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Research paper

Composite Propellant Formulation of Poly (16-, 32- and 64-) Azido Dendritic Esters as Energetic Plasticizer and Evaluation of Properties

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Abstract: 16-, 32- and 64-Polyazido hyperbranched dendrimers were synthesized from hydroxy terminated dendritic ester by following two steps namely, tosylation and azidation. The poly azido dendrimers were incorporated in composite propellant formulations as an energetic plasticizer. The physical, thermal sensitivity and ballistic properties of these composite propellants such as burning rate, Cal-val, density, ignition/decomposition temperature (AET), DSC-TGA, mechanical properties, impact and friction sensitivity were evaluated experimentally while the specific impulse (I_{sp}) and characteristic velocity (C^*) were obtained theoretically. A significant enhancement in heat release was noted in the propellant formulation having 16-azido dendritic ester as an energetic plasticizer compared to 32- and 64-azido dendritic esters and a reference composition.

Keywords: azido dendrimers, energetic plasticizer, composite propellant, burning rate

1 Introduction

The propulsive force of rockets, missiles and launch vehicles is accomplished by the combustion of solid propellants resulting in the ejection of hot gases to produce forward thrust of the rocket. Solid propellants comprise a popular class of composite propellant, composed of an oxidizer, a metallic fuel, binder, plasticizer and other additives such as stabilizers, burning rate modifiers, high energy additives, opacifier, flame suppressant, combustion instability suppressants, bonding agents, *etc.* [1-4]. Plasticizers, an important constituent of composite propellants, are usually liquid organic compounds which help in the processing of propellants and improving their mechanical properties. They are classified into two types, *i.e.* inert and energetic plasticizers. Inert plasticizers have been used as processing aids to reduce the viscosity of the propellant slurry and also to enhance the elongation properties of propellants while energetic plasticizers not only enhance the processability but also the energy output.

Recently, energetic plasticizers have received a great deal of attention due to their high thermal stability and energy content as well as improving the mechanical properties and burning behaviour of propellants [5-8]. Azido [9-11], nitrate ester [12], *N*-nitramine [13] and nitro aromatic oxetane based plasticizers are some examples of energetic plasticizers. However, azido ester based plasticizers have drawn more interest due to their better lubricative properties, good compatibility, low vapour pressure, high boiling point, low viscosity, minimum smoke release, thermally safety and better processability [14]. A few examples of azido plasticizers include, *e.g.* [11]:

- ethylene glycol bis(azidoacetate) (EGBAA),
- diethylene glycol-bis(azidoacetate) (DEGBAA),
- trimethylolnitromethanetriazidoacetate (TMNTA),
- pentaerythritol-tetrakis(azidoacetate) (PETKAA),
- low mol. wt. (400-700) glycidyl azide polymer (GAP), and
- an azide terminated glycidyl azide plasticizer (GAPA).

Ghosh *et al.* [15] have incorporated azido ester plasticizers in place of non-energetic plasticizers, *i.e.* dibutyl phthalate (DBP) with 1,3-bis(azidoacetoxymethyl)-2-(azidoacetoxymethyl)-2-ethylpropane (TAAMP) and 1,3-bis(azidoacetoxymethyl)-2,2-bis(azidomethyl)propane (BABAMP) in triple base propellant formulations and reported an enhancement in energy level as well as improved mechanical properties.

The mono, di-azido ester plasticizers have conspicuous drawbacks in terms of possessing low nitrogen content, which means in turn having a low energy content when used in solid rocket propellants. The present requirement

of energetic formulations, to enhance the nitrogen content of energetic plasticizers so as to offer high heat release rates, has motivated the synthesis of energetic dendrimers as energetic plasticizers [16, 17]. Dendrimers are three-dimensional (3D) repetitively branched structures with all branches originating from a central core. They show low glass temperature (T_g) and low viscosity due to the lack of restrictive interchain entanglement in contrast to most linear polymers [18-20]. The dendrimer additives are reported to act as a lubricant during processing and a toughening agent in the final blend of formulation.

This paper reports the incorporation of 16-, 32- and 64-polyazido hyper branched dendrimers in composite propellant formulations as energetic plasticizers. Further, the physical, mechanical, thermal and ballistic properties such as burning rate, Cal-val, density, AET, impact, friction sensitivity, specific impulse (I_{sp}) and characteristic velocity (C^*) of composite propellants were also evaluated experimentally and theoretically.

