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Efficient purification of Moroccan wet-process phosphoric acid via urea phosphate intermediate: High impurity removal with simultaneous urea nitrate production

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ABSTRACT

Phosphoric acid (PA) is one of the most critical raw materials for global fertilizer production and chemical industries; however, untreated Moroccan phosphoric acid offers significant challenges due to impurities. This study presents a novel two–step purification method for untreated Moroccan PA by incorporating nitrogen fertilizers to produce a high-quality valorized product. The process involved the reaction between untreated Moroccan PA and urea to produce a crystalline urea phosphate (UP), followed by the treatment with concentrated nitric acid (NA) to generate urea nitrate (UN) and recover purified PA simultaneously. An extensive characterization analysis was performed using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), inductively coupled plasma optical emission spectroscopy (ICP-OES), and scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDX), validating substantial P_2O_5 recovery and reduction in metallic impurities. This purification methodology emphasizes an ecologically safe and sustainable process by minimizing waste and implementing safe production reagents. The analytical characterization demonstrated significant removal of contaminants and improved phosphoric acid quality as untreated PA (52.66 wt% P_2O_5) was refined to purified PA (49.80 wt% P_2O_5), highlighting the potential use of this purification method for industrial applications. ICP-OES analysis demonstrated high removal efficiencies for major metallic impurities - Al (99.05%), Fe (97.42%), and Mg (99.26%) – with a global P_2O_5 recovery yield of 71.40% for purified PA, and total product (PA + UN) recovery reach 77.28%. UN by-products can be also efficiently utilized in agricultural applications as a potential nitrogen fertilizer. Furthermore, this process efficiently operates at 50 °C and consumes only 320 MJ per one ton of thermal energy, indicating an 87% reduction in comparison to conventional methods. This dual valorization approach perfectly aligns with the principles of green chemistry, contributing to the circular economy and presenting an environmentally sustainable alternative to conventional purification methods while offering industrial scalability and economic validity.

Keywords: Moroccan phosphoric acid, urea phosphate, urea nitrate, purification, nitrogen fertilizers

NTRODUCTION

Morocco, being the global leader of phosphate rock (PR), retains around 70% of phosphate reserves and produces about 40 million tons of PR in 2022 (Habashi 1985; Kauwenbergh, Stewart, and Mikkelsen 2013; Mubarak 2011). The Moroccan phosphate industry serves as one of the main economic pillars, alongside the potential exportation of raw material as well as value-added products. The phosphoric acid or H_3PO_4 (PA) is mainly produced by the attack of sulfuric acid (H_2SO_4) on phosphate rock through wet process using dihydrate and hemihydrate-dehydrate methods, contributing to 27 to 29% of phosphorous pentoxide (P_2O_5), which is further concentrated by evaporation into merchant-grade acid of 52–54% of P_2O_5 P_2O_5 (Jančaitienė et al., 2023; Slack and Nason 1961). The wet process for the production of PA is done through following reaction;

$$Ca_5F(PO_4)_3 + 5H_2SO_4 + nH_2O \rightleftharpoons$$
$$\rightleftharpoons 5CaSO_4.xH_2O + 3H_3PO_4 + HF\uparrow$$

PA is one of the most essential raw materials for the production of phosphate fertilizers, detergents, and agricultural or industrial applications (Guelfi et al. 2022; Havelange et al. 2022; Leikam and Achorn 2005); however, the wet process PA (WPA) production often contains impurities that hinder its potential use in these applications (Kouzbour et al. 2019).

The contaminants in WPA depend on the chemical composition and physical properties of PR, the type of H_2SO_4, and the type of methods used for production (Hoffmann et al. 2016). The typical contaminants in WPA include mineral compounds of Al, Ca, Fe, Mg, Na, K, F, CL, SO_4, and heavy metals such as Cd, As, Zn, Hg, Pb, Cr, Ni, silica, U, organic substances, carbon, and lanthanide (Hoffmann et al. 2016; Kic et al., 2015; Kouzbour et al. 2019). Due to an increase in the global population and corresponding food production, there is a rising demand for PA production for their further utilization in the synthesis of nitrogen fertilizers and agricultural applications. This makes it necessary for the phosphate industries to efficiently invent novel purification strategies for the production of highly pure and refined PA, ensuring industrial or agricultural sustainability.

Although several conventional purification methods have been employed for decades, these methods exhibit several limitations challenging ecological sustainability and economic feasibility for industrial applications. Solvent extraction, precipitation, ion-exchange, and crystallization methodologies are some of the conventional purification strategies that have been employed for the removal of potential impurities and WPA refining. Solvent extraction requires large volumes of organic solvents, such as tributyl phosphate, resulting in hazardous waste streams generation and the need for extensive solvent recovery systems (Awwad et al., 2013; Hoffmann et al. 2016; Magda et al. 2010). Water-immiscible solvents cause a major energy-consuming distillation to recover solvent and fail to eliminate carbon impurities from WPA, encountering major concerns like phase separation and specific pretreatment of WPA to remove organic contaminants (Awwad et al., 2013; A. Cherrat et al., 2020; Daifullah et al., 2004; Lung et al., 2005; Mersmann 2001). Precipitation utilizes reagents like sodium sulfide or lime and produces substantial solid waste that requires efficient disposal systems (Abdel-Ghafar et al. 2019; Kouzbour et al. 2019).

Meanwhile, ion exchange and crystallization processes are energy-intensive and require high operation costs. The ion exchange purification method is credited with selectivity, but resins bound with these methods become fouled rapidly with organic matter, resulting in frequent regeneration cycles and increased operation costs (Hérès et al. 2018; Ilczyszyn et al., 1992; Kim et al. 2016). Crystallization would probably work on an industrial scale but often requires many stages and usually generates quite dilute products that require further concentration (Anastas and Warner 2000; A. Cherrat et al. 2025; Mullin 2001). These conventional purification technologies present multiple processing stages, high operational costs, significant energy consumption, and the generation of secondary pollutants. This makes these conventional approaches highly inappropriate and incompatible with modern sustainability imperatives and the development of the circular economy.

Nitrogen fertilizers can also be utilized for WPA purification that may offer an ecological and economically rational approach for an

effective synthesis of purified PA and high-quality by-products (Walling and Vaneeckhaute 2022). Nitrogen fertilizers (such as urea, ammonia, and nitric acid) are cheap, commonly available, and have already been used for several agricultural applications, which makes them a potential raw material for an ecological purification method (Babcock-Jackson et al. 2023; Walling and Vaneeckhaute 2022). The implementation of nitrogen fertilizers for PA purification can significantly produce useful nitrogen-containing by-products, which allow a dual valorization strategy, where both the purified acid and the nitrogen compounds are of productive use. This method turns what would normally be waste streams into marketable products, reflecting the concept of the circular economy and possibly low purification costs. The selective reactivity of nitrogen compounds with PA and the differential solubility of the end products are the major reasons to implement this scientific approach. Urea reacts with PA to form urea phosphate, a low-solubility crystalline compound that precipitates from the solution and separates from the dissolved metallic impurities. The nitric acid treatment then decomposes the urea phosphate to recycle PA and transform urea into a urea nitrate, a nitrogen fertilizer with slow-release properties and good agricultural characteristics.

Therefore, the scientific rationale of this study is to develop a two-step nitrogen-based purification strategy with dual valorization for untreated Moroccan PA. While the scientific objectives of this research include: (1) optimized synthesis of urea phosphate from untreated Moroccan PA as the primary purification step; (2) adapted and refine the nitric acid attack methodology to efficiently recover purified PA from urea phosphate; (3) perform analytical characterization of purified PA to analyze the P_2O_5 recovery rates and efficiency of impurity removal; (4) evaluate the urea nitrate (by-product) for its potential as a nitrogen fertilizer; and (5) comprehend the ecological impact, sustainability metrics, and industrial scalability of the proposed purification method. This research not only based on rigorous experimental investigation and analytical characterization but will also aim to provide a scientifically sound, environmentally friendly, and economically feasible alternative to conventional PA purification methods.

METHODS

Raw materials and analytical techniques

The experimental investigation for this research employed a comprehensive suite of advanced analytical instruments to ensure rigorous characterization of raw material, intermediates, and final products throughout the purification process. The sample of natural phosphate, designated as "Natural Phosphate 05-2924" dated February 2017, was sourced from the International Soil Analytics Exchange Program (05-2924-bipea.org/fr/essais-interlaboratoires-sols). This natural phosphate sample underwent comprehensive inter-laboratory characterization analysis to ensure consistency, composition, and traceability of the material, as shown in Table 1. The inter-laboratory characterization analysis revealed the percentage elemental composition of phosphate, containing 29.39% P_2O_5 and 48.93% CaO as the major constituents. The sample of natural phosphate was then used to produce untreated Moroccan phosphoric acid through wet attack of 98% sulfuric acid.

