

Influence of 1,4-butanediol on Hydroxyl-Terminated Poly(butadiene) Based Composite Propellant Binder Characteristics

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1,4-butanediol, as a chain extender, was incorporated into a hydroxyl-terminated poly(butadiene) based composite rocket propellant binder composition. As curing agents, isophorone diisocyanate (IPDI) and toluene diisocyanate (TDI) were used. Composite propellant binder network and mechanical properties, influenced by a presence of 1,4-butanediol, were examined. Network characteristics, sol-gel content and crosslink density have been calculated and successfully correlated to the mechanical uniaxial tensile properties of the tested propellant binders. Differential scanning calorimetry studies showed that 1,4-butanediol content did not influence the glass transition temperature, however the uniaxial tensile properties were shown to be a function of the crosslink density

Key words: butanediol, hydroxyl-terminated polybutadiene, composite rocket propellant fuel, propellant binder component, mechanical properties.

Introduction

COMPOSITE propellants are a mixture of powdered solid particles of inorganic oxidizer, such as ammonium perchlorate (AP), metallic fuel, such as aluminum powder, and/or other solids and additives (plasticizer, antioxidant, and bonding agent), embedded in and held together by an elastomeric matrix that imparts rubber-like elasticity to the propellant grains [1]. The elastomer based matrix serves both as a fuel component and as a binder between the energetic particles, providing the necessary mechanical properties to prevent break-up of the grains or crack formation during ignition or combustion [2]. Once cured, the binder makes the propellant flexible, which decreases the probability of fractures in propellant under stress and pressure. Being a viscoelastic material, solid rocket propellant mechanical properties are highly dependent on temperature and strain rate. Thus, they degrade over time due to chemical aging and damage accumulation, so they have a limited lifetime. The appearance of the fracture of the rocket motor propellant due to different loads acting from the moment of production and continuously during the storage and use, might lead to the undesirable changes in working mode or to the rocket motor explosion [3]. In order to meet the increasing demands of various working environments, the viscoelastic properties of hydroxyl-terminated poly(butadiene) based composite rocket propellants have been thoroughly examined in previous papers [4], with a particular attention to the effects of additives and solid fillers in these properties [5, 6].

The composite propellant binder comprises at least two components. The first one is a liquid prepolymer and the

second one is a curing agent. Polyurethane-based binder systems (hydroxyl-functional prepolymers, such as hydroxyl-terminated polybutadiene, HTPB, cured using multifunctional isocyanates) are extensively used in composite solid propellants, due to convenient reaction conditions and relative lack of adverse side reactions [7]. An essential requirement in the selection of a particular polymeric component is that it must exhibit good mechanical properties. In fact, the major cause of a failure in solid rocket motors is linked to the structural integrity of the propellant [8]. Therefore, the use of different types of polyurethanes is typical for the above-mentioned applications because of the alternating hard and soft segments within their structure [9].

The tensile mechanical properties of a composite propellant depend mainly on the tensile properties of the binder. The ultimate mechanical properties of the binder can be tailored by varying the R-value (equivalents of NCO groups/equivalents of OH groups). Increasing the tensile strength should not be done at the expense of the ultimate elongation, as occurs when the crosslinks density in HTPB is increased by increasing isocyanate/hydroxyl (NCO/OH) equivalent ratio. The amount of urethane bonds can be increased by adding low molecular weight diols to the polymer. These types of diols are called "chain extenders". The most common aliphatic diol for this purpose is 1,4-butanediol (1,4-BD) [10]. The tensile mechanical characteristics of HTPB have been modified by increasing the hard segment content, while 1,4-BD is used as a chain extender. The materials were crosslinked with either 1,6-hexamethylene diisocyanate or dicyclohexylmethane 4,4'-

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diisocyanate. The results showed that the tensile strength strongly increases with the addition of up to two moles of diol per mole HTPB [11].

In this work, the polymer network and mechanical properties of HTPB/1,4-BD cured with aliphatic (IPDI) and aromatic (TDI) diisocyanates were examined. The main goal was to analyze the influence of the 1,4-butanediol content on the composite propellant binder physico-chemical and mechanical properties, i.e. the dependence of mechanical properties on the binder crosslinking density.

