



Section 1. Chemistry

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PHYSICOCHEMICAL CHARACTERIZATION OF SERPENTINITE-MODIFIED AMMONIUM NITRATE

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Abstract

This study investigates the effect of serpentinite as a stabilizing additive to ammonium nitrate (AN) by analyzing its physicochemical properties through XRD, SEM-EDS, and DSC techniques. Serpentinite, rich in magnesium silicates, was added in varying ratios to AN to reduce its thermal sensitivity. The results showed that the addition of serpentinite did not disrupt the crystalline structure of AN, while improving its thermal behavior and particle dispersion. The composition with a 10:2.5 AN to serpentinite ratio exhibited optimal performance, showing delayed decomposition, enhanced thermal stability, and better structural integrity. These findings highlight serpentinite's potential as an effective, low-cost stabilizer for improving the safety of ammonium nitrate-based materials.

Keywords: *Ammonium nitrate, serpentinite, thermal stability, XRD, SEM-EDS, DSC analysis, Physicochemical properties*

Introduction

Ensuring food security for a rapidly growing global population remains one of the most pressing challenges facing governments around the world. According to demographic projections, the Earth's population is increasing at an unprecedented rate – from approximately 1 billion in 1830 to 4.9 billion in 1986, 6.1 billion in 2000, 8.0 billion in 2015, and is expected to reach 9.0 billion by 2050 (Abbasi, T., & Abbasi, S. A.,

2007). This population boom intensifies the demand for food, while simultaneously placing greater strain on the world's finite agricultural resources. One of the most alarming indicators of this pressure is the steady decrease in the amount of arable land available per person. In particular, the area of grain crops per capita has diminished from 0.24 hectares at the beginning of the 20th century to just 0.1 hectares by its end – and is projected to decline further

to 0.08 hectares per capita by 2050 (Abbasi, T., & Abbasi, S. A., 2007).

This sharp decline in per capita agricultural land has numerous causes, including urban expansion, industrial development, soil erosion, desertification, and salinization (Conkling, J. A., & Mocella, C. J., 2010). These challenges are exacerbated by climate change, which alters precipitation patterns and increases the frequency of extreme weather events. The cumulative impact of these factors makes it clear that traditional methods of food production will not be sufficient to meet future global needs. This has led researchers, policymakers, and industry leaders to explore more sustainable and technologically advanced approaches to agriculture, particularly those that maximize yield without expanding land use (Mal'tsev, V. N., Zheltov, V. N., & Polovkov, A. V., 2016).

This trend is also evident in Uzbekistan, where food production is closely tied to land and water availability. The country possesses around 25.7 million hectares of agricultural land, of which 3.73 million hectares are irrigated (Muhammad, N., & Ahmed, I., 2019). Despite comprising a smaller fraction of total land, these irrigated areas contribute more than 97% of the nation's agricultural output, underscoring their critical role in sustaining food production. Given the limited availability of high-quality irrigated farmland and the growing need for intensified agriculture, modern agricultural practices must rely not only on traditional methods but also on technological and chemical advancements (Singh, H., Sharma, R., & Raghunathan, R., 2020).

Uzbekistan, like many other nations in arid and semi-arid regions, faces additional constraints including water scarcity, salinization, and land degradation. To address these issues, national strategies have increasingly emphasized the need for scientific and technological innovation in the agricultural sector. Among the most effective tools for improving productivity and sustainability is the strategic use of mineral fertilizers (Souri, A., & Dastanpour, R., 2020).

One of the most effective means of improving crop productivity is through the rational and balanced use of mineral fertilizers. Studies have shown that an average 40–50% increase in crop yield can be achieved

through proper fertilizer application. Consequently, the demand for mineral fertilizers has been increasing steadily across the globe, including in Uzbekistan, where several major chemical enterprises are engaged in the production of fertilizers specifically for agricultural use (Venkatesan, S., & Mani, N., 2021).

Mineral fertilizers provide essential nutrients that are either unavailable or insufficient in natural soils. The three most critical macronutrients – nitrogen (N), phosphorus (P_2O_5), and potassium (K_2O) – play fundamental roles in plant physiology. Nitrogen promotes vegetative growth, phosphorus is essential for energy transfer and root development, and potassium improves drought resistance and disease tolerance. The consistent supply of these nutrients in optimal proportions is critical to maximizing crop yields and ensuring food security (Wang, L., Zhang, Q., & Yu, X., 2022).

