International Journal of Advanced Research in Science, Engineering and Technology

ISSN: 2350-0328

Vol. 12, Issue 8, August 2025

Innovative Technology for the Synthesis of Suspension-Based Liquid NPK Fertilizers

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ABSTRACT: This study demonstrates the feasibility of producing novel, effective suspended compound fertilizers (NPK) utilizing nitric acid decomposition products of calcium-containing sludge, feed-grade ammonium phosphate (CFA), urea, and potassium chloride sourced from the Dekhkanabad Potash Fertilizer Plant. Through a series of experiments, optimal technological conditions and parameters for synthesizing these new NPK formulations were identified.

KEY WORDS: Calcium sludge, potassium chloride, ammonium nitrate, ammophos, base solution.

I.INTRODUCTION

Following Uzbekistan's independence, significant efforts were directed toward the advancement of scientific research and the development of cost-effective agricultural technologies. These initiatives have led to meaningful progress in the production of nitrogen, phosphorus, and potassium-based fertilizers, as well as other essential nutrients, primarily using local raw materials. Notably, while fertilizers such as ammophos, nitrophos, superphosphate, and ammonium nitrate are widely used to enhance crop productivity, they often fall short in ensuring high yields. Furthermore, the energy-intensive nature of their production processes is frequently overlooked. In developing technologies for suspended fertilizers, several critical scientific considerations must be addressed. These include: devising an efficient method for the partial decomposition of sludge using nitric acid; and optimizing the conditions for producing suspended fertilizers from a mixture of calcium nitrate sludge, ammophos, potassium chloride, and ammonium nitrate.

II. METHODOLOGY

Laboratory experiments were conducted using calcium-containing sludge sourced from the water treatment unit of JSC "Farg'onaazot." The chemical composition of the sludge was as follows (wt.%): CaO – 44.95; MgO – 2.00; CO_2 – 37.51; H_2O – 15.01; and other impurities – 0.85. Additional raw materials used in the study included:

Potassium chloride (K_2O – 60%) from the Dekhkanabad Potash Fertilizer Plant

Urea (N – 46%)

Ammophos (N – 10%, P_2O_5 – 45%)

Nitric acid (HNO_3 – 57%)

Preparation of Base Suspension

To synthesize the suspended complex NPK fertilizers, a base suspension was first prepared. The calcium sludge was decomposed using nitric acid at 35–45 °C for 20–25 minutes, applying nitric acid in a stoichiometric ratio (100%) relative to CaO content. This reaction yielded a calcium nitrate-based solution (hereafter referred to as CNS – Calcium Nitrate Solution). Further chemical analysis of the synthesized suspension-type NPK fertilizers (SSUs) revealed notable differences in composition depending on the nutrient ratios used during synthesis. It was found that the SSU prepared from a base suspension containing ammophos with a nutrient ratio of N: P_2O_5: K_2O = 1:1:1 exhibited a distinct composition. Specifically, 7.85% of the total nitrogen was present in the nitrate form, primarily as calcium nitrate ($Ca(NO_3)_2$ – 34.33%) and magnesium nitrate ($Mg(NO_3)_2$ – 1.91%). Additionally, the mixture contained 16.37% ammophos and 12.00% potassium chloride (KCl). In contrast, SSUs with N: P_2O_5: K_2O ratios of 2:1:1 and 3:1:1 showed elevated nitrogen content, with 10.90% and 12.90%, respectively, existing in both ammonium and nitrate forms. These formulations contained:

Phosphorus (P_2O_5): 5.45% and 4.30%

Potassium (K_2O): 5.45% and 4.30%

Calcium (Ca): 8.87% and 7.00%

These results suggest that increasing the nitrogen content in the nutrient ratio affects not only the total nitrogen levels but also the distribution between nitrate and ammonium forms, which has direct implications for nutrient uptake efficiency in crops. Further formulations using an ammophos base suspension with a 2:1 ratio and varying potassium chloride inputs produced SSUs containing:

Nitrogen: 8.82–14.19%,

mainly from a combination of:

Calcium nitrate ($Ca(NO_3)_2$): 11.27–21.01%

Magnesium nitrate ($Mg(NO_3)_2$): 0.63–1.17%

Ammonium nitrate (NH_4NO_3): 20.04–32.55%

Ammophos: 8.82–10.75%

Potassium chloride (KCl): 7.87–14.70%

These variations highlight the flexibility of the formulation process, allowing targeted adjustment of nutrient concentrations based on agricultural needs. Notably, higher nitrogen levels correspond to increased ammonium nitrate content, while the ratio of calcium and magnesium nitrates varies proportionally to the amount of nitric acid used in the decomposition process.
Overall, these findings demonstrate that precise control of the initial nutrient ratios and base suspension composition enables the design of balanced, highly soluble, and agronomically effective NPK suspension fertilizers.
Subsequently, ammophos granules were added to the CNS at a temperature of 70–80 °C, under continuous stirring. The mixture ratios of CNS to ammophos were varied across the following proportions: 1:1, 1:0.75, 2:1, and 4:1, in order to determine optimal formulation conditions.

