RESEARCH PAPER

Investigation of the Thermal Behavior of Ammonium Dinitramide (ADN) with Metal-Organic Framework, MOF-199 as a Copper-Based Catalyst

Reza Kazemshoar-Duzduzani, Ali Mousaviaza*, Mohammad Jafari, Kamal Ghani

Faculty of Applied sciences, Malek- Ashtar University of Technology, Shahin Shahr, Iran

ARTICLE INFO

Article History:
Received 02 May 2026
Accepted 15 August 2026
Published 01 October 2026

Keywords:
Ammonium dinitramide
Decomposition temperature
Metal-organic frameworks
MOF-199
Thermal decomposition behaviour

ABSTRACT

Ammonium dinitramide (ADN), due to its high oxygen balance, high energy content, and halogen-free combustion products, is a promising new oxidizer for automotive airbags and solid propellants. In this work, we have utilized MOF-199, a well-known copper-based metal-organic framework (MOF), to study the thermal decomposition behaviour of ADN. Different compositions of ADN/MOF-199, *i.e.*, 98:2 (2%), 95:5 (5%), 92.5:7.5 (7.5%), and 90:10 (10%) were prepared by the solvent method and their thermal behaviour has been studied by DSC. The FT-IR and XRD methods were used to investigate the interactions of ADN and MOF-199. Moreover, the dispersion of particles was checked by the FE-SEM/EDX. Our results indicate that MOF-199, despite of its low weight, improves the thermal decomposition behaviour of ADN significantly through Lewis/Bronsted acid sites and porous structure. In addition, DSC studies showed a sharp exothermic peak for ADN/MOF-199 (10%) composite related to the rapid catalytic combustion of the composite Accordingly, MOF-199 can be introduced as an excellent catalyst for modifying the combustion of ADN.

How to cite this article
Duzduzani R., Mousaviaza A., Jafari M., Ghani K. Investigation of the Thermal Behavior of Ammonium Dinitramide (ADN) with Metal-organic Framework, MOF-199 as a Copper-Based Catalyst. J Nanostruct, 2026; 16(4):5095-5106. DOI: 10.22052/JNS.2026.04.050

INTRODUCTION

Understanding the decomposition behavior of composite solid propellants is of significant interest. Nowadays, ammonium perchlorate (AP) is extensively used in composite solid propellants as an oxidizer [1, 2]. AP has the highest effective oxygen content among the oxidizers, i.e., its oxygen balance is up to +34%. However, since AP contains Cl, the combustion of AP-based propellants generates significant quantities of HCl as a reaction product [3], which is a toxic gas and its emission is an environmental concern. Moreover, the HCl produce a large amount of smoke in the rocket's tail [4]. In order to prevent environmental pollution as a universal concern [5, 6], a new green oxidizer as a possible replacement for ammonium perchlorate (AP) has been investigated in recent years. ADN is a potential replacement for ammonium perchlorate [7, 8] or ammonium nitrate (AN) [9]. The main physical properties of ADN are summarized in Table 1.

ADN is a metal-free energetic compound with the chemical formula of $NH_4N(NO_2)_2$ and is composed of ammonium cation (NH_4^+) and the dinitramide anion ($N(NO_2)_2^-$) [10]. It has a high oxygen balance (+25.8%) [11, 12], which is used

* Corresponding Author Email: Kamal.ghani@gmail.com

in automotive airbags, military explosives and smokeless solid propellants or green propellants [13-15]. Moreover, the formation enthalpy, gas production, and combustion heat of ADN are higher than that of AP, and its decomposition temperature (160 °C) is lower than that of AP (400 °C) [16]. ADN has some disadvantages, e.g., the thermal decomposition of ADN is done in two stages and a wide temperature range [17]. The melting of ADN occurs at 93 °C, and two peaks at 155–210 °C (exothermic peak) and 210–230 °C (endothermic peak) relate to the decomposition of AN, which is a product of ADN decomposition [18]. The mechanism of the ADN thermal decomposition is as [19, 20]:

$$NH_4N(NO_2)_2 \rightarrow NH_3 + HN(NO_2)_2$$

$$HN(NO_2)_2 \rightarrow HNO_3 + N_2O$$

$$NH_3 + HNO_3 \rightarrow NH_4NO_3$$

$$NH_3 + HN(NO_2)_2 \rightarrow NH_3 + HNO_3 + N_2O \rightarrow NH_4NO_3 + N_2O$$

