

# Surface Functionalized Ammonium Nitrate Prills with Enhanced Water Resistance Property: Characterizations and its Application as Commercial Explosives

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**Abstract:** Increasing the hydrophobicity of ammonium nitrate is of great significance for its application as commercial explosives. In this article, a surface functionalized ammonium nitrate with enhanced water-resistance property was demonstrated using a new hydrophobic coating agent. Structural properties and thermal stability of the prepared samples were investigated by powder X-ray diffraction, scanning electron microscopy and differential scanning calorimetric analysis. Fourier-transform infrared spectroscopic

results show that a uniform hydrophobic layer is successfully coated on the surface of ammonium nitrate prills. Surface coated ammonium nitrate shows excellent stability in aqueous environment with effective detonation property compared to commonly used ammonium nitrate fuel oil (ANFO) explosives. Thus, our surface modified ammonium nitrate could serve as a potential explosive for watery hole blasting operations.

**Keywords:** Ammonium Nitrate · Surface functionalization · Thermal stability · ANFO · Commercial explosive

## 1 Introduction


Ammonium Nitrate is an important oxidizer; it is widely used in the field of energetic materials such as commercial explosives, propellants and nitrogenous fertilizer [1–3]. Recently ammonium nitrate has gained lot of interest as green propellant due to its eco-friendly plumes, smoklessness and low sensitivity [4,5]. Further, ammonium nitrate is also known to be chemically stable at ambient temperature and pressure. Particularly, ammonium nitrate is a major component in the most widely used commercial explosive, Ammonium Nitrate-Fuel oil (ANFO). Safety, ease of handling, cost efficiency and free-flowing nature makes ANFO a popular explosive for commercial application worldwide. However, poor water resistance and hygroscopic nature of ammonium nitrate restricts its application in wet environment. Moreover, ANFO explosive in its free-flowing form is not useful in watery hole, as ammonium nitrate instantaneously dissolves into water and loses its explosive property.

In the last few decades, significant effort has been put forward by researchers to reduce the hygroscopicity and to improve the water resistance property of ammonium nitrate in order to achieve significant detonation properties of ANFO explosives [6–10]. Surface modification techniques using paraffin wax, mineral oil, and surfactants as hydrophobic coating and encapsulation methods were introduced to reduce the hygroscopicity of ammonium nitrate [6–9]. Many reports significantly contribute in the field on the development of anti-hygroscopic ammonium nitrate with various additives and coating methods under specified condition have been reported in the previous literature

[6, 8, 10–16]. However, studies on the improvement of water resistant property of ammonium nitrate in its free flowing form are scarce in literature. Venter *et al.* reported excellent free flowing water resistant explosive compositions. These compositions required a water resistant emulsion which includes more than one oxidizing salts, fuels and emulsifiers along with few other reagents such as water binding agent, a density reducing agent, parting agent, etc. [17]. Further, White *et al.* introduced a new coating material consisting al-paste by filtering slurry, fatty acid and solvent and used as a part substitute or replacement for conventional fuel oil coatings. This composition also required slurry with aluminum particles and required multiple steps to prepare the product, time consuming, required skilled persons and also increase the overall cost of the product [18]. A. K. Chattopadhyay also described the coated ammonium nitrate prills which increase the storage life and acts as anti-caking agent. But water resistant properties were not studied in

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this report [19]. McNicol *et al.* used multiple chemicals to get the desired product like 5–20% of emulsion as a coating agent and perlite and polystyrene as density controller, which enhanced the overall cost of the product and required more time to prepared desired product [20]. Water resistant ANFO was developed by Reeksting *et al.* However, the composition is difficult to prepare as it consists of different chemicals [21]. This process required a catalyst followed by irradiation to initiate the polymerization process to achieve the product, which is time consuming and also increases the overall cost of the product. Shreeran *et al.* introduced a free flowing granular ANFO explosive with coating of a hydrophilic cold-water swellable thickener, Guar gum. However, solubility of guar gum is known to depend on the temperature and pH of the water [22]. In addition, its tendency to form lums in water may usually impose some practical problems at site. In the reported compositions, along with the gum, a hydrophobic water repelling compound was used which further enhancing the cost of the product. Fox *et al.* described a water resistant explosive composition coated with a waterproofing material and mixed with an anti-agglomeration agent. However, also after 20 min, 48% of AN leached out from the desired product [23].