Synthesis of Suspended NPK Fertilizer

To enhance the concentration of the final NPK suspension, calculated amounts of ammonium nitrate, potassium chloride, and distilled water were added to the base suspension under continuous agitation until complete dissolution.
Analytical Methods Phosphorus content (total P_2O_5, water-soluble P_2O_5, and plant-available P_2O_5) in both raw materials and synthesized products was determined using the photocolorimetric method, forming a yellow phosphorus-molybdenum complex, measured at $\lambda = 440$ nm using a KFK-3 photocolorimeter [1, pp. 16–22; 2, p. 22].Nitrogen content was analyzed using the Kjeldahl distillation method and the chloramine method [3, p. 7]. Potassium content in the final samples was determined using flame photometry techniques according to established procedures [4, pp. 11–18].

Physical Property Measurements

The viscosity of liquid and suspension-type fertilizer samples was measured using a VPZh-2 glass capillary viscometer with a capillary diameter of 0.77 mm across a temperature range of 10–50 °C. This allowed for the assessment of the temperature dependence of flow properties relevant to storage and application.Density measurements were performed using the pycnometric method, ensuring high accuracy in determining the mass-to-volume ratio of the fertilizer solutions.The crystallization temperature of the liquid suspension fertilizers was determined using the visual-polythermal method as described in [5, p. 94]. Due to the turbidity and suspended particles present in suspension fertilizers, direct measurement of freezing points is not feasible. Therefore, only the clarified (supernatant) portion of the liquid fertilizers was used in this analysis.

III. EXPERIMENTAL RESULTS

To obtain a triple-component suspended NPK fertilizer, a pre-prepared NP-based suspension was used as the base. At a temperature of 70–80 °C, and under continuous stirring, a calculated amount of potassium chloride (KCl) was gradually introduced and dissolved in the suspension. This ensured uniform dispersion of the potassium source within the fertilizer matrix. Once a homogeneous suspension was achieved, the mixture was gradually cooled to 20–25 °C under constant agitation to maintain stability and prevent premature crystallization or phase separation. The cooling step was critical to stabilizing the suspension and optimizing the final physical properties of the product. The results of the chemical analysis and the evaluation of physical properties of the synthesized NPK suspension fertilizers are presented in Tables 2.10–2.12.

International Journal of Advanced Research in Science, Engineering and Technology

Vol. 12, Issue 8, August 2025

ISSN: 2350-0328

IJARSET IMPACT FACTOR 7.150

These tables highlight how nutrient ratios, base suspension concentrations, and potassium chloride dosages influenced the final composition, viscosity, crystallization behavior, and overall stability of the fertilizer products. The experimental data revealed that: Increasing the KCl content improved the overall K_2O concentration but also influenced the viscosity and crystallization threshold. Optimal nutrient ratios were achieved at a CNS: ammophos ratio of 2:1, which yielded a stable suspension with desirable flow properties and nutrient availability. Excessive potassium chloride addition led to a slight increase in crystallization temperature and viscosity, which may affect long-term storage but remained within acceptable ranges. These findings suggest that careful optimization of component ratios allows for the production of balanced, stable, and efficient liquid NPK fertilizers suitable for agricultural application.

It was established that in the solid suspension unit (SSU) derived from a basic suspension of ammophos with a nutrient ratio of $N:P_2O_5:K_2O$ = 1:1:1, approximately 7.85% of the total nitrogen is present in the nitrate form, predominantly as calcium nitrate [$Ca(NO_3)_2$] and magnesium nitrate [$Mg(NO_3)_2$]. The composition of this SSU is as follows:

- 34.33% calcium nitrate [$Ca(NO_3)_2$],
- 1.91% magnesium nitrate [$Mg(NO_3)_2$],
- 16.37% ammophos, and
- 12.00% potassium chloride (KCl).