Key enterprises such as “Maxam-Chirchiq,” “Navoiyazot,” “Farg‘onaazot,” “Qo‘qon superfosfat,” “Ammofos-Maxam,” and the “Dehqonobod Potash Fertilizer Plant” manufacture a wide range of nitrogen, phosphorus, and potassium fertilizers. These include products such as ammonium nitrate, carbamide, ammonium sulfate, ammonium phosphate (AFU), ammophos, superphosphate, potassium chloride, and others. In recent years, the volume of production has reached approximately 1.348 million tons of nitrogen fertilizers, 130–140 thousand tons of phosphorus fertilizers, and 150 thousand tons of potassium fertilizers (based on 100% active ingredients). Among nitrogen fertilizers, ammonium nitrate, carbamide, and ammonium sulfate dominate the product range, with ammonium nitrate alone accounting for nearly 1.8 million tons (Zeglin, B., 2015).

Among the primary macronutrients essential for plant development – namely nitrogen (N), phosphorus (P_2O_5), potassium (K_2O), sulfur (S), calcium (CaO), and magnesium (MgO) – nitrogen holds particular significance due to its direct role in vegetative growth and chlorophyll formation. Nitrogen fertilizers such as ammonium nitrate, carbamide, ammonium sulfate, and AFU are widely used to supply this vital nutrient to crops (KCl, M., & Wulandari, R., 2018).

Experimental part

The primary reagent employed in this research was technical-grade ammonium nitrate (NH_4NO_3), widely used as a nitrogen fertilizer, with a certified purity not less than 98%. Serpentine, a magnesium-rich silicate mineral abundantly found in Uzbekistan, served as the stabilizing additive due to its chemical inertness and high thermal resistance. Prior to usage, serpentine was subjected to drying at 105 °C for two hours to eliminate residual moisture. The dried material was subsequently milled and passed through a sieve to obtain a particle size of less than 100 microns.

To assess the influence of serpentine on the thermal behavior and sensitivity of ammonium nitrate, a set of binary compositions was prepared. In each sample, 100 grams of ammonium nitrate was fixed, while serpentine was added in varying quantities ranging from 0.5 grams to 50 grams. All blends were homogenized via dry mixing for 10 minutes at 50 revolutions per minute using a laboratory rotary mixer. The mixing process was conducted in antistatic polyethylene containers under controlled laboratory conditions (ambient temperature of 22 ± 1 °C and relative humidity below 50%) to minimize hazards and ensure uniform dispersion of the additive.

To investigate the thermal stability of the prepared mixtures, Differential Scanning Calorimetry (DSC) and Thermogravimetric Analysis (DTA) were carried out using a NETZSCH STA 449 F3 Jupiter instrument. About 10 mg of each sample was sealed in an aluminum crucible and subjected to a programmed heating sequence from 25 °C to

400 °C at a constant rate of 10 °C/min under nitrogen gas flow (50 mL/min). Key parameters such as the onset temperature of decomposition, enthalpy changes, and total mass loss were systematically recorded for comparative analysis.

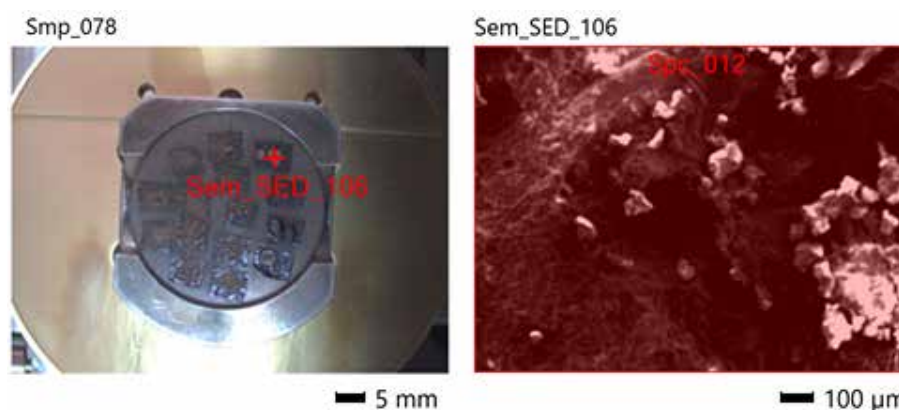
To elucidate the potential physical and chemical interactions between serpentine and ammonium nitrate, a series of characterization techniques were utilized:

