

Article

Thermal Decomposition of Ammonium Dinitramide (ADN) as Green Energy Source for Space Propulsion

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Abstract: The thermal decomposition of an ammonium dinitramide-based energetic compound was conducted for the first time using a dispersive inductively coupled plasma mass spectrometer, DTA-TG analysis, and pyrolysis at a constant temperature. A liquid droplet was injected over synthesized CuO catalytic particles deposited on lanthanum oxide-doped alumina. The thermal behavior of the ADN liquid monopropellant revealed that decomposition in the presence of catalytic particles occurs in two distinct steps, with the majority of ejected gases being detected in real-time analysis using the DIP-MS technique. At a temperature of 280 °C, pyrolysis confirmed the catalytic decomposition behavior of ADN, which occurred in two distinct steps.

Keywords: thermal decomposition; catalyst; propellant; mass spectrometer; real-time gas analysis



Citation: Harimech, Z.; Toshtay, K.; Atamanov, M.; Azat, S.; Amrousse, R. Thermal Decomposition of Ammonium Dinitramide (ADN) as Green Energy Source for Space Propulsion. *Aerospace* **2023**, *10*, 832. <https://doi.org/10.3390/aerospace10100832>

Academic Editor: Angelo Cervone

Received: 6 September 2023

Revised: 23 September 2023

Accepted: 24 September 2023

Published: 25 September 2023



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1. Introduction

The demand for satellites has increased significantly due to the heavy requirements and rapid development of aerospace technologies. In the past decades, conventional propellants, such as hydrazine, have been widely used for attitude and reaction control systems (RCAs) in satellites [1–3]. However, hydrazine propellants pose significant risks to both humans and the environment due to their high toxicity. Therefore, there is a pressing need to develop and replace them with eco-friendly (green) alternatives [4,5].

One promising eco-friendly option is ammonium dinitramide (ADN)-based liquid propellant, which is a blend of ADN [NH₄N(NO₂)₂], methanol, and water. This high-performance green propellant offers a potential solution for replacing hydrazine [6–9]. In recent years, numerous studies have invested in the research and development of ADN-based green propellant thrusters, with a focus on their environmental and eco-friendly benefits. Notably, both Sweden and China have conducted and successfully completed on-orbit tests of ADN-based thrusters, demonstrating their feasibility and effectiveness in space applications.

Typically, ADN-based thrusters rely on a preheating catalytic ignition process. Several studies have examined the operation of ADN-based thrusters using the catalytic ignition method [10–16]. These studies have highlighted advantages, such as high ignition reliability and pulse adaptability. However, there are also notable limitations, as described below:

- The burning temperature of ADN-based liquid propellant exceeds 1800 K, negatively impacting catalyst activity and lifespan at such high temperatures.
- ADN-based thrusters cannot be cold-started because the catalytic bed must be heated. Therefore, there is a need to develop an active ignition technology to replace the catalytic ignition method.
- ADN-based liquid propellants are ionic solutions with high electrical conductivity, making them suitable for ignition through the resistive ignition method. This method

relies on the thermal energy generated from the inherent resistance of the propellant. Notably, an electrical ignition device for ADN droplets has been developed successfully for combustion. Research has investigated the impact of on-load voltage on the combustion characteristics of ADN-based liquid propellants [17,18]. It was found that the evaporation, decomposition, and combustion durations differed from those of other liquid droplets [19–21]. This difference can be attributed to the liquid blend of ADN [$\text{NH}_4\text{N}(\text{NO}_2)_2$], methanol, and water in the propellant. When the circuit is connected, methanol and water (solvents) vaporize, initiating ADN decomposition into NO due to the inherent resistance-induced thermal effect of the droplet. Subsequently, methanol reacts with NO. Overall, while the catalytic ignition method offers advantages, the limitations associated with high temperatures and cold starts necessitate the development of active ignition technologies, such as the resistive ignition method, for ADN-based thrusters. These advancements are crucial for optimizing the performance and reliability of such thrusters in aerospace applications.

