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«ХАЛЫҚ» ЖҚ

ҚАЙЫРЫМДЫЛЫҚ ҚОРЫ
HALYK
CHARITY FOUNDATION

ХАБАРЛАРЫ

ИЗВЕСТИЯ

РОО «НАЦИОНАЛЬНОЙ
АКАДЕМИИ НАУК РЕСПУБЛИКИ
КАЗАХСТАН»
ЧФ «Халық»

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ЧФ «ХАЛЫҚ»

В 2016 году для развития и улучшения качества жизни казахстанцев был создан частный Благотворительный фонд «Халык». За годы своей деятельности на реализацию благотворительных проектов в областях образования и науки, социальной защиты, культуры, здравоохранения и спорта, Фонд выделил более 45 миллиардов тенге.

Особое внимание Благотворительный фонд «Халык» уделяет образовательным программам, считая это направление одним из ключевых в своей деятельности. Оказывая поддержку отечественному образованию, Фонд вносит свой посильный вклад в развитие качественного образования в Казахстане. Тем самым способствуя росту числа людей, способных менять жизнь в стране к лучшему – профессионалов в различных сферах, потенциальных лидеров и «великих умов». Одной из значимых инициатив фонда «Халык» в образовательной сфере стал проект Ozgeris powered by Halyk Fund – первый в стране бизнес-инкубатор для учащихся 9-11 классов, который помогает развивать необходимые в современном мире предпринимательские навыки. Так, на содействие малому бизнесу школьников было выделено более 200 грантов. Для поддержки талантливых и мотивированных детей Фонд неоднократно выделял гранты на обучение в Международной школе «Мирас» и в Astana IT University, а также помог казахстанским школьникам принять участие в престижном конкурсе «USTEM Robotics» в США. Авторские работы в рамках проекта «Тәлімгер», которому Фонд оказал поддержку, легли в основу учебной программы, учебников и учебно-методических книг по предмету «Основы предпринимательства и бизнеса», преподаваемого в 10-11 классах казахстанских школ и колледжей.

Помимо помощи школьникам, учащимся колледжей и студентам Фонд считает важным внести свой вклад в повышение квалификации педагогов, совершенствование их знаний и навыков, поскольку именно они являются проводниками знаний будущих поколений казахстанцев. При поддержке Фонда «Халык» в южной столице был организован ежегодный городской конкурс педагогов «Almaty Digital Ustaz.

Важной инициативой стал реализуемый проект по обучению основам финансовой грамотности преподавателей из восьми областей Казахстана, что должно оказать существенное влияние на воспитание финансовой грамотности и предпринимательского мышления у нового поколения граждан страны.

Необходимую помощь Фонд «Халык» оказывает и тем, кто особенно остро в ней нуждается. В рамках социальной защиты населения активно проводится работа по поддержке детей, оставшихся без родителей, детей и взрослых из социально уязвимых слоев населения, людей с ограниченными возможностями, а также обеспечению нуждающихся социальным жильем, строительству социально важных объектов, таких как детские сады, детские площадки и физкультурно-оздоровительные комплексы.

В копилку добрых дел Фонда «Халык» можно добавить оказание помощи детскому спорту, куда относится поддержка в развитии детского футбола и карате в нашей стране. Жизненно важную помощь Благотворительный фонд «Халык» оказал нашим соотечественникам во время недавней пандемии COVID-19. Тогда, в разгар тяжелой борьбы с коронавирусной инфекцией Фонд выделил свыше 11 миллиардов тенге на приобретение необходимого медицинского оборудования и дорогостоящих медицинских препаратов, автомобилей скорой медицинской помощи и средств защиты, адресную материальную помощь социально уязвимым слоям населения и денежные выплаты медицинским работникам.

В 2023 году наряду с другими проектами, нацеленными на повышение благосостояния казахстанских граждан Фонд решил уделить особое внимание науке, поскольку она является частью общественной культуры, а уровень ее развития определяет уровень развития государства.

Поддержка Фондом выпуска журналов Национальной Академии наук Республики Казахстан, которые входят в международные фонды Scopus и Wos и в которых публикуются статьи отечественных ученых, докторантов и магистрантов, а также научных сотрудников высших учебных заведений и научно-исследовательских институтов нашей страны является не менее значимым вкладом Фонда в развитие казахстанского общества.

С уважением,
Благотворительный Фонд «Халык»!

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ENHANCING THE CONCENTRATION OF NODULAR PHOSPHORITEBY SELECTIVE LEACHING

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Abstract. The focus of this research is the detailed investigation into the selective leaching method specifically designed to concentrate nodular phosphorites, a significant yet challenging phosphate resource to exploit using traditional means. This method employs acetic acid as the principal leaching agent, due to its ability to selectively dissolve phosphorus compounds while leaving behind most of the impurities. This study systematically examines the influence of critical processing parameters - temperature and time, on the consumption rate of acetic acid, the pH value of the resulting solution, and ultimately the P_2O_5 (phosphorus pentoxide) content, which directly indicates the efficiency of phosphorus extraction. Detailed experimental results showed that under the optimal conditions of 30 °C temperature and 30-minute reaction time, the process led to a significant increase in the P_2O_5 content of the treated material, reaching up to 25.52 %. This is an encouraging result, demonstrating the effectiveness of this selective leaching method under these conditions. To gain more insights into the process, X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) analyses were carried out, which revealed interesting changes in both the mineralogical and surface characteristics of the processed nodular phosphorites. These analyses provided essential information on how the selective leaching process affects the physical and chemical properties of the material and how these changes could impact the downstream processing and utilization of the phosphorites. The findings of this study highlight the potential for the selective leaching method to serve as a viable, efficient, and environmentally-friendly option for the concentration and beneficiation of nodular phosphorites. Looking ahead, this research opens up new opportunities for further study, including the possibility of scaling up this selective leaching method to industrial scales. Additional future work could also delve deeper into developing a more comprehensive understanding of the mineralogy and chemical composition of the resulting enriched nodular phosphorites. Such knowledge could offer more insights into optimizing the leaching process and tailoring the downstream processing steps to maximize the utilization and value of these phosphorite resources.