2 Experimental

2.1 Materials and methods

In the present study, 1% of 16-, 32- and 64- hyper-branched dendritic ester plasticizers were incorporated in separate batches by partly replacing dioctyl adipate (DOA) in the compositions and so various propellant formulations were made accordingly. A reference propellant formulation was also made which had only DOA as an inert plasticizer. The NCO content in the toluene diisocyanate (TDI) is 47.8-48.2% and the ratio of NCO/OH used in the present study is 0.7-1. The details of the compositions are shown in Table 1.

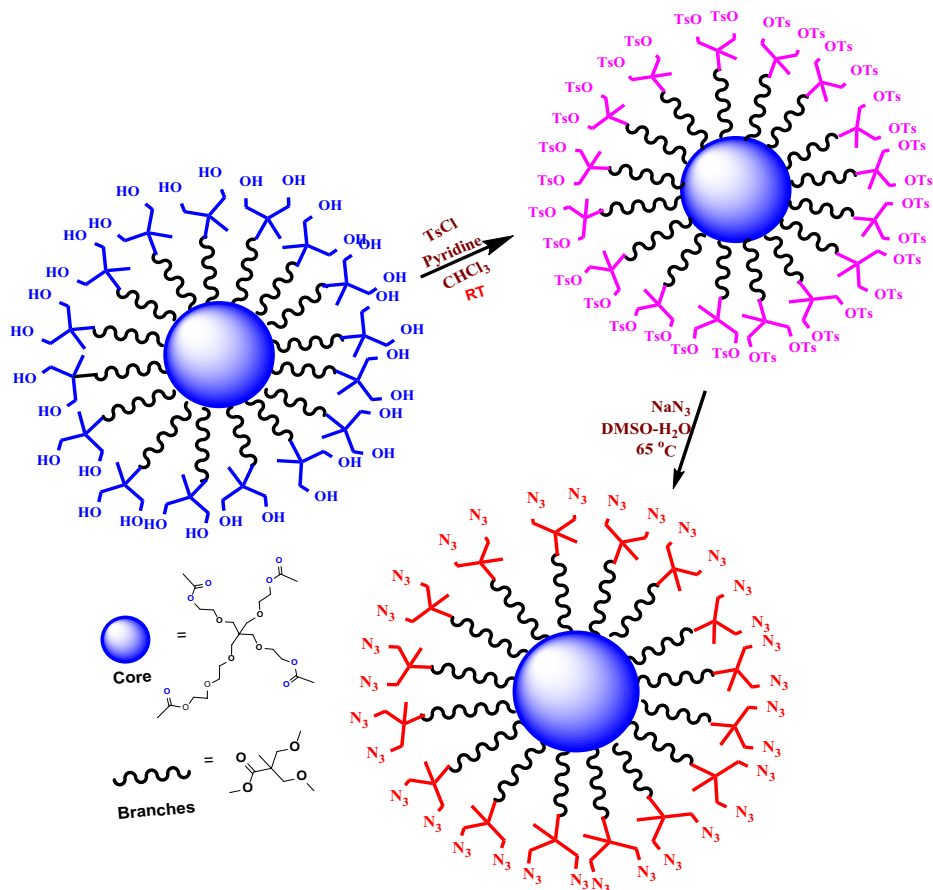
Table 1. Composition details of propellants [26]

Ingredient [%]	Reference composition	Azido Dendritic Ester		
		16-	32-	64-
Ammonium perchlorate (AP)		67		
Aluminum		15		
Hydroxyl-terminated polybutadiene (HTPB)		12		
Cross linking agent		1		
DOA	5	4		
Dendritic Ester	0	1		

