

Current trends in energetic thermoplastic elastomers as binders in high energy insensitive propellants in China

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Abstract

The typical application of energetic thermoplastic elastomers is as energetic binders in solid propellants. The type of energetic binder significantly influences the mechanical properties and consequently the combustion and vulnerability of composite propellants. This review focuses on current advances and future developments in synthesis of some energetic binders and their application in high energy insensitive propellants, especially, in our group and other Chinese researchers. The main topic is on the synthesis and application of energetic thermoplastic elastomers (ETPEs). The application of ETPEs in high energy insensitive propellants increases the energy level with reduction in sensitivity. New approaches to the synthesis of energetic polymer then described, including controlled radical polymerization method of energetic copolymers and supercritical fluid technology in the preparation of energetic prepolymer. Some new products, such as biodegradable energetic elastomer and dendrite energetic polymers, are also introduced.

Keywords : energetic thermoplastic elastomers, binder, high energy insensitive propellants

1. Introduction

When polymer is used as binder in composite propellants and explosives, it encapsulates high-melting crystalline energetic solids loading, e.g. 1,3,5-trinitroperhydro-1,3,5-triazine (cyclotrimethylenetrinitramine, RDX). The performance of propellants and explosives is dependent on the physical shape, surface area and mechanical properties of finished formulation, which are mostly associated with the polymer binder¹⁾.

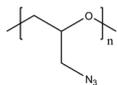
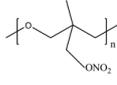
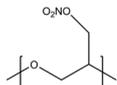
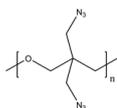
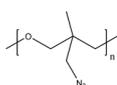
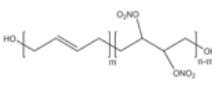
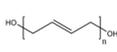
The most commonly used polymer binder in composite propellants is hydroxyl-terminated polybutadiene (HTPB)²⁾. However, HTPB is not energetic and thus the performance of composition is limited unless there is a high solids loading. At very high solids loadings there can be processing as well as vulnerable problems. Energetic polymer binder can be used to add energy and reduce sensitivity to stimulus while enabling a low solids loading, or increase performance while maintaining the same solids loading. Therefore, there are more and more researches focusing on the synthesis and modification of energetic

polymer binder.

Energetic polymer binders are polymers containing energetic groups in the backbones or side chain, such as the nitro (-NO₂), nitrate (-ONO₂), azido (-N₃), etc. The energetic groups can be introduced either to the polymer backbone or side chain³⁾. The most popular energetic polymer binder is nitrocellulose (NC). In the gun propellant chemistry, NC is mainly as a binder to keep the good mechanic and energy performance of propellant and explosive system. However, the NC and nitroglycerine (NG) based gun propellants are highly prone to accidental initiation as a result of external stimulus (fire, shock wave and impact). In order to reduce the risk, high energy insensitive propellants are attractive alternative for conventional NC/NG based propellants. The use of energetic polymer binder in explosive and propellant formulations is regarded as a practical method to formulate high energy insensitive propellants^{4,5)}.

There are several reviews on the recent development and trends on the energetic materials recently⁶⁻⁹⁾.

Table 1 Molecular structure and some properties of energetic polymer binders.

Energetic polymer binder	Structure	Heat of formation [kJ/mol]	Density [g/cm ³]	Oxygen balance [%]	Glass transition temperature [°C]
GAP		+117	1.30	-121	-45
Poly (NiMMO)		-335	1.26	-114	-25
Poly (GlyN)		-285	1.39	-61	-35
Poly (BAMO)		+413	1.30	-124	-45
Poly (AMMO)		+179	1.06	-170	-35
NHTPB		-	1.2	-	-58
HTPB		-52	0.92	-324	-65

However, the application of energetic polymers as binders in propellants system is not focused on in these reviews. Early review¹⁾ in 1994 on the binder application of energetic polymers was just emphasized on the synthesis and modification of typical energetic polymers, such as poly (3,3- nitratomethyl) methyl oxetane [poly (NiMMO)], polyglycidyl nitrate [poly (GlyN)], nitrated HTPB and glycidyl azide polymer (GAP). However, more and more new energetic copolymers and synthesis methods are emerging for now. Especially, in China, many efforts on energetic polymer research are driven by a joint program of government research institution and university^{10,11)}. The state-of-the-art reviews in this field are needed to describe their development and future trends.