Keywords: nodular phosphorites, selective leaching, acetic acid, mineral composition, enrichment

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ІРІКТЕМЕЛІ ШАЙМАЛАУ АРҚЫЛЫ КОНКРЕЦИОНДЫ ФОСФОРИТКОНЦЕНТРАЦИЯСЫН АРТТЫРУ

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Аннотация. Бұл зерттеудің негізгі бағыты дәстүрлі тәсілдермен маңызды, бірақ пайдалану қиын фосфат ресурсы болып табылатын конкреционды фосфориттерді шоғырландыру үшін арнайы әзірленген іріктемелі шаймалау әдісін егжей-тегжейлі зерттеу болып табылады. Бұл әдіс карбонат қосылыстарын селективті еріту қабілетіне байланысты сірке қышқылын негізгі байыту реагенті ретінде пайдаланады. Зерттеуде сыни өңдеу параметрлерінің - температура мен уақыттың, сірке қышқылының жұмсалу деңгейіне, алынған ерітіндінің pH мәніне және сайып келгенде, байыту тиімділігін тікелей көрсететін P_2O_5 (фосфор пентоксиді) мазмұнына әсерін жүйелі түрде зерттейді. Егжей-тегжейлі эксперименттік нәтижелер 30 °С температураның оңтайлы жағдайында және 30 минуттық реакция уақытында өңделген материалдағы P_2O_5 құрамының 25,52 % - ға дейін айтарлықтай артуына әкелетінін көрсетті. Бұл берілген жағдайларда селективті шаймалау әдісінің тиімділігін көрсететін жігерлендіретін нәтиже. Процесс туралы толық түсінік алу үшін рентгендік дифракция (XRD) және сканерлеуші электронды микроскопия (SEM) талдаулары жүргізілді, олар байытылған конкреционды фосфориттерінің минералогиялық және беттік сипаттамаларында өзгерістерді анықтады. Бұл талдаулар селективті шаймалау процесі материалдың физикалық және химиялық қасиеттеріне қалай әсер ететіні және бұл өзгерістер фосфориттерді кейіннен өңдеуге және кәдеге жаратуға қалай әсер ететіні туралы маңызды ақпарат берді. Бұл зерттеудің нәтижелері белгілі бір фосфориттерді байытудың өміршең, тиімді және қоршаған ортаға зиянсыз нұсқасы ретінде іріктемелі шаймалау әдісінің әлеуетін көрсетеді. Сондай-ақ, мұнда әрі қарай зерттеудің жаңа мүмкіндіктерін, соның ішінде осы іріктемелі шаймалау әдісін өнеркәсіптік масштабқа дейін ұлғайту мүмкіндігі бар екендігін ескерген жөн. Оған қоса, алдағы зерттеулерде байытылған конкреционды фосфориттерінің минералогия мен химиялық құрамы туралы толық түсінік қалыптастыруға тереңірек үңілуі мүмкін. Мұндай білім байыту процесін оңтайландыру және осы фосфорит ресурстарының максималды қолданылуы мен құндылығы үшін өңдеудің кейінгі кезеңдерін бейімдеу туралы көбірек ақпарат бере алады.

Түйін сөздер: конкреционды фосфориттер, іріктемелі шаймалау, сірке қышқылы, минералды құрамы, байыту

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ПОВЫШЕНИЕ КОНЦЕНТРАЦИИ КОНКРЕЦИОННОГО ФОСФОРИТА МЕТОДОМ СЕЛЕКТИВНОГО ВЫЩЕЛАЧИВАНИЯ

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Аннотация. В центре внимания этого исследования находится детальное изучение метода селективного выщелачивания, специально разработанного для концентрирования конкреционных фосфоритов, значительного, но сложного в использовании фосфатного ресурса традиционными способами. В этом методе в качестве основного выщелачивающего агента используется уксусная кислота, благодаря ее способности избирательно растворять соединения карбонатов. В статье систематически исследуется влияние критических параметров обработки - температуры и времени - на норму расхода уксусной кислоты, значение pH получаемого раствора и, в конечном счете, содержание P_2O_5 (пятиокиси фосфора), что непосредственно указывает на эффективность обогащения. Подробные экспериментальные результаты показали, что при оптимальных условиях температуры 30 °C и 30-минутном времени реакции процесс приводил к значительному увеличению содержания P_2O_5 в обработанном материале, достигая 25,52 %. Это обнадеживающий результат, демонстрирующий эффективность данного метода селективного выщелачивания в данных условиях. Чтобы получить более полное представление о процессе, были проведены анализы методом рентгеновской дифракции (XRD) и сканирующей электронной микроскопии (SEM), которые выявили интересные изменения как в минералогических, так и в поверхностных характеристиках обогащенных конкреционных фосфоритов. Эти анализы предоставили важную информацию о том, как процесс селективного выщелачивания влияет на физические и химические свойства материала и как эти изменения могут повлиять на последующую переработку и утилизацию фосфоритов. Результаты этого исследования подчеркивают потенциал метода селективного выщелачивания в качестве жизнеспособного, эффективного и безвредного для окружающей среды варианта обогащения конкреционных фосфоритов. Забегая вперед, отметим, что это исследование открывает новые возможности для дальнейшего изучения, включая возможность масштабирования этого метода селективного выщелачивания до промышленных масштабов. В дальнейшем можно добиться более полного понимания минералогии и химического состава полученных обогащенных конкреционных фосфоритов. Такие знания могли бы дать больше информации об оптимизации процесса выщелачивания и адаптации последующих этапов переработки для максимального использования и понимания ценности этих фосфоритных ресурсов.

Ключевые слова: конкреционные фосфориты, селективное выщелачивание, уксусная кислота, минеральный состав, обогащение

Introduction

Nodular phosphorites are sedimentary rocks that are formed through the accumulation of phosphate-rich particles around a central nucleus. The formation process typically occurs in marine environments with low to moderate energy conditions, such as shallow seas and lagoons. The formation of nodular phosphorites begins with the accumulation of organic matter and phosphate-rich sediments on the seafloor. Over time, these sediments become compacted and cemented, forming a hard substrate. Phosphorus in the sediments is derived from various sources, including marine organisms, volcanic activity, and weathering of land-based rocks (Kyser, 2014). As the sedimentation continues, phosphate particles in the water column adhere to the hard substrate, forming small nodules or concretions around a central nucleus. The phosphate minerals in nodular phosphorites are typically composed of carbonate-fluorapatite, which is a type of calcium phosphate mineral. Nodular phosphorites can occur in a range of depositional environments, including continental shelves, lagoons, and oceanic plateaus. They are often associated with other sedimentary rocks, such as shale and sandstone, and can be found in various geological formations (Kolodny, 2009).

The distribution of nodular phosphorites is not uniform, and their occurrence is influenced by several factors, including sea level fluctuations, sedimentation rates, and climate. Some of the largest deposits of nodular phosphorites are located in North Africa, the Middle East, Russia and the United States, although they are found in many other parts of the world as well (Burnett, 1990).