Experimental part

Materials

Eight composite propellant binder compositions (Table 1) selected for this study consisted of 100 phr of HTPB (R-45HT, Sartomer, viscosity at 23°C: 8000 mPas, OH value: 47.12 mg KOH/g, hydroxyl functionality: 2.4 - 2.6, average molecular weight: 2800 g/mol, specific gravity at 23°C: 0.901 g/cm³, glass transition temperature, T_g : -76°C) and 2.5 phr of antioxidant (2,2'-methylene-bis-(4-methyl-6-tertbutylphenol), which is commercially available as a product called AO-2246; Fluka AG, Switzerland). Tested binder samples differed from each other in a 1,4-BD content (average molecular weight: 90.12 g/mol, OH Value: 1245 mg KOH/g, T_g : -42°C). Ingredients mix ratio is expressed by phr (parts per hundred resin based on 100 parts of HTPB).

Curing occurs when the hydroxyl groups of the prepolymer (HTPB) react with the isocyanate groups of the curing agent (IPDI, TDI, purity: 98%, Sigma Aldrich, Germany) to form urethane crosslinks (Fig.1).

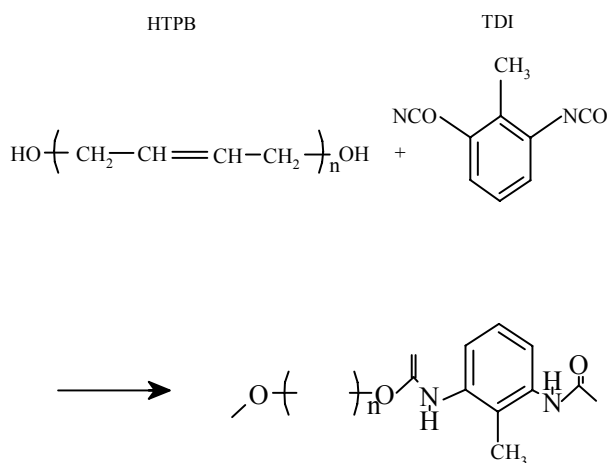


Figure 1. Schema of the urethane reaction

The NCO/OH ratio between the isocyanate groups of the curing agent to the hydroxyl groups of the prepolymer was 0.85. The tested composite propellant binder formulations were mixed in a 1.5 l DRAIS FH planetary mixer at the temperature of 60°C. All of the binder ingredients, except the curing agent, were blended thoroughly for 15 minutes at the ambient pressure and then vacuum mixed for another 15 minutes. Then, the curing agent was added to the mixture, mixed for 10 minutes at the ambient pressure and then vacuum mixed for another 10 minutes. Finally, the mixture was cast and cured into the teflon-coated metal plates to form 4 mm thick slabs. The curing was performed for 5 days at 70°C.

Compositions CPB0 and CPB00 are the baseline propellant binder compositions containing no 1,4-BD, with IPDI and TDI as a curing agent, respectively. 1,4-BD content (ζ) varied from 1 to 3 moles per mole of prepolymer.

Table 1. Tested propellant binder compositions

Sample	ζ , [mol]	Ingredient [phr]			
		R45HT	IPDI	TDI	1,4-BD
CPB0	-	100	8.14	-	-
CPB01	1	100	15.14	-	3.22
CPB02	2	100	21.87	-	6.44
CPB03	3	100	28.61	-	9.66
CPB00	-	100	-	6.31	-
CPB11	1	100	-	11.01	3.22
CPB22	2	100	-	15.71	6.44
CPB33	3	100	-	20.41	9.66

Methods

Determination of the network density of the composite propellant binder samples

The network density of the cured propellant binder samples was estimated by a degree of swelling by an appropriate solvent at equilibrium [12]. As solvent, toluen was used. Swelling test was carried out on the specimens length of 20 mm, width of 20 mm and thickness of 3 mm at the $T=25$ °C. In the preliminary experiment, the sample was allowed to swell for 4 days, but the weight became constant after two days, indicating that equilibrium is reached. In this study, the binder samples were swelled for two days.

Sol-Gel measurements

The sol content of the propellant binder (S_{poly}) is the weight fraction of the propellant, which is obtained by the extraction with the solvent, as described in STANAG 4581 [13]. The extraction was carried out in three Soxhlet extraction units (refluxing with solvent) for 16 hours with approximately 150 ml of dichloromethane (CH_2Cl_2) each.

