process of mixing it with new components, so as to determine the existing compatibility range.

Regarding the methodology proposed in (Mello et al. 2006), in which the diluted polymer solutions are evaluated, Table II shows the viscosity results for diluted resin solutions in 1,4-butanediol and 1,2-propanediol. The volume of the solution was adopted considering the volume of the solvent, based on the calculation of the mass of solvent used and its density informed by the manufacturer. The volume of the solution was approximately the same as the volume of solvent.

Calculations of relative, specific, reduced and inherent viscosity are shown in Table III, presented below.

From the data presented in Table III, it was possible to make the linear adjustment based on the Huggins Equation (Eq. 5) and Kraemer Equation (Eq. 6) and calculate their respective constants. Figures 4 and 5 show the linear adjustments.

For the HTPB resin and BDO system, [η] is equal to 1.816 $dL.g^{-1}$ and Huggins constant equal to -0.1423. For the HTPB Resin and 1,2-propanediol system, [η] was equal to 11.196 $dL.g^{-1}$ and Huggins constant was -0.0553. In the literature (Lucas et al. 2001) it is suggested that an increase in the Huggins constant represents a decrease in the power of the solvent. Thus, BDO showed greater

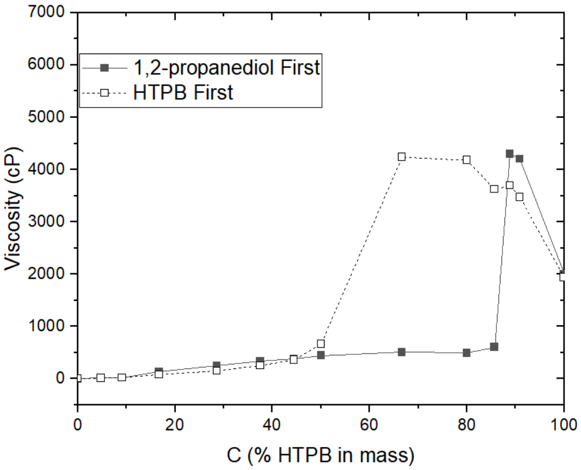

Figure 2. Viscosity profile of 1,2-propanediol and HTPB resin solutions, depending on the resin concentration, for both order of addition.

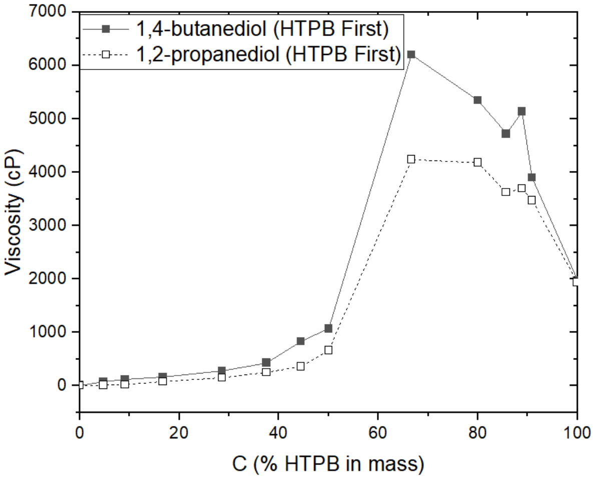

Figure 3. Comparison of the viscosity profile of solutions with 1,4-butanediol and 1,2-propanediol, depending on the resin concentration, adding HTPB resin first.

solvation power for the resin because it had a lower Huggins constant value.

The linear adjustment performed, using the Kraemer method, proved to be relatively satisfactory for the HTPB Resin and 1,4-butanediol and HTPB Resin and 1,2-propanediol systems. The Kraemer constant for each system was -0.2889 and -0.1264, respectively. The intrinsic viscosity of the system with BDO was equal to 1.51 $dL.g^{-1}$, while the intrinsic viscosity of the system with 1,2-propanediol was 4.85 $dL.g^{-1}$. The R^2 with reduced value in both linear regressions for 1,4-butanediol solutions may be due to the approximation of the volume of the solution as the volume of the added solvent, the control of the temperature and viscometer accuracy, since the mixture were low viscosity.

The Kraemer constant depends on the ratio between the hydrodynamic volume occupied by two molecules of solute that interact with each other and the hydrodynamic volume occupied by an isolated molecule. When the Kraemer coefficient is negative, it means that there is a good solvation of the polymer chain and there is a greater polymer-solvent interaction (Schoff 1999, Delpech et al. 2002a, b). In the study developed here, better solvation was observed between HTPB Resin and BDO, proved by the result of the lower Kraemer constant value for this system. Based on the viscometric study performed, the data presented by 1,2-propanediol were less satisfactory when compared to BDO system. This is mainly due to the greater polarity found in the 1,2-propanediol molecule in comparison to the 1,4-butanediol molecule, whose carbon chain is larger, and the hydroxyls are more distant from each other.

Table II. Viscosity data for diluted resin solutions in 1,4-butanediol or 1,2-propanediol.