This paper reviews the current progress in research on the energetic binder in the composite propellants, especially, the advanced high energy insensitivity gun propellants in our group at Nanjing University of Science and Technology and other Chinese researchers. The main topic is on the synthesis and application of energetic thermoplastic elastomers (ETPEs). Most of them are not completely included in other reviews published recently.

2. Energetic thermoplastic elastomers

Early work on energetic binders has been reviewed by Urbanski¹²⁾. More recent reviews on energetic binders have been reported by DRA¹⁾ and India High Energy Materials Research Laboratory^{8,9)}. Energetic binders have been researched extensively over the last 30 years as alternative to HTPB binder for composite propellants and polymer bonded explosive (PBXs). Synthesis and modification routes and the properties of energetic binder, e.g. glycidyl azide polymer (GAP), nitrated HTPB, Poly(3,3-

nitratomethyl) methyl oxetane [poly (NiMMO)] and polyglycidyl nitrate [poly (GlyN)] were widely reported²⁾.

Table 1 lists the molecular structure and properties of main energetic binders used in both explosives and propellants.

The future requirements of recyclability or environmentally compliant disposal of explosives and propellants are very important. The main problem associated with the current binders is that they use an isocyanate cure which is irreversible, toxic, causes gassing, has a finite pot-life and is generally not environment friendly. Such cured rubbers cannot be easily recycled without extensive degradation of polymer and their disposal also poses problems. In order to overcome these problems, use of thermoplastic elastomers (TPEs) has been investigated by many groups¹³⁾⁻¹⁵⁾.

A TPE polymer contains a hard (crystalline) block and a soft (non-crystalline) block. At operational temperatures, thermoplastic elastomers are solid, behaving much like the polymers we use today. At higher temperature they melt. These characteristics would enable the recovery and reuse of the ingredients of a propellant system, and could preferentially lower the life-cycle wastes. Once melted, the ingredients, which are bound together in the TPE, can be separated, recovered, and saved for future use. They can be repeatedly melted and used to manufacture new products. This results in the cost savings and environment friendly demilitarization and disposal.

With the transition of ammunitions to lower vulnerability (LOVA) or insensitive munitions (IM), new thermoplastic elastomers (TPEs) were developed to replace the historical workhorse NC at gun propellants⁴⁾. Thermoplastic elastomers (TPEs) process like