$$NH_4NO_3 \rightarrow N_2O + 2H_2O$$

Xin Li et al. [21] used two high-energy coordination polymers (CPs) $Cu_2(DNBT)_2(CH_3OH)(H_2O)_3{\cdot}3H_2O$ (1) and $[Cu_3(DDT)_2(H_2O)_2]_n$ (2) (H_2DNBT = 3,3′-dinitro-5,5′-bis(1H-1,2,4-triazole and H_3DDT = 4,5-bis(1H-tetrazol-5-yl)-2H1,2,3-triazole) as effective catalysts to improve the thermal decomposition of ADN. Yajin L et al. [22] used copper-series catalysts, including copper chromate ($CuCrO_4$), copper chromite ($Cu_2Cr_2O_5$), copper 2,4 dihydroxybenzoate (*β*-Cu), and 3D energetic MOFs: MOF-Cu, ($[Cu(atrz)_3]$ $(NO_3)_2)_n$, (atrz = 4,4-azo-1,2,4-triazole) to improve the thermal behavior of ammonium dinitramide (ADN). High-energy metal-organic frameworks (E-MOFs) are used extensively as a thermal decomposition catalyst for AP [23, 24]. Zakaria et al. [25] used Carbon nanotubes supported by Ir-CuO as catalysts to enhance the thermal decomposition of ammonium dinitramide (ADN). Kazemshoar-Duzduzani et al. [26] Studied Zinc Oxide (ZnO) with Different Morphologies as an effective Catalytic on the Thermal Decomposition of Ammonium Dinitramide (ADN). Amrousse et al. [27] studied CuO as an effective catalyst to improve the thermal decomposition of ADN, and their results show that CuO plays a vital catalytic role in thermal decomposition of ADN. In order to regulate the burning rates of composite solid propellants, some catalysts are added to their composition so introducing new combustion catalysts, for controlling the thermal decomposition of ADN is essential.

Metal-organic frameworks (MOFs) are a new class of crystalline microporous materials obtained by the covalent linkages between transition metals and organic ligands [28]. To date, a variety of approaches for making the MOFs has been offered and employed like slow diffusion [29, 30], hydrothermal (solvothermal) [31, 32], electrochemical [33, 34], mechanochemical [35-37], microwave-assisted [38-40] heating and ultrasonic approach [41-43]. Metal-organic frameworks (MOFs) due to their fantastic attributes such as high surface area, adjustable pore size, flexibility, and structural diversity [44, 45], good heat stability, and good mechanical hardness and strength [46], and application in gas storage, separation, and sensing [47, 48], drug delivery systems, energy storage like supercapacitors and energy conversion [49], photovoltaic [50, 51], electrochemical application, photocatalysis [50, 52], and combustion catalysis in composite

Table 1. main physical properties of AND.

Property		Ref
Molecular formula	$N_4H_4O_4$	
Appearance	white crystal	
Molecular weight /g mol^{-1}	124.07	
Density/g cm^{-3}	1.82–1.84	[64]
Melting point/ °C	91–93	[65]
Heat of formation/kJ mol^{-1}	-148	[66]
Solubility in water (20 °C)/wt%	78.1	[67]

solid propellants [53] gained much attention. In addition, they are currently gaining attention due to their biomedical applications or use as sensor materials [54]. 1, 3, 5- benzene tricarboxylic acid (BTC) is a well-known ligand that has been used for synthesizing the Cu^{2+} and Fe^{2+} based MOFs