- X-ray Diffraction (XRD) using $\text{Cu-K}\alpha$ radiation was applied to monitor changes in crystalline phases;
- Scanning Electron Microscopy (SEM) (JEOL JSM-IT500) was employed to evaluate surface texture and additive dispersion;
- Energy Dispersive X-ray Spectroscopy (EDS) was conducted to determine the elemental distribution and detect possible minor interactions between components.

Result and Discussion

In order to evaluate the impact of serpentine on the physicochemical behavior of ammonium nitrate, a series of binary mixtures were analyzed using thermal and structural techniques. Among the tested compositions, the formulation with an ammonium nitrate to serpentine ratio of 100: 2.5 demonstrated the most favorable characteristics in terms of thermal stability, phase integrity, and particle dispersion. Based on comparative analysis, this ratio was identified as optimal for reducing sensitivity while maintaining the essential oxidizing properties of the nitrate component.

Figure 1. SEM of 100:0.5 sample



The SEM micrograph of the 100: 0.5 sample, presented in Figure 1, reveals a relatively

uniform dispersion of serpentine particles across the ammonium nitrate matrix. The

fine particles of the additive appear to be well integrated into the host structure, forming a coherent microstructure without notable agglomeration. This morphology suggests enhanced structural compatibility, which contributes to the overall thermal and physical stabilization of the material.

Figure 1 presents the SEM image of the sample prepared with a 100:0.5 ammonium nitrate to serpentinite ratio. The image reveals a relatively uniform distribution of

serpentinite particles within the ammonium nitrate matrix. The bright, irregularly shaped particles correspond to serpentinite, while the background indicates the crystalline structure of ammonium nitrate. Most of the particles are smaller than 100 μm , and minimal agglomeration is observed. This suggests good dispersion and mechanical interaction between the components, contributing to the stability of the composite structure.

Table 1. Elemental composition of the sample (Sp_c_012) based on EDS analysis

Element	Mass % $\pm$ SD	Atom % $\pm$ SD
B	1.34 $\pm$ 0.02	1.90 $\pm$ 0.02
C	0.93 $\pm$ 0.01	1.19 $\pm$ 0.01
N	34.99 $\pm$ 0.09	38.30 $\pm$ 0.10
O	59.18 $\pm$ 0.16	56.71 $\pm$ 0.15
Mg	1.58 $\pm$ 0.01	1.00 $\pm$ 0.01
Al	0.11 $\pm$ 0.00	0.06 $\pm$ 0.00
Si	1.17 $\pm$ 0.01	0.64 $\pm$ 0.01
P	0.03 $\pm$ 0.00	0.02 $\pm$ 0.00
Ca	0.13 $\pm$ 0.01	0.05 $\pm$ 0.00
Fe	0.55 $\pm$ 0.01	0.15 $\pm$ 0.00
Total	100.00	100.00

According to the EDS results, the major elements detected in the sample are oxygen (O – 59.18 mass%) and nitrogen (N – 34.99 mass%), confirming the dominant presence of ammonium nitrate. Minor amounts of magnesium (Mg), silicon (Si), and iron (Fe) – characteristic of serpentinite – were also identified, indicating the additive's uniform dispersion within the matrix. Trace levels of carbon (C) and boron (B) were observed, likely originating from environmental contamination or residual processing components.

The X-ray diffraction pattern shown in Figure 2 corresponds to the optimized 100:0.5 ammonium nitrate–serpentinite composition. The dominant diffraction peaks are attributed to ammonium nitrate (NH_4NO_3), with an intensity contribution of 67.7%, confirming it as the primary crystalline phase in the sample.