The ignition delay time for ADN-based liquid propellants was notably extended. Throughout the ignition and combustion phases, the diameter of the ADN-based liquid droplet exhibited significant variations, distinguishing its combustion behavior from that of most other liquid droplets. To provide some context, when considering a diesel droplet, its diameter decreased with relatively minor fluctuations during the combustion process [19]. In contrast, a methanol droplet exhibited a linear decrease in size during the ignition and combustion stages, which was influenced by varying oxygen levels in the environment. Meanwhile, for petrodiesel and acetylene back mixture droplets, the droplet diameter increased slightly during the heating period and decreased linearly during the combustion period [21].

Furthermore, previous studies have indicated that different combustion phenomena occur when ADN-based liquid propellants ignite in different oxidizing atmospheres. Notably, ADN-based propellants exhibited a more vigorous reaction in a N_2O atmosphere compared to ignition in air, leading to distinct differences in droplet development. In practical applications, ADN-based liquid propellants are ignited in environments devoid of oxidizing gases. During the ADN decomposition process, small molecule oxidizing gases, such as N_2O , NO, and O_2 , are generated [21].

This paper investigates the thermal decomposition behavior of ADN-based monopropellant in the presence of copper particles as a catalyst. The study utilizes a DTA-TG apparatus and DIP-MS analysis technique for analysis. Additionally, the cupric oxide particles are subjected to characterization using various physicochemical techniques.

2. Materials and Methods

2.1. Catalyst Preparation

We utilized alumina doped with lanthanum oxide (La_2O_3) as the catalyst support, synthesized through a sol-gel procedure. The process commenced with the preparation of a colloidal solution at room temperature. Nitric acid was introduced to expedite the dissolution of solid phases and to control the solution's pH. This solution included Disperal boehmite (Sasol, $S_{\text{BET}} = 260 \text{ m}^2 \text{ g}^{-1}$) and urea ($\text{OC}(\text{NH}_2)_2$).

Urea, in the form of white pellets, was added to the beaker containing nitric acid. To ensure proper dispersion of the urea, high-speed mixing at 6200 rpm was maintained for 5 min. Subsequently, boehmite, as fine white powdered crystallites, was gradually added while subjecting the mixture to high-speed mixing of approximately 23,000 rpm for 20 min using an Ultra Turax T25 mixer.

For the lanthanum nitrate hexahydrate $\text{La}(\text{NO}_3)_3$ precursor (Wako, 99%), it was dissolved in a suitable solution of pure ethanol as the solvent. The lanthanum concentration was fixed at 10 wt.% of La_2O_3 .

Furthermore, the doped-colloidal suspension was subjected to thermal treatment at 500 °C in an air environment. This step aimed to create a doped-porous layer, thus enhancing the dispersion of the metal or oxide active phases. The high surface area of the

doped-alumina support is considered crucial for maintaining good catalytic performance in high-temperature applications.

To introduce the CuO active phase onto the calcined support, impregnation was carried out using an excess of water as the solvent. This involved the use of an aqueous solution of copper (II) nitrate trihydrate $\text{Cu}(\text{NO}_3)_2$ (Wako, 99.9%). The $\text{Cu}(\text{NO}_3)_2$ was dried at room temperature for 24 h, followed by an additional drying step at 100 °C for 12 h. Finally, it was treated at 500 °C in an air environment for 8 h [7].

2.2. Catalyst Characterization

2.2.1. Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)

The experimental loading of 10 wt.% CuO on La_2O_3 -doped alumina was determined by using ICP-OES (Inductively Coupled Plasma Optical Emission Spectroscopy) after dissolving the solid catalyst particles. The dissolution was achieved by treating the catalyst with a mixture of nitric acid and hydrochloric acid. The analysis was conducted using the iCAP 6000 apparatus manufactured by Thermo Fisher Scientific in Bremen, Germany. The ICP-OES analysis technique is highly versatile, offering both qualitative and quantitative insights into the elemental composition of substances.