X-ray diffraction (XRD) analysis was performed using a BRUKER D8 Eco diffractometer equipped with Cu Kα radiation operating to provide diffraction patterns on XRD spectra. X-ray fluorescence (XRF) spectroscopy was conducted on an ARL PERFORM'X sequential wavelength dispersive XRF spectrometer, where the samples were prepared as pressed pellets and quantitative analysis was performed using fundamental parameters with certified reference materials for calibration. Inductively coupled optical emission spectroscopy (ICP-OES) was used for the quantitative determination of elemental composition in the sample, using a Thermo iCAP PRO XP DUO instrument and a Sartorius analytical balance (ISO-standard: 22036). Samples were diluted with ultrapure water and then acidified with nitric acid following multi-element calibration standards, depending on the range of concentration. Fourier-transform infrared (FTIR) spectroscopy was efficiently carried out using a Thermo Nicolet iS10 spectrometer equipped with an attenuated total reflectance (ATR) accessory containing a diamond crystal. IR spectra were acquired within the wavelength range from 400 to 4000 cm^{-1}. Scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDX) was employed to determine

Table 1. Chemical composition of natural phosphate sample analyzed through inter-laboratory tests

Parameter	$\%P_2O_5$	$\%K_2O$	%Na	%CaO	%MgO	$\%SO_3$	$\%CO_3$	$\%Cl^-$	$\%F^-$	$\%H_2O$
Results	29.39	0.14	1.0576	48.93	1.28	2.95	17.44	0,03	3.8	0.751
Parameter	Fe (mg/kg)	Al (mg/kg)	Cd (mg/kg)	Cr (mg/kg)	Co (mg/kg)	Cu (mg/kg)	Hg (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Zn (mg/kg)
Results	2776	2377	14.65	170.11	0.73	10.34	0.017	12.79	4.98	131.82

the morphology and elemental composition of the materials by using a MEB SH-5500P field emission scanning electron microscope. For this, samples were mounted on aluminum stubs with carbon adhesive tape and sputter–coated with a thin gold layer to ensure electrical conductivity. Moreover, several other laboratory instruments include standard glassware (volumetric flasks, pipettes, beakers, or filtration apparatus), calibrated pH meters, or laboratory furnaces.

P_2O_5 content was determined using both colorimetric and gravimetric methods. The P_2O_5 colorimetry method was performed using a SHIMADZU UV-1900 spectrophotometer equipped with a Sartorius analytical balance (Mill: Retsch ZM 200), following NF ISO 6598 standard procedures. The gravimetric method involved precipitation of phosphorus as ammonium phosphomolybdate, followed by precipitation and weighing. The P_2O_5 gravimetry was carried out using a drying oven of Memmert and an analytical balance of Sartorius (Mill: Retsch ZM 200). For density measurement, a DMA 35 digital densimeter equipped with a thermometer was utilized following ISO 3675 standards. The total nitrogen and ammonical nitrogen content were determined using a VELP UDK 149 distillation unit equipped with a Sartorius analytical balance by the Kjeldahl method, following ISO 5314 and NF U42-151 standards. Meanwhile, the ROTOFIX 32A centrifuge equipped with a Sartorius analytical balance, a Memmert drying oven, and a STURA-RT mechanical stirrer was employed to determine ureic nitrogen content under ISO 8603 standards. All of these chemical analyses to determine the content of P_2O_5, nitrogen, heavy metals, and trace elements were strictly conducted according to ISO, ASTM, and AFNOR standards.

Moroccan PA production

To maintain global food security, world is currently facing an increasing demand of fertilizers in agriculture as plants acquire nitrogen, phosphorous, and potassium for continuous growth and development. The most commonly used nitrogen and phosphate fertilizers are typically produce from PA, underscoring the strategic importance of efficient PA production through wet attack of H_2SO_4. In Morocco, the production of H_3PO_4 is predominantly done through the wet process, specifically dihydrate process. The process starts with the ore of phosphorus, which has already undergone beneficiation and is now ground to the right particle size (usually 90% passing 200 mesh) in order to increase the reactive surface area. This ground PR is reacted in large agitated reactors at controlled temperature conditions (70–80 °C) with concentrated sulfuric acid (93–98% H_2SO_4). The basic chemical reaction for the wet process PA production is given as follows:

$$Ca_5F(PO_4)_3 + 5H_2SO_4 + nH_2O \rightleftharpoons$$
$$\rightleftharpoons 5CaSO_4.xH_2O + 3H_3PO_4 + HF\uparrow$$

The chemical reaction generates phosphoric acid, while calcium sulfate dihydrate or gypsum ($CaSO_4.2H_2O$) is the major solid by-product (A. Cherrat et al. 2026). This process is accompanied by a large release of heat, and therefore, proper handling of the heat is very important for controlling the process and the quality of the crystals. The reaction mixture is treated in a certain way that encourages the formation of very pure and crystalline gypsum, which in turn makes the separation of the solid from the liquid easier (Ayoub Cherrat et al. 2026). After the reaction is completed, the slurry goes to the filtration units, where gypsum is removed from the PA solution either using vacuum filters or filter presses. The PA that has been filtered usually has a P_2O_5 content of 28-32% and is commonly processed to 54% P_2O_5 by vacuum evaporation in order to cut down the costs of transport and to increase the stability of storage.

The untreated Moroccan PA extracted through the wet process exhibits complicated composition due to the presence of several impurities, reflecting a dynamic relation between the mineralogy

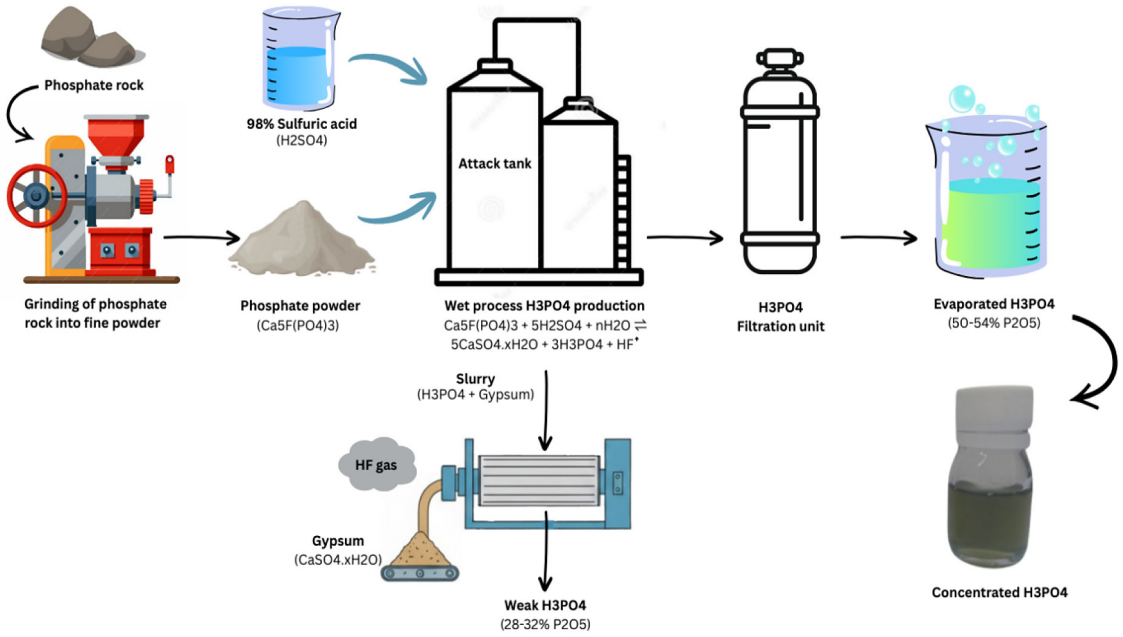

Figure 1. Schematic representation of wet process phosphoric acid production

of the original PR and the chemistry of the extraction process. The usual composition of untreated Moroccan PA contains P_2O_5, ranging from 28–32% for the unconcentrated acid and 52–54% for the concentrated grades. Nonetheless, the acid contains high impurity levels coupled with a lot of contaminants, contributing to significant quality reduction. The most troublesome contaminants are the metallic impurities, with iron usually being present at a level of 1.5–3.0% as Fe_2O_3, aluminum at 0.8–2.0% as Al_2O_3, and magnesium at 0.3–1.5% as MgO among the impurities. These metals come from clay minerals and the phosphate ore gangue particles that are being partially dissolved during the attack of sulfuric acid. Heavy metals are another major problem, with concentration levels of cadmium between 10–50 mg/kg, lead 5–30 mg/kg, chromium 50–200 mg/kg, and arsenic 5–40 mg/kg, depending on the particular ore deposits and processing conditions. Besides these, other elements that may be present to some extent include vanadium, uranium, fluorine, and rare earth elements. Untreated Moroccan PA also contains organic matter, which is the result of the sedimentary origin of the phosphate deposits, suspended solids that consist of unreacted phosphate particles, fine gypsum crystals, and dissolved sulfates resulting from the reaction process.

The impurity load in untreated Moroccan PA gives rise to several problems concerning both environmental and utilization compliance, significantly limiting the use to non-food-grade applications due to the presence of heavy metals. Metal impurities lead to various complications in the downstream process, such as fouling and interference with catalytic reactions in industrial applications, due to their high solubility. The environmental impact of such impurities is persistent throughout the product life, from production waste streams to agricultural runoff, i.e., the part where fertilizers are applied. International regulations and quality standards for PA are getting more and more rigorous, especially in terms of heavy metal content. These challenges reinforce the need to develop environmentally friendly PA purification techniques that are capable of replacing conventional solvent extraction methods to produce high-quality and refined PA meeting both regulatory and environmental standards.

Synthesis of urea phosphate

The synthesis of urea phosphate is considered an important purification stage, which is based on the strong attraction between urea and PA to create a stable crystalline compound that is easily separable from the metallic impurities (Figure 2). The chemical reaction between urea and untreated Moroccan PA under optimal conditions is given as follows:

$$H_3PO_4\,(l) + CO(NH_2)_2\,(s) + \text{Heat} \rightarrow$$
$$\rightarrow CO(NH_2)_2.H_3PO_4(s) + \text{Residual liquor}$$

This reaction is highly exothermic and can be done very easily at room temperature or with slightly higher temperatures. The formation of the urea phosphate complex takes place by hydrogen bonding between carbonyl oxygen/amino groups of urea and the acidic protons/oxygen atoms of PA to develop a well-defined crystalline structure. The compound has a very low solubility in acidic aqueous media compared to most metallic phosphates and sulfates, which allows for selective precipitation while the impurities remain in the mother liquor.