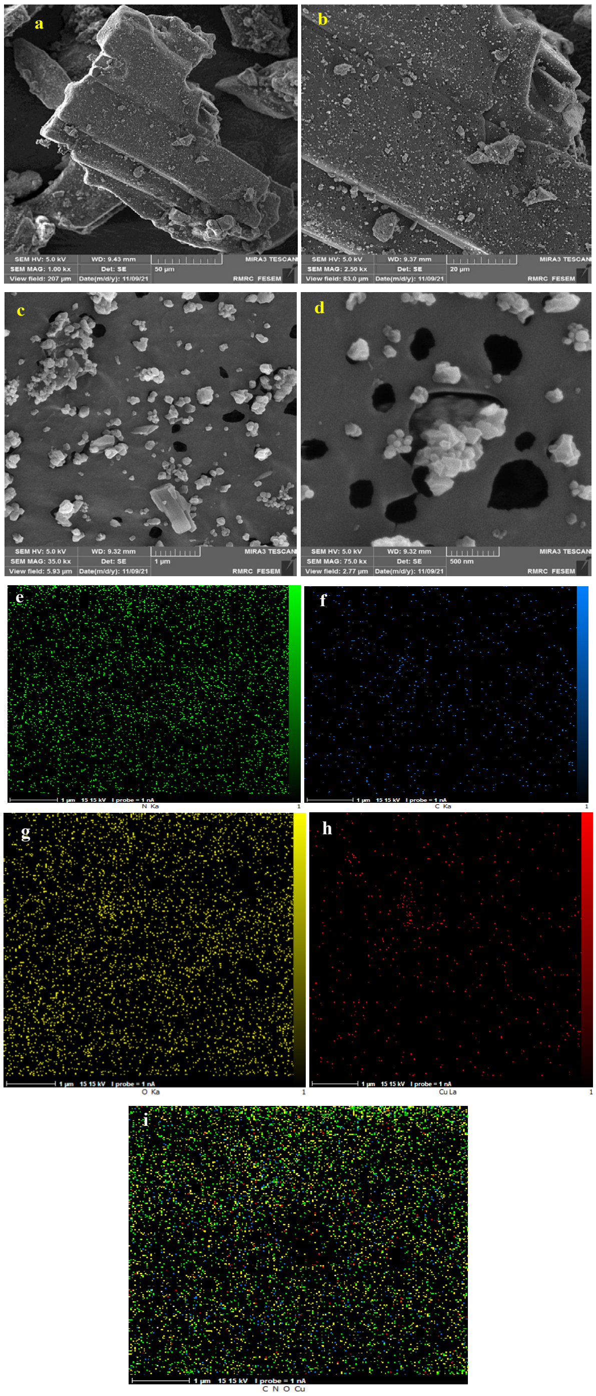

Fig. 1. a-d) FE-SEM image of ADN/MOF-199 10% composite, and e-i) EDX elemental map showing the dispensation of Cu^{2+} ions in the ADN/MOF-199 10% composite (e (N), f (C), g(O), h (Cu^{2+}), and i (N, C, O, and Cu^{2+}).

in order to the phase stabilizing of AN [55] and thermal decomposition of AP [56], respectively. Cu-BTC, i.e., MOF-199 has a high surface area, good thermal stability

This work represents the first example of using a metal-organic framework as an active catalyst in

