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Research paper

Ammonium Nitrate Catalyzed with Copper Chromite Nanoparticles: A Novel Halogen Free Oxidizer for a Solid Propellant with Low Smoke Signature

Sherif Elbasuney1,2,*), **Ahmed Ashraf**3), **Ahmed Fahd**3),
Hosam E. Mostafa2), **Mohamed Attwa**2), **Ali Said**2)
1) *School of Chemical Engineering, Military Technical College, Cairo, Egypt*
2) *Nanotechnology Research Center, Military Technical College, Cairo, Egypt*
3) *Technical Research Center, Cairo, Egypt.*
* *E-mail:* s.elbasuneys@mtc.edu.eg

Abstract: Ammonium perchlorate (AP), the main oxidizer for solid propellants, results in white smoke (HCl); which could induce acidic rain and could damage fertile soil. A chlorine-free oxidizer is highly valued for solid propellants. Ammonium nitrate (AN) emerges as a promising green oxidizer; however it exhibits low performance (*i.e.* specific impulse). The present work describes the synthesis of copper chromite nanoparticles (NPs) of 45 nm. Copper chromite NPs were synthesized through hydrothermal synthesis; the developed nanocatalyst was then integrated into an AN matrix. While virgin AN demonstrated a strong endothermic decomposition of +1707 J/g, catalyzed AN demonstrated a superior exothermic decomposition, with an enthalpy of –1492 J/g. A solid propellant formulation based on AN and copper chromite ($CuCr_2O_4$) was optimized using the Institute for Chemical Technology (ICT) thermodynamic code *via* partial replacement of AP with AN. AN (30 wt.%) was found to decrease the smoke signature by 54.4%, with a minimum decrease in specific impulse. The solid propellant formulation was developed via mixing and vacuum casting. The ballistic performance was assessed using a small-scale ballistic evaluation rocket motor. The AN-based formulation demonstrated a stable combustion process with a slight decrease in the characteristic exhaust velocity and total pressure impulse of 4% and

4.8%, respectively. It may be concluded that a solid propellant with customized ballistic performance and low smoke signature has been optimized and developed.

Keywords: ammonium perchlorate, ammonium nitrate, burning rate modifier, copper chromite, composite propellant

1 Introduction

Most solid propellant formulations rely mainly on ammonium perchlorate (AP) as the primary oxidizer. However, combustion of AP results in gaseous products, including hydrochloric acid (HCl), chlorine (Cl_2), and other chlorinated compounds. Chlorine containing gaseous products have raised environmental concerns due to their corrosivity, toxicity, and acid rain [1, 2]. Additionally, chlorinated gases create a distinct smoke signature [3]. The demand for eco-friendly (chlorine-free) solid propellants has increased attention due to global warming, climate change, and acid rain [4-6], for which eco-friendly and green alternatives to the AP oxidizer have become an urgent need. Recent advances in green solid propellants have focused on low-smoke signatures. Ammonium nitrate (AN) is of interest as the potential substitute for AP [7-10]. AN (the universal soil fertilizer) is a cost-effective oxidizer. However, AN exhibits strong endothermic decomposition, with low oxidizing capability [11-13]; it can exhibit a low burning rate and propellant performance. Much research has been directed at enhancing the AN decomposition characteristics [11-17]. Transition metal oxide catalysts such as manganese oxide and cobalt oxide could have a catalytic effect on AN decomposition [18, 19]. Copper chromite, $CuCr_2O_4$, the most common ballistic modifier for solid propellants, has been employed as a catalyst for AN.

In the present work, copper chromite was developed *via* hydrothermal synthesis. Copper chromite exhibited high-quality particles with an average particle size of 45 nm. Copper chromite nano-catalyst was then integrated into the AN matrix via the anti-solvent technique. The potential impact of copper chromite on AN decomposition enthalpy and activation energy was assessed using Differential Scanning Calorimetry (DSC) and Thermo-Gravimetric Analysis (TGA) respectively. Copper chromite demonstrated a significant impact on the decomposition enthalpy of AN. Catalyzed AN exhibited an exothermic decomposition enthalpy of –1492 J/g, compared with a strong endothermic decomposition of +1707 J/g for virgin AN. The chemical composition of the solid propellant formulation was optimized via partial replacement of AP with AN, using the ICT thermodynamic code. A propellant formulation with AN (30 wt.%)

offered a decrease in HCl gaseous products of 54.4%; the specific impulse (I_{sp}) was comparable to the reference formulation based on AP. The solid propellant formulations were developed *via* mechanical mixing and vacuum casting. The impact of the AN content on the ballistic performance was assessed using a small-scale ballistic evaluation rocket motor; the operating pressure inside the rocket motor was measured during combustion. The AN-based propellant formulation exhibited a stable combustion process with a slight decrease in the characteristic exhaust velocity, and in the total pressure impulse of 4% and 4.8%, respectively. This performance was accompanied by a decrease in the smoke signature of 54.4%.

