

An Extensive Analysis of Potassium Dinitramide (KDN): Synthesis, Properties and Applications

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Abstract- This work focuses on the synthesis, properties, and applications of potassium dinitramide (KDN), a critical intermediate in the development of eco-friendly green propellants like ammonium dinitramide (ADN). KDN is a much safer and environmentally friendlier option than traditional propellants such as ammonium perchlorate and hydrazine which have grave safety and environmental concerns. This study notes KDN's unique thermal expansion anisotropy, greater combustion performance with boron, and higher heat of combustion with carbon black, which strongly suggest the material's advanced propulsion systems potential. Several synthesis methods are presented that address the economics of acid concentration, reaction temperature, and neutralization time for maximum yield. It is concluded that KDN considerably enhances propellant stability, combustion efficiency, and environmental performance, thus supporting the green technology advancements in the aerospace and defense industries.

Keywords: Potassium dinitramide (KDN), Synthesis, Ammonium dinitramide (ADN), Ammonium perchlorate, neutralization, defense technology, energetic materials, nitramine oxidizer, green propellant, thermal stability, oxidizer

I. INTRODUCTION

Using crude gunpowder - a combination of potassium nitrate (saltpeter), sulfur, and charcoal - for rocket propulsion is over a century A.D. in China. At that time, the mixture was packed into bamboo tubes and thrown into fires, which led to some exploding, while others self-propelling into short distance sparking and flashing. With time, explosive filled bamboo tubes began to be used on arrows, leading towards advancements of rocket propulsion. The primary reasoning of rocket propulsion is Newton's third law of motion stating that every action has equal reaction in the opposite direction and with force. The propulsion works by setting on fire which releases gas. The gas expands exponentially and then is sent out, propelled through a high pressure rocket nozzle, which in turn pushes the rocket upwards. By about 1720, Dutch professor Willem Gravesande demonstrated the application of escaping gas for propulsion by designing steam-powered model vehicles. The most significant advance regarding propellants in the very early 20th century was by Robert Goddard. One of the earliest used oxidizers in rocket propellants is potassium nitrate or saltpeter. This compound became common because of its availability, stability, and effectiveness. Sucrose or table sugar acts as a binder and fuel when combined with potassium nitrate. In this particular mixture, potassium nitrate serves as the oxygen donor during combustion and sucrose as the energy donor. Together, they form a clean burning, smokeless, non-toxic propellant which makes this combination ideal for educational and small scale rocket projects. Because of its low cost and availability, potassium nitrate is an often used component in solid propellants. These propellants offer only moderate specific impulse when compared with advanced formulations; however, their ease of use and safety make them ideal for use in amateur rocketry or research projects. These are historically significant propellants and are still used today in applications ranging from model rockets to more advanced military and space systems that require stable and reliable performance.

1.1 ADDITIVES

Additives in solid propellants are intentionally introduced compounds designed to enhance specific properties such as burn rate, stability, energy output, and overall performance. These additives play a crucial role in improving the efficiency and safety of propellants, explosives, and pyrotechnic materials. By modifying

combustion behavior, they help control burn rates, ensure consistent performance, and prevent issues like phase transitions or crystallization. Additives also contribute to higher energy output, better combustion efficiency, and improved material stability under varying environmental conditions. Additionally, they can reduce the environmental footprint of propellants by lowering harmful emissions, thus supporting the shift toward greener and more sustainable propulsion technologies. In this context, potassium dinitramide (KDN) is explored as an additive and compared with potassium nitrate (KNO_3) and boron—compounds commonly used in igniters due to their ability to produce a high volume of hot particles. Furthermore, the inclusion of organic compounds, potassium-based catalysts, and copper catalysts helps stabilize the crystalline phase of ammonium nitrate (AN), thereby enhancing both the stability and performance of the propellant. [Pratim Kumar2(2)] [Pratim Kumar3(2)].

Finally, the the two propellants are relatively sensitive to handling, thus adding

1.2 DRAWBACK OF TRADITIONAL PROPELLENT

Traditional propellants such as ammonium perchlorate (AP) and hydrazine present several significant drawbacks. Both are highly toxic—AP produces environmentally harmful byproducts that contribute to pollution and ozone layer depletion, while hydrazine is a known carcinogen posing serious health hazards. In addition to their toxicity, these propellants suffer from poor storage stability. Hydrazine, being a corrosive liquid and an oxidizer, can degrade its containers over time, complicating long-term storage. AP also contributes to environmental degradation, raising serious ecological concerns. Moreover, both compounds are relatively sensitive to handling, increasing the risk of accidental ignition or explosion during storage or transportation. Although greener alternatives like ammonium dinitramide (ADN) are being explored to overcome these challenges, their widespread adoption remains limited due to high production costs and the early stage of their technological development. [V. Weiser4] [Michael J. Hardie5] [Pratim KUMAR6].