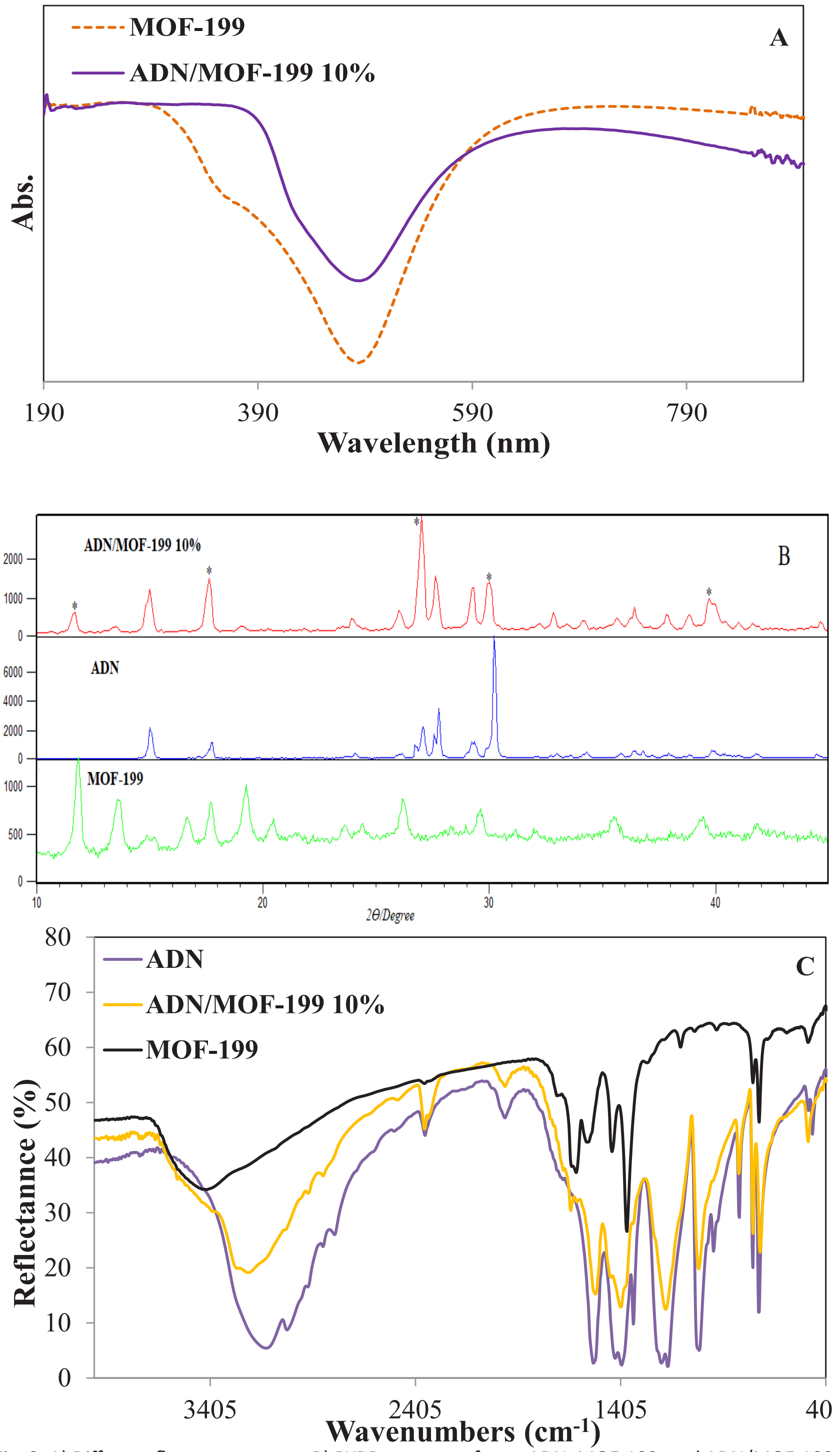

Fig. 2. A) Diffuse reflectance spectra, B) PXRD patterns of neat ADN, MOF-199, and ADN/MOF-199 composite, and (C) FT-IR spectra of neat ADN, MOF-199, and ADN/MOF-199

ameliorate the thermal decomposition behaviour of ADN. For this intention we have chosen MOF-199 as a copper-based MOF because it possesses high surface area, good thermal stability, and an attractive coordination mode [57]. The 3D system of MOF-199 comprises of dimeric cupric paddle wheel nodes coordinated by eight oxygen's, coming from benzene-1, 3, 5-tricarboxylate linkers. Each metal completes its octahedral coordination sphere with an axial ligand (solvent) opposite the Cu-Cu vector [58]. Therefore, it is expected that high specific area and dispersed metallic sites could ameliorate the thermal behavior of ADN.

MATERIALS AND METHODS

All raw materials and solvents were analytical grade, which was obtained from Merck Chemical Co. MOF-199 was synthesized similarly to formerly reported procedures [55]. Then ADN/MOF composites were prepared through a solution-based method with ADN to MOF mass ratios of 0.98:0.02 (2%), 0.95:0.05 (5%), 0.925:0.075 (7.5%), and 0.90: 0.10 (10%) by blending 1 g of the ADN and MOF in 20 ml of acetonitrile. The solvents were evaporated in an open cup at room temperature. The samples were dried at 50 °C for 12 h in a vacuum oven. Differential scanning calorimetry (DSC) was performed for 5 mg of samples by Mettler Toledo (DSC1) with alumina pans an open cell under Ar atmosphere at the heating rate of 5°C min^{-1}. X-ray diffraction (XRD) patterns were taken by an Asenware diffractometer (AW-XDM300) equipped with Cu kα radiation (λ=1.5406Å) using a step size of 0.05° and a counting time of 1s per step. Moreover, the samples were characterized with a field emission scanning electron microscope (FE-SEM) and energy-dispersive X-ray (EDX) spectroscopy using an EDX-equipped MIRA 3-XMU with gold coating. UV–Vis diffuse reflectance (DRS) measurements were obtained using a JASCO (V 670) spectrophotometer. An Infralum FT-08 spectrometer has been used to record the infrared spectrum (from KBr pellets) in the range 400–4000 cm^{-1}.

RESULTS AND DISCUSSION

Several evaluations would be done to prove the influence of MOF-199 in the improves the thermal decomposition behaviour of ADN. As revealed by the FE-SEM, ADN was located in the surface and pores of MOF-199 (Fig. 1 (a- d)). As shown in EDX elemental mapping (Fig. 1 e-i), Cu^{2+} ions were distributed almost uniformly within the composite.