2.2.2. N_2 -Physisorption (BET)

Textural properties, including specific surface area and pore volume, were determined using the Brunauer, Emmett, and Teller (BET) method. These measurements were conducted utilizing a Micromeritics FlowSorb II apparatus under the following conditions: (i) 2 h pretreatment at 250 °C under a nitrogen flow and (ii) nitrogen adsorption in helium with a nitrogen pressure set at 0.3 bar. Indeed, this experimental setup was instrumental in precisely characterizing the textural properties of the catalyst. The BET theory strives to elucidate the physical adsorption of gas molecules onto a solid surface, forming the fundamental framework for a significant analytical method used to determine the textural properties of materials, including surface area, pore volume, pore size, etc.

2.2.3. X-ray Diffraction (XRD)

The CuO-based catalyst was characterized using X-ray diffraction (XRD). Approximately 0.5 g of finely ground powder particles was carefully placed in a standard sample holder. In this arrangement, the crystallites were oriented randomly, and there was no preferred direction of diffraction. The XRD pattern was recorded over a range of 2θ from 5 to 80 degrees, with a step size of 0.06 degrees and an acquisition time of 2 s for each data point.

2.2.4. Transmission Electron Microscopy (TEM)

A transmission electron microscope (TEM), specifically the Philips CM120 Cryo-Electron Microscope, is utilized in conjunction with high-resolution, cooled digital cameras. The catalysts undergo milling, followed by immersion in an ethanol solution. Subsequently, they are dispersed using ultrasound and deposited onto a copper grid before undergoing carbonization.

2.3. Thermal Decomposition of ADN-Based Monopropellant

The thermal decomposition of ADN-based monopropellant, as an environmentally friendly approach, is conducted using three distinct techniques: (i) The classical DTA-TG method is employed under atmospheric pressure conditions. This experiment serves to determine the onset decomposition temperature and to assess the influence of catalytic particles on the thermal behavior of ADN decomposition. (ii) The DIP-MS technique is utilized to decompose ADN at a high heating rate (128 °C min^{-1}), a pioneering approach. (iii) The pyrolysis technique is employed to facilitate the thermal decomposition process with a rapid increase in temperature.

2.3.1. DTA-TG Analysis

We employed a TA Instrument Q600 device to conduct differential thermal analysis and thermogravimetric (DTA-TG) experiments for the decomposition of ADN-based monopropellant. To ensure the precision of our analysis and prevent any potential damage to the equipment, we utilized a minimal quantity of CuO-powdered particles, which were placed within an aluminum sample holder. Additionally, a 2 μ L ADN solution was carefully injected into the holder using a syringe. Subsequently, the system was sealed with an aluminum cap and securely fastened.

During the experiment, we recorded the weight loss and temperature as a function of time. The binary {solution + catalyst} system was subjected to a temperature ramp of 10 $^{\circ}$ C per minute while being maintained in an argon flow (50 mL min⁻¹). Our DTA-TG analysis provided us with the following key information: (i) The onset temperature of decomposition, determined by locating the inflection point on the temperature curve. (ii) The concentration of the ADN solution at the point of decomposition. (iii) The efficiency of the catalyst in influencing the decomposition process. It can be noticed that TG/DTA is a concurrent thermal analyzer capable of assessing various thermal properties of a sample during a single experiment. The TG component gauges temperatures at which decomposition, reduction, or oxidation processes take place. Simultaneously, it tracks alterations in weight linked to decomposition, oxidation, or any other physical or chemical transformations that lead to changes in the sample's weight.

2.3.2. DIP-MS Technique

The DIP-MS L-250G-I A, manufactured by Canon Anelva Co., Ltd. (Kawasaki, Japan), is employed for the examination and advanced thermal analysis of ADN-based samples, with a mass composition of ADN and water. This analytical technique involves introducing the sample into the ionization chamber, followed by its vaporization and subsequent ionization through electronic influence.

The DIP-MS device is regarded as the fastest tool available for real-time gas product identification during thermal decomposition. Notably, it allows for temperature escalation from 40 $^{\circ}$ C to 450 $^{\circ}$ C, employing a higher-temperature ramp of approximately 128 $^{\circ}$ C per minute. This approach is groundbreaking, as it has never been previously reported for ADN-based propellants.