Thus, any modification that enhances the water resistant property of ANFO is of immense importance, as this has a direct relevance for blasting applications in aqueous environment. Commonly used commercial explosives for watery environment are emulsion, slurry explosives and cartridge explosives. However, these explosives are expensive and difficult to manufacture compared to ANFO. Usually, ANFO used in watery hole needs water-proof packaging or dewatering is required before charging the explosive, as small amount of water significantly reduce the ANFO explosive performance. These processes are time consuming, laborious and increase the overall cost of operation.

Considering these limitations, there is an urgent need to develop a functionalized ammonium nitrate/ANFO explosive that is capable of showing better water-resistant property. Such explosives could be very useful for various mining application in watery environment and underwater blasting operations. Considering the scope of such compositions, we report herein a surface modified ANFO explosive for watery environment blasting operations. We discuss the detailed thermal stability, microstructures of the coated ammonium nitrates in comparison with normal ammonium nitrate using various analytical techniques. Importantly, detonation properties of modified ammonium nitrate fuel oil reveal that it could be used in watery environments as an efficient blasting agent. Moreover, surface modification was confirmed by various analytical techniques.

## 2 Experimental Section

### 2.1 Materials & Methods

Ammonium Nitrate prills were obtained from SmartChem Technology Limited, Pune. Long chain soya resin LS50% (R-1); was obtained from Subham Oils & Resins Private Limited, Kolkata. Toluene, AR grade, was procured from S. D. Fine Chem. Limited, India. All commercial chemicals were used as received without further purification unless stated otherwise. Field emission scanning electron microscopy (FE-SEM) images were collected using a JEOL JSM-7100F instrument operated at 18 kV accelerating voltage. NMR (500 MHz) spectra for the R-1 was collected on a Bruker AX 500 spectrometer (500 MHz) instrument. GPC carried out using a WATERS 2695 separations Module apparatus instrument. Thermal analyses (DSC) were carried out on a NETZSCH DSC 204F1 Phoenix analyzer under N<sub>2</sub> atmosphere at a heating rate of 5 °C min<sup>-1</sup> within a temperature range of 28–300 °C. FT-IR spectra on KBr pellet (4000–400 cm<sup>-1</sup>) were recorded with a Perkin-Elmer Spectrum 100. Powder X-ray diffraction (PXRD) patterns were recorded at room temperature on a Philips X'pert X-ray powder diffractometer using Cu-K $\alpha$  radiation ( $\lambda = 1.5418 \text{ \AA}$ ) in the 2 $\theta$  range of 5–50°. Velocity of detonation of modified Ammonium nitrate fuel oil (ANFO) explosive was measured using Dautriche method (IS Code 6609) in confined condition and A MicroTrapVoD/Data Recorder, an instrument by MREL was used for VOD measurement in unconfined condition. Details of <sup>1</sup>H NMR, <sup>13</sup>C NMR, FT-IR, GPC analysis of R-1 were provided in supporting information.

### 2.2 General Procedure for Ammonium Nitrate Hydrophobic Coating

The hydrophobic coating on ammonium nitrate prills was prepared by gentle mechanical stirring of ammonium nitrate prills with long chain resin (R-1; LS50%) in the presence of toluene at room temperature using standard methods [6]. In brief, the long chain resin (R-1) (LS50%), 1 wt% was dissolved in 1 wt% of toluene and the mixture was mixed with Ammonium nitrate prills with gentle stirring condition for 10 min. With these coated ammonium nitrates, diesel oil (D/O) was used as fuel phase to prepare modified ANFO. 94.5% of modified Ammonium nitrate and 5.5% of diesel oil was used to prepare modified ANFO mixture and used for experiment unless mention otherwise.