The urea phosphate production reaction was explained in our previous research (A. Cherrat et al. 2020; 2025). The synthesis reaction was carried out by the chemical reaction between 46% urea and untreated Moroccan PA under optimal conditions at a temperature of 50 °C for 90 minutes and a 1:1 stochiometric concentration. Due to the slow reactivity of urea during this exothermic reaction, it takes time for urea to completely dissolve, and after the reaction, the urea phosphate precipitation was done at room temperature, followed by the extraction of urea crystals in dry form. The purified urea crystals were then filtered through a Buchner. The whole process of urea phosphate synthesis was optimized to determine ideal parameters and reaction conditions by utilizing the design of experiments (DOE on Software Nemrodw).

Purified PA and urea nitrate synthesis

The second purification step was established with the help of techniques adapted from the McCullough research (US 3967948) and mainly focused on the nitric acid attack methodology to disintegrate urea phosphate and purify untreated PA in Morocco. The patent involved a detailed account of a series of magnificent processes for purified PA and nitrogen fertilizers production; however, our work particularly modified nitric acid treatment to make it applicable for untreated Moroccan PA purification based on green chemistry. The nitric acid attack of urea phosphate involves the following reaction:

$$CO(NH_2)_2 \cdot H_3PO_4 + \\ + HNO_3 \rightarrow H_3PO_4 + CO(NH_2)_2 \cdot HNO_3$$

The urea phosphate complex obtained in the previous section is decomposed to release purified PA, and urea is turned into urea nitrate (a nitrogen

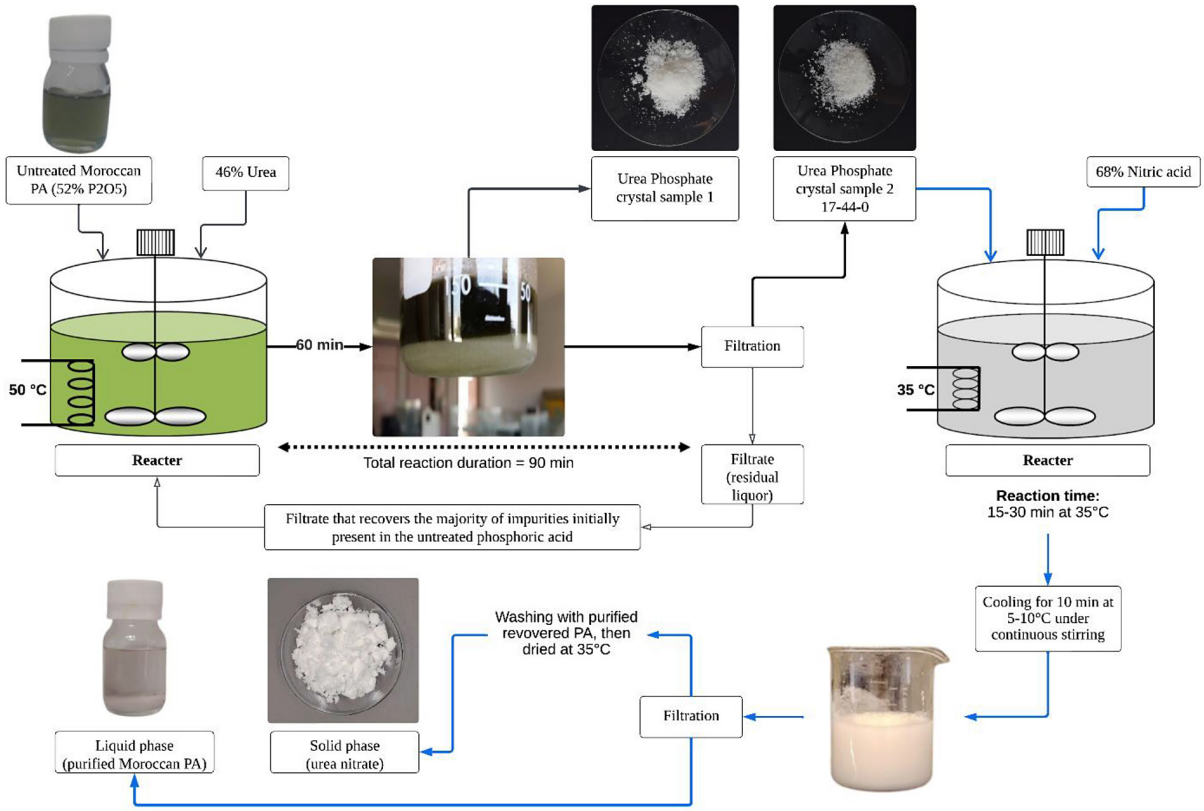

Figure 2. Schematic representation of process flow adapted for the purification of PA

fertilizer). The reaction generates a little heat but is very effective when the conditions are properly monitored. Both products, such as regenerated PA and urea nitrate, are of great importance with exceptional commercial value, which provide strong evidence contributing to the recycling of waste into valorized products and supporting the principles of the circular economy.

The nitric acid treatment was done following the optimized protocol to get an effective PA purification, as shown in Figure 2. The urea phosphate was dried and put in a glass reaction vessel that had mechanical stirring, a thermometer, and a reflux condenser. The urea phosphate was treated in the glass vessel while concentrated nitric acid (68% HNO_3) was added slowly and stirred continuously at room temperature. A molar ratio of nitric acid to urea phosphate (1:1 or 1.05:1) was set with a slight excess to make sure that the reaction is complete. The reaction mixture was stirred nonstop at room temperature for half an hour to an hour, and urea phosphate dissolved on reaction with nitric acid. The temperature was kept at 35 °C for 15 minutes by controlling the addition rate and using external cooling when necessary to prevent the decomposition of urea nitrate. After the completion of the reaction, the liquid was treated to isolate the two main products.

The separation strategy relied on the different solubility and thermal stabilities of PA and urea nitrate. The crystallization of the urea nitrate started as the solution was concentrated and kept at a lower temperature, because of its lower solubility compared to phosphoric acid. The mixture was gradually cooled down to a temperature range of 5–10 °C in order to facilitate the urea nitrate crystallization to the extent that PA remained in the solution. The urea nitrate crystals were isolated by using either vacuum filtration or centrifugation, then the urea nitrate was washed with small amounts of cold water in order to remove any attached phosphoric acid, and finally, the urea nitrate was dried at 35 °C. The liquid portion, which contained purified PA, was gathered and further evaporated under vacuum to get the desired P_2O_5 concentration (normally 52–54%).

To enhance the purification efficiency, several parameters were modified such as; (1) nitric acid concentration and addition rate for reaction temperature control and urea nitrate decomposition prevention; (2) reaction time and mixing to be sure of uncomplete decomposition of urea phosphate; (3) concentration and cooling methods to product separation maximize and energy consumption minimize; and (4) washing operations to product purity high and water usage low. The process incorporates some of green chemistry principles: atom economy is very high because both main products are not only utilized but also rather than discarded; energy consumption is all the time less with the use of ambient or low temperatures; hazardous solvents are not used at any stage and generation of waste is very low, as the only significant by-products are the impurities removed in the first step. The use of nitrogen fertilizer reagents (urea and nitric acid) that generate a nitrogen fertilizer product (urea nitrate) creates a coherent nitrogen cycle that supports agricultural sustainability.

RESULTS

Purified PA characterization

The purified PA obtained through two step purification steps as explained in the methods section was then subjected to comprehensive physiochemical characterization to assess the effectiveness of impurity removal and evaluate product quality. The characterization analysis for purified PA includes the determination of P_2O_5, density, solid content, mass balances, % recovery yield of P_2O_5, heavy metals, and trace elements.

Determination of P_2O_5 content

Input analytical data, such as mass fractions and trace elements, were used to quantify (i) P_2O_5 recovery yields across the purification process and (ii) removal efficiencies of impurities, comparing untreated Moroccan PA and purified PA. The two-step purification process resulted in the recovery of significant amounts of P_2O_5, where the untreated Moroccan PA included 52.66 wt% of P_2O_5, corresponding to a weight of 0.5266 for the raw PA. The purified PA contains 49.80 wt% P_2O_5, with a weight of approximately 0.4980 for PA after the purification process (Table 2). The normalization factor based on P_2O_5 was given as: $f = w(P_2O_5, raw) / w(P_2O_5, pur)$, and is equal to 1.057430. The small decline in P_2O_5 concentration in the finalized product reflects the dilution effect of nitric acid treatment, and the selective removal of impurities occurred during the different stages of purification. The first purification

step, which involved the synthesis or recovery of urea phosphate, yielded a P_2O_5 recovery of 84%. While the second step, wherein urea phosphate was treated with 68% HNO_3, led to an overall product recovery of 92%, out of which 85% was attributed to purified PA and 7% to urea nitrate formation. The global P_2O_5 recovery yields, which were calculated in accordance with the initial untreated PA, were determined as follows: purified PA yielded 71.40% ($\eta_1 \times \eta_2$, PA = 0.84 × 0.85), whereas total products, including urea nitrate, reached 77.28% ($\eta_1 \times \eta_2$, prod = 0.84 × 0.92). These findings demonstrated that approximately 5.88% of the initial P_2O_5 was transformed into urea nitrate, thus proving the effective dual valorization of the raw material. Therefore, the recovery yield of 71.40% for purified PA shows a favorable ratio of impurity removal efficiency to phosphorus retention, which is crucial for implementation on an industrial scale.