Uniaxial tensile properties of the composite propellant binder samples

The uniaxial tensile properties of the cured propellant binder samples were evaluated at 20°C using the INSTRON 1122 tensile test machine fitted with a 5000 N load cell. «JANNAF C» dog bone samples (120.65 mm length, 25 mm width, 8 mm thickness) were used. Crosshead speed of the tensile machine was 50 mm/min, while the effective gauge length of the JANNAF C samples was 68.6 mm.

Differential scanning calorimetry (DSC)

DSC analyses were carried out using the DSC Q20 manufactured by TA Instruments with a liquid nitrogen cooling. The temperature scale is calibrated using the melting temperature of a high purity indium. These measurements were made with the purpose to investigate the thermal based glass transition temperature (T_g) of the tested samples. The small amount of the samples (~5 mg) was scanned with a heating rate of 10°C min⁻¹, in a range from -90°C to +50°C. The T_g of the samples were determined from the midpoints of the transitions.

IR Spectroscopy

Infrared spectroscopy was performed by using the Perkin Elmer IR spectrometer, spectrum range 4000-200 cm⁻¹. A thin layer of the sample (sol content) was placed on a plate of potassium bromide.

Results

Composite propellant binder network density

The gravimetric technique is used for measuring the swell ratio of the crosslinked polymeric propellant binder networks [14]. A sample is carefully weighed (W_0), then immersed in

toluen as a solvent at the required temperature for 24 hours. At the end of this period, the sample is again carefully weighed (W_g), and the swell ratio is computed from this and ratio of the densities of the solvent to the polymer, K , as

$$q = \frac{W_d + W_g - W_d \cdot K}{W_d} \quad (1)$$

The density of toluene is 0.86 g cm^{-3} , and those of cured binder samples were in the range of 0.927 to 0.971 g cm^{-3} [15].

The network density ($N \times 10^{-5} \text{ mol cm}^{-3}$) of the polymer can be estimated by the value of v_r as follows:

$$\ln(1 - v_r) + v_r + \chi v_r^2 = -N V_1 (v_r^{\frac{1}{3}} - \frac{V_r}{2}) \quad (2)$$

where: V_r - volume fraction of the tested binder sample in the swollen gel fraction, χ - Flory-Huggins polymer-solvent interaction parameter, V_1 - molar volume of the solvent, N - network density of the tested binder sample.

Knowing soluble fraction content (S_{poly}), the crosslink density (C) of the propellant binder (Table 3) can also be estimated by following the modified version of the Charlesby-Pinner [16] equation

$$C = \frac{(1 - S_{poly}) \cdot (2 - (S_{poly} + \sqrt{S_{poly}}))}{(S_{poly} + \sqrt{S_{poly}})} \quad (3)$$

The crosslink densities obtained in this manner for various 1,4-BD levels are compared with the corresponding values obtained using the swelling test (Fig.2).

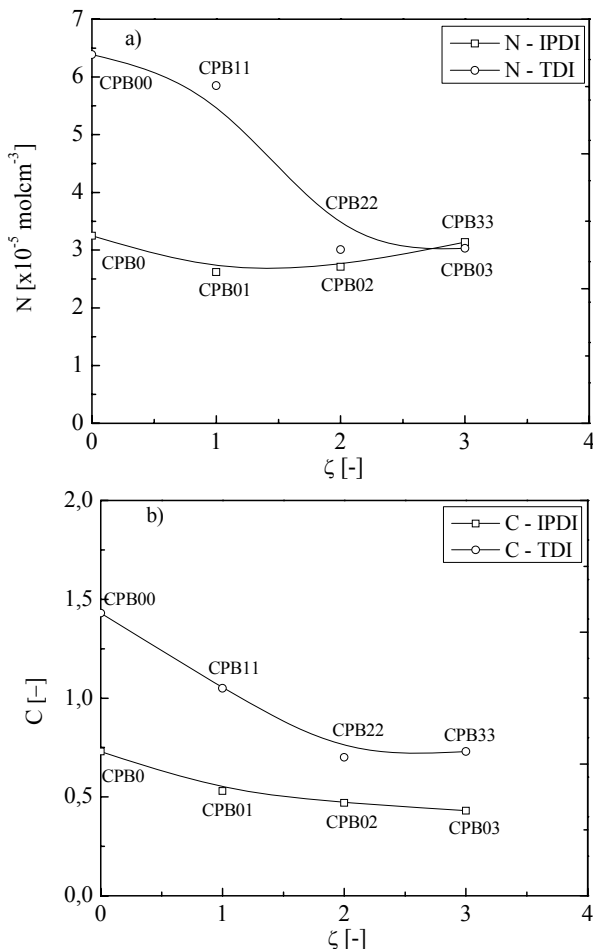


Figure 2. Crosslink density values dependencies on 1,4-BD content obtained by a) a swell method and b) a sol-gel analysis