## 3 Results and Discussion

### 3.1 XRD Analysis

Ammonium nitrate is known to have P<sub>mmn</sub> space symmetry group having two molecules per unit cells and have C<sub>2v</sub>

symmetry for  $\text{NH}_4^+$  and  $\text{NO}_3^-$  ions [24–26]. A detailed Powder X-ray Diffraction (PXRD) investigation was performed to characterize the prepared (R-1) resin coated ammonium nitrates pills. As shown in the Figure 1, the diffraction signals at  $2\theta$  are  $17.9^\circ$ ,  $22.5^\circ$ ,  $24.65^\circ$ ,  $28.8^\circ$ ,  $31.3^\circ$ ,  $32.8^\circ$ ,  $36.1^\circ$ ,  $37.7^\circ$  and  $39.9^\circ$  were assigned for the 100, 011, 010, 111, 002, 020, 102, 201 and 112 lattice planes of ammonium nitrate crystal structure. The position of the signals remains almost same with the R-1 coated AN prills in comparison with the uncoated ammonium nitrates prills, except the change in peak intensity, which is mainly due to the preferred orientation of the coated resin on the ammonium nitrate prill, which resulted in some changes in the diffraction pattern.

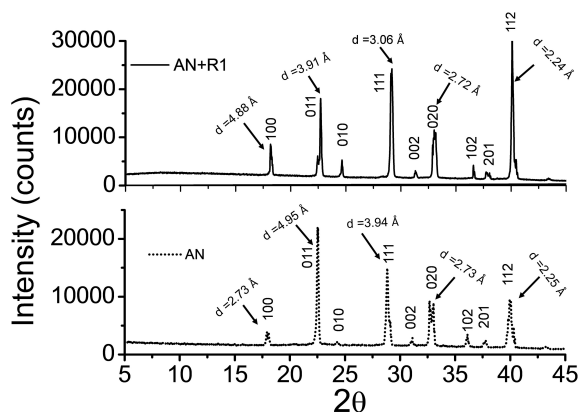
The increased intensity of the R-1 coated ammonium nitrates was because of the better absorption of the resin on the surface cracks, pores and void of AN Prills. Reports also indicate an ordered crystal structure has larger crystallite size and less disordered structure compare to the disordered crystal structures [27].

Full-Width Half Maximum (FWHM) and crystallite size of the resin coated ammonium nitrates prills and control ammonium nitrates prills, were calculated from the intense PXRD diffraction peaks using Scherrer formula. The diffraction peak at  $2\theta = 28.8^\circ$  was selected for the comparative study as it has strong signal in the R-1 coated ammonium nitrate (AN) samples and uncoated AN prills.

The calculated crystallite size was larger for the R-1 coated AN prills compared to the uncoated AN. For the R-1 coated AN, it was found to be 47 nm and uncoated sample it is 44 nm. Therefore, based on the analysis, we speculate that the crystal structure of R-1 coated AN crystal is more ordered than the uncoated AN crystal.

### 3.2 Thermal Analysis

The thermal behavior of ammonium nitrate plays a significant role on the crystal structure. AN can exist in several



**Figure 1.** XRD patterns of Ammonium nitrate in presence and absence of R-1.

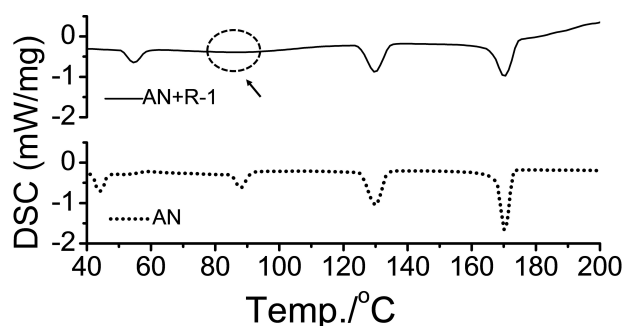
polymeric forms such as  $\beta$ -rhombohedral,  $\alpha$ -rhombohedral, tetragonal and cubic form under ambient pressure and these solids samples exhibit different physical & thermodynamic properties; specific volume, heat capacity, solubility etc [28].

In this study, DSC curves recorded at a heating rate of  $5^\circ\text{C min}^{-1}$ , were used to study the thermochemical behavior of the non-coated and R-1 coated ammonium nitrate samples shown in Figure 2 and the characteristics peak of DSC were summarized in Table 1. Non-coated and R-1 coated ammonium nitrate has similar kind of DSC spectral pattern, which confirms the coated samples does not affect the thermal behavior of AN under specified experimental conditions.