2 Experimental Part

2.1 Materials

AP and AN were employed as oxidizers (Alpha Chemica, India). Aluminum (Al) particles of 20 µm were employed as a fuel (Aldrich Chemical Co., England). Hydroxyl-terminated polybutadiene (HTPB) was employed as a prepolymer (Iverise Co., Brazil). Dioctyl azelate (DOZ) was employed as a plasticizer (Aldrich Chemical Co., UK). Hexamethylene diisocyanate (HMDI) was used as a curing agent (Aldrich Chemical Co., England).

2.2 Synthesis of copper chromite nanocatalyst

Copper chromite nanoparticles were developed *via* batch hydrothermal synthesis. $Cu(NO_3)_2{\cdot}3H_2O$ (1 mmole), and $Cr(NO_3)_3{\cdot}9H_2O$ (2 mmol) were dissolved in deionized water (200 mL) under magnetic stirring for 1 h to develop a homogeneous solution. The solution was transferred to a hydrothermal batch reactor and the reaction was carried out at 300 °C for 8 h to form a crystalline structure. The developed colloidal particles were centrifuged off and dried at 60 °C for 12 h.

2.3 Nano-catalyst characterization

The morphology of the dry copper chromite powder was investigated using scanning electron microscopy (SEM; ZEISS SEM EVO 10 MA supported with an EDAX detector). Elemental mapping using the EDAX detector was used to assess the uniform dispersion and quantification of the main copper chromite elements.

2.4 Integration of copper chromite into AN

The copper chromite particles were precipitated from their synthesis medium and re-dispersed in methanol. Subsequently, AN particles were dissolved in the methanol colloid. The weight ratio of copper chromite to AN was 3:97. Toluene was employed as an anti-solvent to integrate the copper chromite particles into the AN. A schematic diagram of the integration process is presented in Figure 1.

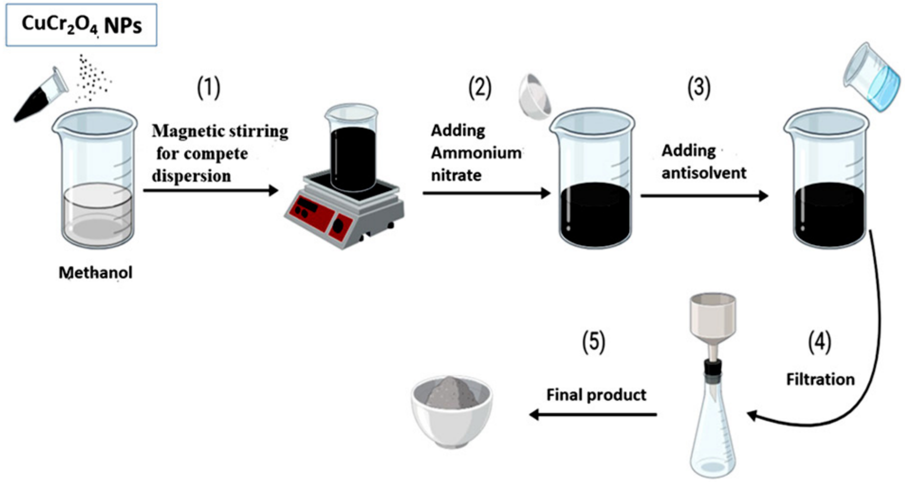


Figure 1. Integration of copper chromite into the AN matrix

2.5 Thermal behaviour of AN

The thermal behaviour and decomposition enthalpy of AN was investigated, assessed and compared with AP using DSC. DSC Q20 (TA Instruments) was used for the thermal analysis study. The tested sample was heated from 25 °C to 500 °C at 10 °C/min, under nitrogen gas flow (20 mL/min). The variation of weight loss of AN with temperature was investigated using TGA. The tested sample was heated at 10 C /min from 25 to 500 °C using a TGA 55 thermal analyzer (TA Instruments).