The soluble or extractable content is a useful parameter to characterize the extent of cross-linking within a cross-linked polymer [17]. The greater the fraction of not cross-linked polymer chains, the higher is the soluble fraction. Both N and C values are higher for the propellant binder compositions containing TDI as a curing agent. The baseline formulations, containing TDI and IPDI (CPB0 and CPB00), have the higher soluble fraction values, whereas the formulation containing 1,4-BD are characterized by higher S_{poly} content and lower cross-link density values. Decrease of the network density can be explained by an increase in physical crosslinking due to a high concentration of the linear 1,4-BD polymer chains.

The parameters obtained by a sol-gel analysis are very useful and will be used for correlation with the results obtained by a mechanical characterization.

DSC measurements

As an essential ingredient, the binder largely determines the mechanical properties of the composite solid propellants. Low glass transition temperature is preferred for the composite propellant binder, so that it can withstand a large spectrum of stress transients in an operation. Therefore, the glass transition temperature (T_g) of the elastomer bonded composite propellant is one of the most important property determining their in-service application [18]. T_g values of the tested propellant binder samples determined through DSC are shown in Table 2.

Table 2. Glass transition temperature values of the tested propellant binder samples

Sample	T_g , [°C]
CPB0	-73.41
CPB01	-73.68
CPB02	-73.14
CPB03	-71.48
CPB00	-72.97
CPB11	-73.15
CPB22	-73.28
CPB33	-73.07

Polyurethane based elastomers contain soft and rigid segments [10]. The soft segments consist of large flexible non-crystalline polymer chains, while the rigid segments consist of diisocyanate residues, chain extenders and urethane groups. Glass transition as a relaxation process involves the release of the cooperative motions of HTPB prepolymer main chains between the crosslinks within the soft segment regions. These polymer network segments are flexible at the room temperature because of the urethane group's low polarity.

The differences between T_g values of the tested binder samples determined through DSC are within an experimental error. Obtained T_g values show that any regularity cannot be observed in its change with increasing 1,4-BD content in the propellant binder formulations. Also, the values are lower than the minimum service temperature (usually -40 to -50 °C, depending on the application). The T_g of HTPB cured with IPDI determined by DSC is higher than that of IPDI cured HTPB (reported as -83 °C by Bhagawan et al. [19]). Thus, 1,4-BD has very little influence on the glass transition temperature, which may be due to the fact that for the elastomers, glass transition temperature depends more on segmental motion of the polymer chains than on the concentration of crosslink points [20]. This indicates that, for the tested propellant binder networks, the distance between the crosslink points is such that any reduction in local

constraints caused by a presence of the 1,4-BD during crosslinking does not affect T_g .

Uniaxial tensile mechanical characteristics

The uniaxial tensile mechanical properties: tensile strength (σ_m), strain at maximum load (ε_m), strain at break (ε_b) and the Young's modulus (E) for the tested propellant binder samples are shown in Table 3. Because of the different structures of used curing agents, the measured tensile mechanical properties are a function of the original polymer matrix structure, also of the secondary bonds formed in the polymer. Considering the baseline binder compositions, the results revealed that TDI based propellant binders have higher stress and lower strain at the maximum load compared to one with IPDI. This behaviour is caused by the TDI's stiff and rigid benzene ring, which is absent in IPDI.