1.3 WHAT ARE GREEN PROPELLENT

To reduce the environmental risks and harmful emissions associated with traditional rocket propulsion, green propellants are being developed as more eco-friendly alternatives. Compounds such as ammonium dinitramide (ADN) and potassium dinitramide (KDN) represent promising efforts to replace conventional propellants like hydrazine and ammonium perchlorate (AP) with less toxic, higher-efficiency oxidizers. ADN, in particular, shows significant potential due to its lower environmental impact and enhanced performance in solid rocket formulations. These green propellants serve as intermediates in ADN synthesis and help stabilize the phase transitions of ammonium nitrate (AN), effectively addressing issues related to hygroscopicity and phase instability. This leads to improved burn rates and greater energy efficiency. By avoiding the formation of harmful chlorine-based emissions typical of AP, propellants such as AN and KDN support the advancement of cleaner, more sustainable technologies in space exploration and missile systems. [Martin Rahm7] [Pratim KUMAR6].

1.4 WHAT IS KDN?

Potassium dinitramide (KDN), with the chemical formula $\text{KN}(\text{NO}_2)_2$, serves as a key intermediate in the synthesis of ammonium dinitramide (ADN)—a more environmentally friendly oxidizer. KDN plays an important role in the production of ADN, which is widely regarded as a promising alternative to ammonium perchlorate (AP) in rocket propellants due to its reduced environmental impact. However, KDN is thermally unstable and must be stored in cool, dry conditions to prevent decomposition. Its primary application lies in its use as a precursor for ADN, which offers high performance, produces cleaner exhaust gases, and contributes significantly to the advancement of green and efficient propulsion technologies. [Kyeongmin Jang][Tomasz Golofit].

1.5 ADVANTAGES

Potassium dinitramide (KDN) is highly beneficial to the development of high-energy compositions and green propellants because of several important characteristics. As a precursor to ammonium dinitramide (ADN), KDN minimizes the environmental impact of rocket and missile technology by enabling green propellants to produce significantly lower toxic combustion products than the existing systems of hydrazine and ammonium perchlorate (AP). As a material that can deliver high heat of formation, good oxygen balance, and high density, KDN brings about better energetic performance of propellants as a result of better combustion efficiency and energy production. Phase transition stabilization of ammonium nitrate (AN), said to exhibit crystallographic transformation that may cause hygroscopicity, structural instability, and cracking in propellant blends, is one of the primary functions of KDN. To ensure the maintenance of the mechanical strength and homogeneity of the propellant, particularly under conditions of storage and under changing environmental conditions, such phase transitions need to be stabilized. Furthermore, KDN-based propellants also exhibit increased burn rates and improved combustion properties, especially when formulated with catalysts such as copper-cobalt oxides and

cupric oxide (CuO). The ability of KDN to be produced from comparatively low-cost raw materials renders it a low-cost precursor for the manufacture of ADN, yet another plus point. These combined benefits make KDN a pivotal component in the advancement of safer, more efficient, and environmentally sustainable energetic materials. (*Kyeongmin Jang, Pratim Kumar*).

1.6 FUTURE SCOPE

As an intermediate in green propellant production, namely ammonium dinitramide (ADN), a less toxic and more eco-friendly alternative to ammonium perchlorate in rocket propulsion, potassium dinitramide (KDN) plays a key role. With the significant reduction of chlorine emissions, ADN-based propellant systems are advantageous owing to their less environmentally harmful effects. Additionally, by preventing problems such as hygroscopicity and unwanted phase transitions, KDN stabilizes ammonium nitrate (AN), enhancing its energy density and structural strength. Due to these properties, KDN is a key component in the development of sustainable, high-performance propellants, explosives, and pyrotechnics, which is pertinent to the global trend toward green chemistry and eco-friendly technologies in the energy, aerospace, and defense industries.

2. EXPERIMENTAL APPROACH

2.1 MATERIALS

The chemicals required for this experiment include a variety of acids, alcohols, and potassium salts. Sulfamic acid (H_2NSO_3) is used to facilitate nitration reactions, while sulfuric acid (H_2SO_4), an effective dehydrating agent, is essential for the synthesis of nitro compounds. Nitric acid (HNO_3) plays a key role in the production of nitrated compounds and is also necessary for nitration processes. Potassium hydroxide (KOH) serves as a base in neutralization and ion-exchange operations. Ethyl alcohol ($\text{C}_2\text{H}_5\text{OH}$) and isopropyl alcohol ($\text{C}_3\text{H}_7\text{OH}$) are utilized as solvents in various stages of the experiment, primarily for purification and crystallization. Acetone (CH_3COCH_3) is also needed in the purification process due to its properties as a polar solvent. Potassium salts, including potassium chloride (KCl) and potassium sulfate (K_2SO_4), are employed in ion-exchange reactions during the synthesis of the desired compounds.

2.2 NITRATION PROCESS

In this experiment, the effects of varying acid concentrations, reaction temperatures, and neutralization conditions on the process are investigated. For the acid concentration experiment, nitric acid is tested at concentrations of 60%, 70%, 80%, 86%, 90%, and 99%, while sulfuric acid is available at concentrations of 95% or 98%. All tests are conducted at a constant temperature of -38°C with a fixed neutralization time of 30 minutes, and the neutralizing solution is maintained at 50%. The temperature reaction experiment is carried out at three different temperatures: -40°C , -38°C , and -35°C , using 99% nitric acid and 98% sulfuric acid, along with a neutralizing solution at 50% and a neutralization period of 30 minutes. The neutralization condition test is performed at a constant temperature of -38°C , using 98% sulfuric acid and 99% nitric acid. This test examines three different neutralization times (10, 20, and 30 minutes) and three concentrations of the neutralizing solution (12.5%, 25%, and 50%). The goal is to assess how variations in acid concentration, temperature, and neutralizing conditions affect the resulting products of the reaction.