2.6 Thermal behaviour of catalyzed AN

The significant impact of copper chromite particles on the AN decomposition enthalpy was investigated *via* DSC. The thermal behaviour of the catalyzed AN nanocomposite was investigated using DSC Q20 by TA. The impact of the copper chromite particles on the AN weight loss was assessed using TGA 55 by TA.

2.7 Chemical composition optimization of the solid propellant

The impact of partial replacement of AP with AN on the propellant performance I_{sp} and smoke gaseous products (HCl) was investigated using the ICT thermodynamic code. Table 1 lists the different formulations developed *via* partial replacement of AP with AN. The impact of AN on I_{sp} and % emission of gaseous HCl was evaluated.

Table 1. Formulations of composite propellants

<table>
<tr><th colspan="3" rowspan="2">Component</th><th colspan="8">Composition [wt.%]</th></tr>
<tr><th>1</th><th>2</th><th>3</th><th>4</th><th>5</th><th>6</th><th>7</th><th>8</th></tr>
<tr><td colspan="2" rowspan="2">Oxidizer</td><td>AP</td><td>59</td><td>49</td><td>44</td><td>39</td><td>29</td><td>19</td><td>9</td><td>0</td></tr>
<tr><td>AN</td><td>0</td><td>10</td><td>15</td><td>20</td><td>30</td><td>40</td><td>50</td><td>59</td></tr>
<tr><td rowspan="4">Binder</td><td>Pre-polymer</td><td>HTPB</td><td colspan="8">10.5</td></tr>
<tr><td>Plasticizer</td><td>DOZ</td><td colspan="8">2.6</td></tr>
<tr><td>Curing agent</td><td>HMDI</td><td colspan="8">0.55</td></tr>
<tr><td>Bonding agent</td><td>MAPO^{a)}</td><td colspan="8">0.3</td></tr>
<tr><td colspan="2">Metallic fuel</td><td>Al</td><td colspan="8">17</td></tr>
<tr><td>Burning modifier</td><td>Catalyst</td><td>Copper chromite</td><td colspan="8">0.03</td></tr>
<tr><td>Special component</td><td>Nitramine</td><td>HMX</td><td colspan="8">10</td></tr>
</table>

a) Tris(1-(2-methyl)aziridinyl)phosphine oxide

2.8 Ballistic performance assessment

The reference solid propellant formulation based on 59 wt.% AP and the optimized solid propellant formulation based on AN (30 wt.%) were developed *via* mechanical mixing and vacuum casting. The chemical compositions of the developed formulations are listed in Table 2. The developed formulations were cast under vacuum into small scale ballistic evaluation rocket motors (Figure 2). The ballistic performance of the developed propellant was assessed *via* static firing; the operating pressure inside the rocket motor was recorded with time.

Table 2. Chemical composition of developed solid propellant formulations

Component			Composition [wt.%]	
			1	2
Oxidizer		AP	59	29
		AN	0	30
Binder	Pre-polymer	HTPB	10.5	
	Plasticizer	DOZ	2.6	
	Curing agent	HMDI	0.55	
	Bonding agent	MAPO	0.3	
Fuel	Metallic fuel	Al	17	
Special component	Nitramine	HMX	10	
Burning modifier	Catalyst	Copper chromite	0.03	

Figure 2. Development of propellant grain for ballistic performance evaluation

3 Results and Discussion

3.1 Characterization of copper chromite nanoparticles

TEM micrographs demonstrated the copper chromite nanoparticles were of 45 nm average particle size (Figure 3). The morphology of dry copper chromite particles was investigated by SEM. The SEM micrographs showed high quality particles with uniform size and shape (Figure 4). They also demonstrated that the copper chromite NPs were mono-dispersed with a spherical shape. Elemental mapping of the copper chromite NPs was conducted using an EDAX detector, and revealed uniform dispersion of copper and chromium within the material. The ratio of Cr/Cu was 2/1 (Figure 5). The developed copper chromite was a balanced composition free from any interfering substances.