Table 3. Uniaxial tensile properties as a function of 1,4-butanediol content and crosslink density

Sample	ζ , [mol]	C , [-]	σ_m , [MPa]	ε_m , [%]	E , [MPa]
CPB0	-	0.73	0.29	663.77	0.15
CPB01	1	0.54	0.29	623.15	0.21
CPB02	2	0.47	0.41	545.27	0.29
CPB03	3	0.43	0.39	355.43	0.40
CPB00	-	1.43	0.36	277.60	0.32
CPB11	1	1.05	0.48	525.06	0.27
CPB22	2	0.70	0.42	814.52	0.12
CPB33	3	0.72	0.40	718.20	0.13

The number of urethane bonds is affected by the choice of the curing agent and the hard segment content (ζ , calculated as the mass fraction of 1,4-BD and curing agent in the binder composition) is altered to only a slight extent. Figure 3 shows σ_m values as a function of a hard segment content. IPDI achieves a similar number of urethane groups as TDI, but due to its cyclic structure the orientation of polymer segments is weaker. This leads to the difficulties in establishing the secondary bonds, weakening the interchain forces and lowering the values of stress at the maximum load.

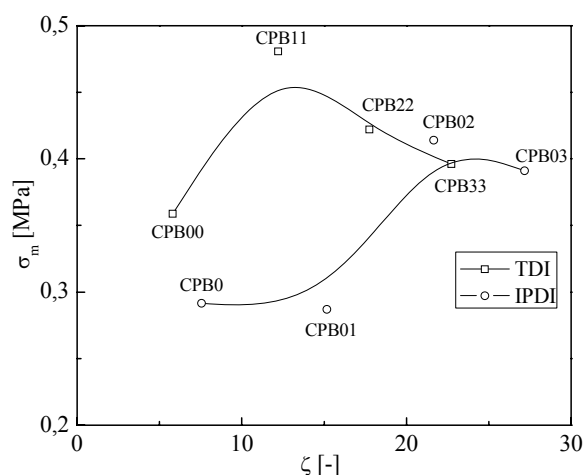


Figure 3. Tensile strength (σ_m) dependence on a hard segment content for the tested propellant binder samples

The level of 1,4-BD ($\zeta=1$, sample CPB11) contributes to the increase in tensile strength value by 34% compared to the baseline composition with TDI. Composition CPB02 ($\zeta=2$) has an increase in this value by 45%. Percentage-wise, this corresponds to an increase in the hard segment content.

The effect of the 1,4-BD on ε_m value of tested binder

samples is dependent on the applied curing agent. Considering the ε_m value of the baseline compositions, and the trend of changes, the addition of 1,4-BD contributes to a decrease of the value by 46% for CPB03 ($\zeta=3$), and an increase of 193% for CPB22 ($\zeta=2$).

As can be seen, for binder samples containing IPDI as a curing agent, an increase in the modulus is realized at the expense of the decreased ε_m value. The modulus increases linearly with increasing amount of 1,4-BD.

It is evident, for HTPB/TDI binder samples, with a decrease in the crosslink density, the ε_m value increases. Explanation is as follows: incorporation of 1,4-BD into the binder increases the molecular weight of polymer chains between crosslinks, which further increase the ε_m value (Table 3). Conversely, for the HTPB/IPDI binder samples, a decrease in the crosslink density also decreases the ε_m value. The explanation could be as follows: the N-H group in polyurethane could develop a hard segment by hydrogen bonding with the oxygen of carbonyl groups. Such strong hydrogen bonding acts as physical crosslinks restricting the motion of segmental of the polymer chain. So, a significant phase separation occurred between the hard and soft segments and decreased the elasticity of the propellant binder [21].

IR measurements

Fig.4 shows the IR spectra of the propellant binders with IPDI as a curing agent, without and with 1,4-butanediol as a chain extender.