The diffuse reflectance spectrum (Fig. 2A) showed a hypochromic alteration for both d–d transition and ligand to metal charge transfer (LMCT) bands, attributable to the change in the symmetry around octahedral Cu^{2+} ions due to the adsorbed $N(NO_2)_2^-$ and NH_4^+ ions [59, 60]. On the other hand, the XRD patterns taken from ADN, MOF-199, and ADN/MOF-199 composite (Fig. 2B) reveal the presence of $[Cu(NH_3)_2]N(NO_2)_2$ in the sample. As shown, five new peaks appeared in ADN/MOF-199, which indicates different patterns compared to ADN. In this case, peaks labeled by hashtag (*) are identified as the characteristic peaks of $[Cu(NH_3)_2]N(NO_2)_2$, indicating the coordination Cu^{2+} with ammonium ion NH_4^+ and the dinitramide $N(NO_2)_2^-$ (17). The FT-IR spectra (Fig. 2C) were used to study the intermolecular interactions in composites of ADN with MOF-199. As can be seen in (Fig. 2C), when MOF-199 was added, the IR spectrum of the composite exhibited some differences, which may be due to the interactions of Lewis/Bronsted acid sites of MOF-199 with ADN. The main spectral distinction between these results appeared in the wavelength ranges of 2900-3500 cm^{-1} and 949-1643 cm^{-1}. Those peaks in the spectral range of 2900-3500 cm^{-1} are due to the vibrations of NH_4^+. Another change was the appearance of two absorption peaks at 1423 and 1315 cm^{-1} in the composite. Obtaining a single crystal of ADN/MOF-199, due to the low solubility of MOF-199 in organic solvents, was not possible. Also, the interactions between ADN and MOF-199 in the ADN/MOF-199 cannot be studied by solid-state NMR spectroscopy.

DSC Study thermal behaviour of ADN, ADN/MOF-199

To prevail the problems of thermal decomposition and facilitate the practical use of ADN in the gas generator systems, different percentages of ADN/MOF-199 were prepared and their thermal decomposition behaviour have been investigated.

DSC was used to assess the thermal decomposition behaviour of the ADN and ADN/MOF-199 composites [61, 62]. Fig. 3 shows the DSC curves for ADN under the Ar atmosphere. In the pure ADN, the endothermic peak, which is observed at 92.87 °C related to its melting point and other two major peaks, which were related to the thermal decomposition of ADN appeared in 155–210 °C and 210–230 °C ranges. The third peak, in the range of 210-230 °C, is related to the decomposition of AN (17).

According to the DSC curves of ADN/MOF-199 (2, 5, 7.5, and 10%) in Fig. 4-8, two peaks were seen. The first peaks (endothermic peaks) are related to the melting of the sample; the maximum melting temperature for the samples of 2, 5, 7.5, and 10% are equal to 93.03, 92.99, 93.15 and 93.23 °C, respectively. Accordingly, the melting points of all ADN/MOF-199 samples are very close to the melting point of pure ADN, which is 92.87 °C. The second peaks (exothermic peaks) are related to the sample decomposition.

The maximum decomposition peaks of 2, 5,

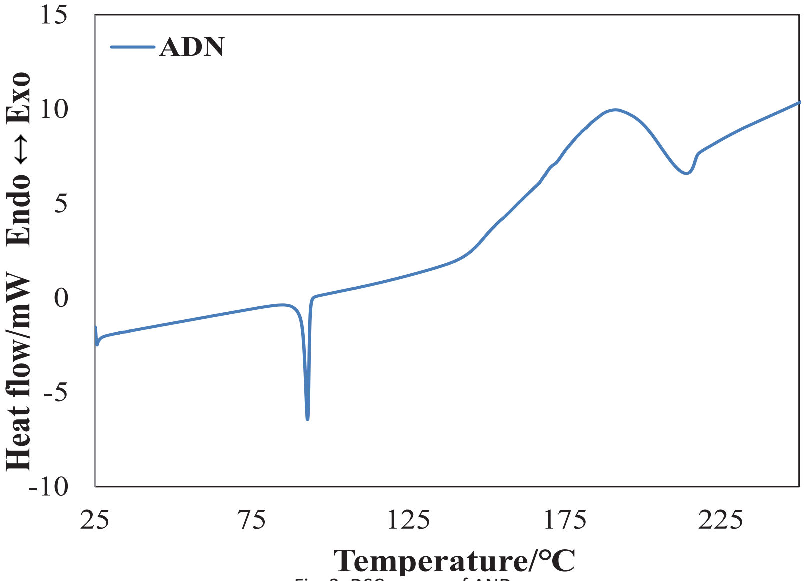

Fig. 3. DSC curves of AND.