The probe temperature program was configured to increase from room temperature to 450 $^{\circ}$ C at a rapid temperature ramp of 128 $^{\circ}$ C per minute. During the analyses, we employed an ionization voltage of 70 eV, maintained a source temperature of 250 $^{\circ}$ C, and examined a mass range spanning from $m/z = 17$ to 100. We conducted scans at a rate of 2 scans/s, ensuring a vacuum level of superior quality, better than 7×10^{-5} mbar.

2.3.3. Pyrolysis

The pyrolysis of the sample following the procedure (Figure 1) is carried out using the JCI-22 pyrolyzer instrument, developed by Japan Analytical Industry Co., Ltd. (JAI, Tokyo, Japan). This process involves utilizing pyrofoils that are set to a temperature of 280 $^{\circ}$ C. The objective is to rapidly achieve this temperature within a short duration of 3 to 5 ms, as demonstrated by Souagh et al. [22].

In this method, we employed the Curie point-portable injector, a cutting-edge Curie point pyrolyzer continually striving for enhanced usability. It stands as the world's inaugural portable pyrolyzer, designed to inject solid samples, liquid samples, outgas, or VOC samples into GC/MS using the same procedure as a micro syringe injection. Additionally, its washable/disposable sample tube and needle ensure a consistently clean flow line.

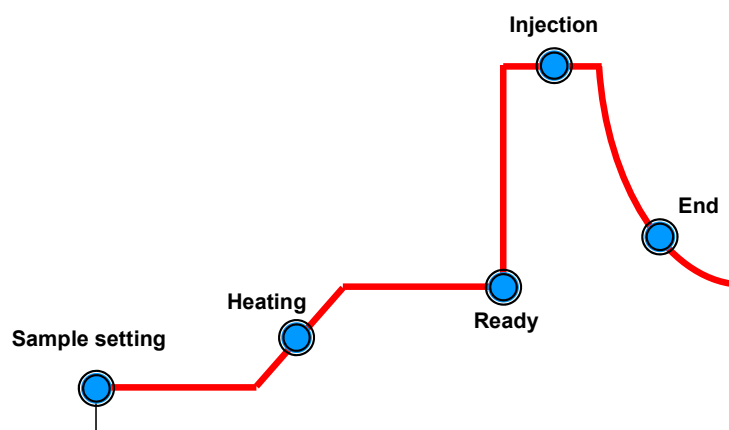


Figure 1. Pyrolysis procedure.

3. Results and Discussion

3.1. Catalyst Characterization

The loading of CuO particles, deposited via the impregnation method, was determined using the ICP-OES analytical technique. The experimental value obtained was 9.8 wt.% of CuO oxide. This value aligns with the desired target due to the thermal treatments and potential experimental errors during processes, such as washing and measurement.

The impregnation of doped-alumina with La_2O_3 using CuO crystallites has a minimal impact on its textural properties. Specifically, the BET surface area (S_{BET}) remains close to the value obtained for the support before impregnation, with $S_{\text{BET}} = 252 \text{ m}^2 \text{ g}^{-1}$ (as shown in Table 1), compared to the original value of $S_{\text{BET}} = 260 \text{ m}^2 \text{ g}^{-1}$. This suggests that the specific surface area of the CuO-based catalyst remains largely unchanged from that of the initial support. This phenomenon may be explained by the fact that the impregnation and heat treatment processes do not significantly alter the texture of the alumina, and the active phase particles contribute to the overall surface area of the catalysts.

Table 1. N_2 -physorption of CuO-based catalyst (BET).

	Doped-Alumina	CuO-Based Catalyst
Surface area ($\text{m}^2 \text{ g}^{-1}$)	260	252
Pore volume ($\text{cm}^3 \text{ g}^{-1}$)	32	15

Furthermore, it is observed that the pore volume decreases compared to that of the support after the incorporation of the active phase. In this case, the blocking or occupation of the porous material by CuO crystallites results in the reduction of the pore volume. This decrease is attributed to the partial filling of the pores and the saturation of the support catalyst by the salt precursor during the impregnation process.