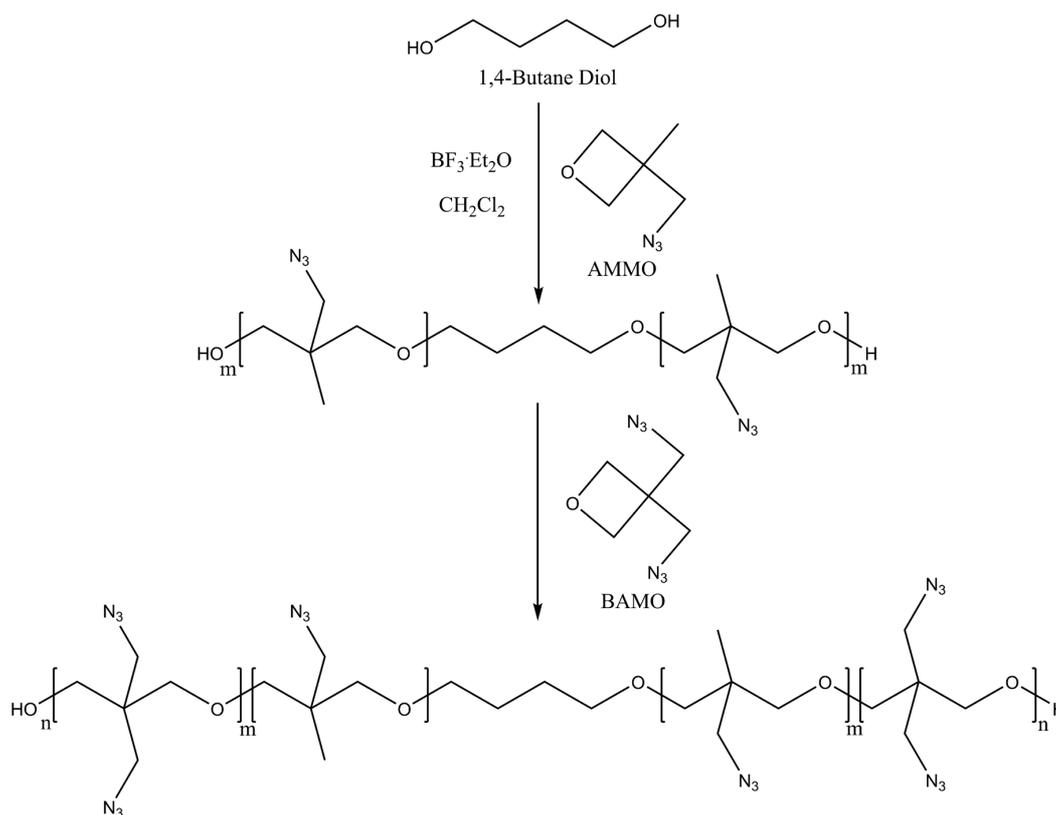


Figure 1 Synthesis route for BAMO/AMMO tri-block copolymer.

thermoplastics but behave like elastomers at gun operating temperatures, while offering the opportunity for ready inclusion of energetic fillers, good physical properties, and reprocessibility. Early LOVA formulations were based on energetic fillers in nonenergetic binders, but the quest for higher performance led to a concentration on the use of energetic TPEs^{10, 11, 16–18}) such as GAP, poly [3,3-bis (azidomethyl) oxetane] [poly (BAMO)] and poly (3-azidomethyl-3-methyl oxetane) [poly (AMMO)], etc.

Energetic copolymer consists of block energetic polymers such as poly (BAMO), poly (NiMMO), and poly (AMMO), and variants thereof. The synthesis of energetic copolymer based on BAMO was reviewed by the recent book written by High Energy Material Research Laboratory in India²). It also listed the gun propellant formulations and performance level of some BAMO-AMMO thermoplastic elastomers used in propellants.

Some of the energetic thermoplastic elastomers studied in China include BAMO based copolymer¹⁹) and GAP based TPEs²⁰). Their application in solid propellants has been studied based on the laboratory experiments^{21, 22}).

Chu et al.²³) synthesized azido-typed thermoplastic energetic binder using melting-prepolymerization method with 3,3-bis(azidomethyl)oxetane-tetrahydrofuran (BAMO-THF) as soft segments, and isophorone diisocyanate (IPDI) and 1,4-butanediol (BDO) as hard segments.

However, the introduction of inner THF segments in the copolymer of BAMO-THF may decrease the energy level of TPEs. More BAMO based copolymer with energetic segments of GAP, AMMO, NiMMO et al. were

studied.

Gan et al.²⁴) at Xi'an Modern Chemistry Research Institute synthesized the energetic thermoplastic elastomer (ETPE) based on BAMO/AMMO of number average molecular weight about 25000 polymerization. The BAMO/AMMO copolymer was prepared by one-step solution polymerization using tetrahydrofuran as solvent, poly (BAMO) as hard block, Poly (AMMO) as soft block, 2,4-toluene diisocyanate (TDI) as diisocyanate monomer, butane-1,4-diolurethane oligomer as linking compound. The mechanic property of synthesized ETPE Based on BAMO/AMMO was tested on a universal material testing machine (Instron 6022) with a tensile speed of 500 mm/min. Its tensile strength and elongation at room temperature were about 5MPa and 400%, respectively.

Furthermore, Song et al.²⁵) at Xi'an Modern Chemistry Research Institute studied the thermal behaviors of BAMO-AMMO and its compatibility with some energetic materials. The results indicated that thermal decomposition of BAMO-AMMO can be divided into two stages. The compatibilities of BAMO-AMMO with RDX, cyclotetramethylene tetranitramine (HMX), hexanitrohexaazaisowurtzitane (CL-20), AP, Al, NG and 1,5-diazo-3-nitrazapentane (DIANP) were good. It can be used as components in propellants.