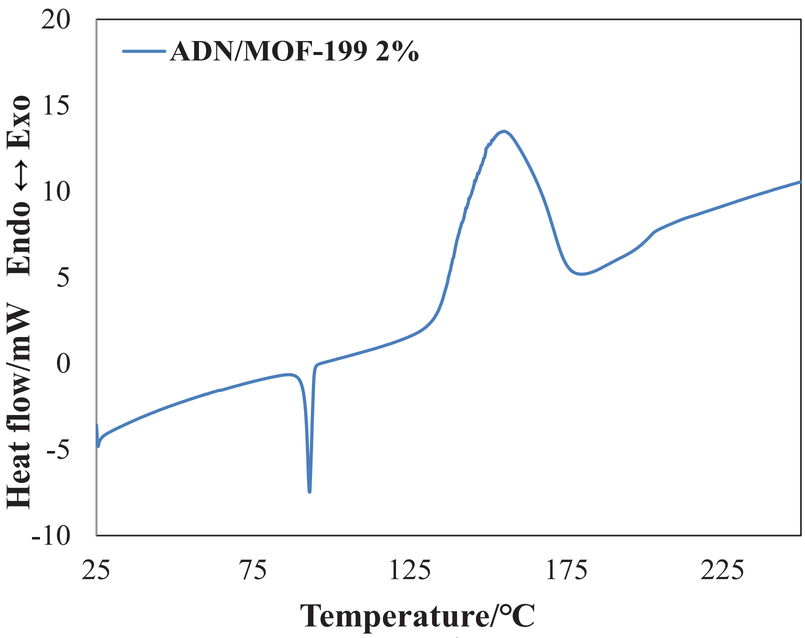

Fig. 4. DSC curves of ADN/MOF-199 2%.

7.5, and 10% samples were appeared in 154.96, 154.69, 152.33, and 146.92 °C, respectively. In 2, 5, and 7.5% samples, the decomposition temperatures reduced by the same amounts from 192 to about 152.33-154.96 °C. However, in 10% sample, the decomposition temperature reduced

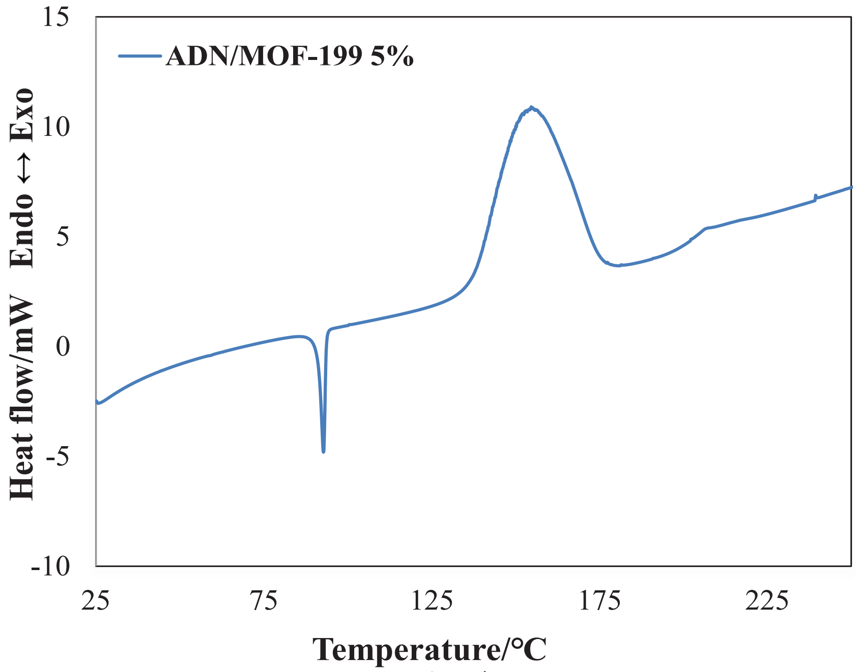

Fig. 5. DSC curves of ADN/MOF-199 5%.