Additionally, the broadening of the diffraction peaks corresponding to CuO on the doped-alumina support is clearly evident in Figure 2. The presence of amorphous phases and larger patterns with low intensities suggests that the CuO crystal particles are highly dispersed on the doped-alumina support, resulting in smaller sizes and crystallite forms. This observation leads us to conclude that nano-sized crystallites are deposited, which can be associated with a higher density of crystal lattice defects. Such defects are generally considered to be highly active sites for catalytic processes.

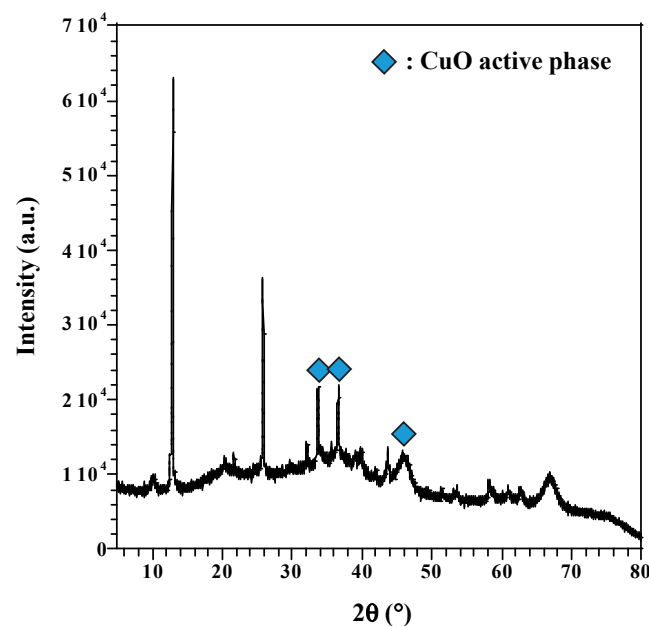


Figure 2. X-ray diffraction of CuO-based catalyst.

The size of the CuO crystallites was determined using the Debye–Scherrer equation, which yields a value of approximately 22 nm. This size determination is based on the analysis of three characteristic patterns of CuO crystallites, and it is noteworthy that there is no interference or disturbance from the support phase, indicating that the CuO crystallites are well-dispersed and maintain their distinct characteristics.

Figure 3 displays a TEM micrograph of the CuO-based catalyst. This image reveals that CuO particles exhibit heterogeneous distribution, primarily because of the limited interaction between the oxide active phase and the doped support. Specifically, some nano-sized particles are found to be embedded within the porous doped-alumina. However, owing to the relatively weak interaction between the metal and support, CuO crystallites are also observed to be deposited on the surface. It is worth noting that a detailed investigation of CuO adsorption on La₂O₃-alumina will be thoroughly explored in a separate publication.

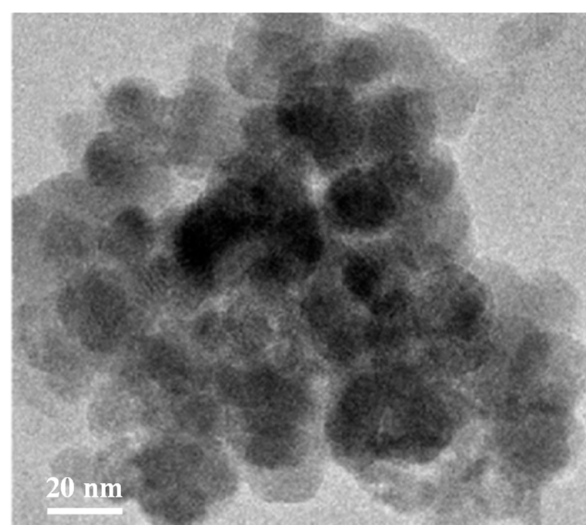


Figure 3. TEM micrograph of CuO particles.