Several secondary phases were also identified, indicating interactions or minor crystalline impurities likely introduced from the serpentinite additive. These include:

- $\text{Mg}_3\text{MgSi}_4\text{O}_{10}(\text{OH})_2$ (commonly part of serpentinite group minerals) – 4.3%;

- Calcium tetraboride (B_4Ca) – 4.1%;
- Magnesium oxide (MgO) – 0.2%;
- Siderazot (Fe_3N) and FeAl alloy ($\text{Fe}_{0.95}\text{Al}_{0.05}$) – trace levels ($\sim 0.1\%$);
- Calcium oxide (CaO) and calcium-related phases – minor presence.

The presence of nitrogen (N_2) and magnesium-rich silicates further confirms serpentinite decomposition products or reaction intermediates during processing.

The sharp, well-defined peaks indicate a crystalline and phase-separated structure, with no signs of amorphous or glassy phases. This suggests that serpentinite addition did not disrupt the crystalline nature of ammonium nitrate, but rather resulted in partial integration of stabilizing phases, potentially contributing to improved thermal stability.

The DSC thermogram presented in Figure 3 corresponds to the thermal behavior of the optimized 100:0.5 ammonium nitrate–serpentinite mixture. The graph displays both endothermic (green) and exothermic (red) events over the heating range of 20 $^{\circ}\text{C}$ to 180 $^{\circ}\text{C}$.

Figure 2. XRD Analysis Interpretation

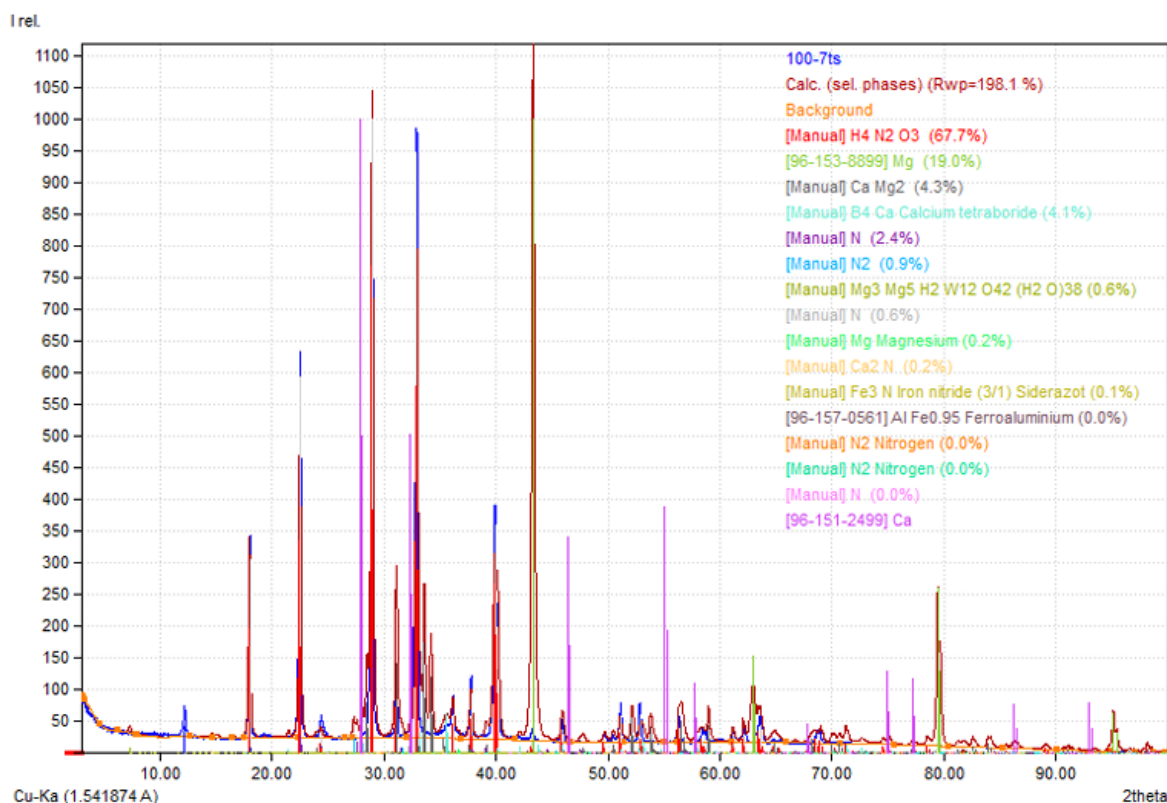
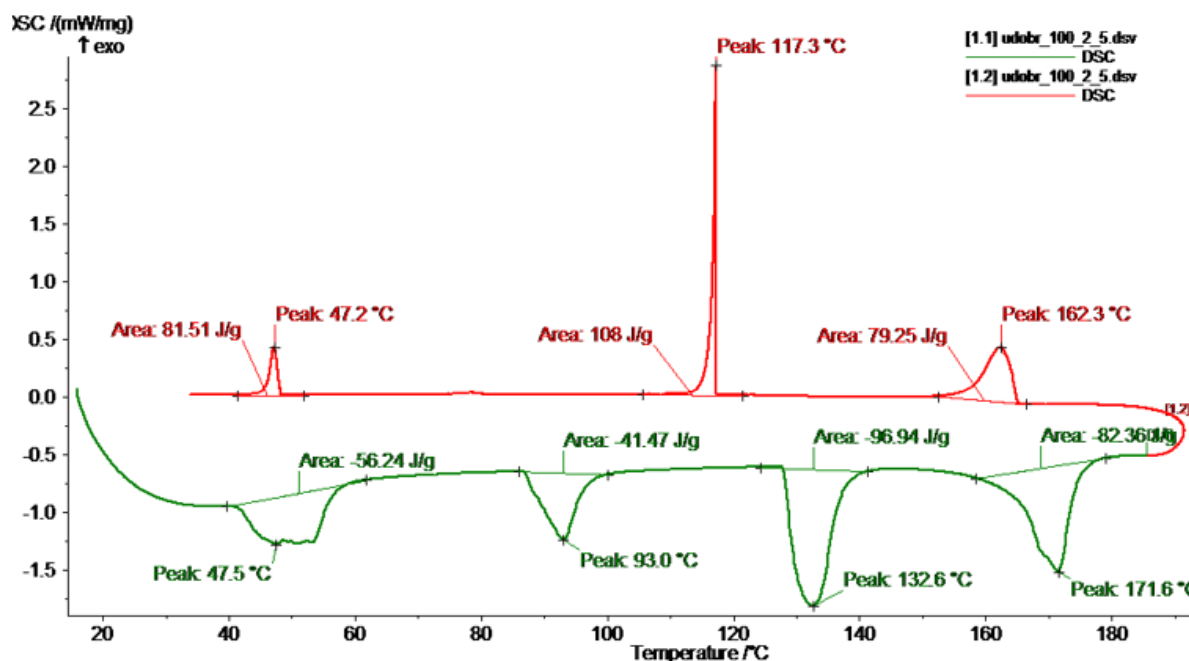