Nodular phosphorites are found in various parts of the world, with some of the largest deposits located in North Africa, the Middle East, and the United States. Here are some of the main deposits of nodular phosphorites in the world:

- Western Sahara: This region is home to some of the largest deposits of nodular phosphorites in the world, with estimated reserves of around 50 billion tons. The deposits are located in the Bou Craa mine and are heavily mined for the production of phosphate rock (Van Kauwenbergh, 2010);

Morocco: Morocco is the largest producer of phosphates in the world and has significant reserves of nodular phosphorites. The deposits are located in the Khouribga and Benguerir regions and are an important source of phosphorus for the global market (El Asri, 2009);

- Tunisia: Tunisia has extensive deposits of nodular phosphorites in the Gafsa basin, which is located in the southern part of the country. The deposits are mined by the Compagnie des Phosphates de Gafsa (CPG) and are an important source of phosphorus for the country's agriculture and industry (Boujlel, 2019);

- Florida, USA: Florida is home to one of the largest deposits of nodular phosphorites in the United States, with reserves estimated at around 1.4 billion tons. The deposits are located in the Bone Valley formation and are mined for the production of fertilizer (Beavers, 2013);

- Peru: Peru has significant deposits of nodular phosphorites in the Bayovar region, which is located in the northern part of the country. The deposits are mined by several companies, including Mosaic, and are an important source of phosphorus for the global market (Ramos, 2016);

China: China has several deposits of nodular phosphorites, including the Qilianshan and Wengfu deposits. The country is one of the largest producers of phosphorus in the world and relies heavily on its domestic deposits of nodular phosphorites (Jiang, 2018).

As it is known, the most common types of phosphorites in the world are nodular and microgranular phosphorites, which are two different types of sedimentary rocks that contain high concentrations of phosphate minerals. While they share some similarities, they also have several key differences (Ahmed, 2022; Krasilnikova, 1993; Yang, 2019):

- appearance: nodular phosphorites are characterized by their nodular or concretionary appearance, which is caused by the accumulation of phosphate-rich particles around a central nucleus. Microgranular phosphorites, on the other hand, have a fine-grained or granular texture, with the phosphate minerals distributed evenly throughout the rock;

- mineralogy: nodular phosphorites are typically composed of carbonate-fluorapatite, which is a type of calcium phosphate mineral. Microgranular phosphorites can contain various types of phosphate minerals, including francolite, collophane, and wavellite;
- formation: nodular phosphorites are formed through the accumulation of phosphate-rich particles around a central nucleus in low to moderate energy marine environments. Microgranular phosphorites are often associated with upwelling currents and are formed through the precipitation of phosphate minerals in the water column;
- occurrence: nodular phosphorites are found in a range of depositional environments, including continental shelves, lagoons, and oceanic plateaus. Microgranular phosphorites are often associated with ancient marine basins and can be found in sedimentary rocks such as shale and sandstone;
- use: nodular phosphorites are an important source of phosphorus for various industries, including agriculture and chemical manufacturing. Microgranular phosphorites are less commonly used and are often considered to have lower economic value due to their lower concentrations of phosphate minerals;

The most important difference between the two types of phosphorites is the high degree of isomorphic substitution of a phosphate group for a carbonate one (Yanshin, 1979). The carbon dioxide entering into the molecule loosens it, as it were, makes phosphate more soluble, and therefore the nodular phosphorites are a good raw material for the production of phosphate rock, which has long been used in Russia and other countries as the simplest environmentally friendly phosphorus-containing fertilizer (Lygach, 2019). In addition, another feature of the nodular phosphorites is their difficulty in enriching from the thin germination of phosphate with other minerals and, as a result, the high content of iron and aluminum oxides in the nodules. To date, several methods of enrichment of nodular phosphorites are known: flotation, magnetic separation, mechanical enrichment, the use of heavy suspensions, acid enrichment. However, each of the above-mentioned methods has its drawbacks (Al-Fariss, 2013; Mohammadkhani, 2011; Ptacek, 2016; Abbes, 2020). The most common method of enrichment is flotation. However, each of the above-mentioned methods has its drawbacks. The most common method of enrichment is flotation, but its use is also limited. The main disadvantages of flotation are the high cost of flotation reagents, complex hardware design, contamination of the phosphate part with organic compounds, dependence on the availability of water. In this regard, the search for new or improvement of known enrichment methods is relevant. Scientists from different countries where phosphate ore is mined call the method of selective leaching the most promising.

Selective leaching is a process used to concentrate valuable minerals from ores or rocks that contain multiple minerals or compounds. It involves dissolving the target mineral using a chemical solution, while leaving the other minerals intact. The process is based on the differences in the reactivity and solubility of the minerals in the ore or rock, which allows for the selective extraction of the desired mineral. Selective leaching can be used to extract a wide range of minerals. It is often used in hydrometallurgical processes, where minerals are extracted from ores using liquid solutions, and can also be used as a pre-treatment step to enhance the recovery of target minerals.

The process of selective leaching involves several steps, including the preparation of the ore or rock, the selection of the appropriate leaching solution, and the implementation of the leaching process. The leaching solution can be an acid, a base, or a complexing agent, depending on the mineral being targeted. Selective leaching has several advantages over conventional mineral processing methods, including reduced energy consumption, lower costs, and higher recovery rates (Du, 2020).

At the same time, organic acids can be used as a leaching solution. Organic acids, such as citric acid, oxalic acid, and humic acid, offer several prospects and benefits for the enrichment of low-grade phosphorites (Zafar, 1996; Zafar, 2007; Ashraf, 2005). These naturally occurring compounds are biodegradable, environmentally friendly, and readily available. Enrichment of low-grade phosphorites with organic acids is a cost-effective method as organic acids are relatively inexpensive compared to traditional chemical reagents used in mineral processing. This makes the process more affordable and accessible. Additionally, organic acids are environmentally sustainable as they are biodegradable and do not produce toxic byproducts that can harm the environment. This makes the process more sustainable and environmentally friendly. Organic acids are able to selectively leach carbonates from low-grade phosphorites, which means that they can be used to target specific minerals and increase the concentration of phosphorus in the ore. They can also enhance the efficiency of the enrichment process by increasing the rate and extent of carbonates dissolution (Bakry, 2015). This results in a higher quality phosphate product that has a lower impurity content and a higher concentration of phosphorus. Enrichment of low-grade phosphorites with organic acids can also lead to higher yields of phosphate, which can result in increased profitability for mining and processing companies. This makes the process more economically viable and attractive for companies operating in the industry.

As a whole, the use of organic acids for the enrichment of low-grade phosphorites offers several benefits, including cost-effectiveness, sustainability, selective leaching, increased efficiency, improved quality, and higher yields.

Nodular phosphorites in Kazakhstan is mainly represented by the Chilisai deposit (Aktobe phosphorite-bearing basin, Western Kazakhstan). Kazakhstan is a significant exporter of phosphorus-containing products to countries in Europe and Asia, which has led to a rise in both domestic and external demand for these products. However, the development and operation of new phosphorus deposits have not been carried out. As a result, the known deposits of phosphorites are being depleted, which necessitates the use of new resources. To address this issue, the concept of geological development of the mining industry of the country highlights the search for new methods of enrichment of nodular phosphorites and further processing into phosphorus products as one of the primary tasks.