The sensitivity to impact was measured by the Fallhammer method using a 2 kg drop weight and the data obtained from the samples analysed in terms of the height for 50% probability of explosion (H_{50}) [22]. The friction sensitivities of the formulations were determined using a Julius Peter apparatus [23]. The tensile strength, E-modulus and percentage elongation were evaluated using a Hounsfield universal testing machine (UTM) using dumbbells conforming to ASTM-D-638 type IV at a cross head speed of $50 \text{ mm}\cdot\text{min}^{-1}$ at ambient temperature. A Gas Pycnometer was used to measure the density of the propellant formulations under helium gas. The calorimetric values of the samples were measured using a Parr isoperibol bomb calorimeter 6200 (in an inert nitrogen atmosphere). The acoustic emission technique was used to determine the burning rates of samples under a nitrogen atmosphere at 7.0 MPa using a 750 cm^3 stainless steel bomb [24]. Acoustic emission refers to the generation of a transient wave by the rapid release of energy within a material. In the acoustic emission technique, an acoustic sensor converts the mechanical energy carried by the elastic wave into an electrical signal. The automatic explosion test (AET) was determined using an automatic explosion tester (model No. AET-402, 02M, Czech Republic) by placing 0.02 g of sample in a glass test tube using a rose metal's bath at a heating rate of $5 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$. The temperature at which the sample ignited/decomposed was recorded [25]. DSC studies were performed using a Perkin-Elmer, Pyris DSC-7 with an ultra high pressure (UHP) nitrogen atmosphere with a flow rate of $50 \text{ mL}\cdot\text{min}^{-1}$ and a heating rate of $10 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$. TGA was carried out using a Perkin Elmer STA 6000 in nitrogen atmosphere at a heating rate of $10 \text{ }^\circ\text{C}\cdot\text{min}^{-1}$.

2.2 Synthesis of 16-, 32- and 64-polyazido dendritic ester

The synthesis of poly azido dendritic ester from poly hydroxy dendritic ester has been reported earlier as a two step process (Scheme 1) [21]. At the outset, poly hydroxy dendritic ester has been treated with tosyl chloride in the presence of chloroform and pyridine for 48 h at room temperature. The reaction mixture containing the product was washed with water and extracted in dichloromethane and concentrated under reduced vapour pressure.



Scheme 1. Synthetic scheme of poly(16-, 32- and 64-)azido dendritic esters [22]

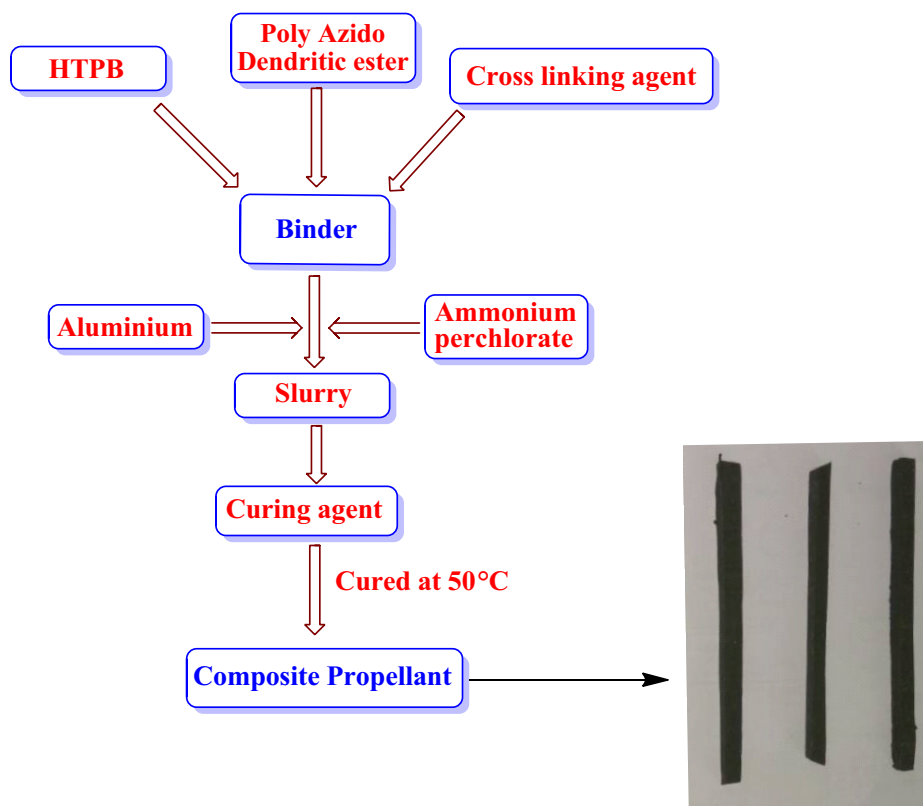
The crude product was de-tosylated using sodium azide in DMSO at 65°C for 72 h. Thereafter, the reaction mixture was poured into cold water and extracted in dichloromethane, washed 2-3 times resulting in a yield obtained of $\sim 80\%$. The product was analyzed by IR, ^1H NMR and confirmed by CHN analysis. By repeating this process, 16-, 32- and 64-polyazido dendritic esters were scaled up to ~ 15 g and then utilized individually in composite propellant formulations as energetic plasticizers.