A significant amount of laboratory equipment must be used in order to get this experiment done. Conical flasks, standard flasks, and measuring flasks are needed in the experiment in order to measure the chemicals correctly and mix them. Glass droppers are utilized for extremely accurate liquid handling, and a stirring rod is utilized to mix the reagents thoroughly. A weighing machine and spatulas are utilized for the transfer of solid chemicals to get accurate material measurements. Two crucibles are utilized in order to heat or calcine the chemicals at high temperatures, and two glass dishes are utilized for the evaporation of solvents or the separation of the precipitates. In addition to these materials and the proper chemicals, an elementary set of apparatus is thought to be necessary in order to carry out the investigation.

Some laboratory equipment is required to finish the experiment. To measure and mix chemicals accurately, conical flasks, normal flasks, and measuring flasks are required. To measure the liquids accurately, glass droppers are required, and a stirring rod will be required to make sure that all of the chemicals have been mixed well. Spatulas will be required to transfer solid chemicals, and a weighing device will be required in order to weigh the materials accurately. Two glass dishes will be required to evaporate the solvents and precipitates, and two crucibles will be required for higher heat or calcination of chemicals. The basis for conducting the investigation is formed by this equipment and the correct substances.

2.3 SYNTHESIS CONDITIONS

This experiment involved various tests to ascertain optimal KDN synthesis parameters like sulfuric and nitric acid concentration, synthesis temperatures, concentrations of neutralization solution, and neutralization time. Synthesized ones were compared. Experimental conditions in all cases are shown in Table No. 1.