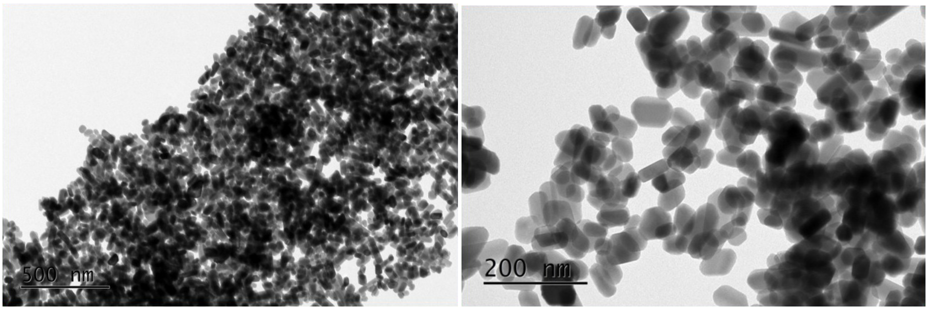


Figure 3. Morphology of catalyst nanoparticles using TEM

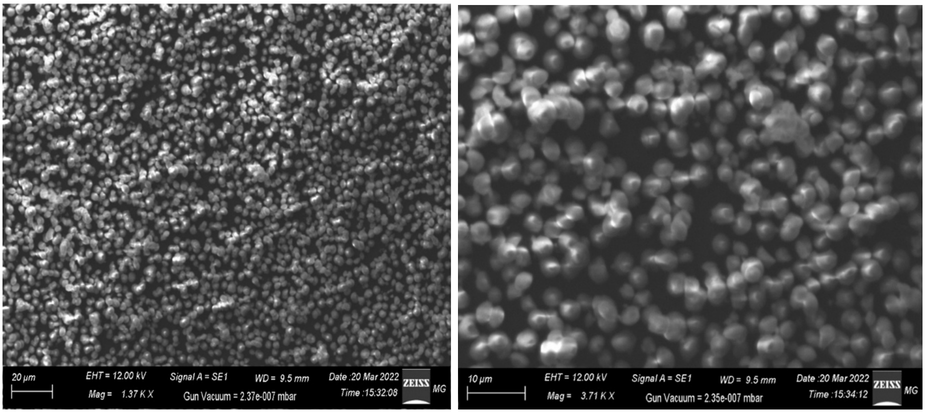
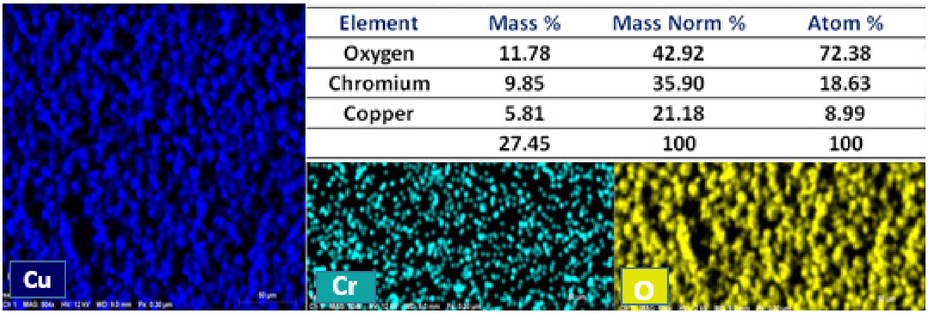


Figure 4. Morphology of catalyst nanoparticles using SEM

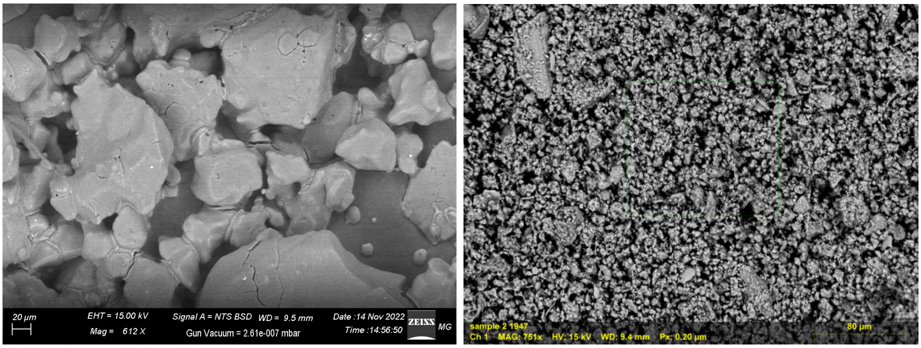


Element	Mass %	Mass Norm %	Atom %
Oxygen	11.78	42.92	72.38
Chromium	9.85	35.90	18.63
Copper	5.81	21.18	8.99
	27.45	100	100