In contrast, SSUs prepared with $N:P_2O_5:K_2O$ ratios of 2:1:1 and 3:1:1 were found to contain:

- 10.90% and 12.90% total nitrogen, in both ammonium and nitrate forms,
- 5.45% and 4.30% phosphorus (P_2O_5),
- 5.45% and 4.30% potassium (K_2O),
- and 8.87% and 7.00% calcium, respectively.

Furthermore, SSUs derived from ammophos suspensions with a 2:1 base ratio, depending on the specific ratio of nutritional components, contained 8.82% to 14.19% total nitrogen. The nutrient content of these SSUs varied as follows:

- Calcium nitrate [$Ca(NO_3)_2$]: 11.27% to 21.01%,
- Magnesium nitrate [$Mg(NO_3)_2$]: 0.63% to 1.17%,
- Ammonium nitrate [NH_4NO_3]: 20.04% to 32.55%,
- Ammophos: 8.82% to 10.75%, and
- Potassium chloride (KCl): 7.87% to 14.70%.

These results indicate that by altering the $N:P_2O_5:K_2O$ ratio in the base suspension, it is possible to tailor the composition of SSUs, especially in terms of nitrogen form and content, enabling the development of targeted fertilizers suitable for specific agronomic requirements.

Table 1. Chemical Composition of Complex Suspension NKP Fertilizers Based on Ammophos, Ammonium Nitrate, and Potassium Chloride (% by mass)

$N:P_2O_5:K_2O$ Ratio	NKS:Ammophos Ratio	N (%)	P_2O_5 (%)	CaO (%)	K_2O (%)	MgO (%)	H_2O (%)
1:1:1	4:1	7.85	7.20	11.71	7.20	0.51	35.20
2:1:1	4:1	10.90	5.45	8.87	5.45	0.39	36.37
3:1:1	4:1	12.90	4.30	7.00	4.30	0.31	37.13
1:1:1	2:1	8.82	8.82	7.17	8.82	0.32	34.12
2:1:1	2:1	12.31	6.16	5.01	6.16	0.22	35.90
3:1:1	2:1	14.19	4.73	3.84	4.73	0.17	36.85
1:1:1	1:0.75	9.39	9.40	5.09	9.40	0.23	33.74
2:1:1	1:0.75	12.87	6.44	3.49	6.44	0.15	35.71
3:1:1	1:0.75	14.69	4.90	2.65	4.90	0.12	36.74
1:1:1	1:1	9.71	9.71	3.95	9.71	0.17	33.52
2:1:1	1:1	13.16	6.58	2.67	6.58	0.12	35.61
3:1:1	1:1	14.92	4.97	2.02	4.97	0.08	36.69
1:2:1	—	6.37	12.80	5.20	6.37	0.23	31.50

ISSN: 2350-0328

International Journal of Advanced Research in Science, Engineering and Technology

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Table 2.11. Salt Composition of Complex Suspension NKP Fertilizers Based on Ammophos, Ammonium Nitrate, and Potassium Chloride (% by mass)

$N:P_2O_5:K_2O$ Ratio	NKS:Ammophos Ratio	H_2O (%)	$Ca(NO_3)_2$ (%)	$Mg(NO_3)_2$ (%)	NH_4NO_3 (%)	Ammophos (%)	KCl (%)
1:1:1	4:1	35.20	34.33	1.91	—	16.37	12.00
2:1:1	4:1	36.37	25.99	1.45	14.56	12.40	9.07
3:1:1	4:1	37.13	20.51	1.14	24.14	9.78	7.16
1:1:1	2:1	34.12	21.01	1.17	13.99	8.82	14.70
2:1:1	2:1	35.90	14.67	0.82	20.04	24.27	10.26
3:1:1	2:1	36.85	11.27	0.63	32.55	10.75	7.87
1:1:1	4:3	33.74	14.93	0.83	13.39	21.36	15.66
2:1:1	4:3	35.71	10.22	0.57	28.09	14.62	10.73
3:1:1	4:3	36.74	7.77	0.43	35.80	11.13	8.15
1:1:1	1:1	33.52	11.57	0.59	15.90	22.08	16.19
2:1:1	1:1	35.61	7.84	0.44	30.12	14.96	10.97
3:1:1	1:1	36.69	5.92	0.33	37.42	11.31	8.28
1:2:1	—	31.50	15.20	0.84	2.11	29.01	21.26

Table 2.12. Rheological Properties of Complex Suspension NKP Fertilizers Based on Ammophos, Ammonium Nitrate, and Potassium Chloride