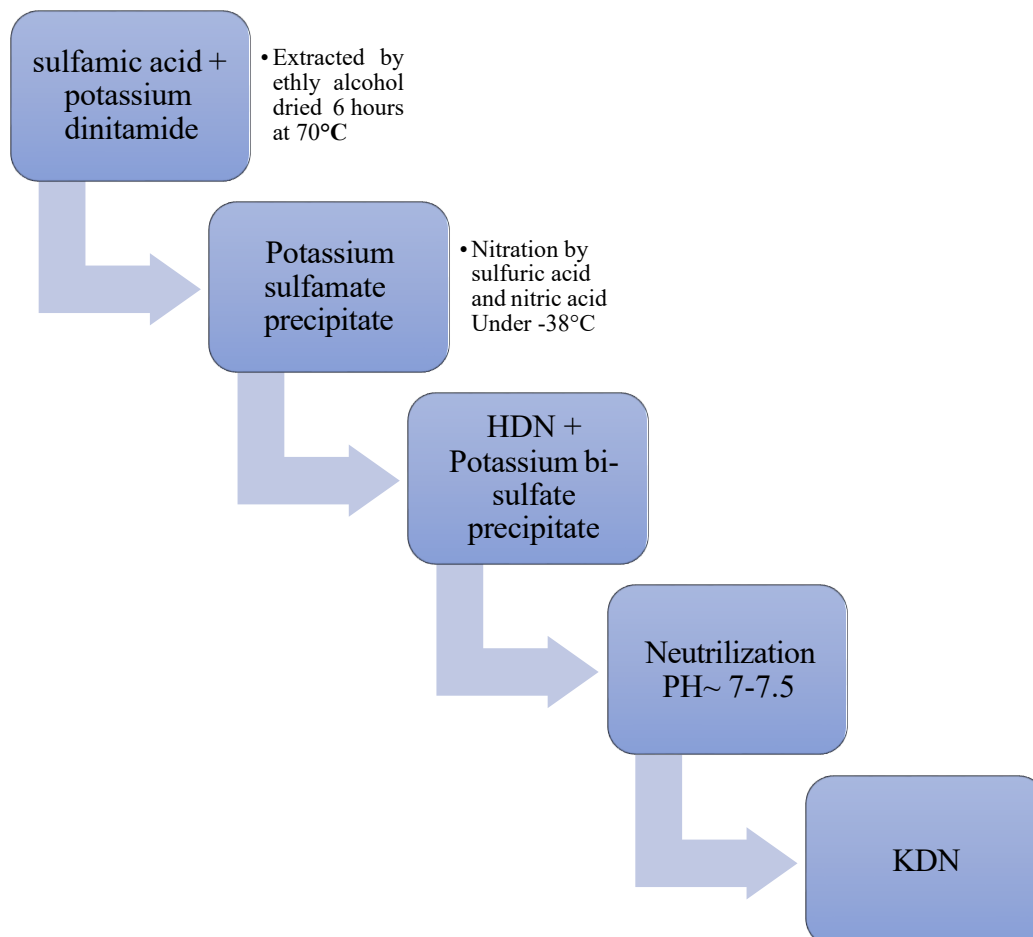


Figure 1: KDN synthesis flow diagram

Acidity level, temperature of reaction, and condition under neutralization experiment conditions are all thoroughly explained in the table. Each experiment includes varying amounts of factors and measuring the effects. Three experimental conditions were used in attempting to study the effect of temperature, acid concentration, and neutralizing conditions on the reaction process. Sulfuric acid concentration is 95% or 98%, and nitric acid concentration is 60% to 99% in the acid concentration experiment. The neutralizing time is always 30 minutes, but the neutralizing solution concentration is 50%. 99% nitric acid and 98% sulfuric acid are used in the reaction temperature experiment, which is roughly testing three temperatures: 40°C, 38°C, and -35°C. The neutralizing solution is 50% for 30 minutes. Lastly, the neutralizing condition experiment test was carried out at -38°C with 99% nitric acid and 98% sulfuric acid, 10, 20, and 30 minutes neutralization times, and 12.5%, 25%, and 50% concentrations of the neutralizing solution. The conditions offer the facility to experiment with different variables that influence the response process. Minimal lab equipment is needed to carry out the experiment effectively. Conical flasks, normal flasks, and measuring flasks are needed to obtain accurate measurements of chemicals and mix. A stirring rod is needed to enable equal mixing of the reacting chemicals, and glass droppers are needed to offer accurate handling of liquids. A weighing machine is needed to provide an accurate measurement of the substance, and spatulas are needed when handling solid chemicals. Two glass dishes evaporates solvent or separates a precipitate from another chemical. Using two crucibles will have to heat calcined at high temperatures, these material plus the specific chemicals will therefore become the very important equipment toward performing this study adequately.

Synthesis yields were then compared. Table No. 1 contains the complete experimental conditions.

Table No. 1: KDN Synthesis Experimental Conditions (Kyeongmin Jang) (Tomasz

Experiment	Variables	Values
Acid concentration	Temperature [°C]	-30/-20
	Nitric acid concentration [%]	60/70/80/90/99
	Sulfuric acid concentration [%]	95/98
	Neutralizing time [min]	30
	Neutralize solution concentration [%]	50
Reaction Temperature	Temperature [°C]	-40/-35/-30/-25
	Nitric acid concentration [%]	99
	Sulfuric acid concentration [%]	98
	Neutralizing time [min]	30
	Neutralize solution concentration [%]	50
Neutralizing Condition	Temperature [°C]	-35/-30/-25
	Nitric acid concentration [%]	99
	Sulfuric acid concentration [%]	98
	Neutralizing time [min]	10/20/30
	Neutralize solution concentration [%]	12.5/25/50

Gołofit) (Kumar & Joshi)

2.4 NITRIC ACID SYNTHESIS

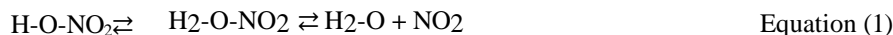
Preparation of fuming nitric acid is a delicate process. It can easily burn and evaporate instantly in shock. The compound is prepared only in the laboratory by the process of distillation. For the experiment, 110 g of potassium nitrate was combined with 60 mL of sulfuric acid, heated to 175°C, and left for two hours. As a result, 50 milliliters of primary nitric acid with a concentration of more than 95% were obtained. For the preparation of secondary nitric acid, 50 milliliters of primary as well as sulfuric acids were combined. Then, the resulting mixture was cooked at 120°C for one hour. Then the resultant mixture was fried for one hour at a temperature of 120°C. In the presence of 40 milliliters of extraction, the secondary concentration of nitric acid exceeded the purity of nitric acid is higher than 99.5%, and its density is 1.511 g/cm³.

2.5 POTASSIUM SULFAMATE SYNTHESIS

Sulfamic acid is utilized to neutralize potassium hydroxide for nitrating the potassium sulfamate precursor. This involves the addition of a solution containing 70.35 g of 98% sulfamic acid in 100 mL of distilled water. addition of 42.8 g of 95% potassium hydroxide in the same volume of distilled water to that solution. Due to the fact that potassium hydroxide dissolves exothermically and sulfamic acid dissolves endothermically, the potassium hydroxide solution was cooled to 25°C in a water jacket prior to mixing with the sulfamic acid. 250 milliliters of ethyl alcohol were added with the hope of precipitating the potassium sulfamate due to its solubility in water.

3. CHEMICAL REACTION DURING POTASSIUM DINITRAMIDE SYNTHESIS

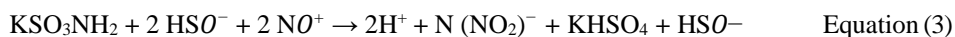
99.9%. Each of the nitrates collected was weighed on a density meter. From these data, this work utilized different experiments to adjust the conditions for synthesizing KDN. These were the concentration and % of nitric acid, the concentration and % of sulfuric acid, the synthesis temperatures, the concentration in the neutralizing solution, and the neutralizing time. The corresponding synthesis yields in each of these experiments were compared.