Evaluation of density and solid content

The purified PA was efficiently evaluated, which shows a tapped density of 1.581 g/cm³ at 20 °C, while the density of the untreated PA was 1.642 g/cm³ (Table 2). This decline in density can be linked to the removal of metallic salts and the reduction in P_2O_5 concentration that took place during the purification process. The solid content tests showed 0.50% for total nitrogen (Namm) and 0.80% for total nitrogen (Ntotal) in purified PA, which means that there were very few traces of urea or urea derivatives left over from the purification agents. The percentages of nitrogen claim that the UN was effectively separated from the acid during crystallization and filtration.

Mass balance and % recovery yield of P_2O_5

The reaction yield was evaluated based on the mass balance of P_2O_5 between the reactants and recovered products, along with their respective P_2O_5 mass concentrations, where the reaction involved UP and concentrated AN as reactants for formation of PA and UN as products (Table 3). The mass of P_2O_5 was associated with each stream was calculated to provides additional evidence of the efficiency of the process. On the reactant side, the process involves 71.5 g of AN with 0% P_2O_5 and 63.5936 g of UP with 44.2 wt.% P_2O_5. The total mass of P_2O_5 introduced into the reaction originates exclusively from UP and is calculated as follows:

$$m(P_2O_5, \text{reactants}) = \\ = 63.5936 \text{ x } 44.2 / 100 = 28.1084 \text{ g}$$

After the completion of the reaction, two recovered products containing PA and UN were obtained. PA contains a recovered mass of 48.1393 g with 49.8 wt.% P_2O_5 and mass P_2O_5 of 23.9734 g, while UN contains recovered mass of 41.8 g with 5.1 wt.% P_2O_5, corresponding to mass P_2O_5 of 2.1318 g. The initial P_2O_5 mass represented by m_0 = 26.1088 kg (which was equivalent to 28.1088 kg of UP), the products purified PA had 23.9734 kg of P_2O_5 mass and UN contained 2.1318 kg of P_2O_5. The total mass of P_2O_5 recovered in the products is therefore:

$$m(P_2O_5, \text{products}) = \\ = 23.9734 + 2.1318 = 26.1052 \text{ g}$$

The P_2O_5 recovery yield of the reaction is defined as the ratio between mass P_2O_5 of recovered products and mass P_2O_5 in the initial reactants was calculated as:

$$\text{Recovery Yield (\%)} = (m(P_2O_5, \text{products}) / \\ / m(P_2O_5, \text{reactants})) \times 100$$

$$\text{Recovery Yield (\%)} = \\ = (26.1052 / 28.1084) \times 100 = 92.87\%$$

The total P_2O_5 mass recovered of 26.105 kg was very close to the theoretical input, resulting in a mass recovery ratio (products/reactants) of 92.87%, thereby affirming the accuracy of experimental measurements and the sturdiness of purification methodologies. These results demonstrate a high P_2O_5 recovery efficiency, confirming the effectiveness of the nitric acid attack of urea phosphate. The efficiency of purification process can also be observed through comparison between untreated Moroccan PA and purified PA, as shown in Figure 3. Meanwhile, the remaining loss estimated at about 7% is mainly attributed to process –related factors such as material retention on reactor walls, adhesion to the vessel, filtration surfaces, and minor mechanical losses either during handling, transferring, and product recovery related steps.

Heavy metal and trace element analysis

ICP-OES analysis was implemented to evaluate the removal of metallic impurities in all the elements, where the purification process indicated removal efficiency of over 95% for most of the major contaminants, as shown in Table 4. AS the

P_2O_5 content differs between the two acids, impurity concentrations (mg/kg) were normalized to a P_2O_5 basis and for each element i:

$$E_i(\%) = [1 - (C_i,pur / C_i,raw) \times f] \times 100$$

where: C_i,raw and C_i,pur are the measured concentrations (mg/kg) in raw and purified acids, and $f = w(P_2O_5,raw)/w(P_2O_5,pur)$.

Negative E_i values indicate an apparent increase (enrichment) in the purified acid and may reflect analytical variability, contributions from reagents, or low-level quantification limits.

The aluminum (Al) concentration plunged from 2,011 mg/kg to 18.00 mg/kg in the purified PA, with 99.05% removal efficiency. Iron (Fe), the major impurity in wet process PA, was decreased from 3,357 mg/kg to 81.89 mg/kg, which means 97.42% was removed. Magnesium (Mg) was 9,558 mg/kg in first place and was lowered to 67.00 mg/kg with an extraordinary 99.26% removal efficiency. Heavy metals that are of great concern to the environment and are often the subject of regulations showed an excellent removal rate by the purification process. The amount of cadmium (Cd) in the treated sample went from 9.97 mg/kg to 0.04 mg/kg (E_i(%) = 99.58%), chromium (Cr) from 266 mg/kg to 1.08 mg/kg (E_i(%) = 99.57%), and nickel (Ni) from 62.78 mg/kg to 1.43 mg/kg (E_i(%) = 97.59%). The arsenic (As) level was lowered from 2.381 mg/kg to 0.550 mg/kg, which corresponds to an impurity removal efficiency of 75.57%. Other elements, such as cobalt (E_i(%) = 92.51%), copper (E_i(%) = 84.32%), and vanadium (E_i(%) = 99.85%), also showed high purification efficiencies.

The removal of alkaline and alkaline earth metals has been done quite effectively, for calcium (Ca), the E_i of 77.95%, potassium (K) of 98.94%, and sodium (Na) of 77.31%. Silicon (Si), which usually causes problems in downstream applications, was reduced from 1,028 mg/kg to 1.13 mg/kg with 99.88% removal efficiency. The concentration of sulfur (S), which is mainly due to the presence of sulfate ions from the wet process, was reduced from 5,643 mg/kg to 130.80 mg/kg, a 97.55% removal rate.

Most of the trace elements showed efficient removal in the final purified PA (typically >95%), which has been synthesized through a purification process. Antimony (Sb) showed 100% removal efficiency, molybdenum (Mo) of 99.44%, vanadium (V) of 99.85%, and boron (B) of 90.14%, resulting in excellent results for purification. Interestingly, three elements (Be, Pb, or Se) showed negative removal efficiencies. This may be due to numerous reasons, such as analytical variability at

Table 2. Comparison of analytical calculations between untreated Moroccan PA and purified PA obtained after the purification process

Parameters		Untreated PA 54%	Purified PA
		wt%	wt%
P_2O_5	%	52.66	49.80
Al	mg/kg	2011	18.00
As	mg/kg	2.381	0.55
B	mg/kg	223	20.90
Ba	mg/kg	11.32	3.46
Be	mg/kg	0.025	0.03
Ca	mg/kg	4288	964.0
Cd	mg/kg	9.97	0.04
Co	mg/kg	1.13	0.08
Cr	mg/kg	266	1.08
Cu	mg/kg	20.71	3.07
Fe	mg/kg	3357	61.69
K	mg/kg	747	7.46
Li	mg/kg	26.98	4.45
Mg	mg/kg	9588	67.00
Mn	mg/kg	30.91	2.98
Mo	mg/kg	15.24	0.09
Na	mg/kg	742	159.2
Ni	mg/kg	62.78	1.43
Pb	mg/kg	1.841	1.66
S	mg/kg	8643	130.6
Sb	mg/kg	4.237	0.00
Se	mg/kg	0.095	0.74
Si	mg/kg	1028	1.13
Sn	mg/kg	0.555	0.04
Sr	mg/kg	13.65	4.27
Te_2	mg/kg	0.092	0.07
Ti	mg/kg	64.38	2.25
Tl	mg/kg	0.999	0.07
V	mg/kg	230	0.33
Zn	mg/kg	472	10.95
Density	*	1.642	1581
	Tapped	*	*
	Bulk	*	*
N_{amm}	%	*	0.50
N_{total}	%	*	0.80

Note: PA – phosphoric acid, Wt – weight, N_{amm} – total ammonium nitrogen, N_{total} – total nitrogen concentration.

Table 3. Mass balance and P_2O_5 recovery yield calculated during the reaction

Parameters	Reactants		Recovered products	
	AN	UP	PA	UN
Mass	71.5	63.59	48.13	41.8
$\%P_2O_5$	0	44.2	49.8	5.1
Mass P_2O_5	0	28.10	23.97	2.131
Total mass P_2O_5	28.108		26.105	

Note: AN – nitric acid, UP – urea phosphate, PA – phosphoric acid, UN – urea nitrate, P_2O_5 – phosphorous pentoxide.