2.3 Preparation of composite solid propellants

The preparation of composite propellants involves the mixing of fuel, oxidizer cross-linking agents and other ingredients followed by casting of the resulting slurry into the mould and heating under vacuum for solidification. All the ingredients were of analytical grade and used without any further purification.

2.4 Procedure

Prepolymer resins consisting of hydroxyl-terminated polybutadiene (HTPB), DOA with one of 16-, 32- or 64-azido dendritic ester as a plasticizer along with the antioxidant 2-phenyl naphthylamine (Nonox-D) and a crosslinking agent (a 2:1 mixture of 1,4-butanediol and 1,1,1-trimethylol propane (TMP)) except curative were well mixed for 30 min in a vertical planetary mixer (500 mL). After this, these ingredients were mixed under vacuum for another 30 min to drive out entrapped air. Then Al powder ($\sim 15 \mu\text{m}$) was added to the mixture. After the Al powder had been completely added, mixing was resumed for another 20 min. Then bimodal AP (particle sizes 300 and 50 μm) was added and mixed in such a way that homogenous mixing took place. The overall mixing temperature was maintained at $40 \pm 2 \text{ }^\circ\text{C}$. After addition of all the solid ingredients, mixing was continued under vacuum for 30 min. At this point, toluene di-isocyanate (TDI) was added and mixed in for another 40 min. The composition was then cast into 100 mm inner diameter (ID) moulds using the vacuum casting technique and cured at $50 \text{ }^\circ\text{C}$ for 5 days. By using the above method, three different composite propellant formulations were made with 16, 32 and 64 azido dendritic esters as energetic plasticizers. Subsequently, the propellant moulds were cooled and trimmed into specific sizes which were then tested to evaluate different properties. The flow diagram for the preparation of composite solid propellant is depicted under Scheme 2.



Scheme 2. Flow diagram for the preparation of propellant

The HTPB used in the above procedure falls into the category of a functionally terminated or telechelic polymer. A large number of hydroxyl telechelic polymers are known, but HTPB is one of the most versatile, cost effective, exotic in application and particularly suitable as a fuel binder in solid propellants as it has excellent properties for use in networks as a matrix resin with elastomeric behaviour. It is less viscous in nature and contains a proportion of the hydroxyl KOH in the range of 40-42 $\text{mg}\cdot\text{g}^{-1}$. The telechelic hydroxyl groups react with NCO of TDI to provide structural strength to the propellant grain.

3 Results and Discussion

3.1 Physical properties of composite propellant formulations

After solidification, various physical properties of the propellants such as density, sensitivity and mechanical properties were evaluated. It was observed that the density of the propellants having 16-, 32- and 64-azido dendritic ester were higher than that of the reference (see Table 2). This may be due to the higher densities of the poly azido dendritic esters compared to DOA. The data presented in Table 2 also show that the impact and friction sensitivities were enhanced compared to the reference propellant due to the increase in the number of azido groups in the composition. The data on mechanical properties given in Table 2 show that partly replacing DOA with increasing numbered azido groups (*i.e.* 16-, 32- and 64-) results in an increase in the tensile strength and elastic modulus of the compositions, while the percentage elongation decreases. Thus, the value of the tensile strength and E-modulus for compositions containing 16-, 32- and 64- azido dendritic esters increases from 0.66 and 4.3 to 0.77 and 4.9 MPa, respectively compared to 0.54 and 3.5 MPa for the reference composition. However, the opposite trend was obtained in the case of percentage elongation of the compositions studied. This may be due to enhancement of the interstitial forces created by azido groups

Table 2. Physical properties of composite propellants (16-, 32- and 64-azido dendritic esters and reference propellants)

Composite propellants	Density [g·cm ⁻³]	Impact H_{50} [cm]	Sensitivity [N]	Mechanical Properties		
				T.S. [MPa]	E-mod [MPa]	%E
16 Azido	1702	55	160	0.66	4.3	38
32 Azido	1702	52.5	128	0.70	4.6	37
64 Azido	1703	47.5	84	0.77	4.9	35
Reference	1697	60	182	0.54	3.5	41

T.S. – Tensile strength, E-mod – E- modulus, %E – percentage of elongation

3.2 Ballistic characteristic

After analyzing the physical properties of the propellant compositions studied, the ballistic properties such as burning rate [27, 28], Cal-val [29] and auto explosion temperature (AET) were also determined. The data obtained are presented in Table 3. The data show that the burning rate, AET and Cal-val were on the lower side compared to the reference counterpart due to the higher nitrogen content in azido dendritic esters. The theoretical specific impulse

of the compositions studied showed a marginally higher value for the I_{sp} of about 1 s compared to the reference composition. The opposite trend was observed in the case of the characteristic velocity (C^*). This may be due to high nitrogen containing propellants releasing high nitrogen gases during combustion thus reducing the heat of the propellant [30].