Mass HTPB (g)	**Mass 1,4-butanediol (g)**	**Volume 1,4-butanediol (L)**	**C ($g.L^{-1}$)**	**Viscosity (cP)**
0.060	25.014	0.0246	2.439	19.2
0.099	25.304	0.0249	3.971	24.0
0.095	19.999	0.0197	4.806	28.8
0.155	20.315	0.0200	7.764	28.8
0.205	19.978	0.0196	10.413	33.6
Mass HTPB (g)	Mass 1,2-propanediol (g)	Volume 1,2-propanediol (L)	C ($g.L^{-1}$)	Viscosity (cP)
0.062	19.924	0.0192	3.234	19.2
0.107	20.445	0.0197	5.417	24.0
0.172	19.468	0.0188	9.164	24.0
0.251	19.908	0.0192	13.062	14.4
0.273	19.849	0.0192	14.249	19.2

Table III. Relative, specific, reduced and inherent viscosity data for diluted 1,4-butanediol or 1,2-propanediol resin solutions.

Diol	C ($g.L^{-1}$)	Viscosity (cP)	η_r	η_{sp}	η_{red} ($dL.g^{-1}$)	η_{inh} ($dL.g^{-1}$)
1,4-butanediol	2.439	19.2	1.33	0.33	1.37	1.18
	3.971	24.0	1.67	0.67	1.68	1.29
	4.806	28.8	2.00	1.00	2.08	1.44
	7.764	28.8	2.00	1.00	1.29	0.89
	10.413	33.6	2.33	1.33	1.28	0.81
	C ($g.L^{-1}$)	Viscosity (cP)	η_r	η_{sp}	η_{red} ($dL.g^{-1}$)	η_{inh} ($dL.g^{-1}$)
1,2-propanediol	3.234	19.2	4.00	3.00	9.276	4.29
	5.417	24.0	5.00	4.00	7.384	2.97
	9.164	24.0	5.00	4.00	4.365	1.76
	13.062	14.4	3.00	2.00	1.531	0.84
	14.249	19.2	4.00	3.00	2.105	0.97

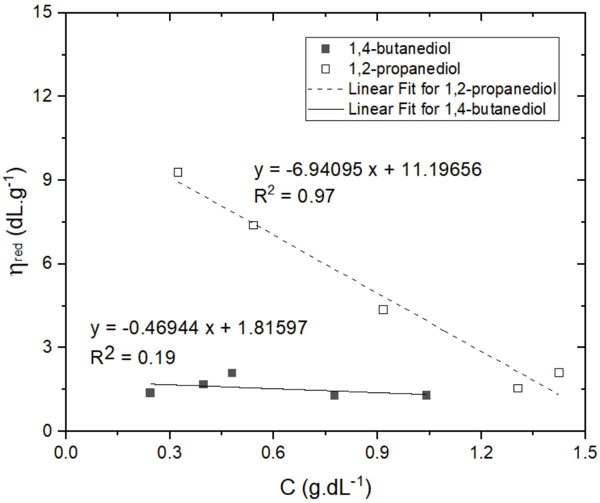

Figure 4. **Data adjustment of Huggins equation for reduced viscosity versus concentration for diluted HTPB resin solutions in 1,4-butanediol and 1,2-propanediol.**

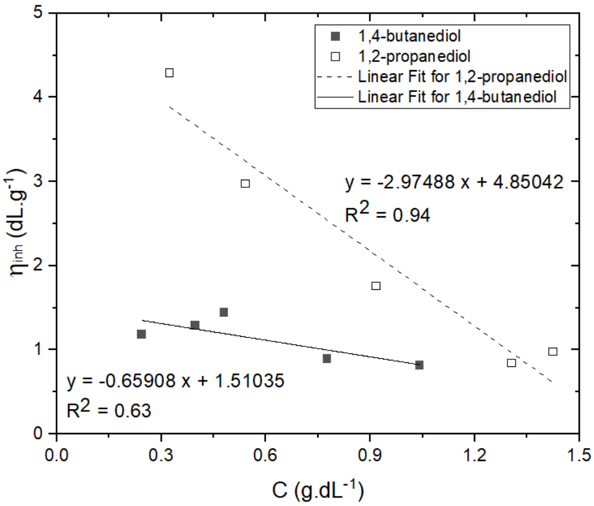

Figure 5. **Data adjustment of Kraemer Equation for inherent viscosity versus concentration for diluted HTPB resin solutions in 1,4-butanediol and 1,2-propanediol.**

CONCLUSION

The viscometric study carried out, using concentrated and diluted mixtures, was important to determine the compatibility of short chain diols with the HTPB resin used in the processing of propellants. The results show that 1,4-butanediol has greater compatibility with the resin, considering the evaluation of mixtures when different concentrations were used and, also, by the values of the Huggins and Kraemer constants determined for diluted resin solutions. On the other hand, the data suggest that the greater polarity of the 1,2-propanediol molecule, when compared with the 1,4-butanediol molecule, makes it difficult to mix with the resin. Moreover, the order of addition interferes with the final viscosity of the mixture. It is considered that both evaluations can be widely used to predict the rheological behavior of mixtures involving diols and resin. Furthermore, they may be useful in the processing of propellant formulations.

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Bruno Cesar Christo da Cunha planned and carried out the experiments, in addition to writing the draft of the article. Marcela Galizia Domingues reviewed and proposed improvements in the writing and in the structure of the article. José Atílio Fritz Fidel Rocco is the supervisor teacher of the research group.