The reaction is the formation and dissociation of nitronium ion (NO_2^+) in the presence of sulfuric acid. Nitric acid (HNO_3) reacts with sulfuric acid (H_2SO_4), where H_2SO_4 protonates HNO_3 , forming the nitronium ion (NO_2^+) and water (H_2O). This ion can then participate in electrophilic substitution reactions, such as nitration. The equilibrium suggests a reversible process where water and nitronium ion are in balance.



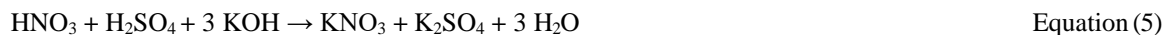
In this reaction, nitric acid (HNO_3) and sulfuric acid (H_2SO_4) react to give water (H_2O), bisulfate ion (HSO_4^-), and nitronium ion (NO_2^+). A proton from sulfuric acid would protonate the nitric acid, thereby paving the way to the formation of the nitronium ion, one of the strong electrophiles in nitration reactions.



In this reaction, it involves the conversion of sulfamic acid (KSO_3NH_2), reacting with bisulfate ions (HSO_4^-) and nitronium ions (NO_2^+), into the dinitramide anion ($\text{N}(\text{NO}_2)_2^-$), potassium bisulfate (KHSO_4), and sulfite ion (HSO_3^-). The reaction liberates protons in the transformation of reactants to products.



The process produces potassium dinitramide ($\text{K}_2\text{N}(\text{NO}_2)_2$), potassium sulfite (K_2SO_3), and water (H_2O) when the protons interact with the dinitramide anion ($\text{N}(\text{NO}_2)_2^-$), sulfite ion (HSO_3^-), and potassium hydroxide (KOH). In the reaction, ion exchange and neutralization result in the products.



(H_2O), potassium nitrate (KNO_3), and potassium sulfate (K_2SO_4) are the resultant products of this reaction between nitric acid (HNO_3), sulfuric acid (H_2SO_4), and potassium hydroxide (KOH). Due to the neutralization of the acids by the potassium hydroxide, salts and water are the end products. There were numerous contaminants in the KDN solution, including potassium nitrate, potassium sulfate, potassium bisulfate, and potassium sulfite. Using acetone and isopropyl alcohol for solvent extraction, these contaminants can be eliminated. Solvent extraction was used to remove the KDN mixture that contains KHSO_4 , K_2SO_4 , KNO_3 , and other contaminants after neutralization.

Among the contaminants were water, acetone, and isopropyl alcohol. It produced the matching green-yellow solution with KDN, acetone, and water throughout the extraction procedure. The pure solution's vivid green-yellow hue served as a reliable marker of successfully produced KDN.

Solvent	Water	Acetone	Isopropyl Alcohol
COMPONENTS			
KDN	Soluble	Soluble	InSoluble
KHSO_4	Soluble	Soluble	Soluble
K_2SO_3	Soluble	InSoluble	Sparingly
K_2SO_4	Soluble	InSoluble	InSoluble
KNO_3	Soluble	Soluble	Soluble

Table No. 2 KDN Solubility and Contaminants (1)

3.1 THE PROPERTIES OF DINITRAMIDE

Dinitramide ion is created from the interaction between two molecules of nitrogen dioxide and one nitrogen molecule during nitration. As posited by Nazeri et al., such a reaction takes place even when the temperature is low, as -20°C . Incidentally, as the temperature ranges from -45°C to -30°C , the reaction is activated. Dinitramide ions are stable even under weak basic conditions (7–8 pH), but are destroyed under acidic conditions. Besides, they undergo hydrolytic decomposition quickly when heated above 5°C . Hence, solutions of dinitramide ion need to be stored in weakly basic conditions at low temperatures. Dinitramide ions are identifiable through UV-Vis spectroscopy with absorbance at 284 nm. Thermal characteristics of the ions are investigated by methods such as Thermogravimetric Analysis and Differential Scanning Calorimetry (TGA-DSC). Melting point of salts of dinitramide varies according to the corresponding cations. Typically, these salts decompose readily above 90°C , giving off different nitrogen oxide gases. DSC-TGA serves to authenticate the identity and purity of dinitramide salts by determining their melting point, pyrolysis temperature, and the type of evolved gas.

3.2 KDN SYNTHESIS AND ANALYSIS RESULTS

Sulfuric acid is a catalyst for the nitration reaction, though it is not very efficient at inducing the decomposition of nitric acid into nitro groups at low concentrations. Sulfuric acid concentrations of 95% or higher are usually needed to enable effective nitration. Both sulfuric and nitric acid concentrations are important in determining the yield of the reaction during KDN synthesis. It was noted that KDN synthesis was not possible when concentration of nitric acid was maintained at 60%, even though the concentration of sulfuric acid was raised from 95% to 98%. A successful synthesis of KDN was, however, obtained when nitric acid concentration was more than 70%, and the yield was better as the concentration was higher. Interestingly, once the concentration of nitric acid was more than 93%, there was a dramatic improvement in the synthesis yield. At a concentration of about 86%, nitric acid is white and gives off nitrogen dioxide gas, also known as "white fuming nitric acid." When the concentration is above 93%, the acid becomes yellow and gives off a heavy, dark yellow nitrogen dioxide gas, referred to as red fuming nitric acid (RFNA). At this concentration, nitric acid easily decomposes to produce nitro groups and nitrogen dioxide into the reaction mixture and hence enhance nitrating significantly and converting the overall yield of the synthesis to a much higher level. The employment of very concentrated sulfuric acid also had this impact. In addition, the concentrated acids had fewer impurities—stabilizers or excess water—present, so byproducts such as HDN (hexanitrodiazapentalene) and other possible impurities remained in their molecular form or precipitated out from the low water content. Also, the used strong acids could not hydrolyze water under these conditions. Therefore, strong acidic conditions were not necessary to initiate the synthesis or precipitation of HDN prior to the neutralization step.