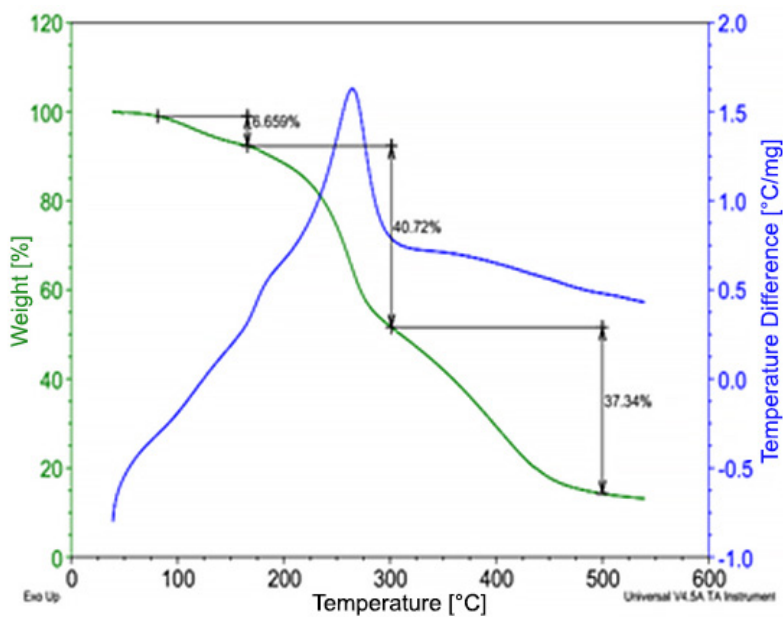
Table 3. Studies of 16-, 32- and 64-azido dendritic esters and reference propellant by experimental and theoretical methods

Composite propellants	Experimental ^a			Theoretical ^a		
	Burning rate at 7 MPa, [mm·s ⁻¹]	Cal-val [cal·g ⁻¹]	AET [°C]	I_{sp} [s]	C^* [m·s ⁻¹]	OB [%]
16 -Azido	4.905	1415	347.55	261.867	1545.34	-132.79
32 -Azido	4.895	1396	326.07	261.867	1550.46	-131.57
64 -Azido	4.72	1379	320.45	261.510	1556.02	-130.70
Reference	5.02	1450	365.41	260.5	1562.04	–

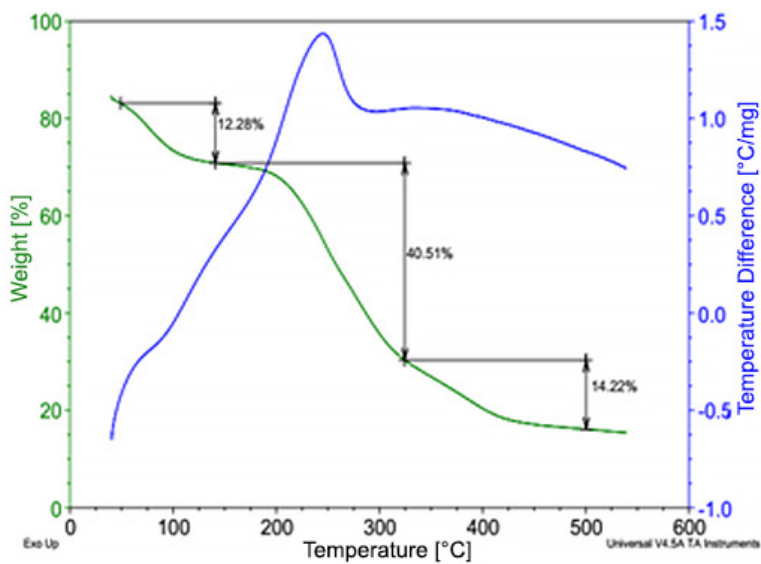
^a Cal-val – Calorimetric value, AET – Auto explosive temperature, I_{sp} – Specific impulse, C^* – Characteristic velocity