Figure 3. DSC Analysis Interpretation



Endothermic Events:

- Peak at 47.5 °C (Area: -56.24 J/g) indicates a solid-solid phase transition, likely within the ammonium nitrate crystal structure;
- Peak at 93.0 °C (-41.47 J/g) and 132.6 °C (-96.94 J/g) suggest pro-

gressive melting or decomposition stages;

- A final endothermic event at 171.6 °C (-82.36 J/g) is associated with the main decomposition or melting of ammonium nitrate.

Exothermic Events:

- Peak at 47.2 °C (Area: 81.51 J/g) likely corresponds to recrystallization or rearrangement of the additive phase;
- A sharp exothermic peak at 117.3 °C (Area: 108 J/g) is the most intense, indicating major decomposition or oxidation reaction;
- Additional exothermic responses at 79.25 J/g (162.3 °C) highlight further thermal events, possibly linked to the reaction of serpentinite components or secondary reactions.

Conclusion

The addition of serpentinite to ammonium nitrate was found to influence its physicochemical properties significantly. Based on XRD, SEM-EDS, and DSC analyses, the 10:2.5 ratio was identified as optimal, showing improved thermal stability, uniform particle distribution, and reduced decomposition sensitivity. These results confirm that serpentinite can effectively stabilize ammonium nitrate and enhance its safety for practical applications.

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