Figure 5. Elemental composition of catalyst particles

3.2 Characterization of the AN nanocomposite

The morphology of virgin AN and the developed copper chromite/AN was investigated using SEM. While virgin AN demonstrated polydispersed particles with a particle size of 80 µm, the copper chromite/AN nanocomposite demonstrated monodispersed particles of 5 µm particle size (Figure 6). The dispersion of copper-chromite particles into the AN matrix was investigated by EDAX analysis. Uniform dispersion of the copper and chromium elements within the AN matrix was verified as shown in Figure 7. It is obvious that there was no agglomeration of the copper chromite within the AN matrix. This finding confirmed the good mixing and distribution of the nano-particles and thus validated the preparative method.



(a) (b)

Figure 6. SEM of starting AN (a) and copper chromite/AN nanocomposite (b)

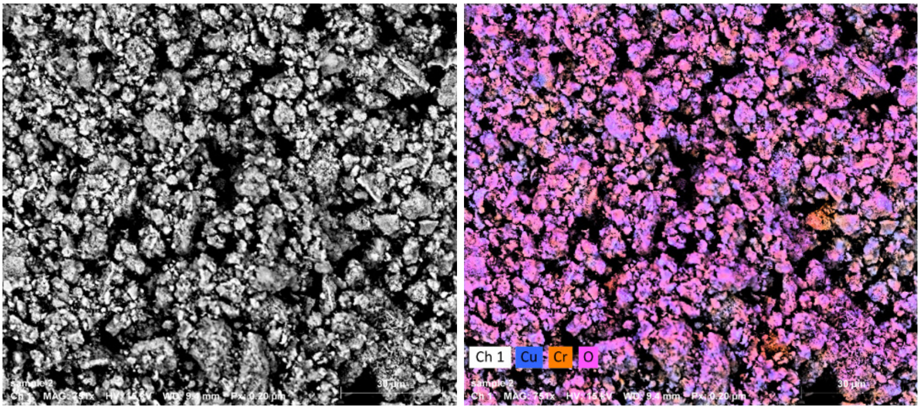


Figure 7. Chemical composition of AN nanocomposite

3.3 Thermal behaviour of virgin AN

The decomposition enthalpy and thermal behaviour of virgin AN was investigated and compared to that of AP (universal oxidizer) using DSC. Virgin AP exhibited an exothermic reaction with a total decomposition enthalpy of –733.0 J/g. AN exhibited endothermic phase changes at 52 and 127 °C and endothermic melting at 170 °C, with subsequent endothermic decomposition at 283 °C. AN exhibited strong endothermic decomposition with a total decomposition enthalpy of +1734.9 J/g (Figure 8). The thermal behaviour of AN could facilitate a high burning rate and propellant performance.