AN exhibited four endothermic peak at  $40^\circ\text{C}$ ,  $84^\circ\text{C}$ ,  $130^\circ\text{C}$  and  $170^\circ\text{C}$ , were assigned for the phase  $\text{IV} \rightleftharpoons \text{III}$  change,  $\text{III} \rightleftharpoons \text{II}$  and  $\text{II} \rightleftharpoons \text{I}$  changes, which was further supported by the literature reports [5].

DSC spectra for the coated AN shows almost similar kind of thermal properties. However, for R-1 coated ammonium nitrate sample signal at  $\sim 42^\circ\text{C}$  was disappeared (Figure 2 & Table 1). This may be due to the interaction of the polar functionalities of R-1 and ammonium nitrate by forming H-bonding [5].

Porosity & oil absorption ratio of ammonium nitrate plays an important role in ANFO explosive efficiency. Therefore, Porosity and oil absorption ratio of coated ammonium nitrate samples were analyzed using standard methods. Porosity of uncoated ammonium nitrates is usually found in between 18% to 20% (wt.%). The porosity of coated ammonium nitrate samples was found similar compared to uncoated ammonium nitrate as shown in Table 2. Also calculated densities and oil absorption ratio of uncoated sample were closely related to the ammonium nitrate prills properties (see Table 2).



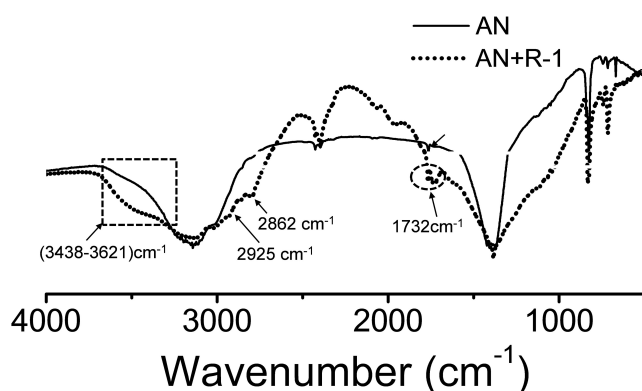
**Figure 2.** DSC curve of Ammonium Nitrate (AN) and AN with R-1.

**Table 1.** Characteristic DSC Peaks of studied AN samples.

four characteristic peaks of DSC profiles ( $^\circ\text{C}$ )				
samples	first	second	third	fourth
AN	43.82	88	130	170
AN + R-1	54	disappear	130	170

**Table 2.** Physical Properties of Modified ANFO.

Parameters	Units	Temperature °C	
		30	40
Poured Density	g/cm <sup>3</sup>	0.87 ± 0.01	0.87 ± 0.01
Settled Density	g/cm <sup>3</sup>	0.89 ± 0.01	0.89 ± 0.01
Porosity (%)	(%)	19.35	20.21
Oil absorption & retention	(% by wt.)	7.86	8.29



**Figure 3.** FT-IR spectra of ammonium nitrate (AN) and AN with R-1.

### 3.3 FT-IR Analysis

Fourier-transform infrared spectroscopy (FT-IR) spectral analysis was performed to monitor the surface modification of ammonium nitrate with R-1 as shown in Figure 3. The band at 3140 cm<sup>-1</sup> and 3014 cm<sup>-1</sup> were belongs to the asymmetric NH<sub>4</sub><sup>+</sup> stretching and asymmetric vibrational frequency of NH<sub>4</sub><sup>+</sup> ion [7,29,30]. The bond observed at 1753 cm<sup>-1</sup> was due to the NO<sub>3</sub><sup>-</sup> in plane deformations. Further, the signal at 1392 cm<sup>-1</sup>, 831 cm<sup>-1</sup> were assigned as doubly degenerate NO<sub>3</sub><sup>-</sup> stretching frequency and out of plane deformation of nitrate (V) anion [7,29].

Importantly, the new broad peak appears at (3438–3621) cm<sup>-1</sup> in coated AN sample was due the the O–H straching of the R-1 [30]. Also an intense signal observed at 1732 cm<sup>-1</sup> for coated AN sample was assign to the stretching of with –C=O group of COOH and overlapped of ester functionality of the R-1. Weak but observable signal at 2925 and 2862 cm<sup>-1</sup> were due to the C–H stretching mode (Figure 3). The FT-IR analyses clearly indicate that the presence of R-1 on the ammonium nitrate surface in modified AN samples.