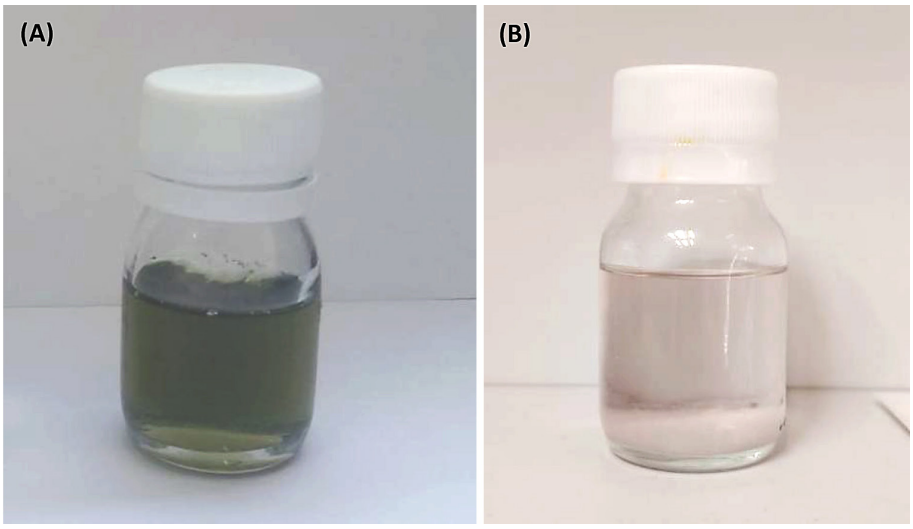

Figure 3. Comparison of visual representation between (A) untreated Moroccan PA and (B) purified PA obtained through purification process between urea phosphate and nitric acid, indicating significant refining of PA with clear color and impurity removal

very low concentrations near the limits of detection of the instruments, possible trace contamination within the NA reagent, concentration changes during the evaporation, or simply that these elements behaved differently during the urea phosphate precipitation. Pb (E_i(%) = -26.89%), Be (E_i(%) = -7.98%), and Se (E_i(%) = -721.09%) are still at such low concentrations that they do not negatively affect the overall quality of the purified acid for most industrial uses.

The normalization of impurity concentrations to a P_2O_5 basis (using the factor f = 1.057430) guarantees that accurate comparisons can be made between the untreated and the purified acids even though they exhibit different P_2O_5 contents. This normalization also takes into account the dilution effect of the purification process and thus gives us a measure of impurity removal rather than mere concentration changes. In summary, the global recovery of P_2O_5 in the purified PA was 71.4% and in the total products (purified PA and urea nitrate) was 77.28%, indicating efficient removal of impurities following purification process.

Urea nitrate characterization

X-ray diffraction (XRD) analysis

X-Ray diffraction (XRD) analysis of urea nitrate synthesized was found to be consistent with standard pattern of urea nitrate explained in the bibliography under the JCPDS number 00-006-0332, as explained in Figure 4. The reaction conditions for the XRD analysis of the urea nitrate include 35 °C for 15 minutes. Therefore, the XRD pattern confirms that the synthesized urea nitrate under optimized reaction conditions is pure as the standard $CO(NH_2)_2.HNO_3$ (JCPDS number: 00-006-0332). The consistent results with reference urea nitrate completely indicate the high purity of the valorized product with empirical formula of $CH_5N_3O_4$ and chemical formula of $CH_4N_2O.HNO_3$, validating synthesis of refined urea nitrate.

Table 4. Percentage efficiency (E_i) of impurity removal for all the elements was calculated

Element	Unit	C_raw	C_pur	C_pur/C_raw	(C_pur/C_raw)×f	E_i (%)
Al	mg/kg	2.011	18.00	0.008	0.009	99.05
As	mg/kg	2.381	0.550	0.230	0.244	75.57
B	mg/kg	223.0	20.80	0.093	0.098	90.14
Ba	mg/kg	11.32	3.480	0.307	0.325	67.49
Ca	mg/kg	4 288	894.0	0.208	0.220	77.95
Cd	mg/kg	9.970	0.040	0.004	0.004	99.58
Co	mg/kg	1.130	0.080	0.070	0.074	92.51
Cr	mg/kg	266.0	1.080	0.004	0.004	99.57
Cu	mg/kg	20.71	3.070	0.148	0.156	84.32
Fe	mg/kg	3.357	81.89	0.024	0.025	97.42
K	mg/kg	747.0	7.480	0.010	0.010	98.94
Li	mg/kg	26.98	4.450	0.164	0.174	82.56
Mg	mg/kg	9.558	67.00	0.007	0.007	99.26
Mn	mg/kg	36.91	2.880	0.078	0.082	91.75
Mo	mg/kg	15.24	0.080	0.005	0.005	99.44
Na	mg/kg	742.0	159.2	0.214	0.226	77.31
Ni	mg/kg	62.78	1.430	0.022	0.024	97.59
S	mg/kg	5 643	130.8	0.023	0.024	97.55
Sb	mg/kg	4.237	0	0.000	0.000	100.0
Si	mg/kg	1 028	1.130	0.001	0.001	99.88
Sn	mg/kg	0.555	0.040	0.072	0.076	92.38
Sr	mg/kg	13.65	4.270	0.312	0.330	66.92
Te	mg/kg	0.092	0.070	0.756	0.800	19.98
Ti	mg/kg	64.38	2.260	0.035	0.037	96.29
Tl	mg/kg	0.998	0.070	0.070	0.074	92.60
V	mg/kg	230.0	0.330	0.001	0.001	99.85
Zn	mg/kg	472.0	10.85	0.022	0.024	97.57

Note: C_i,raw and C_i,pur are the measured concentrations (mg/kg) in raw and purified acids, and f = w(P_2O_5,raw)/w(P_2O_5,pur).

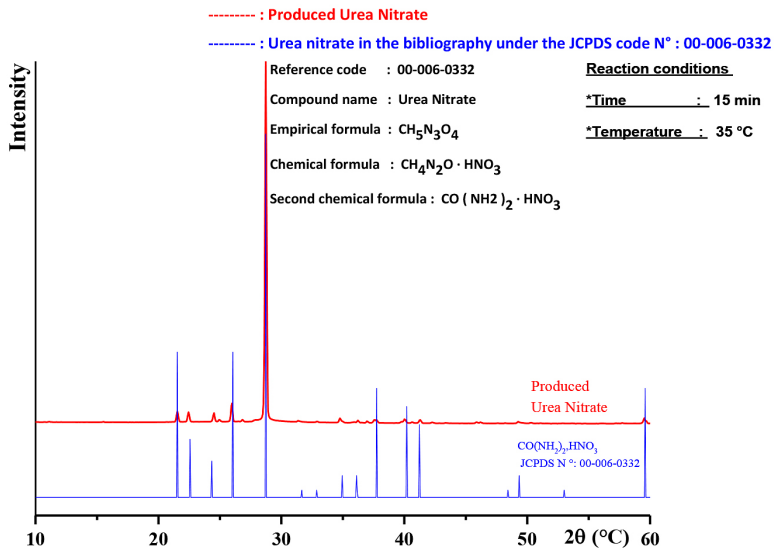

Figure 4. XRD analysis of the synthesized urea nitrate sample observed under optimized reaction conditions of temperature 35 °C for 15 minutes, confirming the purity of the synthesized urea nitrate

Inductively coupled plasma-optical emission spectroscopy (ICP-OES) analysis

The elemental characterization of UN was subjected to ICP-OES analysis, which not only confirmed the purity of the synthesized nitrogen fertilizer but also revealed extremely low levels of metallic impurities (Table 5). The wt% concentration of P_2O_5 in UN was 5.1%, which corresponds to the theoretical mass of P_2O_5 recovered in this product stream (2.1318 kg from the mass balance calculations). The major metallic impurities were detected at minor levels for Al (47.00 mg/kg), Ca (11.10 mg/kg), Fe (235.20 mg/kg), Mg (0.56 mg/kg), and Na (608.20 mg/kg). Meanwhile, the environmental hazards, heavy metals, were confirmed at very low levels, including Cd (0.04 mg/kg), Cr (0.16 mg/kg), Cu (1.93 mg/kg), Ni (0.37 mg/kg), and Pb (0.64 mg/kg). The impurity/P_2O_5 ratios for UN showed a range from 0.0001 to 0.0113, which means less than 1.13% of the P_2O_5 content is represented by the most concentrated impurity. UN was calculated to have a P_2O_5 recovery of 8.0 wt%, which shows that urea nitrate is mainly a nitrogen fertilizer product rather than being a source of phosphorus. The very low levels of metallic impurities was observed in the UN which are indicative of the preferential retention of these contaminants in the mother liquor during the crystallization of UP, followed by their removal in the washing steps. For the UN product, nitrogen content was determined as 0.93 wt% for ammonical nitrogen and 36.09 wt% for total nitrogen concentration. The nitrogen content in the UN product is very close to the theoretical nitrogen content of pure urea nitrate (33.6% N) and the small excess is probably due to absorbed nitric acid on the crystal surfaces or residual ammonium salts.

Infrared spectrometry

Infrared spectroscopy (IR) analysis for synthesized urea nitrate was performed through a Thermo Nicolet iS10 spectrometer that works along with the ATR technique. The frequencies of the vibrational bands of the functional groups found in the synthesized urea nitrate are presented in Table 6. The synthesized product was observed through FTIR spectra, which validates that characteristic bands of the functional groups in the product correspond to urea nitrate (Figure 5). Therefore, each of the absorbance peaks seen in the IR spectra represents specific bond vibrations, which is further attributed to the indication of the compound present in higher quantities found in the crystal samples. While interpreting FT-IR spectra, the peak observed at a wave number of 1048 cm^{-1} indicates the PO_4^{3-} group, PO at 1295 cm^{-1}, NO_3^- at 1321 cm^{-1}, CN at 1430 cm^{-1}, and NH at 1565 cm^{-1}. Moreover, the bond vibrations at a wave number of 1701 cm^{-1} correspond to C=O, and 2400 cm^{-1} indicate the C=O···$HONO_2$ group. The peak of the NH_2 or NH group was seen at a wave number range of 3187 to 3400 cm^{-1}. Therefore, the results of FT-IR spectra confirm the synthesis of urea nitrate, which is highly pure.