Therefore, the purpose of this research is to explore the enrichment of nodular phosphorites by selective leaching using acetic acid. This method is being investigated as a potential alternative to traditional mineral processing methods to increase the concentration of valuable minerals, such as phosphate minerals, in low-grade ores. The use of selective leaching with acetic acid has shown promise in enhancing the efficiency and selectivity of the mineral processing process while reducing the environmental impact associated with traditional methods.

Overall, the research aims to contribute to the sustainable management of phosphorus resources in Kazakhstan and to promote the development of more environmentally friendly and efficient methods for the enrichment of nodular phosphorites.

Materials and Methods

Sampling and grinding

In order to conduct the research on the enrichment of nodular phosphorites, samples were obtained from the Chilisai deposit in Western Kazakhstan. To prepare the raw materials for further analysis, a laboratory ball mill MSHL-1 was used. The mill operates by filling the drum with raw materials and balls made of durable steel material. As the drum rotates, the impact of the balls crushes the material to a finer consistency. After crushing, the raw materials were sifted using a "Analysette" vibro-sieve.

This process of crushing and sifting the raw materials was necessary to prepare the samples for further testing and analysis. By reducing the size of the nodular phosphorites and separating them into finer particles, the samples were made more amenable to selective leaching and other mineral processing techniques.

Analysis

Several analytical methods were used to study the properties and composition of the nodular phosphorite samples.

To measure the concentration of phosphorus in the sample used a photometric method with Trilon B as the reagent. This method involved a reaction between Trilon B and phosphorus, which formed a complex with a characteristic color. The intensity of the color was proportional to the concentration of phosphorus and was measured using a spectrophotometer at a specific wavelength of 440 nm. This photometric method was chosen because of its sensitivity and reliability, making it suitable for this study.

To obtain high-resolution images of the sample surface used the JSM6490 LV scanning electron microscope (SEM). The SEM was equipped with a backscattered electron detector, an energy-dispersive X-ray spectroscopy (EDS) system, and a cathodoluminescence detector. The backscattered electron detector provided information on the surface and sub-surface structure of the sample, while the EDS system was used for chemical analysis. The cathodoluminescence detector was used to visualize light emission from the sample in response to electron bombardment.

In addition to the SEM analysis performed X-ray diffraction (XRD) analysis using the D8 Advance equipment from Bruker. This method allowed for the characterization of the crystalline structure of the sample, providing information on crystal structure, crystallinity, and preferred orientation.

The IR-Fourier spectrometry was performed on the IR-Prestige 21, a modern device equipped with a quick-release Michelson interferometer and a spectral range of 7800–350 cm^{-1}. The IRsolution software package was used for processing the received analytical materials. This method provided information on the chemical composition and functional groups present in the samples.

To determine the mineralogical structure of the phosphorites, the NEOPHOT-21 metallographic microscope was used. This microscope, from Germany, is equipped with computer software for processing the received micrographs. The raw materials were first prepared using installations from the STRUERS brand, from Denmark. This method provided information on the mineralogical composition and crystal structure of the samples.

Additionally, the Q-1500 D derivatograph from Hungary was used for differential thermal analysis of the materials. This technique allowed for the study of the heat resistance of the materials and the processes of hydration and dehydration.

To better understand the processes of enrichment of low-grade phosphorites with acetic acid, a thermodynamic analysis was performed using the HSC 9.3 software package. This multifunctional software is based on the principle of maximum entropy and minimization of Gibbs energy. The software's Reaction Equations module was used to calculate the change in the values of enthalpy (ΔH), entropy (ΔS), and Gibbs free energy (ΔG) for the heterogeneous reactions involved in the process.

Description of the experimental process

In the experimental studies, crushed raw materials were utilized. The process was carried out within a temperature range of 20–40 °C, and the enrichment time was set to be between 15-45 minutes. The liquid/solid ratio was set to be 3/1, which was determined by calculating the amount of acetic acid required for the complete decomposition of carbonate compounds in phosphate raw materials and the volume of water required for dilution. The solubility of acetic acid in water (40 g/l) was also taken into account. The dilute acetic acid had a density of d=1.030, a pH value of 2.33, and $pK\alpha=6.2\cdot10^{-2}$.

To determine the concentration of the dilute acid and the resulting pulp, the pH values were used to calculate the concentration (C, mol/l) using the expression $[H^+]=10^{-pH}$. These values were then utilized to calculate the level of consumption of acetic acid for the enrichment process (α):

$$\alpha = \frac{(C_1-C_2)}{C_1}\cdot 100, \tag{1}$$

where: C_1 – concentration of dilute acetic acid, mol/l; C_2 – pulp concentration, mol/l.

Based on this value, the reaction rate is calculated using the following formula (2):

$$\nu = \frac{\Delta n}{V\Delta\tau}, \tag{2}$$

where: Δn – change in the number of moles of starting substances, mol; V – volume of the mixture, l; $\Delta\tau$ – time, min.

The efficiency of the process under study was determined by P_2O_5,%. The degree of increase in P_2O_5, %, is calculated by the following expression:

$$P_2O_{5\,DI} = \left[\frac{B\cdot b}{P\cdot p}\right]\cdot 100\,, \tag{3}$$

where: B – weight of enriched raw materials, g; b – P_2O_5 in enriched raw materials, %; P – weight of the initial raw material, g; p – P_2O_5 in initial raw material, %.

Processing of experimental data

To evaluate the significance of the results conducted statistical analysis using Microsoft Excel. Normal distribution tests were conducted to ensure that the data met the assumptions of parametric statistical analysis. A p-value of less than 0.05 was considered statistically significant, indicating a 95 % confidence level that the results were not due to chance.

Results and Discussion

Material composition of nodular phosphorites

Studying the material composition of nodular phosphorites is essential to selective leaching as it provides insights into the properties and reactivity of the mineral components. The mineralogical composition of nodular phosphorites can vary significantly depending on the deposit, and the concentration of the desired phosphate minerals can be low. Understanding the mineralogy and chemical composition of the raw materials is critical to developing effective and efficient selective leaching processes.

The chemical composition of nodular phosphorites is shown in Table 1.