Luo's group at Beijing Institute of Technology²⁶) synthesized BAMO/AMMO tri-block copolymer by cationic ring opening polymerization. The results showed that molecular weight of BAMO/AMMO tri-block copolymer is controllable and molecular weight distribution is relatively narrow. The synthesized copolymer exhibited the characteristics of an energetic

thermoplastic elastomer. The schematic synthesis route for BAMO/AMMO tri-block copolymer is showed in Figure 1.

Luo's group also studied the synthesis and properties of copolymer including GAP-PET²⁷⁾⁻²⁹⁾, GAP-PEG^{30),31)}. When PET or PEG was introduced in the elastomer, the tensile strength of elastomer increased. The mechanic properties of pure GAP can significantly improved by introduces of PET and PEG segments.

In order to apply the energetic thermoplastic elastomer (ETPE) to solid propellants, Zhao *et al.*³²⁾ at Xi'an Modern Chemistry Research Institute studied the effect of particle size and types of nitramine on combustion performance of ETPE gun propellants based on BAMO -AMMO by closed bomb test. Results showed that the pressure exponent of ETPE gun propellant is about 1.2 under 50 - 260 MPa, while the pressure exponent turns down from 1.2 to 1.0 as the pressure range changes from 75 - 175 MPa to 175 - 260 MPa. Burning rate and pressure exponent of the ETPE gun propellant increases with increasing of the particle size of RDX. The sample of ETPE gun propellant using HMX as oxidizer has lower burning rate and higher pressure exponent than the sample of ETPE gun propellant using RDX as oxidizer.

Research of Luo's group³³⁻³⁵⁾ at Beijing Institute of Technology realized that BAMO and GAP based energetic thermoplastic elastomers have good prospective application in solid rocket propellants with higher mechanic strength and low sensitivities.

Although there are many researches on the application of ETPE to the solid rocket propellants, few efforts are devoted to studies of synthesis and application of ETPE on high energy insensitive propellants.

Our group led by Prof. Xu at Nanjing University of Science and Technology also studied the GAP based thermoplastic elastomers to explore their application in high energy insensitive propellants^{10),11)}.

The initial efforts are derived from the studied of ETPE on solid rocket propellants. Nitrate ester plasticized thermoplastic elastomer Polyethylene glycol (PEG-TPE) for gun propellant binder were synthesized according to the strength and processing demand of gun propellant, and the properties of PEG-TPE are studied. PEG-TPE was synthesized by lower molecular weight PEG as soft segment, which is different from higher molecular weight PEG in rocket propellant, 4, 4'-diphenylmethane diisocyanate (MDI) and ethylene glycol as hard segment. Polyethylene glycol (PEG) as soft segments has good miscibility with nitrate ester, 4,4'-methylenebis(phenyl isocyanate) and ethylene glycol as hard segments³⁶⁾. DSC, FTIR and DMA were applied to characterize the synthesized elastomer. Results showed that the elastomer have structure characteristic of Polyurethane. The tensile stress of PEG-TPE is 3.75MPa, and elongation at break is 469% when hard segment is 50% in weight and the molar ratio of -NCO to -OH (R) is 1. The microstructure of PEG-TPE is homogeneous.

In order to improve the mechanical properties of PEG-TPE, polymethyl methacrylate (PMMA) was introduced

into PEG-TPE network to synthesize interpenetrating polymer networks (semi-IPN).

PMMA/PEG-TPE was synthesized by sequence interpenetrating technique to enhance the tensile stress of PEG-TPE. The tensile stress and elongation at break of PMMA/PEG-TPE (60/40, mass ratio) are 24.2MPa and 4.6%, respectively, whereas the tensile stress and elongation at break of PMMA/PEG-TPE 3.75MPa and 469%, respectively, showing that PMMA/PEG-TPE is a kind of strong but brittle binder. Dynamic Thermomechanical Analysis (DMA) showed that PMMA/PEG-TPE has two individual glass transition temperature (T_g), the lower T_g and the higher T_g are corresponding to those of PEG-TPE's soft segment and PMMA's, respectively.

Semi-interpenetrating polymer network of crosslinked-PMMA/PEG-TPE was synthesized using EGDMA as the crosslinking agent of PMMA, in order to improving the interpenetrating degree of PMMA and PEG-TPE polymer networks. DMA testing results showed that the crosslinked-PMMA/PEG-TPE (10/90) has only one glass transition temperature. The results showed that PMMA can make tensile strength of s-IPN improved significantly, but the elongation-to-break of s-IPN decreases greatly.