Scanning electron microscopy

Scanning electron microscopy (SEM) analysis was performed to determine the crystalline structure and particles found in the synthesized urea nitrate, as shown in Figure 6. The SEM microscopic images depict the existence of crystal structures that are well-defined, counterproductive to the design and shape of the quasicrystals. Technically, the images of the source materials being visualized with the lower magnifications are the same highly-appealing granules of the materials with compact and very dense surfaces, and the same could be suggestive of the fact that the crystal clusters were being made of more cohesively-formed crystallites during the valorization of the material. Conversely, the brighter, higher quality images that are of higher magnifications are the ones that show again the same crystal faces previously mentioned, but with such high reflective qualities, and also on top of that, very regular and uniform distribution and size of the formed particles. The high-resolution images are the ones that show the presence of crystal growing in layers and also the structures being edged very sharply over the whole length of the crystal, which are two factors that are indicative of the impurities being largely removed during the purification process and the crystal growth being under control. As the solvated surface and dried material present some voids and irregularities, the areas are probably affected by the evaporation of the solvent. The process was confirmed through the SEM analysis to have yielded a uniform and highly crystalline urea nitrate, which is suitable for subsequent industrial applications, if any, of the material, and at the same time showing an increase in the morphological uniformity and the structural integrity of the material.

Table 5. Elemental characterization of reactants and products was evaluated through ICP-OES analysis for nitric acid, urea phosphate, urea nitrate, and phosphoric acid

Parameters		AN		UP		UN			PA			
		Wt%	Impurity / P_2O_5	Wt%	Impurity/ P_2O_5	Wt%	Impurity/ P_2O_5	P_2O_5 wt%	Wt%	Impurity/ P_2O_5	Impurity reduction wt%	P_2O_5 wt%
P_2O_5	%	0.00	*	44.20	**	5.10	**	8.00	49.80	**		85.00
Al	mg/kg	0.00	*	47.00	0.00	50.77	0.0010	**	18.00	0.36	99.97	**
As	mg/kg	0.00	*	2.04	0.00	2.46	0.00	**	0.55	0.01	99.96	**
B	mg/kg	0.00	*	53.96	0.00	57.97	0.0011	**	20.90	0.42	99.97	**
Ba	mg/kg	0.00	*	9.96	0.00	11.15	0.0002	**	3.46	0.07	99.97	**
Be	mg/kg	0.00	*	0.05	0.00	0.04	0.00	**	0.03	0.00	99.98	**
Ca	mg/kg	0.00	*	1198.00	0.003	711.40	0.0139	**	964.00	19.36	99.99	**
Cd	mg/kg	0.00	*	0.13	0.00	0.16	0.00	**	0.04	0.00	99.96	**
Co	mg/kg	0.00	*	0.14	0.00	0.12	0.00	**	0.08	0.00	99.98	**
Cr	mg/kg	0.00	*	4.03	0.00	4.87	0.0001	**	1.08	0.02	99.96	**
Cu	mg/kg	1.00	*	2.40	0.00	1.83	0.00	**	3.07	0.06	99.99	**
Fe	mg/kg	4.00	*	196.80	0.00	235.20	0.0046	**	61.69	1.24	99.96	**
K	mg/kg	0.00	*	19.38	0.00	20.81	0.0004	**	7.46	0.15	99.97	**
Li	mg/kg	0.00	*	17.08	0.00	20.82	0.0004	**	4.45	0.09	99.96	**
Mg	mg/kg	0.00	*	155.90	0.00	159.90	0.0031	**	67.00	1.35	99.97	**
Mn	mg/kg	0.00	*	6.66	0.00	6.68	0.0001	**	2.98	0.06	99.97	**
Mo	mg/kg	0.00	*	0.19	0.00	0.18	0.00	**	0.09	0.00	99.98	**
Na	mg/kg	9.00	*	510.70	0.001	608.20	0.0119	**	159.20	3.20	99.96	**
Ni	mg/kg	0.00	*	2.07	0.00	1.49	0.00	**	1.43	0.03	99.98	**
Pb	mg/kg	1.00	*	0.55	0.00	0.64	0.00	**	1.66	0.03	100.00	**
S	mg/kg	16.00	*	423.00	0.001	520.50	0.0102	**	130.60	2.62	99.96	**
Sb	mg/kg	0.00	*	0.01	0.00	0.01	0.00	**	0.00	0.00	99.97	**
Se	mg/kg	0.00	*	0.62	0.00	0.07	0.00	**	0.74	0.01	99.99	**
Si	mg/kg	0.00	*	35.78	0.00	53.13	0.0010	**	1.13	0.02	99.64	**
Sn	mg/kg	0.00	*	0.11	0.00	0.12	0.00	**	0.04	0.00	99.97	**
Sr	mg/kg	0.00	*	10.42	0.00	10.93	0.0002	**	4.27	0.09	99.97	**
Te2	mg/kg	0.00	*	0.10	0.00	0.07	0.00	**	0.07	0.00	99.98	**
Ti	mg/kg	0.00	*	7.05	0.00	8.11	0.0002	**	2.25	0.05	99.96	**
Tl	mg/kg	0.00	*	0.07	0.00	0.03	0.00	**	0.07	0.00	99.99	**
V	mg/kg	0.00	*	0.46	0.00	0.32	0.00	**	0.33	0.01	99.98	**
Zn	mg/kg	0.00	*	25.57	0.00	26.26	0.0005	**	10.95	0.22	99.97	**
Density	*	1.32	*	*	*	*	*	**	1581	31.75	*	**
	Tapped	*	*	1.07	0.024	*	*	**	*	*	*	**
	Bulk	*	*	0.80	0.018	*	*	**	*	*	*	**
N_{amm}	%	0.00	*	0.99	0.00	0.93	0.00	**	0.50	0.01	99.98	**
N_{total}	%	10.80	*	17.50	0.396	36.00	*	**	0.80	*	*	**

Note: AN – nitric acid, UP – urea phosphate, UN – urea nitrate, PA – phosphoric acid, P_2O_5 – phosphorus pentoxide, Wt – weight, N_{amm} – total ammonium nitrogen, N_{total} – total nitrogen concentration.

Energy dispersive X-ray (EDX)

EDX analysis was performed by using MEB SH-5500P field emission scanning electron microscope. EDX analysis results in the depiction of the analysis outcome in a graph, showing different energy peaks corresponding to different types of elements present in the urea nitrate. The energy is represented by the horizontal x-axis in keV, while the count rate or intensity of the peak

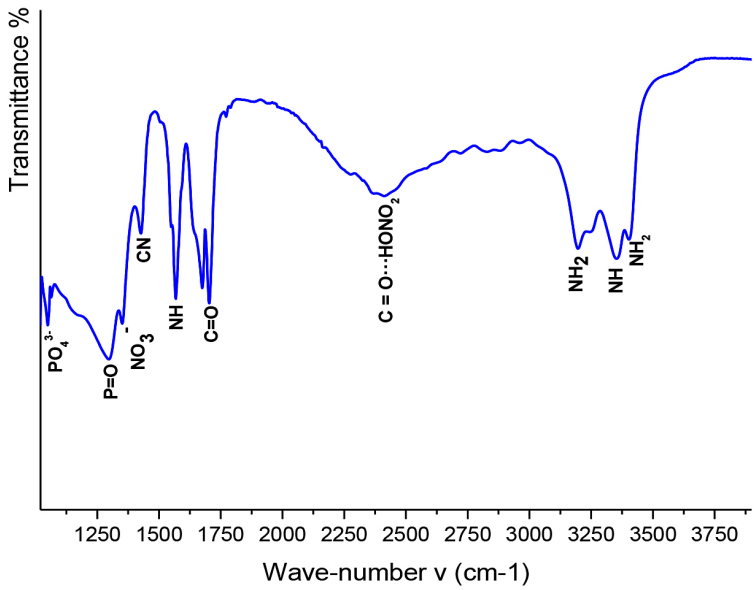

Figure 5. IR spectroscopy analysis of the product produced by the purification of urea phosphate depicts the bond vibrations of urea nitrate

Table 6. Frequencies of the vibrational bands of functional groups found in the urea nitrate

Assignment of bond vibrations		Wave number v (cm^{-1})
NH_2	vas	3400
NH	v	3338
NH2	vs	3187
$C{=}O\cdots HONO_2$	vs	2400
C=O	v	1701
NH	v	1565
CN	v	1430
NO_3^-	v	1321
PO	v	1295
PO_4^{3-}	vs	1048

is characterized by the vertical y-axis in cps/eV, as shown in Figure 7. The EDX spectra validated the presence of carbon, nitrogen, oxygen, and phosphorus elements in the synthesized product, i.e., urea nitrate.

The findings from these characterization analyses, therefore, demonstrate that the purified PA and urea nitrate synthesized in this research form untreated Moroccan PA are refined and of high quality.

Life cycle assessment

The life cycle assessment (LCA) analysis assesses the environmental performance of the nitrogen-based PA purification process developed in this research and compares it to conventional solvent extraction technology. The objectives of LCA are: (1) to evaluate environmental impact through multiple impact category evaluations of the nitrogen-based purification process; (2) to pinpoint environmental impact areas combined with process improvement possibilities; (3) to assess environmental efficiency of existing industrial purification methods; and (4) to develop sustainable production methods for PA through research-based recommendations. The assessment aims to identify the main environmental hotspots, evaluate resource efficiency, and determine scientifically rigorous environmental benefits for this nitrogen-based purification process, supporting transparent decision-making processes for industrial implementation.