Table 1. Chemical composition of nodular phosphorites

Compounds	P_2O_5	CaO	MgO	K_2O	Al_2O_3	Fe_2O_3	SiO_2
Composition, %	18.12±0.91	31.19±1.13	1.72±0.24	0.13±0.09	2.04±0.31	0.97±0.37	23.10±1.24

The chemical composition of nodular phosphorites typically includes a relatively high concentration of phosphorus pentoxide (P_2O_5) at 18.12±0.91 %. The determined amount of phosphorus anhydride indicates that this particular raw material can be categorized as low-grade phosphorites. Other important components include calcium oxide (CaO) at 31.19±1.13 %, and silica (SiO_2) at 23.10±1.24 %. Additionally, small amounts of magnesium oxide (MgO), potassium oxide (K_2O), aluminum oxide (Al_2O_3), and iron oxide (Fe_2O_3) are also present in nodular phosphorites, with values ranging from 0.13±0.09 % for K_2O to 2.04±0.31 % for Al_2O_3 and 0.97±0.37 % for Fe_2O_3. The exact chemical composition of nodular phosphorites can vary depending on the specific deposit and geological conditions. To compare the chemical composition, the method of energy dispersive analysis was used, the results of which are shown in Table 2 and Figure 1.

Table 2. Element-weight composition of nodular phosphorites

Element	Spectrum 1		Spectrum 2		Spectrum 3		Spectrum 4		Average, %	
	Weight comp., %	In terms of oxides, %	Weight comp., %	In terms of oxides, %	Weight comp., %	In terms of oxides, %	Weight comp., %	In terms of oxides, %	Weight comp., %	In terms of oxides, %
C	2.93	-	-	-	-	-	1.76	-	2.34	-
O	43.26	-	54.86	-	54.53	-	52.94	-	51.39	-
F	2.32	-	0.66	-	1.71	-	-	-	1.56	-
Mg	3.37	5.58	1.19	1.97	1.37	2.27	0.42	0.69	1.58	2.61

Al	1.60	3.02	0.87	1.64	0.11	0.20	1.38	2.60	0.99	1.87
Si	10.17	21.75	14.21	30.39	11.08	23.70	12.43	26.58	11.97	25.60
P	8.08	18.51	6.31	14.45	7.43	17.02	8.17	18.71	7.49	17.17
K	0.13	0.15	-	-	0.27	0.32	0.10	0.12	0.16	0.19
Ca	27.72	38.78	21.09	29.50	23.16	32.40	22.63	31.65	23.65	33.08
Fe	0.42	0.60	0.81	1.15	0.34	0.48	0.17	0.24	0.43	0.61

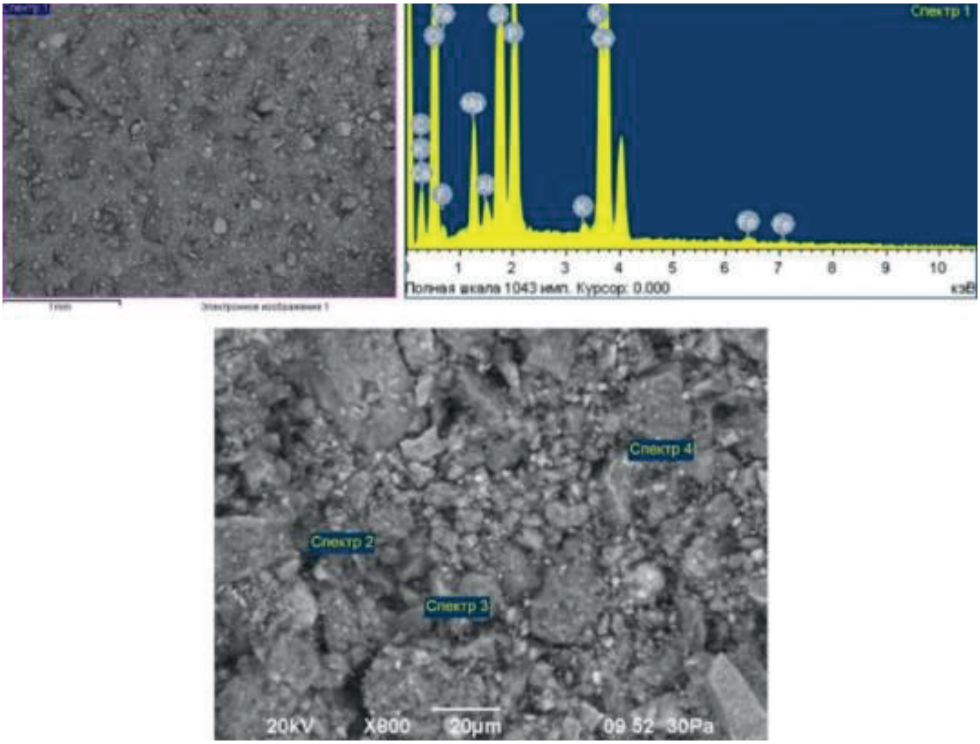

Figure 1. Micrograph of the surface of samples of nodular phosphorite and its elemental-weight composition

The surface of the nodular phosphorite sample was analyzed using a scanning electron microscope (SEM) at a low voltage to minimize damage to the delicate surface. The resulting image showed a highly textured surface with numerous spherical nodules ranging in size from a few micrometers to several millimeters in diameter. The nodules appeared to be composed of small crystalline structures, with irregular shapes and sizes. The surface also exhibited several areas of mineral deposition and accumulation, suggesting a complex geological history. Overall, the SEM analysis provided valuable insights into the surface morphology and mineralogical composition of the nodular phosphorite sample. In addition, after analyzing the results of chemical and elemental-weight analyzes, one can see that the given composition of nodular phosphorites is the same.

To accurately study the composition of the research object, the method of X-ray diffraction analysis was used, the results of which are shown in Figure 2.

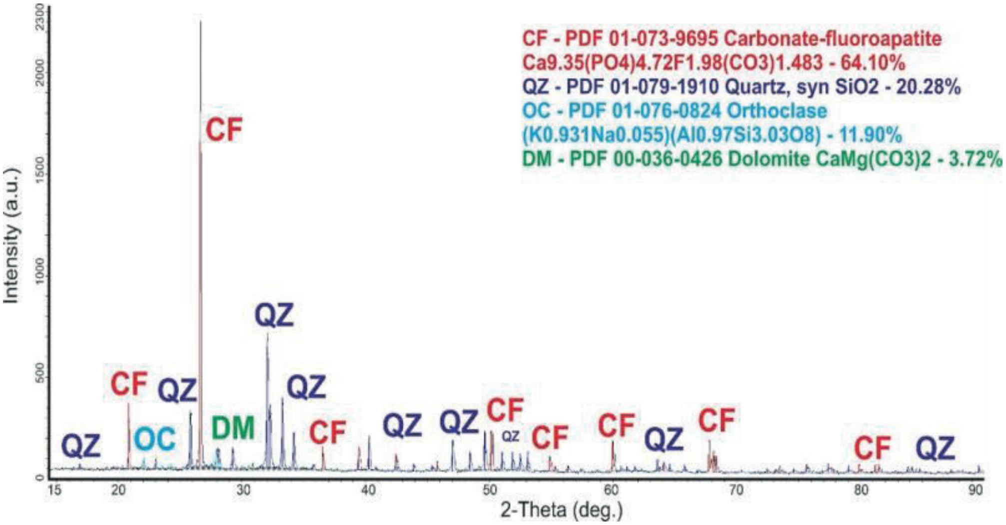

Figure 2. X-ray diffraction peaks of nodular phosphorites

The XRD analysis of the nodular phosphorite sample revealed the presence of several mineral phases. The dominant phase was carbonate-fluoroapatite (NR), which accounted for 64.10% of the sample. This mineral is a major source of phosphate in the sample and is commonly found in nodular phosphorites. As mentioned above, the phosphate part in nodular phosphorites is represented by the mineral kurskite (Yudovich, 2018). This is a variety of fluorapatite, in which there was a partial isomorphic replacement of the phosphate group by the carbonate group. In terms of mineralogical composition, the ore of this deposit is close to the phosphates of the Egorievskoe and Vyatskoe deposits in Russia (Kurbaniyazov, 2016).