3.3 YIELD OF KDN SYNTHESIS BASED ON SULPHURIC AND NITRIC ACID CONCENTRATIONS

The Catalyst for the process of nitration is sulfuric acid, though it is not highly effective at degrading nitric acid into nitro groups when the concentration is low. Sulfuric acid is usually in excess of the concentration required for the proper nitration when it is concentrated to a level of 95% and above. The synthesis and yields of KDN depend on the concentrations of sulfuric and nitric acids. KDN was not obtainable if the concentration of nitric acid remained at 60%, even when the concentration of sulfuric acid was increased from 95% to 98%. Nevertheless, it was possible to synthesize KDN when the concentration of the nitric acid was above 70%, and the yield increased as the concentration of the nitric acid increased. It should be noted that when the concentration of nitric acid was increased above 93%, the yield of synthesis was greatly enhanced.(1) One of the very effective salts that hold potential to be an environmentally friendly space propellant is ADN. Direct synthesis is however difficult, and KDN, its precursor, has a remarkable influence on its productivity and yield. The aim of the present work is to enhance the productivity of ADN by exploiting the synthesis typical of KDN, synthesized using potassium sulfamate, nitric acid, and sulfuric acid. (1) Several variables, such as temperature, neutralization time, concentration of nitric acid, and concentration of the neutralizing agent, had a great impact on this yield. The yield of KDN increased at higher values above 93% when the concentration of nitric acid was increased further. Nitric acid concentration was the principal factor affecting the yield of synthesis of KDN. Since highly concentrated nitric acid is volatile and rapidly forms nitro groups, more amounts of KDN are formed.

With an optimal yield of 45.04% for the synthesis of KDN, the ideal neutralizing conditions thus seemed to be at about 38°C and for 30 minutes. In conclusion, By improving KDN synthesis, the production rate of KDN, ADN, and various other dinitramide salts could be increased. It thus opens opportunities for thrusters based on larger ADN, which will, in turn, pave the way for a safer propulsion technology in space research that is more

environment-friendly.

3.4 TEMPERATURE-DEPENDENT YIELD OF KDN SYNTHESIS One of the most important parameters during KDN and ADN synthesis is reaction temperature. Although nitration at temperatures under 20°C is possible but becomes vigorous at around 35°C. The synthesis yields at 35°C, 38°C, and 40°C are 43.51%, 45.04%, and 44.72%, respectively. Higher temperature generally favors the incorporation of nitro groups. But in this experiment, the best temperature for the highest yield is 38°C since dinitramide was produced. Because dinitramide synthesis is a thermally exothermic reaction, higher yields were expected when carried out at lower temperatures. Nevertheless, the optimum yield was noted at 38°C. This is due to the fact that, at temperatures lower than 38°C, the mixture freezes, thus making it hard to carry out vigorous agitation and, therefore, poor efficiency during the reaction

3.5. YIELD OF KDN SYNTHESIS DEPENDING ON NEUTRALIZING CIRCUMSTANCES

During the synthesis of KDN and other dinitramide salts, the neutralization temperature needs to be kept below 5 °C. Because the neutralization is an exothermic reaction, the mixture employed in this study needed to be neutralized between 5 °C and 10 °C. The HDN mixture, having a high acid strength, interacted with the highly basic neutralizing agent (usually KOH), liberating a lot of heat within a short period of time. This made it difficult to sustain an even reaction temperature and to properly control the time of neutralization.

It was noticed that with the longer the period of the neutralization process, the yield of the final KDN product progressively increased and stabilized at 30 minutes, which became the best neutralization time. To regulate the temperature of the reaction, the mixture was agitated continuously and cooled with dry ice. Regardless of these measures, the inner temperature was usually more than 5 °C as a result of the heat generated during neutralization. Lengthening the period of neutralization permitted lowering of the central temperature slightly, and this minimized KDN's thermal degradation, besides improving general yield.

At 30 minutes, no additional variations were noted in the extracts, indicating that the blend had attained thermal equilibrium, assisted by the coolant removing enough heat to keep the temperature between 5 °C and 10 °C. Initially, the flow rate of KOH solution was increased from 0.1 mL/s to 1 mL/s stepwise in order to prevent thermal degradation of HDN.