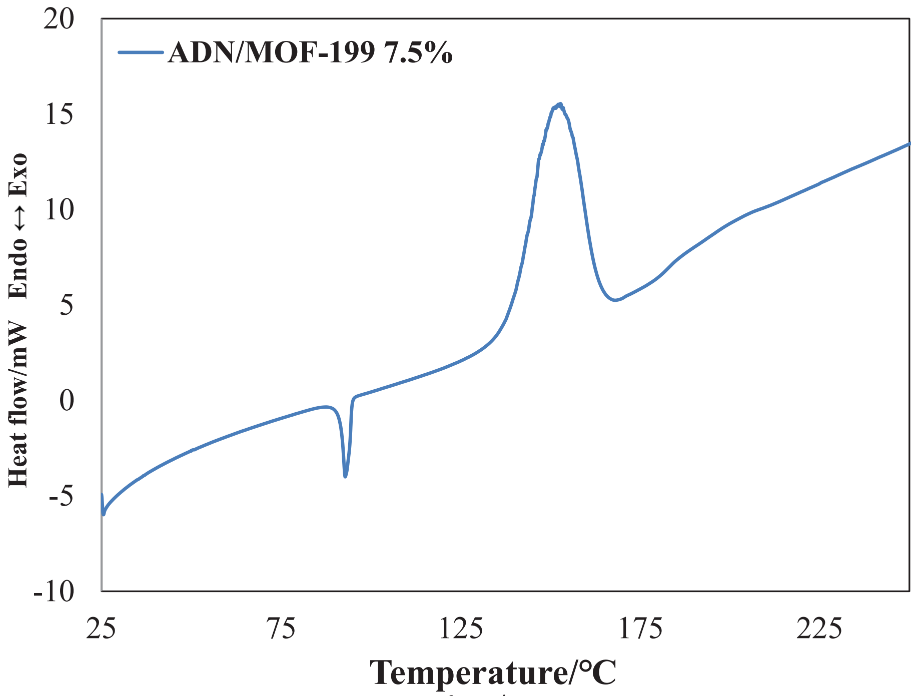

Fig. 6. DSC curves of ADN/MOF-199 7.5%.

to 146.92 °C, which is a significant difference from the other samples. Therefore, the decomposition of ADN/MOF-199 samples was done in much lower temperature ranges and in shorter times.

Moreover, in the 10% sample, the decomposition peak is explosively very sharp.

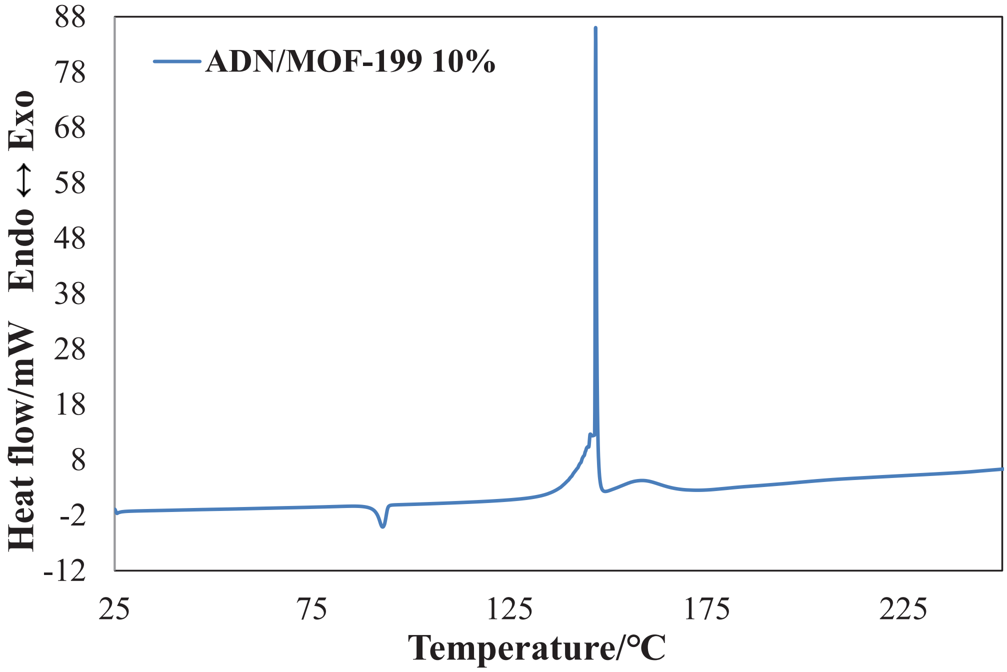

Fig. 7. DSC curves of ADN/MOF-199 10%.