Functional unit and system boundaries

The functional unit for this LCA is defined as the production of 1 metric ton of purified PA, which is equivalent to 54% P_2O_5 concentration. This functional unit was selected to reflect the composition of the actual product obtained, enable direct comparison with conventional industrial purification processes, and account for phosphorous concentration rather than simply total acid mass. Therefore, all of the environmental impacts are normalized to this functional unit, ensuring comparability across different process configurations and technologies. The LCA employs a cradle-to-grave approach, encompassing the production of wet-process PA from phosphate

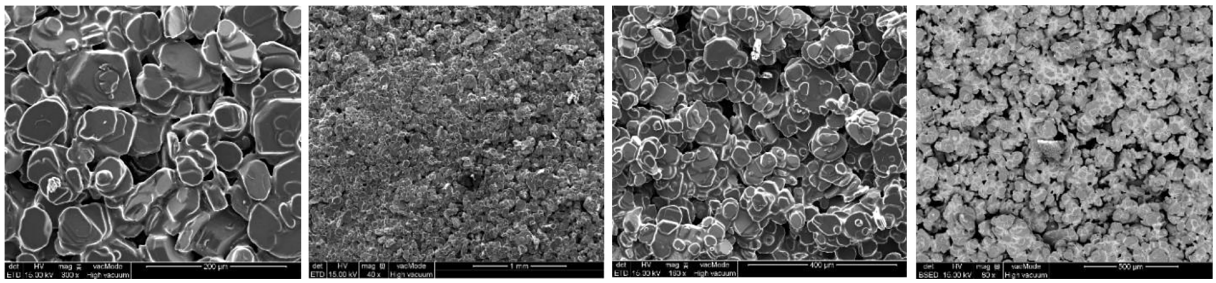

Figure 6. SEM microscopy analysis of the valorized urea nitrate produced from the purification of urea phosphate

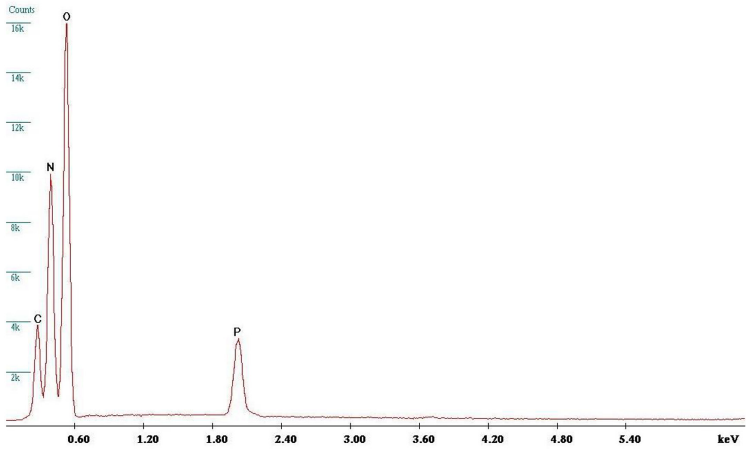

Figure 7. EDX analysis for the characterization of the elemental composition of the urea nitrate, indicating elemental peaks (C, N, O, or P) on the EDX spectra

rock, synthesis of UP, nitric acid decomposition of UP, crystallization/separation of UN, and concentration of purified PA. The application of downstream fertilizer and transportation were not included in this LCA assessment.

Inventory analysis

The life cycle inventory includes the consumption of urea, nitric acid (68%), water for washing or crystallization, and electricity required for mixing, filtration, centrifugation, and mild temperature control (<50 °C). The process involved intermediate temperature conditions because it does not need either high-temperature distillation or the track system of regeneration and reactivation processes. The absence of organic solvents eliminates emissions associated with volatile organic compounds (VOCs) because all organic solvents have been removed, lead to decreased operational cost for solvent collection systems. Additionally, the exothermic nature of UP formation partially offsets external heating requirements and contribute to overall energy efficiency.

The main output of the process produces purified PA which weighs 1,000 kg for each operational unit. The process produces UN as a valuable co-product containing 330 kg of product per ton of purified PA while serving as a high-quality nitrogen fertilizer with 36% of total nitrogen content. The process generates one main waste stream which consists of concentrated impurity residue of about 220 kg for every ton of purified PA and contains both removed metallic impurities and heavy metal contaminants (Fe, Al, or Mg) and heavy metals (Cd, Pb, Cr, or As). These materials can be disposed of through waste management or metal recovery operations.

Life cycle impact assessment

Global warming potential (GWP) quantifies total greenhouse gas emissions expressed as kg CO_2 equivalents, accounting for the radiative forcing of different greenhouse gases. The nitrogen-based purification process is expected to exhibit lower greenhouse gas emissions due to elimination of energy-intensive solvent distillation

and regeneration processes found in solvent extraction methods. The reaction operates at low temperatures while it obtains energy through exothermic heat release, which results in less need for external energy sources. Conventional solvent extraction requires heating to 120-180 °C for solvent stripping and distillation for solvent recovery, consuming 2,000-2,500 MJ of thermal energy per ton of purified PA. In contrast, this purification strategy operates at ambient temperature of 50 °C, requiring only 320 MJ of thermal energy per ton, representing an 87% reduction in use of energy. Furthermore, the co-production of UN provides environmental advantages through avoided production of equivalent nitrogen fertilizer via the energy-intensive Haber-Bosch ammonia synthesis process. Preliminary estimates suggest that the nitrogen-based process could reduce greenhouse gas emissions by 40-50% compared to conventional solvent extraction, contributing to climate change mitigation objective while maintaining high purification efficiency.

Atom economy – the process achieves an extremely high atom economy because it transforms both purified PA and UN into highly valorized products. Nearly, all atoms from the reactants (urea and nitric acid) are incorporated into valuable products that minimizes the production of waste products. The theoretical atom economy exceeds 85%, which is calculated as:

$$\textit{Atom economy} = (\textit{mass of useful products} / \textit{mass of all reactants}) \times 100$$

However, reagents are consumed to generate waste precipitates or utilization of solvents that further require disposal in conventional purification methods. The nitrogen-based PA purification process evades waste generation by transforming purification reagents into marketable fertilizer product, supporting the principles of green chemistry and circular economy.

E-factor (waste generation index) (kg waste per kg product) – decreases significantly because precipitation-based methods that produce excessive sludge and results in high level of waste products. The proposed process achieves impurity elimination through its design, creating restricted residual streams that recover urea nitrate as a commercial fertilizer. The 68–76% reduction in waste generation represents a major environmental advantage for reducing disposal costs, environmental liability, and land requirements for waste management facilities in comparison to conventional technologies.

Toxicity reduction – the purification step achieves removal efficiencies above 95% for most heavy metals (Cd, Cr, V, Fe, or Mg), which leads to a significant reduction of potential environmental toxicity during downstream fertilizer application. The reduced levels of heavy metals in soil enable safer compliance with international fertilizer regulations (EU 2019/1009) which establishes stringent contamination limits to decrease the risk of hazardous substance accumulation.

Comparative environmental assessment

This novel PA purification method offers several environmental benefits over conventional methods of solvent extraction and ion exchange because it eliminates organic solvents, requires less thermal energy, produces minimal secondary waste, and develops a marketable nitrogen fertilizer as a by-product. As discussed above, the purification process uses only aqueous solutions that help in eliminating the environmental impacts associated with the production, handling, loss, and disposal of organic solvents. However, the utilization of reagents in solvent extraction exhibits several toxicity concerns as it requires energy-intensive production from petroleum feedstocks and generates VOC emissions during processing. Regarding the above interpreted findings, life cycle impact assessment (LCIA) suggests valuable consequences over conventional solvent extraction treatment methodologies due to elimination of organic solvents, utilization of reduced thermal energy requirements, minimal secondary waste generation, production of high value nitrogen fertilizer, and efficient removal of heavy metals. Overall, this purification method integrates purification and fertilizer production, which results in decrease material losses and increase in resource efficiency.

Integration of circular economy principles

The process establishes an integrated nitrogen–phosphorus circular recovery. Phosphorus is recovered in purified form for high-value applications, while nitrogen reagents are transformed into urea nitrate fertilizer. The dual valorization strategy guarantees that both primary nutrients are essential for agriculture and remain within the productive cycle to minimize resource dissipation and support sustainable nutrient management.

The process embodies circular economy principles through multiple mechanisms, which is particularly significant for phosphorous sustainability phosphorus is essential, non-substitutable nutrient for food production, yet PR reserves are geographically concentrated and exhibits concerns regarding long-term resource security. Therefore, purification strategies that maximize phosphorus recovery enable access to premium marketable products with high value and minimize loss throughout the chain, contributing sustainable utilization of phosphorous. Similarly, efficient management of nitrogen reduces reliance on energy-intensive Haber-Bosch ammonia synthesis, accounting for ~1–2% of global energy consumption and emission of associated greenhouse gases.

Industrial sustainability and implications

From an industrial perspective, the process utilizes widely available reagents and conventional chemical engineering equipment (reactors, crystallizers, filters, dryers). The system needs lower operational expenses as it operates at moderate conditions compared to solvent-based systems acquiring high temperatures or use of disposable solvents. Preliminary environmental assessment suggests that integration of this purification strategy within existing fertilizer production facilities could lead to reduced carbon emissions, improved regulatory compliance, and enhanced economic sustainability. Therefore, this LCA demonstrates that the nitrogen-based purification process represents an eco-friendly purification alternative which competes with traditional PA purification methods, strengthening the circular economy principles for environmental-friendly fertilizer production.