### 3.4 Morphology Analysis

To understand the surface microstructures and morphologies of the ammonium nitrate prills with and without R-1, field emission scanning electron microscopic (FESEM) studies were performed. In the FESEM images of R-1 coated am-

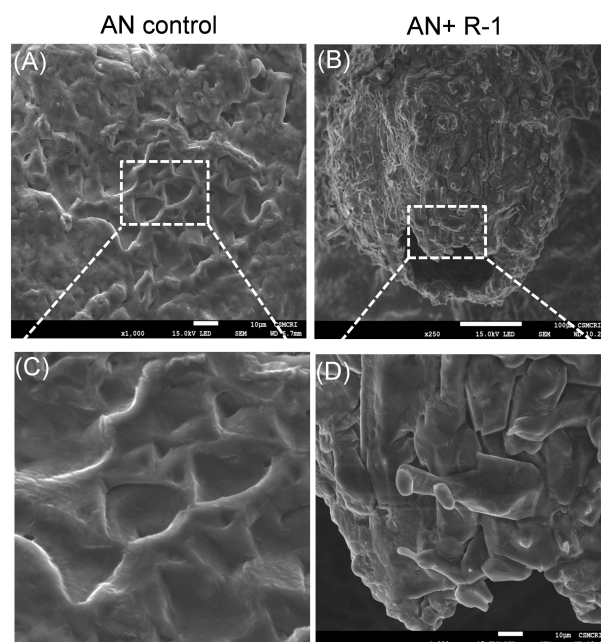
monium nitrate prills, surface looks identical with surface deformations and cracks on the entire surface shown in Figure 4. These kinds of surface deformations allow a better absorption of fuel phase which provide the enhanced blasting efficiency of ANFO explosives.

### 3.5 Nitrate Dissolution Studies

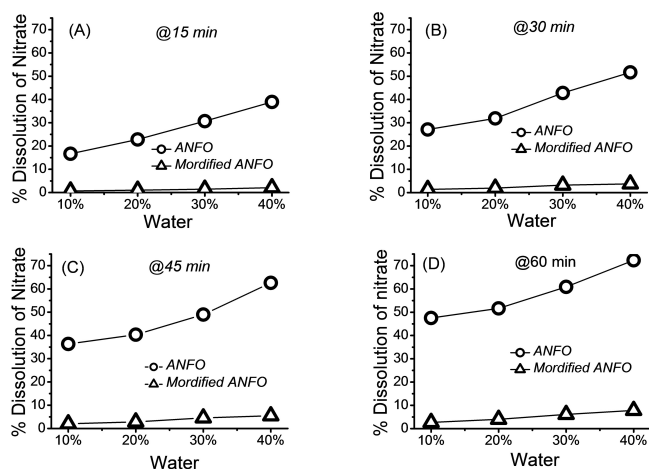
Stability in water is very important parameter for any commercial explosives prior to use it in aqueous environment. In order to verify the water stability of the modified ANFO explosive prepared using coated ammonium nitrate and diesel oil was poured into water in a measuring cylinder having various amount of water and uncoated ANFO was also poured into the water under identical experimental condition as control. Uncoated ANFO start dissolving in water as soon as it was added into water and it loses its explosive property. Interestingly, R-1 coated ANFO remain stable up to 2 h under aqueous condition.

After 2 h, only 12% ammonium nitrate was dissolved in water whereas normal ANFO dissolve completely under identical condition (Figure 5).