The analysis also detected the presence of quartz, which accounted for 20.28 % of the sample. Quartz is a common mineral in sedimentary rocks. Quartz is mainly presented in the form of glauconite. After all, it is known that nodular phosphorites are sandy phosphorites with a high content of glauconite in the composition (Yanshin, 1979). Also, the mineralogy of these phosphorites is represented by orthoclase, which is a rock-forming mineral from the silicate class. Orthoclase was also present in the sample, accounting for 11.90 % of the total. Orthoclase is a potassium-rich feldspar mineral that is commonly found in igneous and metamorphic rocks.

Finally, dolomite was identified in the sample, accounting for 3.72 % of the total. Dolomite is a calcium-magnesium carbonate mineral that is commonly found in sedimentary rocks.

In the case of nodular phosphorites, DTA was used to determine the thermal behavior of the carbonate and phosphate minerals present in the sample (Figure 3).

Figure 3. Derivatogram of nodular phosphorites

The DTA analysis of the nodular phosphorites sample revealed several important findings. The results showed that the destruction of structural water in the sample was characterized by endothermic effects occurring at temperatures between 70–180 °C. The transformation of quartz occurred at temperatures of 200 °C and above. Decomposition of carbonate-containing compounds began at temperatures above 700 °C (Manly, 1950). The sample lost 6.67 % of its mass in 82 minutes. These findings suggest that the nodular phosphorites sample is composed of complex compounds that undergo significant changes at different temperatures. The information obtained from the DTA analysis is useful in understanding the thermal behavior and stability of the sample, which can be valuable in determining the most effective processing methods.

Mechanism of the selective leaching method of nodular phosphorites

As described above, the phosphate part in nodular phosphorites is presented in the form of fluorocarbonate apatite with the formula $Ca_5(PO_4)_3CO_3F$. The reaction of these phosphorites with acetic acid can be written as follows:

$$4Ca_5(PO_4)_3CO_3F + 37CH_3COOH \rightarrow 18Ca(CH_3COO)_2 + 6CO_2\uparrow + 12H_3PO_4 + 2CaF_2 + 2H_2O \quad (1)$$

The given chemical reaction involves the reaction between four molecules of fluoroapatite carbonate ($Ca_5(PO_4)_3CO_3F$) and 37 molecules of acetic acid (CH_3COOH) in the presence of water. The products formed in the reaction are 18 molecules of calcium acetate ($Ca(CH_3COO)_2$), 6 molecules of carbon dioxide (CO_2), 12 molecules of phosphoric acid (H_3PO_4), 2 molecules of calcium fluoride (CaF_2) and 2 molecules of water (H_2O).

The reaction proceeds as follows (Figure 4):

First, the acetic acid (CH_3COOH) reacts with the fluoroapatite carbonate ($Ca_5(PO_4)_3CO_3F$) to produce calcium acetate ($Ca(CH_3COO)_2$) and carbon dioxide gas (CO_2). This reaction is exothermic in nature and releases energy in the form of heat. Next, the phosphoric acid (H_3PO_4) and calcium fluoride (CaF_2) are produced through the decomposition of fluoroapatite carbonate ($Ca_5(PO_4)_3CO_3F$) in the presence of acetic acid (CH_3COOH).

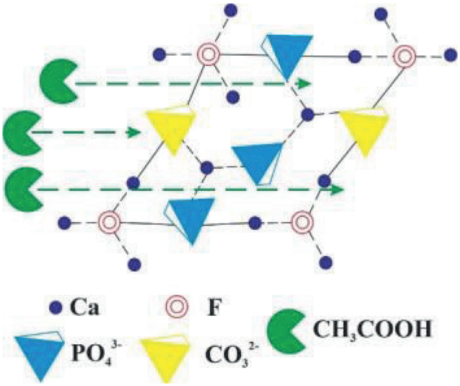

Figure 4. Illustrative reaction (1) mechanism

However, according to the reaction, the phosphate part is carried away into the solution in the form of phosphoric acid. This phenomenon is undesirable for the process under study, since according to studies (Gharabaghi, 2009), the phosphate part should not be affected by organic acid. For example, in the well-known reaction between fluorapatite and acetic acid, from a thermodynamic point of view, the reaction does not occur (Xie, 2019):

$$Ca_5(PO_4)_3F + 10CH_3COOH{=}5Ca(CH_3COO)_2 + 3H_3PO_4 + HF\ (\Delta G^0{=}362.9\ kJ/mol) \quad (2)$$

As can be seen from the above reaction, the Gibbs free energy takes on a positive value, which indicates the impossibility of the reaction. According to the method under study, the carbonate part of phosphorite, represented in our case by dolomite, undergoes selective leaching. Calcium and magnesium carbonates react with acetic acid in the following reactions:

$$CaCO_3{+}2CH_3COOH{=}Ca(CH_3COO)_2{+}H_2O{+}CO_2\uparrow\ (\Delta G^0{=}{-}2.7\ kJ/mol) \quad (3)$$

$$MgCO_3{+}2CH_3COOH{=}Mg(CH_3COO)_2{+}H_2O{+}CO_2\uparrow\ (\Delta G^0{=}{-}31.1\ kJ/mol) \quad (4)$$

From these reactions and calculated values, ΔG indicates that the reaction is thermodynamically favorable or spontaneous.

The silicate part of nodular phosphorites is presented in the form of quartz and orthoclase. Acetic acid is a weak acid and does not have the ability to react with or decompose quartz, which is a chemically inert mineral. Quartz is made up of silicon and oxygen atoms arranged in a crystalline structure, and its bonds are too strong to be broken by weak acids like acetic acid. Therefore, in the presence of acetic acid, quartz remains stable and does not undergo any significant chemical reactions. However, it is reported that acetic acid is able to partially decompose minerals in the form of nepheline (Dorfman, 1959). Nepheline, like orthoclase in our case, is a rock-forming mineral from the feldspar class. Their difference is only in the lower content of silicon. Although there is no information on the dissolution of orthoclase with acetic acid.