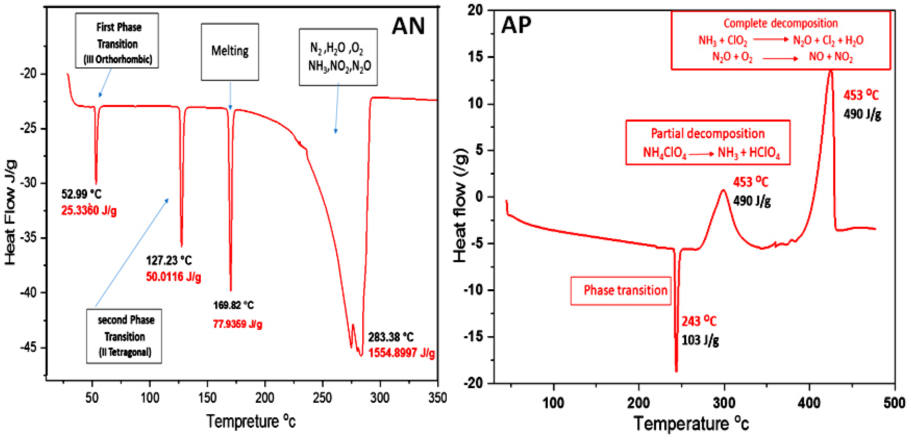


Figure 8. Comparison of the decomposition enthalpy of AN and AP using DSC

The crystallographic transformations of AN IV → II and II → I are observed as two endothermic peaks at 53 and 127 °C, respectively. The endothermic peak at 169.26 °C corresponds to the melting point of AN (solid to liquid phase transition) [12]. The endothermic peak at 283 °C is caused by the decomposition of AN to gaseous products [20]. It is clear that AN decomposes endothermically with a total heat of +1707 J/g; therefore AN exhibits endothermic decomposition and could augment propellant performance. The thermal behaviour of AN was further investigated and compared to that of AP using TGA. The weight loss of the tested samples was recorded against temperature (Figure 9).

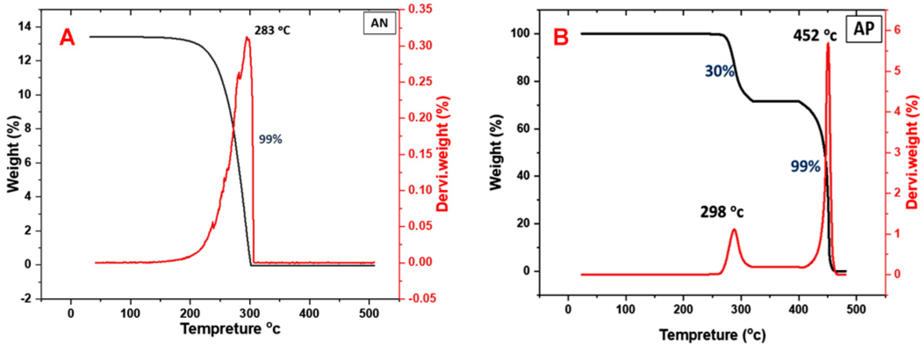


Figure 9. Weight loss vs temperature of AN and AP using TGA

AP decomposition took place in two separate stages at 298 °C (mass loss of 30 wt.%), and 452 °C (mass loss of 70 wt.%). Virgin AN exhibited a total mass loss (99%) in one stage at 283 °C; this peak was correlated with the final decomposition. The TGA results were found to be in good agreement with the DSC results.

3.4 Thermal behaviour of catalyzed AN

The impact of copper chromite (3%) on AN thermal decomposition and decomposition enthalpy was investigated using DSC. Copper chromite caused a significant change on AN thermal decomposition. Copper chromite/AN exhibited an exothermic decomposition enthalpy of –1492 J/g; compared with an endothermic decomposition of +1707 J/g for virgin AN (Figure 10).

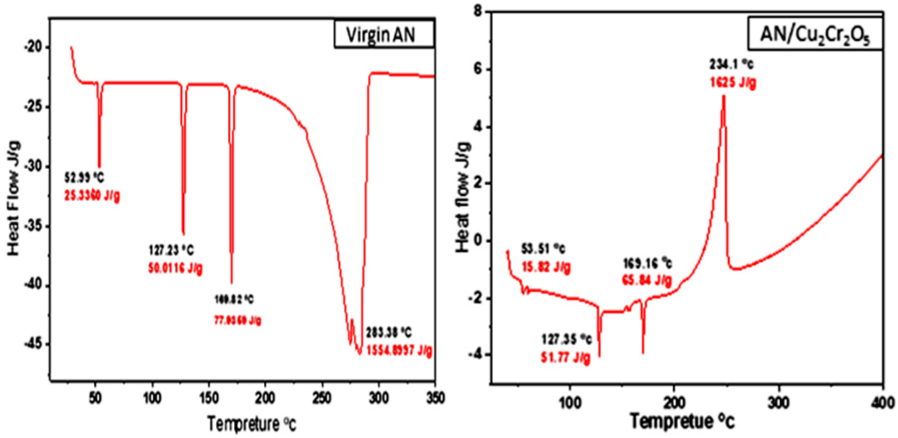


Figure 10. Impact of catalyst particles on AN decomposition enthalpy (DSC)

Copper chromite had a significant impact on the AN decomposition process. AN decomposition was completely changed from being a strongly endothermic reaction of +1554 J/g to a strongly exothermic reaction of –1625 J/g. The integration of copper chromite as a catalyst offered a novel exothermic decomposition reaction. AN decomposition can be summarized in the following steps [21, 22]:

- AN could decompose into ammonia (NH_3) and nitric acid (HNO_3).
- HNO_3 could dissociate into hydrogen ions (H^+) and nitrate ions (NO_3^-).
- Release nitrogen gas (N_2), water (H_2O), and oxygen gas (O_2) as products.