3.3 Thermal properties of hyper branched dendritic esters

The thermal behaviour of synthesized 16-, 32- and 64-azido dendritic esters were studied using DSC/TGA and the data obtained is presented in Figures 1(a)-1(c), respectively. It is clear from the figure that a single broad exotherm peak was observed in the temperature range 245-248 °C for 16- and 32-azido dendritic esters whereas in the same temperature range a sharp exotherm peak was observed for 64-azido dendritic esters. This may be due to the presence of a higher number of $-N_3$ groups in the molecules where thermal decomposition took place instantly. The DSC study further revealed that the heat released by the studied azido dendritic esters are in decreasing order, *i.e.* high in the case of 16-azido ($-1476 \text{ J}\cdot\text{g}^{-1}$) followed by 32-azido ($-1126 \text{ J}\cdot\text{g}^{-1}$) and 64-azido dendritic esters ($-778 \text{ J}\cdot\text{g}^{-1}$), respectively indicating higher energy content in the case of 16-azido dendritic esters (Table 4).



(a)



(b)

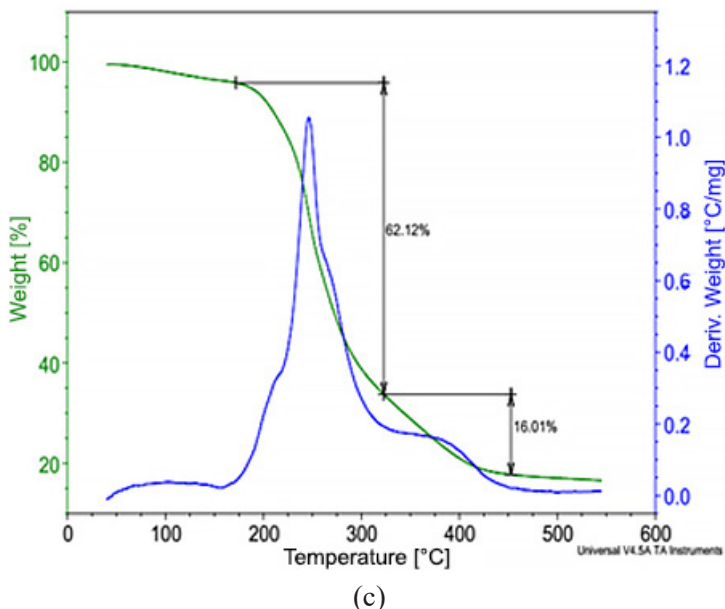


Figure 1. DSC and TGA of 16- (a), 32- (b) and 64- (c) azido dendritic esters

Table 4. Thermal and physical properties of pure 16-, 32- and 64-azido dendritic esters

Azido dendritic ester compound	Formula	DSC [°C]	ΔH [$J \cdot g^{-1}$]	Carbon [%]	Nitrogen [%]	Density [$g \cdot cm^{-3}$]
16-Azido	$C_{75}H_{112}N_{48}O_{29}$	245	-1476	41.90	31.27	1250
32-Azido	$C_{155}H_{224}N_{96}O_{61}$	246	-1126	42.23	30.50	1260
64-Azido	$C_{315}H_{448}N_{192}O_{125}$	248	-778	42.40	30.14	1270

The TGA analysis (Figure 1(a)) revealed that 16-azido dendritic ester demonstrated a weight loss which started at a temperature of about ~ 50 to ~ 150 °C which accounted for about 6.65 weight percentage loss and beyond 150 to 300 °C the weight loss was 40.72% followed by a 7.34% weight loss in the temperature range of 300 to 500 °C. On the other hand 32-azido dendritic esters exhibited a weight loss which also started at ~ 50 to ~ 150 °C which accounted for about 12.28 weight percentage loss and 40.51% weight loss which was observed in the temperature range of 320-500 °C. However, the weight loss pattern in the case of 64-azido dendritic esters was found to start at 150 °C which accounted for 62.12% up to 320 °C and a weight loss of 16.01% was observed in the temperature range of 320 to 450 °C. The above findings revealed that

16-azido dendritic ester is more thermally stable in terms of weight loss compared to 32- and 64-azido dendritic esters.