Presentation of the results of the selective leaching experiments

Numerous reports have demonstrated the use of various organic acids in leaching phosphate rock (Lazo, 2017). Acetic acid, in particular, has been proven to enhance the dissolution of calcium carbonate (Sengul, 2006). Other organic acids have also been found to have the ability to dissolve carbonate materials in low-grade phosphate rock (Haweel, 2013; Arroug, 2021). As previously stated, it has been observed that acetic acid has the ability to dissolve some minerals, including nepheline, to a certain extent. These research studies suggest that the type of organic acid used is closely linked to the enrichment rate of P_2O_5. There is currently no information available on how organic acids affect the enrichment of nodular phosphorites. Figure 5-6 presents the outcomes of experiments conducted to investigate this topic.

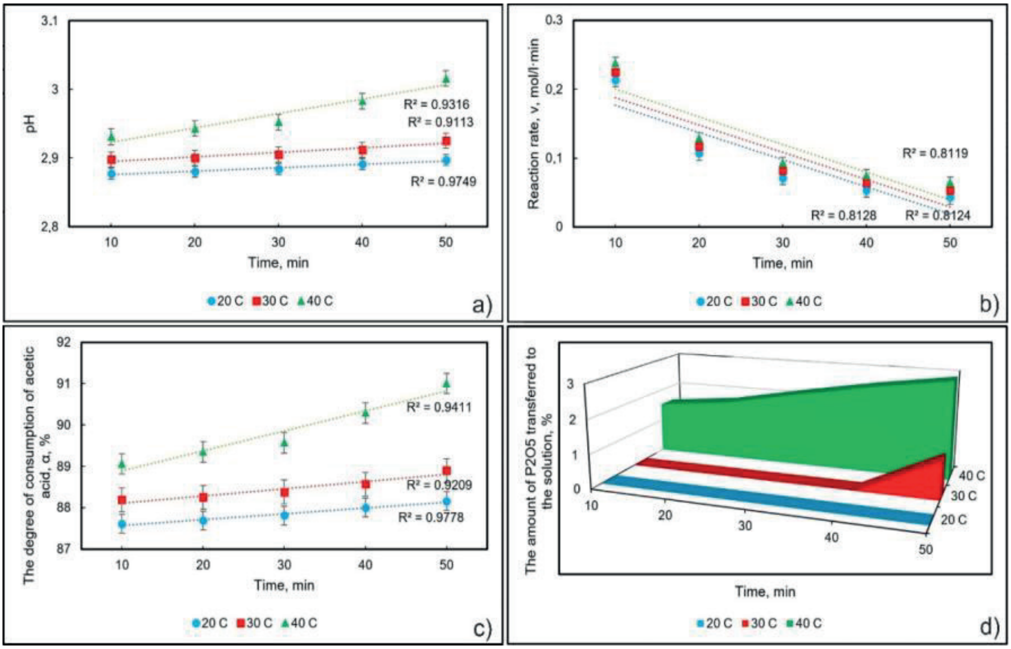

Figure 5. Acetic acid enrichment of nodular phosphorites: a - change in pH over time; b - change in reaction rate over time; c - change in the degree of consumption of acetic acid over time; d - the amount of phosphoric anhydride passed into solution.

The experimental results indicate an increase in pH over time. Equilibrium in the system is observed at 20 and 30 °C, but at 40 °C, a considerable alteration in pH is observed. The reaction rate at all temperatures decreases with increasing time because the concentration of reactants decreases as the reaction progresses, which leads to a decrease in the number of collisions between reactant molecules and a consequent decrease in the rate of reaction. Additionally, the formation of reaction products can also inhibit the reaction by slowing down the rates of intermediate steps or by decreasing the availability of reactants.

The observation that the degree of consumption of acetic acid reaches a maximum value of up to 91 % at 40 °C confirms this statement. However, it also suggests that the reaction between acetic acid and the carbonate part of the phosphorite is not complete, as some acetic acid remains unused.

Selective enrichment refers to the process of separating and extracting the desired component from a mixture while leaving unwanted components behind. In the case of nodular phosphorites, the desired component is the phosphorus-containing mineral, while the unwanted component is the carbonate mineral. If the phosphate part is carried away into the solution at a temperature as low as 30 °C, it means that the selective enrichment is not occurring effectively, and there is a risk of losing the desired component. Additionally, carrying away the phosphate part can lead to an incomplete reaction and lower the overall efficiency of the enrichment process. Therefore, it is highly undesirable for the phosphate part to be carried away into the solution during selective enrichment. The experimental results indicate that a rise in temperature results in the removal of the phosphate component. Some studies (Xie, 2019) suggest that elevating the reaction temperature may enhance the concentration of P_2O_5. However, the dissolution of carbonates does not increase at temperatures beyond 40 °C. This suggests that higher temperatures may cause the evaporation of water and acetic acid. Additionally, the higher temperature may also cause other undesired reactions to occur, such as the breakdown of organic acids or the formation of byproducts, which could interfere with the selective enrichment process.

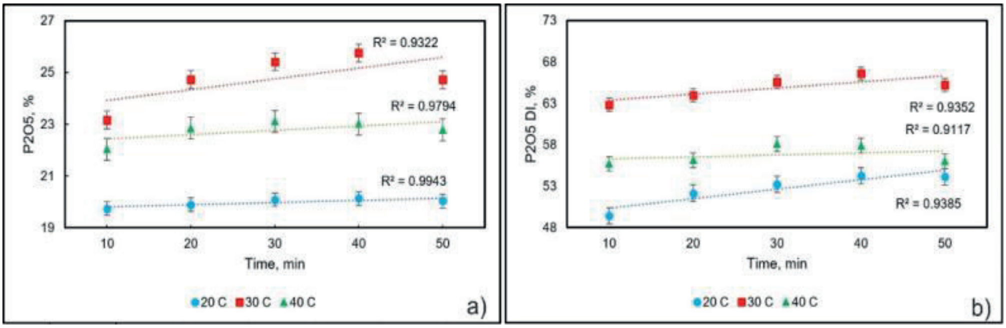

Figure 6. Change in the amount of P_2O_5 (a) and the degree of its increase (b) over time

The enrichment of the main component P_2O_5 occurs actively at temperatures of 20–30 °C, as revealed by the experiment. However, the increase in phosphoric anhydride is only slight upon raising the temperature to 40 °C, and up to 3 % of it is transferred into the solution. This phenomenon is attributed to the selective reaction of acetic acid with open carbonate minerals in the sample particles during the first 30 minutes at 20-30 °C. Upon completion of this reaction, the first exposed carbonate minerals fully react with the acid. Analysis of nodular phosphorite by XRD indicated that the sample contained about 3.72 % dolomite. Gradually increasing the temperature and reaction time leads to an acid attack of the crystal lattice of the phosphate part, and a significant increase in the degree of pH at these values confirms the rapid reaction between acetic acid and carbonate-fluorapatite.