Furthermore, various factors have been identified as influencing the particle sizes of CuO in the catalyst. These factors include the preparation method of the support, the

heat treatment temperatures, and the gas flow rates. While the histogram of the particle size distribution of the CuO-based catalyst is not presented here, it is worth noting that a number of relatively large CuO particles were measured. The presence of these larger particles contributes to an average particle size of approximately 20 nm for CuO, which aligns with the crystallite size ($d = 22$ nm) determined from the XRD patterns.

Importantly, active components with smaller particle sizes tend to exhibit higher catalytic activity. Therefore, the presence of nano-sized CuO particles increases the catalyst's surface area and provides a high density of surface-active sites. This, in turn, enhances the catalytic decomposition of ADN liquid monopropellant, making it a promising avenue for improving the catalytic performance of the system.

3.2. Thermal Decomposition of ADN-Based Monopropellant

3.2.1. DTA-TG Analysis

The catalytic decomposition of ADN liquid monopropellant over (10% CuO)-based catalysts was initially monitored through thermal analysis. Specifically, 2 μ L of ADN liquid was analyzed in the presence of 3 mg of the CuO-based catalyst, as depicted in Figure 4.

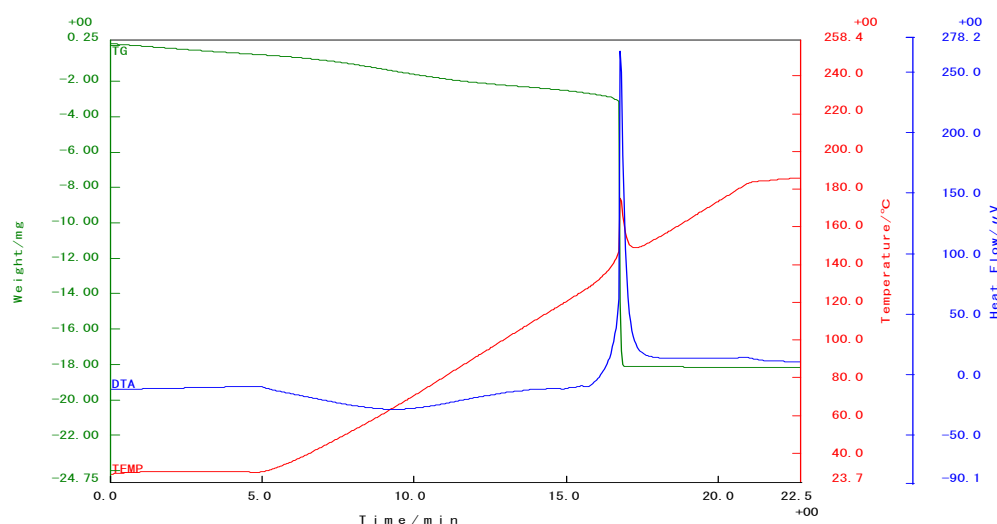


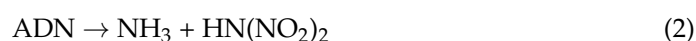
Figure 4. DTA-TG thermogram of ADN-based liquid monopropellant over CuO particles.

It is worth noting that the weight loss observed at the conclusion of the reaction process can be attributed to the significant exothermic nature of the decomposition. This exothermic reaction can result in a small amount of catalyst being expelled from the sample holder. In fact, a prominent exothermic peak is observed, representing a rapid decomposition process, followed by a smaller endothermic peak. This endothermic peak indicates a relatively low energy release towards the end of the process, likely due to the presence of water as one of the decomposition products.

3.2.2. DIP-MS Technique

In this experiment, the decomposition of ADN liquid monopropellant was conducted using a 10%-CuO-based catalyst, and the results obtained are presented in Figure 5.