Both the concentration and the duration of the KOH solution were important variables affecting the yield. Increased KOH concentrations produced better yields. Prior to neutralization, the HDN mixture—nitric acid, sulfuric acid, and impurities—contained low water content and was mostly unionized. But when low-concentration neutralizing solutions with excess water were applied, the HDN mixture ionized, resulting in highly acidic conditions that disrupted the complete conversion of HDN to KDN. This dilution of dinitramide ions lowered the efficiency of synthesis.

Thus, to avoid HDN degradation and achieve maximum KDN yield, a very concentrated, cold KOH solution and a neutralization time of at least 30 minutes are required.

4. COMPARISON

• Phase Transition and Stability Improvements

KDN (potassium dinitramide) stabilizes ammonium nitrate (AN) efficiently, with a great decrease in its phase transition propensity. This stabilization is particularly significant at increased levels, significantly at 50% as well. Copper oxide (CuO) also supports thermal stability and phase stabilization. The Cu-Co* catalyst maximizes combustion efficiency through higher burning rates.

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• Catalytic Efficiency

The Cu-Co* catalyst shows excellent performance in enhancing the energetic properties of propellants and facilitating sustained burning, even at low pressure. Cu-Co* improves combustion properties while CuO excels at enhancing thermal stability, thus being best suited for high-thermal-resistance applications.

• Environmental Benefits

Green oxidizers like KDN and ammonium dinitramide (ADN) produce fewer chlorine-based by-products than conventional oxidizers like ammonium perchlorate (AP), which makes them potential substitutes for environmentally friendly propellant formulations. AN and KDN have fewer toxic emissions, making them better choices for future propulsion systems.

Parameter	Thermal Expansion (KDN)	Burning Behavior (B/KDN)	Heat of Combustion (KDN)
Material Studied	Potassium Dinitramide (KDN)	Potassium Dinitramide (KDN) mixed with Boron	Potassium Dinitramide (KDN) mixed with Carbon Black (CB)

• Burning Rate and Combustion Stability

KDN, when blended with Cu-Co*, serves to alleviate such universal issues as the phase instability and hygroscopicity of AN. Both decomposition and combustion performance are improved by this blend. Addition of Cu-Co* to AN-based propellant systems not only enhances stability but also raises energy density, and thus these systems find application in next-generation rocket propulsion.

• Thermal and Combustion Analysis

This research addresses burning rate experiments performed under pressure-controlled conditions. Companion analyses consist of decomposition behavior and thermal stability evaluations with methodologies such as kinetic modeling, thermogravimetric analysis (TGA), and differential scanning calorimetry (DSC).

• Applications and Innovations

The inclusion of catalysts such as Cu-Co* within AN-based propellants is a strategic improvement toward the compromise between stability and energetic performance. This methodology offers considerable promise toward the development of high-efficiency and environmentally sound propulsion technologies.

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• Application Innovation

The addition of catalysts such as Cu-Co* in AN-based propellants is a balanced approach to enhancing stability and energetic performance. This innovation has potential applications in environmentally friendly and high-performance propulsion

Temperature Range	85–298 K	Combustion observed under controlled pressure at 1 MPa and 7 MPa	Combustion tested using a bomb calorimeter setup
Thermal Behaviour	Anisotropic thermal expansion, along b-axis	N/A	Heat release depends on AP/KDN/CB ratios
Combustion Behaviour	N/A	Rapid ignition; higher pressure and temperature for B/KDN	KDN burns with CB; stable combustion requires AP addition
Pressure Generation	N/A	Peak ~2.8 MPa for B/KDN (15:85), compared to ~1.6 MPa for B/KNO ₃	N/A
Peak Combustion Temp.	N/A	4900 K for B/KDN; significantly higher than B/KNO ₃ (~3900 K)	Not directly mentioned; AP/KDN mixtures release higher heat
Oxidizing Efficiency	N/A	KDN produces more gas and sustains higher temperatures	Lower efficiency unless mixed with AP
Applications	Structural properties, sensitivity to temperature changes	Igniters for propellants	Potential use in green propellants

Table No. 3 Comparative description of KDN

The table consolidates the performance and properties of potassium dinitramide (KDN) under three experimental conditions: thermal expansion, combustion characteristics, and heat of combustion. Thermal expansion studies indicate that KDN is anisotropic, with the highest expansion observed along the b-axis between 85 K and 298 K. This anisotropy plays a significant role in the material's structural stability and temperature sensitivity. In combustion characteristics research, boron (B) doped KDN exhibits fast ignition and much greater pressure and temperature yields than typical oxidizers such as potassium nitrate (KNO₃). A 15:85 B/KDN composition yields a maximum combustion pressure of about 2.8 MPa and achieves a combustion temperature of 4900 K, significantly higher than the 3900 K found in B/KNO₃ compositions. These conclusions highlight the efficiency of KDN as an aggressive oxidizer, which can produce large amounts of gas and can resist high temperatures, thus it is suitable for application in propellant igniters.

For combustion heat, tests with KDN in conjunction with carbon black (CB) in stable combustion were carried out through bomb calorimetry. Ammonium perchlorate (AP) had to be added to the mix, and heat output depended on the ratio of AP/KDN/CB. These findings confirm KDN's viability as an environmentally benign high-energy oxidizer for inclusion in green propellant compositions. In summary, the information emphasizes KDN's multi-variable versatility and high-performance attributes in numerous thermal and combustion parameters, validating its potential for sophisticated, environmentally friendly propulsion systems, particularly in boron- or carbon-based additive formulations.