The catalytic effect of copper chromite could be ascribed to the facile reaction of chromium ions with the decomposition reactants. This condensed phase reaction could facilitate AN decomposition, and could boost the decomposition enthalpy. The catalytic effect of copper chromite can be summarized in the following steps:

- Excess nitrate ions (NO^{-3}) can oxidize chromium ions (Cr^{+3}) to chromate ions (CrO_4^{+2}) in the presence of hydrogen ions (H^+).
- The chromate ions (CrO_4^{+2}) can further react with oxygen gas (O_2) to form dichromate ions ($Cr_2O_7^{-2}$).
- Nitrate ions react with $Cr_2O_7^{-2}$ ions to produce nitrite ions (NO^{+2}) and CrO_4^{+2}.

The chromium ion can adopt different oxidation states and could induce different catalytic condensed phase reactions (Figure 11).

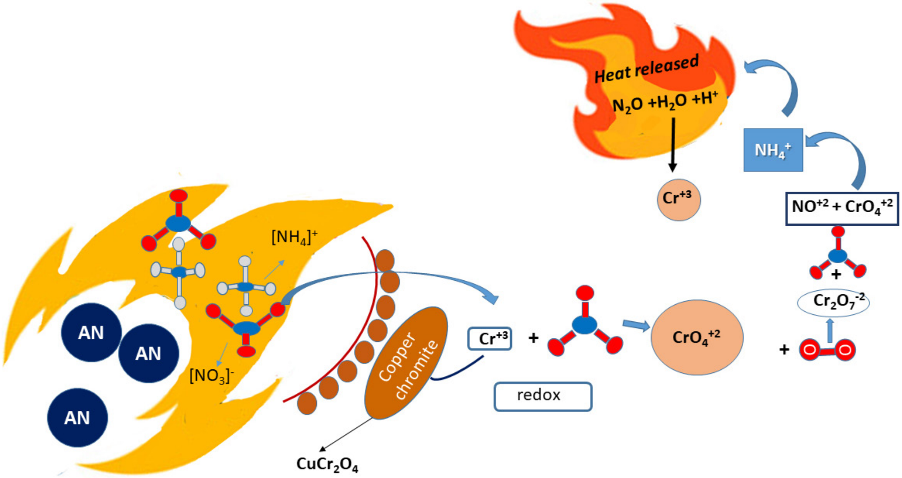


Figure 11. Catalytic mechanism of copper chromite on AN decomposition

3.5 Chemical composition optimization

A propellant formulation based on 59 wt.% AP was considered as reference. The potential impact of partial replacement of AP with AN was assessed using the ICT thermodynamic code. Thermochemical parameters including I_{sp}, combustion temperature, gaseous exhaust velocity and HCl gaseous are tabulated (Table 3). Formulation 5 (29 wt.% AP and 30 wt.% AN) offered a decrease in HCl smoke by 54.4%. This formulation offered an acceptable I_{sp} of 259.9 compared with 265.6 s.

Table 3. Theoretical ballistic parameters and HCl percent by the ICT-Code

Formulation	AN [wt.%]	I_{sp} [s]	Combustion temperature [K]	Density [g/cm^3]	Exhaust velocity [m/s]	HCl [%]
1	0	265.6	3322	1.772	2603	12.64
2	10	264	3219	1.75	2589	10.2
3	15	263.1	3165	1.74	2568	9.03
4	20	262.1	3109	1.73	2546	7.89
5	30	259.9	2992	1.73	2531	5.712
6	40	257.5	2871	1.71	2875	3.64
7	50	254.8	2748	1.7	2500	1.6
8	59	252.3	2636	1.66	2475	0

3.6 Ballistic performance evaluation

The ballistic performance of formulation 5 based on 30 wt.% AN was compared to the reference formulation based on 59% AP. The propellant grain was developed *via* mechanical mixing and vacuum casting. The propellant grain was cast into a two-inch small-scale ballistic evaluation test motor. The ballistic performance was assessed *via* precise measurement of the pressure inside the rocket motor under firing, and revealed promising results. The partial substitution of AP with AN led to a noticeable reduction in smoke emission without a significant impact on I_{sp}. The ballistic performance remained within the acceptable range for various applications, as shown in Figure 12.