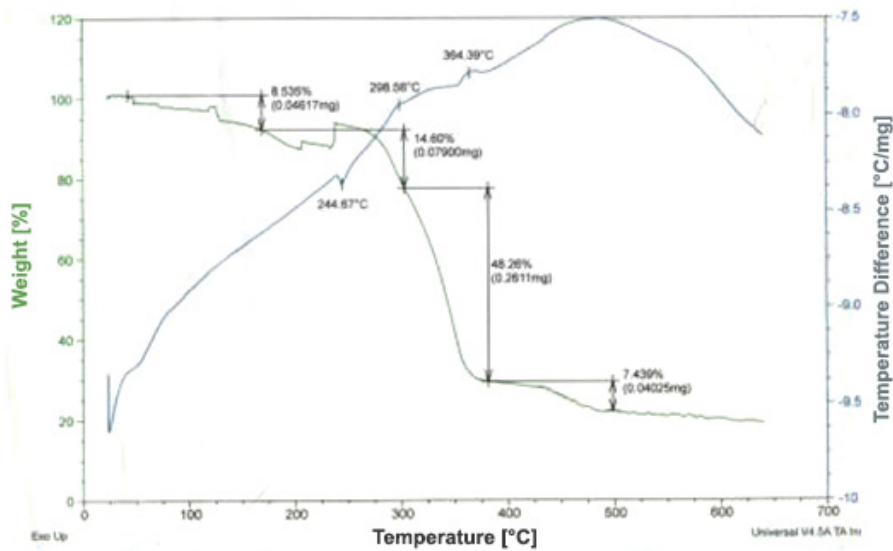
3.4 Thermal properties of composite propellant formulations

The thermal properties of 16-, 32- and 64-azido dendritic esters incorporated in propellant compositions by partly replacing DOA were also studied using DSC/TGA and the data is presented in Figure 2 and Table 5. It is clear from Figure 2 that there is a sharp endotherm at 243-244 °C indicating the phase change of AP from orthorhombic to cubic. Further, two small exotherm peaks were also observed in all the studied cases, *i.e.* 297 and 368 °C, indicating the thermal decomposition of AP.

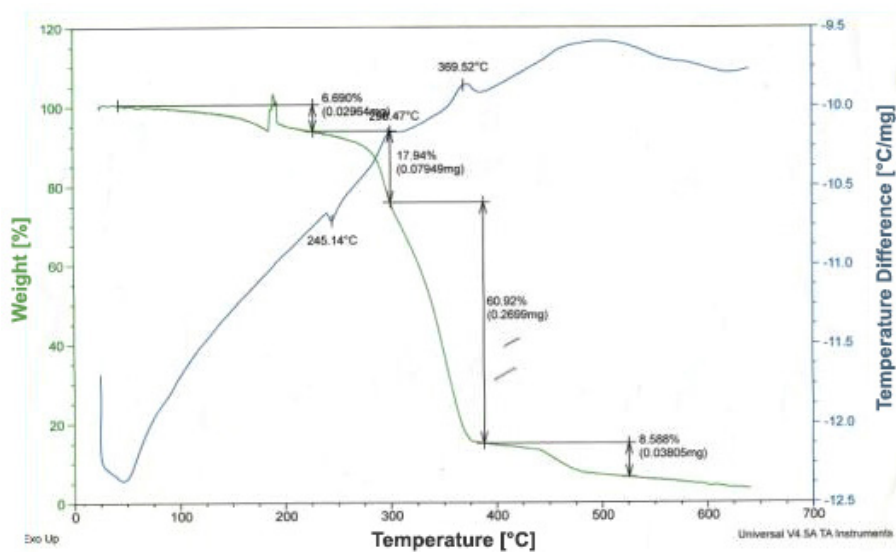
A TGA study of the reference propellant composition revealed that the weight loss initiated at a temperature of 2 °C which accounted for 5.84% up to 240 °C followed by a 2.51% weight loss in the temperature range of 240-300 °C. Further, a 60.52% weight loss was observed in the temperature range 300-370 °C followed by a 4.7% weight loss in the temperature range of 372-615 °C. The same loss pattern was observed in all the studied compositions containing 16-, 32- and 64-azido dendritic esters except in the 4th step where the maximum temperature range was in between 380-565 °C inferring that decomposition of the composition containing dendritic esters completed at a lower temperature compared to the reference composition due to their energetic ($-N_3$) groups.