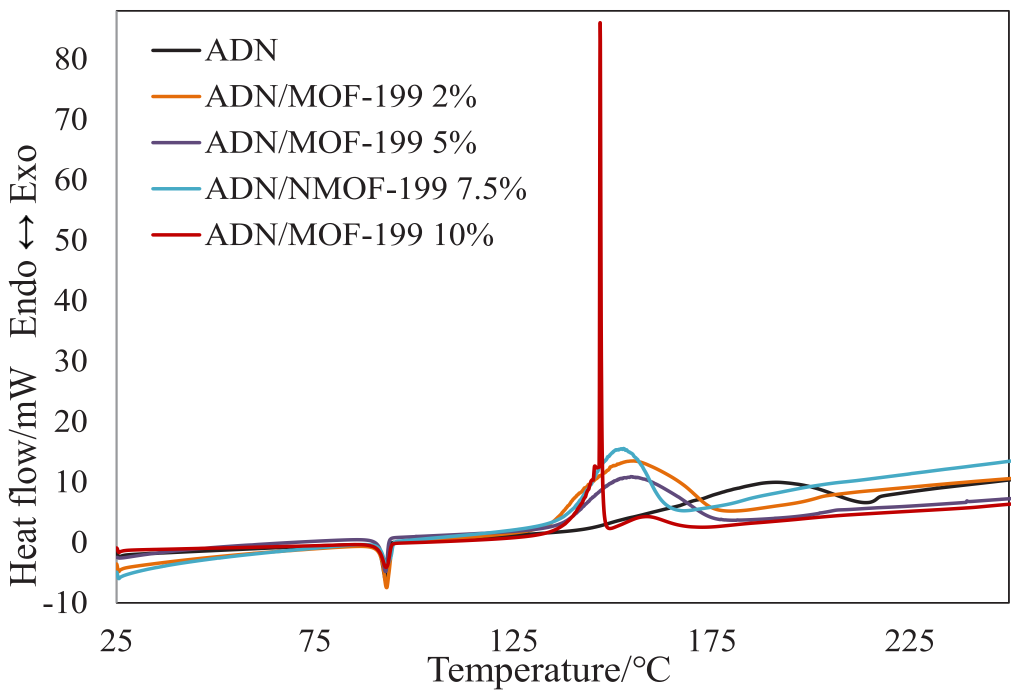

Fig. 8. DSC curves of ADN and ADN/MOF-199 composites with various MOF contents.

The significant difference between ADN/MOF-199 mixture and pure ADN in terms of exothermic onset temperature, maximum peak temperature, and exothermic end temperature indicates that strong interactions occurred between MOF-199 and ADN. Results of thermal analysis of ADN and ADN/MOF-199 (2, 5, 7, and 10%), such as the melting point, exothermic onset temperature,

Table 2. Thermal decomposition behavior of ADN and ADN/MOF-199.

Sample	Melting point ℃	Onset temp. /℃	Peak temp. /℃	End temp. /℃	Ref
ADN	92.87	155	192.18	230	This work
ADN/MOF-199 2%	93.03	129.83	154.96	178.4	This work
ADN/MOF-199 5%	92.99	129.33	154.69	178.75	This work
ADN/MOF-199 7.5%	93.15	131.58	152.33	166.75	This work
ADN/MOF-199 10%	92.33	131.08	146.92	148.5	This work
ADN/ZnO-Nanorods* 5%	90.46	154	171.85	198.16	[68]
ADN/ZnO-Microrod* 5%	90.30	152	171.71	198.33	[68]
ADN/ZnO- Sub-Microrods* 5%	90.27	151	171.13	198	[68]
ADN/ZnO-Decorated-Nanoparticles* 5%	90.27	151	171.80	201	[68]
ADN/ZnO-Nanorods# 5%	90.79	148	172.39	180	[68]
ADN/ZnO-Microrod# 5%	90.72	155	170.86	175	(68)
ADN/ZnO- Sub-Microrods# 5%	90.50	157	170.02	178.33	[68]
ADN/ZnO-Decorated-Nanoparticles# 5%	91.33	154	171.52	174	[68]
ADN/$CuCrO_4$	93.38	-	141.6	-	[69]
ADN/$Cu_2Cr_2O_5$	94.5	-	152.6	-	[69]
ADN/*β*-Cu	93.3	-	149.4	-	[69]
ADN/MOF-Cu	93.3	-	177.7	-	[69]
AND/NC	93	145	172	180	[70]
AND/18C6	151.5	175	213.2	230	[71]

maximum peak temperature, and exothermic end temperature, were summarized in Table 2, and results are compared with the results described in previous articles. Interestingly the thermal decomposition behavior of ADN was improved due to the introduction of small amounts of MOF-199. Two possible explanations can be expressed for the observed thermal behavior of the ADN/ MOF-199 composite: The first void coordinate sites at the Cu^{2+} centers in the MOF-199, known as Lewis acid sites, can be activated to supplant the solvent molecules with ADN. Moreover, the Bronsted acid sites are defects on the outer surface of MOF-199 microcrystals, which can interact with ADN molecules [63]. The second porous system synergizes by bringing ADN molecules into contact with the Lewis/ Bronsted acid sites.

CONCLUSION

In this work, MOF-199 has been utilized as a well-known copper-based MOF, to study the thermal decomposition behavior of ADN. Our results showed that MOFs, due to their porous structure, their Lewis/Bronsted acid sites, and the dispersed Cu^{2+} ions sites, can be introduced as an excellent catalyst for modifying the combustion of ADN-based solid propellants. Accordingly, due to the catalytic effect of copper centers despite the low weight of MOF-199, the ADN decomposition process takes place in a very low-temperature range and in a shorter time. Furthermore, the ADN/MOF-199 10% sample has the best influence in reducing the ADN decomposition temperature from 192 to 146 °C. According to the results, we believe that the results of this work will open new perspectives in the field of using ADN as a chlorine-free oxidizer in automotive airbags and solid propellants.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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