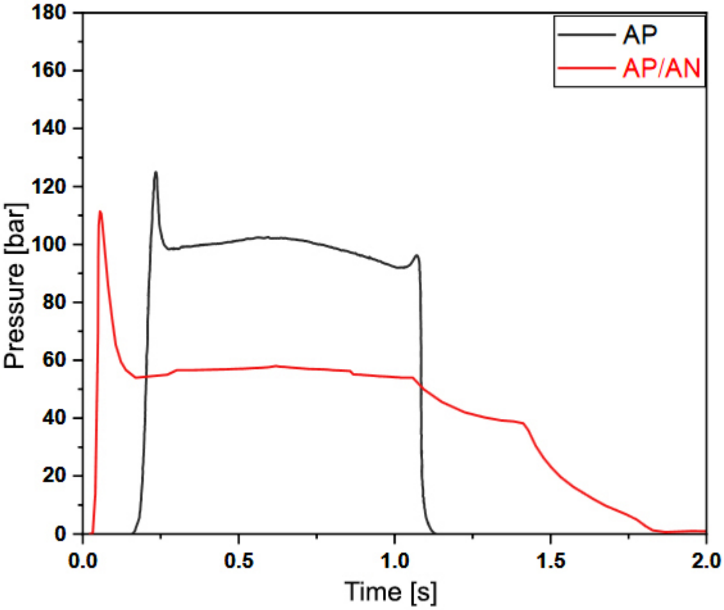


Figure 12. Pressure-time curve for the developed solid propellant formulations

Formulation 5, based on AN, demonstrated a low operating pressure and extended burning time compared with the reference formulation. However, the total pressure impulse of the AN-based formulation was found to be comparable to that of the reference formulation. Table 4 summarizes the main ballistic performance parameters.

Table 4. Main ballistic performance parameters of the developed propellant formulations

Oxidizer	TEMP. [°C]	THROAT [mm]	WT [kg]	TB [s]	IPT [bar·s]	Pav [bar]	P50% [bar]	R50% [mm/s]	C^* [m/s]
AP	20	8.2	0.327	0.91	91.5	100.4	106.0	14.95	1464
AP/AN	20	8.2	0.327	1.67	87.1	52.14	60.8	9.08	1405

The AN-based formulation exhibited a decrease in the average operating pressure, burning rate and an increase in the total burning time. The total pressure impulse was decreased by only 4.8%. This ballistic performance could be considered for developing propellants with a low smoke signature.

4 Conclusions

- The above work concluded that AN could be a green substitute for AP, to develop a halogen-free oxidizer based propellant formulation. However, AN exhibited endothermic decomposition with copper chromite nanoparticles of 45 nm (which were developed *via* hydrothermal synthesis).
- The developed nano-catalyst was integrated into the AN matrix *via* the anti-solvent technique.
- Copper chromite exhibited an advanced catalytic effect on AN decomposition. While virgin AN demonstrated an endothermic decomposition of +1708 J/g; copper chromite/AN demonstrated an exothermic decomposition with a total decomposition enthalpy of –1492 J/g.
- The chemical composition of a low signature solid propellant was optimized *via* partial replacement of AP with AN; the impact of AN on the ballistic performance was assessed using the ICT thermodynamic code. A solid propellant formulation based on 30 wt.% AN exhibited a decrease in smoke signature of 54.4%, with a minimal decrease in I_{sp} of 2.1%. The solid propellant formulation based on AN demonstrated a decrease in average operating pressure and burning rate; however, the total pressure impulse was comparable to an AP-based formulation. It may be concluded that a customized propellant smoke signature and ballistic performance were optimized.

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Authorship contribution statement

Sherif Elbasuney: conception, foundations, methods, performing the statistical analysis
Ahmed Ashraf: foundations, methods, performing the experimental part
Ahmed Fahd: conception, foundations, performing the experimental part
Hosam E. Mostafa: foundations, methods, performing the experimental part
Mohamed Attwa: foundations, methods, performing the experimental part
Ali Said: foundations, methods, performing the experimental part

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