Aspect	Process 1: Using Sulfamic Acid, Nitric Acid, and Sulfuric Acid (KDN Synthesis (2) [7])	PROCESS 2: Langlet's Method (Modified) (Optimization of Potassium Dinitramide Preparation [8])	Process 3: Using Potassium Sulphamate and Fuming Nitric Acid (Kumar & Joshi [9])
Precursor	Potassium sulfamate synthesized from sulfamic acid and KOH	Potassium sulfamate synthesized from sulfamic acid and KOH	Potassium sulfamate synthesized from sulfamic acid and KOH
Nitrating Agents	Nitric acid (HNO ₃) and sulfuric acid (H ₂ SO ₄)	Nitric acid (999%) and sulfuric acid (95%) in specific molar ratios	Nitric acid (100%) and sulfuric acid (95%)
Reaction Temperature	-35 °C to -40 °C	-40 °C	-40 °C
Reaction Duration	30 minutes for nitration	25 minutes for nitration	30 minutes for nitration
Neutralization Conditions	KOH solution at 0 °C to -10 °C	KOH solution at -50 °C, gradually raising temperature during neutralization	KOH solution at 0 °C to -10 °C
Purification Method	Solvent extraction using acetone and isopropyl alcohol	Solvent extraction with acetone; reduced water for cost efficiency	Solvent extraction using acetone
Yield	>40%	~55.8%	~48%
Key Observations	Moderate cost; nitration occurs at higher temperature compared to organic methods	Optimized for commercial scalability; precise control of acid ratios and reaction times	Uses pure nitric acid; acetone preferred for KDN extraction
Notable Challenges	Requires strict temperature control and additional purification	High viscosity of reaction mixture creates challenges at low temperatures	Slightly hygroscopic KDN crystals; KN forms over time, affecting purity
Advantages	Uses inexpensive raw materials; higher temperature reduces cooling needs	Higher yield; optimized for large-scale production	Purity of ~99%; straightforward neutralization and extraction

Table No. 4 Comparative description of synthesis process of KDN

The table presents a comparative analysis of three distinct synthesis methods for potassium dinitramide (KDN), highlighting variations in precursors, nitrating agents, reaction conditions, purification techniques, and overall outcomes. All three methods begin with potassium sulfamate, synthesized from sulfamic acid and KOH. While the nitrating agents used are generally similar, Process 1 utilizes a combination of nitric acid and sulfuric acid. In contrast, Processes 2 and 3 employ more concentrated forms, with Process 3 specifically using pure nitric acid for enhanced reactivity.

Reaction temperatures also vary: Process 1 is carried out between -35°C and -40°C, while Processes 2 and 3 are maintained at a consistent -40°C. Reaction duration differs slightly, with Process 2 requiring 25 minutes and Processes 1 and 3 taking approximately 30 minutes each. Neutralization approaches show further variation. In Process 2, KOH is added at -50°C in a controlled manner, with the temperature incrementally increased. Conversely, Processes 1 and 3 carry out neutralization between 0°C and -10°C. All three processes use solvent extraction for purification; however, Process 2 is more water-efficient, which contributes to lower production costs. In terms of yield, Process 2 achieves the highest at around 55.8%, followed by Process 3

at approximately 48%, and Process 1 yielding just over 40%. Key differences also emerge in scalability, cost-efficiency, and product purity—Process 2 is best suited for large-scale manufacturing, while Process 3 delivers the highest purity at roughly 99%. Despite challenges such as precise temperature control, increased viscosity during reaction, and the hygroscopic nature of KDN crystals, the synthesis methods—especially Process 2—demonstrate potential for cost-effective production with reduced cooling requirements, depending on the chosen protocol.

5. CONCLUSION

Being a very energetic compound, ammonium dinitramide (ADN) is still a potential green propellant for space travel. Nevertheless, direct synthesis of ADN is difficult, and yield and productivity are highly dependent on the availability and purity of potassium dinitramide (KDN), which is an important precursor. To maximize ADN production, research was carried out intensively on the synthesis parameters of KDN. It was synthesized with potassium sulfamate, nitric acid, and sulfuric acid. Some variables were determined to be the most important drivers of the synthesis yield—viz., reaction temperature, neutralization time, the concentration of nitric acid, and the concentration of the neutralizing agent. The most essential aspect was found to be nitric acid concentration. Increased concentration led to easier formation of nitro groups with improved efficiency, thereby increasing the yield of KDN substantially under the best of circumstances to a figure greater than 93%. Optimum temperature and time of neutralization were identified as -38°C and 30 minutes, respectively, providing a highest of 45.04%. Overall, the research reaffirmed that synthesis of KDN in optimized conditions not only enhances ADN production efficiency but also performance of KDN and related salts of dinitramide. This improvement advocates for wider utilization of thrusters based on KDN, a safer and ecologically more viable alternative to traditional propulsion systems.

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