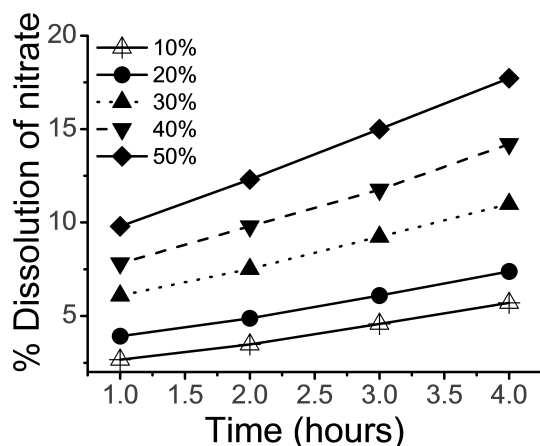
Further, to evaluate the water resistance property of modified ANFO; we have carried out the time dependent nitrate dissolution studies depicted in Figure 6. Results clearly suggest that the modified ANFO was stable up to 4 h. Only 17.8% of ammonium nitrate dissolves into water under 50% watery environment after 4 h, whereas it was



**Figure 4.** Scanning electron microscopy images: (A) uncoated ammonium nitrate (AN) prills; (B) AN coated with R-1. Images C and D are the enlarged view of A and B images.



**Figure 5.** Nitrate dissolution of normal ANFO and modified ANFO at various time interval (A: 15 min; B: 30 min; C: 45 min and D: 60 min) with water content.



**Figure 6.** Modified ammonium nitrate dissolution in various proportion of water with time.

found to be more effective with lesser amount of watery environment.

### 3.6 Detonation Characteristics

Encouraged by the nitrate dissolution studies we prepared explosive compositions with coated ammonium nitrate and diesel oil and tested its explosive properties. Sensitivity test of the prepared ANFO composition was performed. The modified ANFO compositions were prepared with coated ammonium nitrate and Diesel oil was initiated with No. 6 and 8 detonators. NO detonations were observed. Results confirmed non-cap sensitive nature of the prepared explosives.

Velocity of detonation (VOD) is the speed with which the detonation shock wave travels through the explosive. It

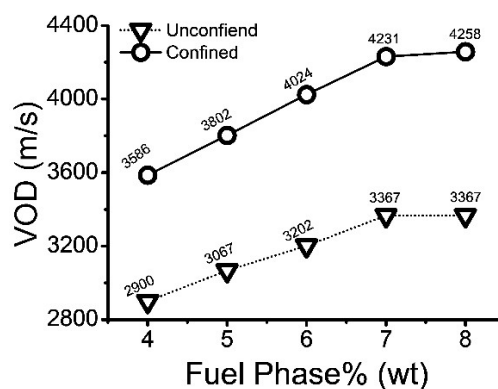
is an important property of explosive materials and a measurable indicator of explosive performance in real time and its overall energy.

Velocity of detonation of the modified ANFO having cartridge diameter 125 mm and individual length of 47 cm, was measured by using Dautriche method (IS Code 6609) and electronic timer method with variable proportion of fuel phase in confined and unconfined conditions. VOD of normal ANFO usually in the range of 2700 m/s [31]. In order to determine the effect of fuel oil content on the detonation properties of prepared ANFO mixture with coated ammonium nitrate, various proportion of fuel phase were chosen.

Velocity of detonation (VOD) of modified ANFO mixture initially increases from 2900 m/s to 3367 m/s with increase in fuel phase up to 7% (wt%) and remain unaltered at 9% fuel phase in unconfined condition. Velocity of detonation in confined condition also follows the similar trend, shown in Figure 7. The results clearly indicate the coated ammonium nitrate based fuel oil explosive mixture retain its explosive property in watery environment and shows enhanced velocity of detonation in comparison with normal ANFO.

## 4 Conclusion

In summary, we have developed a new surface modified ammonium nitrate prills with enhanced water resistivity for watery hole blasting operations. SEM, XRD and FT-IR studies confirmed the well coated hydrophobic layer of R-1 on ammonium nitrate prills. Studies also confirmed that the modified ammonium nitrates have remarkable effects on improving the hydrophobicity. Notably, only 17.8% of the modified ammonium nitrate dissolves into water at 50% watery environment after 4 h with significant detonation properties. Moreover, this modified ammonium nitrate-fuel oil (ANFO) mixture could even be used as a blasting agent in watery hole. This work provides a unique example of sur-



**Figure 7.** Velocity of detonation of modified ANFO with various fuel phases under confined and unconfined condition.



face modification of ammonium nitrate/ANFO for underwater blasting operations.

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## Data Availability Statement

The data that supports the findings of this study are available in the supplementary material of this article.

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