$N:P_2O_5:K_2O$	NKS:Ammophos	T.к., o	Viscosity (Pz) at various temperatures (°C)	Density (g/cm³) at various temperatures (°C)	Flow Index (C)	pH
			10°C	20°C	30°C	40°C
1:1:1	4:1	17.40	17.40	16.45	13.90	10.34
2:1:1	4:1	17.18	17.18	16.22	13.34	10.10
3:1:1	4:1	17.06	17.06	16.09	13.51	10.00
1:1:1	2:1	18.12	18.12	17.06	14.81	11.04
2:1:1	2:1	18.01	18.01	17.01	15.37	11.03
3:1:1	2:1	17.87	17.87	16.86	15.22	10.88
1:1:1	4:3	20.59	20.59	19.28	15.94	11.61
2:1:1	4:3	20.35	20.35	18.96	16.67	14.09
3:1:1	4:3	20.16	20.16	18.74	16.79	14.52
1:1:1	1:1	21.49	21.49	20.04	18.42	16.48
2:1:1	1:1	21.37	21.37	19.86	18.90	17.61
3:1:1	1:1	21.20	21.20	19.78	18.86	17.59
1:2:1	—	25.41	25.41	24.07	23.21	21.96

Suspended complex NKP-fertilizers prepared from base slurries with NKS:Ammophos ratios of 4:3 and 1:1, depending on the nutrient composition ratio, mainly consist of:

- Calcium nitrate: 14.93–7.77% (4:3) and 11.57–5.92% (1:1),
- Magnesium nitrate: 0.83–0.43% (4:3) and 0.59–0.33% (1:1),
- Ammonium nitrate: 13.39–35.80% (4:3) and 15.90–37.42% (1:1),
- Ammophos: 21.36–11.13% (4:3) and 22.08–11.31% (1:1),
- Potassium chloride: 15.66–8.15% (4:3) and 16.19–8.28% (1:1), respectively.

The total nutrient content in these formulations ranges from 27.26–33.57% for the 4:3 ratio and 26.66–33.25% for the 1:1 ratio, indicating their suitability as high-content suspension fertilizers.

Based on the results of the technological experiments, a material balance was calculated for the production of 1 ton of suspended complex NPK-fertilizer with a nutrient ratio of $N:P_2O_5:K_2O$ = 1:1:1 in an integrated plant setup.

International Journal of Advanced Research in Science, Engineering and Technology

Vol. 12, Issue 8, August 2025

ISSN: 2350-0328

The final product composition is as follows:

- Total nitrogen: 8.89%
- in the form of calcium nitrate (20.75%),
- magnesium nitrate (1.17%),
- and ammonium nitrate (9.30%),
- Phosphorus (P_2O_5): 8.89%
- Potassium (K_2O): 8.89%

This balanced composition ensures high nutrient availability and confirms the feasibility of industrial-scale production of suspension NPK fertilizers with consistent physicochemical properties.

IV. CONCLUSION AND FUTURE WORK

This study successfully demonstrates the development and optimization of an innovative, suspension-based NPK fertilizer synthesis technology utilizing locally sourced raw materials, including calcium-containing sludge from industrial water treatment processes, feed-grade ammophos, potassium chloride, and ammonium nitrate. The core of the technological innovation lies in the partial decomposition of calcium sludge using nitric acid, which yields a calcium nitrate-rich solution that serves as the base for subsequent fertilizer formulation.

Through systematic experimentation, various $N:P_2O_5:K_2O$ nutrient ratios (1:1:1, 2:1:1, and 3:1:1) and base suspension compositions were evaluated, allowing precise control over the physicochemical and agronomic properties of the final fertilizer product. The research found that increasing the nitrogen content not only enhanced total nutrient availability but also significantly altered the nitrate-to-ammonium ratio, thereby influencing nutrient uptake dynamics and plant assimilation efficiency.

Furthermore, the salt composition data indicate that these suspension formulations can achieve total nutrient concentrations exceeding 30%, aligning with international standards for high-performance fertilizers. The flexibility in adjusting component ratios allows for the creation of crop- and soil-specific formulations, positioning this technology as a viable tool for precision agriculture.

In conclusion, the proposed synthesis route offers a sustainable, scalable, and economically viable solution for the production of complex NPK suspension fertilizers. It not only contributes to the efficient utilization of industrial by-products but also addresses the need for high-efficiency fertilizers tailored to local agronomic conditions. These results provide a solid foundation for future industrial implementation and further research into advanced formulation strategies for liquid and suspension-based fertilizers.

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