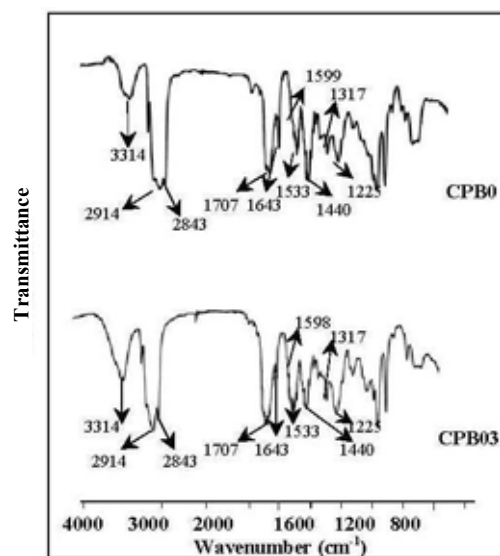


Figure 4. IR spectrum of the propellant binders with IPDI as a curing agent

IR spectra show characteristic bands of the urethane groups at 3314 cm^{-1} (N-H stretching). The other peaks observed were assigned as: 2914 cm^{-1} (CH symmetric stretching vibrations of CH_2); 2843 cm^{-1} (CH asymmetric stretching vibrations of CH_2 groups); 1707 cm^{-1} , 1643 cm^{-1} (C=O bond); 1599 cm^{-1} (NH deformations); 1533 cm^{-1} (aromatic ring) 1440 cm^{-1} (CH_2 bending vibration); 1317 cm^{-1} (CH_2 wagging). By extending prepolymer with 1,4-BDO, the IR spectra showed a very strong peak at about 1707 cm^{-1} which was assigned to C=O stretching of urethane. Peaks corresponding to the absorption of NH, C=O and C-O were observed at 3314 cm^{-1} , 1707 cm^{-1} (non-hydrogen bonded), 1643 cm^{-1} (hydrogen bonded) and 1225 cm^{-1} , respectively, which indicate the propellant binders with the urethane (NHCOO) group. The observed N-H bending vibrations at 1598 cm^{-1} , C-O-C stretching absorption band corresponding to the linkage between OH and NCO groups to form an

urethane bond in the range of 1057-1130 cm^{-1} also provide a strong evidence for the formation of PU [22].

It is valuable to mention the N-H group in polyurethane could develop a hard segment to hard segment H-bonding with the oxygen of carbonyl groups. Such a strong H bonding acts as physical crosslink, restricting the motion of the polymer's segments [23].

Fig. 5 shows the IR spectra of the propellant binders with TDI as a curing agent, without and with 1,4-butanediol as a chain extender.

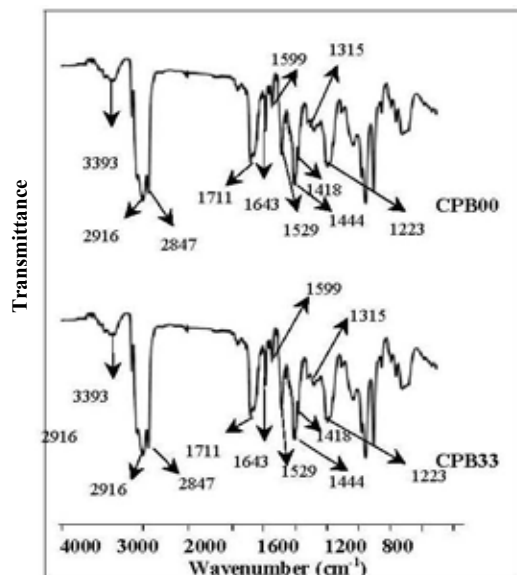


Figure 5. IR spectrum of the propellant binders with TDI as a curing agent

IR spectra show characteristic bands of the urethane groups at 3393 cm^{-1} (N-H stretching). The other peaks observed are: 2916 cm^{-1} (symmetric stretching); 2847 cm^{-1} (asymmetric stretching vibrations of CH_2) groups; 1711 cm^{-1} , 1649 cm^{-1} (C = O bond); 1599 cm^{-1} , 1529 cm^{-1} (NH deformations); 1444 cm^{-1} and 1418 cm^{-1} (CH_2 bending vibration); 1315 cm^{-1} (CH_2 wagging). Peaks corresponding to the absorption of -N-H and -C=O groups at 3393 cm^{-1} and 1711 cm^{-1} , respectively indicate that the propellant binders have -NHCOO (urethane group). Further is evident that a new peak at 1223 cm^{-1} for NHCO provides a strong evidence for the formation of PU [22].

Conclusion

Uniaxial tensile mechanical characterization, sol-gel analysis, differential scanning calorimetry and IR measurements were employed together with a gravimetric swell technique method to elucidate the behaviour of the HTPB-based composite propellant binder containing 1,4-butanediol as a chain extender. Crosslink density values of the polymer network evaluated by a sol-gel analysis indicates that the specified 1,4-BD level sol fraction of the tested binder sample is 20 to 45 percent. The obtained network densities trend values are similar meaning that with different analytical techniques the same phenomena was investigated and characterized.