P(MMA/EA)/PEG-TPE interpenetrating polymer networks were synthesized by PEG-TPE and copolymer P(MMA/EA) of methyl methacrylate and ethyl acrylate, considering that the crosslinked-PMMA/PEG-TPE is not suitable to the extrusion processing of gun propellant. The DMA testing shows that the P(MMA/EA)/PEG-TPE (10/90, 30/70) have only one glass transition temperature.

Although the introduction of TPE to the propellant system can improve the mechanic properties of gun propellants, it can't meet completely the high energy requirements of propellant. Glycidyl azide polymer (GAP) is one of the most widely studied energetic polymers and was first synthesized in 1972 by the reaction of NaN_3 with polyepichlorohydrin (PECH-triol) in DMF. GAP possesses a high density (1.3 g cm^{-3}) compared with other polymers. It is found to be very insensitive. It is currently the most readily available energetic binder due to its relatively straightforward and low cost synthesis and shows excellent binder properties in its pure form.

A GAP based energetic thermoplastic elastomer (ETPE) was synthesized by melt prepolymerization using prepolymerized (GAP) as soft segments, 4,4'-diphenylmethane diisocyanate (MDI) and 1,4-butanediol (BDO) as hard segments for the purpose of high energy insensitive propellants³⁷⁾. The tensile strength of the GAP-based ETPE is 6.12 MPa, and the elongation is 71% when hard segment is 40% in weight and the mole ratio of -NCO to -OH (R) is 0.98.

Energetic thermoplastic elastomer GAP/MDI/ethylene glycol (DEG)-ETPE by melt prepolymerization method using different diols as chain extender is also synthesized for the purpose of using as high energy binder of insensitive propellants^{38),39)}. The energy parameters of the designed propellant formulations based on GAP energetic thermoplastic elastomer (ETPE) are calculated by inner

energy method⁴⁰). The results show that the RDX/ETPE propellant has an impetus about $1170 \text{ kJ}\cdot\text{kg}^{-1}$, and replacing the RDX in RDX/ETPE propellant formulation with CL-20, TNAZ (3,3-trinitroazetidine) etc. makes the impetus change in linear law and can reach an ideal impetus over $1300 \text{ kJ}\cdot\text{kg}^{-1}$.

In summary, in order to increase the energy and mechanic strength of ETPE, extension researches on the synthesis and properties of above ETPE and their application in solid propellant are conducting in different groups for new generation rocket propellant and composite gun propellant with high energy and insensitive performance. Significant progress on the small-scale test related with combustion and energy is made in the development of high energy and insensitive propellants including ETPE at Xi'an Modern Chemistry Research Institute and Nanjing University of Science and Technology.

3. New approaches to the synthesis of energetic polymers

There are two kinds of chemical methods employed to prepared energetic polymers. One is polymerization of an energetic monomer, and the other is modification of an existing polymer to introduce energetic groups to the polymer backbone.

Polymer modification is easier to make energy copolymer by introduce energy to the existing polymer. On the other hand, polymerization of energetic monomers requires careful control of reaction conditions since initiators may not be compatible with the energetic groups and high-risk during the synthesis process¹).

BAMO-AMMO and BAMO-GAP energetic thermoplastic elastomers are synthesized widely used cation living polymerization method. Their synthesis route and methods were reviewed by Chinese researchers and abroad²⁰). Practical requirements for living polymerization include very stringent reaction conditions, such as high vacuum or an inert atmosphere, and the absence of water and other impurities.

Therefore, controlled radical polymerization method was introduced into the preparation of energetic binders. It can synthesize polymers, where wide range of monomers can undergo radical polymerization under relatively simple conditions. In addition, it has been successfully used to synthesize polymers of well defined chemical structures, controlled molecular weight and narrow distribution of molecular weight⁴¹⁾⁻⁴⁶).

Controlled free radical polymerization method is developed to synthesize GAP based TPE to improve its mechanic performance. GAP is one of the most widely studied energetic binders. However, due to the weak mechanic properties of pure GAP, there are more efforts to synthesize the GAP based copolymer or TPE to overcome this shortcoming.