DISCUSSION

This research work presented a two-step nitrogen-based purification process that successfully converts untreated Moroccan PA into a high-purity product while simultaneously producing

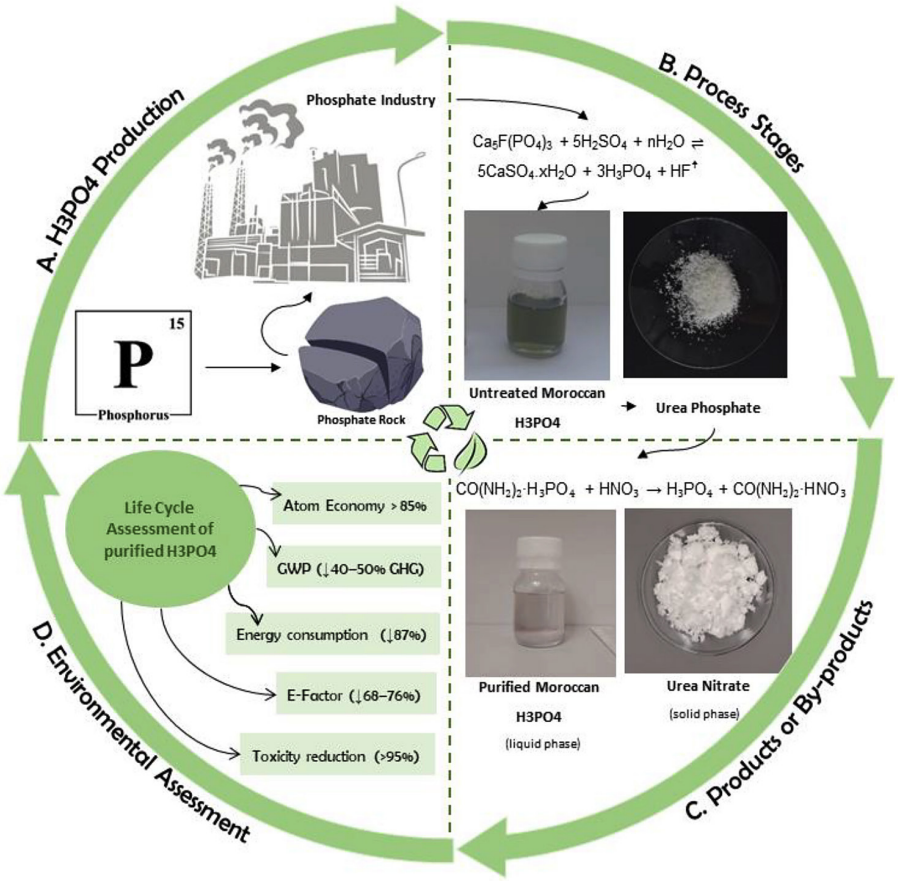

Figure 8. Life cycle assessment for the purification of untreated Moroccan phosphoric acid obtained from the Moroccan phosphate industries treated through a two-step nitrogen purification process

UN fertilizer, as a marketable nitrogen fertilizer. This approach provides multiple advantages because it enables better economic outcomes, reduced environmental impacts, and increased sustainable development compared to traditional purification methods. The purification process achieved remarkable improvements in acid quality, with P_2O_5 content increased from 52.6 wt% in the untreated Moroccan PA to 49.8 wt% in the purified Moroccan PA. Critically, the global P2O5 recovery yield was 71.40% in purified PA and 77.28% across total purified products (purified PA and UN), demonstrating that phosphorous is effectively retained within the valorized product streams rather than lost to waste. These findings provide effective outcomes in comparison to solvent extraction methods, which typically achieve overall recovery of 60–70% (Awwad, El-Nadi, and Hamed 2013; Kouzbour et al. 2019). Therefore, this purification process enhances concentration, which results in lower transportation expenses, reduced storage space needs, and decreased processing requirements for high-purity phosphoric acid applications.

By implementing this purification strategy, more than 90% removal of all major contaminants was achieved, such as 93% reduction of iron and aluminum, disrupting chemical reactions or decreasing product quality, and 94% magnesium removal because it prevents precipitation problems in both concentrated acid solutions and downstream products. These elements form stable complexes with phosphate in the wet process and their selective incorporation into the UP crystal lattice during the first purification step is consistent with the lower affinity of metallic impurities for the UP structure compared to the strong urea-PA hydrogen bonding (Jančaitienė, Medekšaitė, and Šlinkšienė 2023; Ayoub Cherrat et al. 2025). The mechanism aligns with the co-precipitation exclusion implemented for Jordanian wet process PA, demonstrating >95% Al and Fe rejection under similar crystallization conditions (Mubarak 2011).

The commercial and environmental benefits of heavy metal reductions represent a major accomplishment of this research. The purified acid achieves food-grade PA standards and environmental requirements through a reduction in 95-97% concentrations of cadmium, lead, and chromium impurities. The European Union regulations currently impose restrictions on heavy metal content in fertilizers, which require cadmium levels to remain below 60 mg per kg of P_2O_5 in specific product types. Meanwhile, the purified acid contains 0.04 mg/kg cadmium efficiently meets the standards and allow producers to sell products in high-end markets. North America, Japan, and other developed regions have established regulations that require businesses to attain reduced heavy metal levels, thus making purification processes essential for the market (Kouzbour et al. 2019). The efficiency of phosphorus recovery through the two-step process provides another important consideration. The overall P_2O_5 recovery reached 85% because phosphorus loss occurred during the crystallization, washing, and separation processes, which indicated better performance than conventional solvent extraction methods. The concentration effect results in volume loss, but the valuable phosphorus content remains mostly undamaged because it exists in a more valuable, purified state.

This purification process is also of great importance as it produces a beneficial UN by-product which exhibits commercial value due to the presence of low moisture content and decreased concentration of heavy metal impurities. UN ($CO(NH_2)_2 \cdot HNO_3$) serves as a premium nitrogen fertilizer, which contains 33.6% nitrogen by weight because both of its components (urea and nitrate) provide nitrogen. The nitrogen content of UN was consistent with standard nitrogen fertilizers, which include ammonium nitrate (34% N) and urea (46% N), because it provides competitive value in the fertilizer industry. UN provides dual nitrogen forms, which are further useful in agricultural applications. The nitrate component supplies instant nitrogen for plants, resulting in fast plant growth and development. Urea converts into forms that plants can use through soil urease activity at a gradual pace, which results in nitrogen release that lasts longer while reducing nitrogen loss through leaching as compared to sodium nitrate fertilizers. The system uses two nitrogen release methods, which result in efficient nitrogen utilization while decreasing how often farmers need to apply fertilizer and stopping environmental losses through leaching and nitrogen volatilization. The UN produced through this purification method achieves a higher fertilizer value because of its crystalline structure and pure composition. The well-formed crystals with good particle size distribution (10–100 μm) provide excellent physical properties for blending, spreading, and soil incorporation. Therefore, the product obtained through this purification process

is of great quality, making it a potential fertilizer for agricultural applications.

From a green chemistry perspective, the nitrogen-based process demonstrates several advantages over conventional solvent extraction methods. The theoretical atom economy exceeds 85%, as both purified PA and UN fertilizer constitute commercially valuable products and the only significant by-product stream is the concentrated impurity residue (~220 kg per ton of purified PA), which is amenable to metal recovery processing rather than requiring disposal as hazardous waste (Anastas and Warner 2000). The thermal energy requirement of 320 MJ per ton of the purified PA represents an 87% reduction relative to solvent recovery at 120–180 °C. The E-factor (kg waste per pkg product) is substantially lower than solvent-based processes as the purification reagents (urea and nitric acid) are incorporated into the UN product rather than generating spent solvent streams requiring disposal. The complete elimination of organic solvents removes VOC emission concerns, occupational exposure risks, and the need for explosion-proof process equipment, thereby reducing both capital expenditure and regulatory compliance costs for industrial implementation.

Purified PA obtained through this purification process meets or exceeds international standards requirements by leading global standards organizations. Food Chemicals Codex (FCC) specifications for food-grade PA require a minimum P_2O_5 content of 75% for the reference 85% H_3PO_4 basis, which, when adjusted for concentration, corresponds to approximately 52% P_2O_5 for the typical commercial concentrations produced in this study. The purified product achieves all required FCC heavy metal restrictions, including lead levels below 1.66 mg/kg, arsenic levels below 0.55 mg/kg, and mercury levels below 1 mg/kg. The European Pharmacopoeia and United States Pharmacopeia definitions for pharmaceutical-grade phosphoric acid, which represent the highest global purity requirements, will become achievable through minor process modifications that will decrease trace contaminant levels. The research results demonstrate that the purified acid quality meets all technical-grade industrial standards that apply to detergent production, metal treatment, and chemical synthesis operations. The European Union Fertilizer Regulation (EU 2019/1009) establishes maximum contaminant limits for various elements in fertilizers, including cadmium (currently 1.5 mg/kg P_2O_5, with future reductions to 40 mg/kg planned), lead, and other heavy metals. In this research, the obtained UN by-product contains negligible heavy metal contamination, positioning it favorably for European markets and other regions with stringent environmental standards. The fertilizer product achieves worldwide market distribution through its compliance with AAPFCO standards in North America, EU 2019/1009 in Europe, and equivalent standards in Asia, Africa, and South America.

CONCLUSIONS

A sustainable two-step nitrogen-based purification process for untreated Moroccan phosphoric acid through urea phosphate followed by nitric acid decomposition was developed. The process achieved high removal efficiencies for major impurities (Al, Fe, and Mg), while producing purified PA with a P2O5 recovery yield of 71.40%. Simultaneously, a valuable urea nitrate fertilizer by-product was generated which demonstrates an effective resource valorization. This approach offers lower energy consumption, avoids the use of organic solvents, and aligns with green chemistry and circular economy principles as compared to conventional purification methods. These findings highlight the potential of this technology as an environmentally friendly and industrially relevant alternative for PA purification, while future studies should focus on pilot-scale validation, process optimization, and integration with phosphogypsum valorization technologies.

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