Table 5. Thermal properties of composite propellants having 16-, 32- and 64-azido dendritic esters and reference propellant

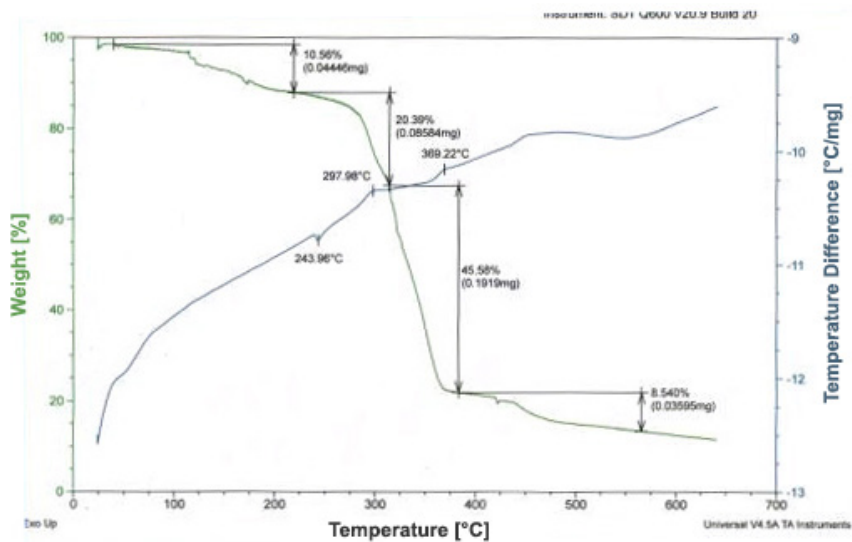
Composite propellant	TGA, in step								DSC		
	1 st		2 nd		3 rd		4 th		1 st peak	2 nd peak	ΔH
	[°C]	[wt.%]	[°C]	[wt.%]	[°C]	[wt.%]	[°C]	[wt.%]	[°C]	[°C]	[J·g ⁻¹]
16-azido	50-170	5.53	170-305	14.60	305-380	48.26	380-500	7.49	244	364	388.3
32-azido	50-227	6.69	227-300	17.94	301-385	60.92	385-525	8.56	245	369	249.7
64-azido	52-225	10.56	225-320	20.39	320-382	45.58	382-565	8.54	243	369	230.6
Reference	52-240	5.84	240-302	12.51	302-372	60.52	372-615	4.70	245	337	186.4



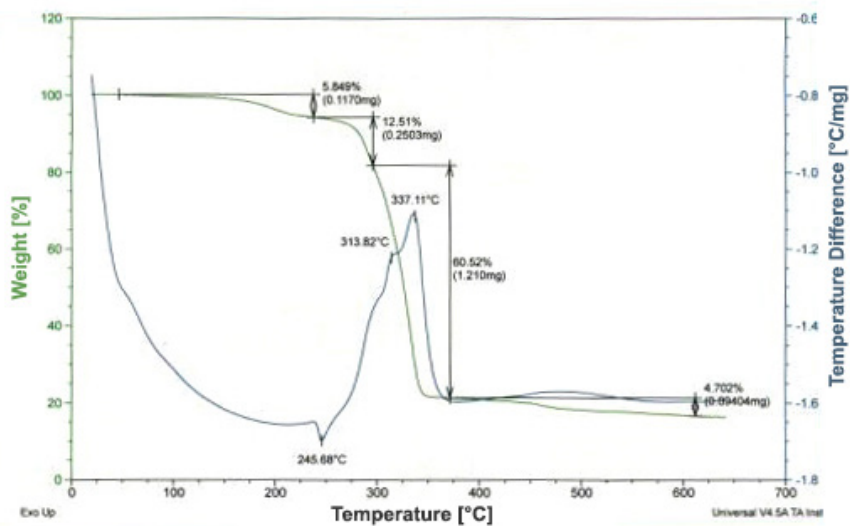
(a)



(b)



(c)



(d)

Figure 2. DSC and TGA of 16, 32, 64-azido dendritic ester as plasticizer in propellant ((a)-(c), respectively) and reference propellant (d)

4 Conclusions

A successful attempt has been made to incorporate three energetic plasticizers (16-, 32- and 64-azido dendritic esters) in propellant formulations and to evaluate their different properties. The results revealed a significant increase in the heat release in propellant formulations containing 16-azido dendritic ester ($388.3 \text{ J}\cdot\text{g}^{-1}$) compared to the reference propellant ($186.4 \text{ J}\cdot\text{g}^{-1}$). Properties such as burning rate, Cal-val, oxygen balance, impact, friction and ignition/decomposition temperature (AET) were observed to decrease with an increase in the azido groups whereas the density, mechanical properties and theoretical I_{sp} increased. Based on the above results of three azido dendritic propellants, 16-azido dendritic azido ester demonstrated better ballistic properties compared to 32- and 64-azido dendritic esters.

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