GAP-poly (methyl methacrylate) (PMMA) block and graft copolymer were prepared by free radical polymerization of MMA monomer in the presence of GAP macro-azo-initiator (MAI). The MAIs were prepared by

the polycondensation reaction of hydroxyl terminated groups of elastomers with 4,4'-azobis (4-cyanopentanoyl chloride) (ACPC). GAP was identified as suitable soft segment, and PMMA as a thermoplastic hard segment⁴⁴).

In addition, GAP based TPEs include GAP-PEG, GAP-PET, GAP-NC, GAP-BMMO/THF and GAP-THF et al. in many reports²⁰). The mechanic properties of pure GAP can be improved significantly.

Another new approach is the application of supercritical CO_2 in the formation and processing of energetic polymers²). Cationic ring opening polymerization of various energetic oxetanes and oxiranes was carried out in super critical CO_2 to form energetic prepolymer for use as binders in propellants.

Further application of supercritical CO_2 in the microfoamed NC processing and NC/NG system are carried out in our group⁴⁷). Preliminary experimental results showed that the obtained propellant system composed of microporous energetic polymer indicates a significant high burning rate characteristic.

4. Future vision

In propellant chemistry, though GAP, BAMO, AMMO and their copolymers are energetic, they exhibit certain drawbacks like poor processibility, low solid loading, and inferior mechanical properties. In order to overcome these problems, there is a need to develop suitable additives which can act as processing aid, improve solid loading, and also impart additional energy to the formulations. The dendrimer energetic polymer can be as binder additives due to its branched three-dimensional structure, low glass transition temperature T_g and low viscosity. They are able to behave as lubricants during processing and toughening agent in the final blend formulation to improve comprehensive performance of solid propellants.

A new class of first generation azido-terminated dendritic esters have been synthesized by four step processes in India High Energy Materials Research Laboratory⁴⁸). A second-generation [G-2] terminal azido functionalized dendritic ester has been synthesized from a commercially available dendritic polyol BOLTORN H 20⁴⁹). The azidated BOLTORN H 20 has 16 terminal azide groups.

Luo et al.⁵⁰⁻⁵²) reviewed the dendrimer polymer and their application in solid propellants. It showed an application prospective in propellant composition interface interaction and easily process properties.

Over the last few decades, the expected depletion of oil resources and a greater awareness for the environmental impact of plastic products have created a strong interest toward polymers that are not only biodegradable but also obtainable from renewable resources⁵³⁾⁻⁵⁶). Various natural and synthetic biodegradable elastomers have been developed by means of a cross-linked network to satisfy specific mechanical and physical requirements and to improve the product properties.

Cossu et al.⁵⁷) designed and synthesized a biodegradable and energetic copoly (ether/ester-urethane) network from polyepichlorohydrin (PECH) and sebacoyl chloride (SC)

derivatives able to release only nontoxic degradation products and compatible with contemporary ingredients found in propellants and gas generators. They chose thermoset polyurethane elastomers because they can present excellent mechanical properties over a large range of temperature. At this early stage of development, the biodegradability of the synthesized products is evaluated using standardized composting experiments, but the bio/ecotoxicity of the decomposition products is not assessed yet.

Zhao⁵⁸) also reviewed the research progress of green energetic materials. The novel green raw materials mainly include high energy density materials, energetic thermoplastic elastomers and non-toxic catalysts. It represents an important research field in future. Green and safety synthesis will be at the heart of chemistry of energetic binder.

5. Conclusions

Extensive research and novel applications in energetic polymer as binders in propellant and explosives are carrying out. Many emerging energetic polymers are aimed at new application in the propellant development. High energy and insensitivity of energetic polymers are forever goals to be pursued.

With improved mechanic strength and energy level, ETPE can apply to most of solid composite propellants. The results of small-scale experiments of ETPE in high energy insensitive gun propellants showed the application of ETPE increases the energy level with reduction in sensitivity.

The controlled radical polymerization method is introducing to synthesize GAP based TPE to improve its mechanic performance. Moreover, the synthesis of energetic prepolymer can carried out in supercritical CO₂. Supercritical CO₂ technology can also be applied to the processing of energetic materials.

Future development of energetic polymers aims to the green and safety synthesis and the improvement of comprehension performance of propellant system. Biodegradable energetic elastomer will become rapid development research fields in energetic polymers.

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