It is evident from the data that the temperature jump observed when ADN is heated in the presence of CuO particles suggests that the decomposition of ADN may occur in two distinct zones. This finding aligns with previous research conducted by Mebel et al. (Equations (1) and (2)) in 1995 [23]:



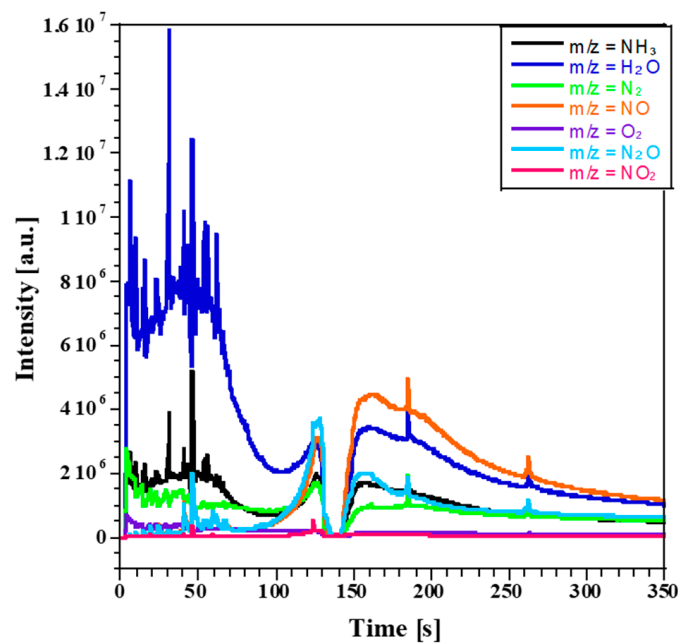
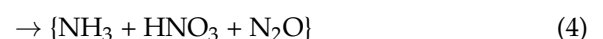


Figure 5. Online analysis via DIP-MS technique of ADN thermal decomposition over CuO particles.

During the ADN decomposition process, several stable products have been identified, including ammonia (NH_3), NO_2 , NO , N_2O , HNO_3 , and water vapor as major products. Additionally, there is the presence of NH_4NO_3 , as well as trace amounts of nitrogen and oxygen. The mechanism of thermal decomposition for this propellant and the specific pathways leading to the formation of these products, predominantly governed by radical species like H , OH , NO , and NH_x (where $x \leq 2$), remain unclear, as indicated by previous studies [23].

In light of the thermograph results, the primary thermal decomposition steps of ADN can be outlined as follows:

- The ADN decomposition begins with its dissociation into ammonia (NH_3) and hydrogen dinitramide ($\text{HN}(\text{NO}_2)_2$) as stable species. This behavior is typical of ammonium salts.
- Subsequently, the thermal dissociation of $\text{HN}(\text{NO}_2)_2$ leads to the production of N_2O and nitric acid (HNO_3) in an exothermic process.
- Once present, nitric acid and ammonia quickly recombine to form ammonium nitrate (NH_4NO_3 , AN) as a stable species.
- Ammonium nitrate (AN) decomposes at higher temperatures in the second step of ADN decomposition, yielding N_2O and H_2O . This mechanism is in line with the findings of Löbbecke et al. [24], who developed a detailed understanding of this highly energetic process. The mechanism can be described by the following equations (Equations (3)–(6)) [24]:



In addition to the major species previously described, minor entities have been observed and detected during the thermal decomposition of ADN liquid propellant. Real-time mass spectrometry analysis has confirmed the formation of elementary species, such as NO , O_2 , and N_2 .

Given the significance of replacing hydrazine with ADN-based monopropellant, gaining a comprehensive understanding of its thermal behavior, despite its complexity and challenges, is crucial. Therefore, in addition to the exothermic reaction of hydrogen dinitramide to produce N_2O and HNO , it appears that the oxidation of NH_3 by NO_2 is also responsible for the exothermic nature of ADN decomposition, as previously reported [24].

Furthermore, the vaporization profile of NO_2 as a stable gas supports the idea of its vapor-phase reaction, as it demonstrates a decrease in NO_2 intensity at higher temperatures [25]. This observation lends further insight into the complex mechanisms at play during the ADN decomposition process.

3.2.3. Pyrolysis

For the pyrolysis of ADN monopropellant over the CuO catalyst, we employed aluminum pyrofoils specifically designed for this instrumentation. The pyrolysis was carried out at a temperature of $T = 280^\circ\text{C}$. During this process, both {ADN + CuO crystals} were rapidly heated to the desired temperature within a mere 3 ms, reflecting extremely rapid temperature rise conditions. This experimental setup enabled us to investigate the behavior of the binary solution/catalyst system under both standard and challenging conditions. The resulting data are presented in Figure 6.