Baseline compositions, HTPB cured with IPDI or TDI, gave the propellant binders with a quite different mechanical characteristics. Adding 1,4-BD into the propellant binder composition affects increasing or decreasing the strain at a maximum stress and the Young's modulus, but the influence on a tensile strength cannot be easily recognized. The highest value of the strain at the maximum stress was found in the CPB22 composition (TDI as a curing agent and 2 moles of

1,4-BD per mole of HTPB). The T_g values of the tested binder samples were approximately -73°C , without any influence of 1,4-BD on this value. In essence, the desired uniaxial tensile properties require the optimum combination of the chain extender and curing agent. From all the shown data, it can be concluded that the composition of the network forming agents can be optimized to achieve the desired uniaxial tensile properties for a specified application.

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Uticaj 1,4-butandiola na karakteristike gorivno-vezivne komponente kompozitnog raketnog goriva na bazi hidroksiterminiranog poli(butadiena)

1,4-butandiol, kao proizvođač lanaca, korišćen je za izradu sastava gorivno-vezivne komponente kompozitnog raketnog goriva na bazi hidroksiterminiranog poli(butadiena). Kao umreživači korišćeni su izoforondiizocijanat (IPDI) i toluendiizocijanat (TDI). Ispitivane su karakteristike polimerne mreže kao i mehaničke karakteristike dobijenog sistema. Određeni su parametri nastale polimerne mreže, sol-gel sadržaj i gustina umreženosti. Izvršena je korelacija ovih parametara i mehaničkih karakteristika dobijenih ispitivanjem jednoosne zavisnosti napon-deformacija. Rezultati dobijeni diferencijalnom skenirajućom kalorimetrijom pokazali su da sadržaj 1,4-butandiola ne utiče na vrednost temperature ostakljivanja, dok vrednosti jednoosnih mehaničkih karakteristika zavise od gustine umreženosti ispitivanih sastava gorivno-vezivne komponente.

Ključne reči: butandiol, hidroksiterminirani polibutadien, kompozitno raketno gorivo, gorivno-vezivna komponenta, mehaničke karakteristike.

Influence du butanediol 1,4 sur les caractéristiques de la composante propergol – liant du propergol composite à la base de poly (butadiène) hydroxyle terminé

On a utilisé butanediol 1,4 comme prolongateur des chaînes pour la fabrication de la composante propergol- liant, composante du propergol composite à la base du poly(butadiène) hydroxyle terminé. Comme les durcisseurs on a employé isophorone diisocyanate (IPDI) et toluène diisocyanate. Les caractéristiques du réseau polymère ainsi que les propriétés mécaniques du système obtenu ont été examinées. On a déterminé les paramètres du réseau polymère réalisé, contenu sol gel et la densité du durcissement. On a établi la corrélation des ces paramètres et les propriétés mécaniques obtenus par l'examen de la dépendance uniaxe de la tension – déformation. Les résultats obtenus par la calorimétrie différentielle scannée ont démontré que le contenu de butanediol 1,4 ne faisait pas l'influence sur la valeur de température de vitrification alors que les valeurs de propriétés mécaniques uniaxes étaient dépendantes de la densité du durcissement des compositions propergol liant de la composante examinée.

Mots clés: butanediol, poly(butadiène) hydroxyle terminé, propergol composite, composante propergol – liant, caractéristiques mécaniques.

Влияние 1,4-бутандиола на характеристики топливно-связующих компонентов композитных ракетных топлив на основе гидроксигтерминированного поли (бутадиена)

1,4-бутандиол в качестве удлинителя цепи был использован для изготовления состава топливно-связующей компоненты композитного ракетного топлива на основе гидроксигтерминированного поли(бутадиена). В роли агентов использованы изофорондиизоцианат (ИФДИ) и толуолдиизоцианат (ТДИ). Мы исследовали характеристики полимерных сеток, а в том числе и механических свойств полученной системы. Здесь определяются параметры полученной полимерной сети по содержанию золь-гель и плотность сшивки. Здесь выполнена корреляция этих параметров и механических характеристик, полученных исследованием одноосной зависимости напряжение-деформация. Результаты полученные методом дифференциальной сканирующей калориметрией показали, что содержание 1,4-бутандиола не влияет на значение температуры стеклования, в то время как значения одноосных механических характеристик зависят от плотности сшивки испытанных составов топливно-связующей компоненты.

Ключевые слова: бутандиол, гидроксигтерминированный полибутадиен, композитное ракетное топливо, топливно-связующие компоненты, механические характеристики.