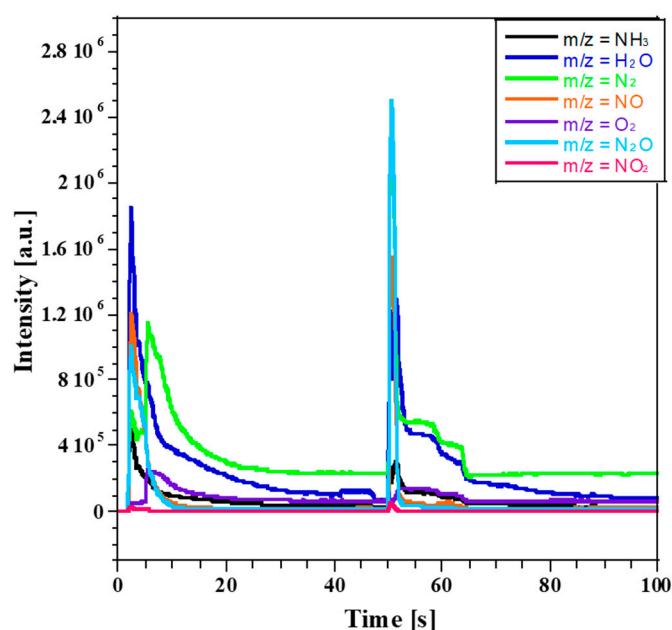


Figure 6. Online analysis via pyrolysis process of ADN thermal decomposition over CuO particles.

Figure 6 illustrates that the decomposition of ADN likely occurs in two distinct phases, consistent with the thermal decomposition behavior observed at the high heating rate of 128°C per minute conducted by DIP-MS. This decomposition process is characterized by two sequential steps in the gas phase. The major gases detected during this process include ammonia, N_2 , N_2O , NO_2 , and water vapor.

The decomposition initiates with the vaporization of solvents, which is an endothermic process, and the pure ADN solutions decompose subsequently, in the best-case scenario. It is noteworthy that the decomposition process of the liquid propellant typically begins before the complete evaporation of water. As the temperature rises, the dissociation of water into radicals becomes favorable, potentially leading to detonation phenomena.

In summary, through these analytical techniques, it can be inferred that ADN liquid monopropellant undergoes a two-step decomposition process over copper oxide crystals. The gas-phase products and the underlying decomposition mechanism have also been investigated and discussed.

4. Conclusions

In this paper, we investigated the thermal decomposition of ADN-based liquid, which serves as an environmentally friendly propellant for satellite attitude control and in-space operations. Our study employed a multi-pronged approach:

- DTA-TG Thermal Analysis: We conducted DTA-TG thermal analysis to elucidate the thermal behavior and determine the onset temperature of the decomposition process.
- DIP-MS Online Analysis: To understand the nature of the gas species released during the thermal decomposition, we utilized DIP-MS online analysis. This allowed us to monitor and identify the gases produced in real time.
- Pyrolysis at Constant Temperature: We subjected the ADN liquid to pyrolysis at a constant high temperature to examine its behavior under extreme thermal conditions.

Additionally, we prepared and characterized CuO particles using physicochemical techniques. These CuO particles exhibited promising performance in facilitating the thermal decomposition of this environmentally friendly monopropellant. Overall, our study contributes to the understanding and potential applications of ADN-based liquid propellants for space operations.

Author Contributions: Conceptualization, R.A. and Z.H.; software, R.A.; resources, R.A.; writing—original draft preparation, R.A. and Z.H.; writing—review and editing, R.A., K.T., M.A. and S.A. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: We declare that the data and materials of this paper are available.

Acknowledgments: The authors express their gratitude to Kobe Material Testing Laboratory, located in Ueno, Tokyo, Japan, for their valuable assistance in conducting the DIP-MS and pyrolysis experiments. Their support has been instrumental in the successful execution of this research.

Conflicts of Interest: The authors declare no conflict of interest.

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