Despite conducting the experiments at 20–30 °C, the utilization of acetic acid only reaches 88–89 %, indicating that 10 % of the acid remains unreacted in the reaction mixture. This could be because the reaction products in the form of acetate salts hinder further reaction. The accumulation of reaction products in the form of acetate salts can create a diffusion barrier around the unreacted phosphate rock particles, preventing further reaction. As a result, the unreacted acid is not able to effectively reach the remaining carbonate minerals in the phosphate rock, leading to a decrease in the degree of consumption of acetic acid. Additionally, the accumulation of reaction products can also shift the equilibrium of the reaction towards the formation of these products, further inhibiting the reaction between acetic acid and the remaining carbonate minerals.

The optimal condition of temperature and time at 30 °C and 30 minutes, respectively, allowed for the highest degree of consumption of acetic acid while minimizing the loss of phosphate material into the solution. As a result, the increase in the content of P_2O_5 was maximized, reaching up to 25.58 %. This indicates that at these conditions, the selective dissolution of the carbonate minerals was more efficient, leading to a higher concentration of the valuable phosphate component in the solid product.

Chemical and mineralogical composition of enriched nodular phosphorites

Table 3 and Figure 7 display the chemical and mineralogical composition of the enriched nodular phosphorites that were analyzed in this study.

Table 3. Chemical composition of enriched nodular phosphorites

Compounds	P_2O_5	CaO	MgO	K_2O	Al_2O_3	Fe_2O_3	SiO_2
Composition, %	25.58±0.47	26.94±0.84	-	0.04±0.02	1.12±0.14	1.39±0.29	28.24±1.05

The chemical composition of enriched nodular phosphorites is characterized by an increase in the percentage of P_2O_5 and a decrease in CaO compared to the initial raw material. The percentage of P_2O_5 increased from 18.12±0.91 % in the initial raw material to 25.58±0.47 % in the enriched nodular phosphorites. The percentage of CaO decreased from 31.19±1.13 % in the initial raw material to 26.94±0.84 % in the enriched nodular phosphorites. The MgO content was reduced to 0%, while the K_2O content remained minimal at 0.04±0.02 %. The percentage of Al_2O_3 increased slightly from 2.04±0.31 % in the initial raw material to 1.12±0.14 % in the enriched nodular phosphorites. The reduction in the quantity of potassium and aluminum-containing compounds indicates that acetic acid has the capability to selectively dissolve minerals other than carbonates.

The percentage of Fe_2O_3 increased from 0.97±0.37 % in the initial raw material to 1.39±0.29 % in the enriched nodular phosphorites. The SiO_2 content increased slightly from 23.10±1.24 % in the initial raw material to 28.24±1.05 % in the enriched nodular phosphorites. Overall, the enrichment process resulted in a higher concentration of P_2O_5 and a lower concentration of CaO, which is desirable for the production of phosphorus-containing products.

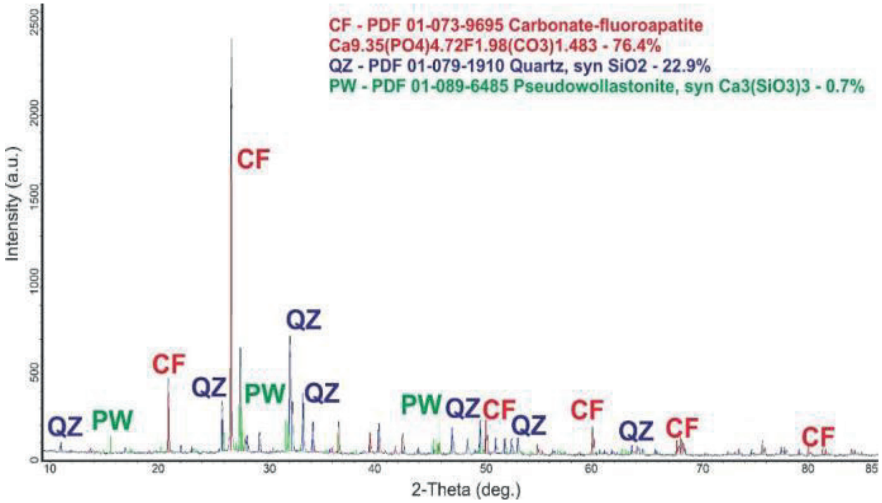

Figure 7. X-ray diffraction peaks of enriched nodular phosphorites

The XRD analysis of enriched nodular phosphorites showed a significant change in the mineral composition compared to the initial raw material. The main mineral phase is carbonate-fluoroapatite, which is present in a higher percentage of 76.4 %. Quartz is the second most abundant mineral phase, with a lower percentage of 22.9 %. Pseudowollastonite is present in a very small percentage of only 0.7 %. These results indicate that the selective leaching process effectively removed some of the non-phosphate mineral phases and enriched the phosphate content in the sample.

Phosphoric anhydride, also known as P_2O_5, is the main component needed for the production of phosphoric acid, which is a crucial ingredient in the production of mineral fertilizers. The higher the amount of P_2O_5 in the phosphate raw material, the higher the yield of phosphoric acid and, consequently, mineral fertilizers. Therefore, to ensure high-quality and cost-effective production of mineral fertilizers, the minimum amount of P_2O_5 in phosphate raw materials is set at 25 % (Samreen, 2019). This requirement has been adopted in world practice to maintain consistency and efficiency in the production of mineral fertilizers. When choosing a supply of phosphate rock for the production of wet-process acid, various factors are taken into consideration. One of these factors is the CaO/P_2O_5 weight ratio, which determines the amount of sulfuric acid needed to acidulate the phosphate rock in the manufacture of wet-process acid. The CaO/P_2O_5 weight ratio is a widely used measure of the quality of phosphate rock, with a ratio of 1.32 for pure apatite, while commercially available rock may have a CaO/P_2O_5 ratio of up to 1.6 (Gilmour, 2013; Ryszko, 2023). In our case, this ratio is 1.05, which may require the least amount of sulfuric acid to process. Additional research is required to validate this assertion.

Conclusions

The study aimed to evaluate the effectiveness of selective leaching using acetic acid to increase the content of P_2O_5 in nodular phosphorites. The results showed that the increase in P_2O_5 content was highest at 30°C and 30 minutes of reaction time, with a maximum increase of up to 25.52 %. Chemical and XRD analysis revealed that the main component in the enriched sample was carbonate-fluoroapatite, with a decrease in the amount of potassium and aluminum-containing compounds. The use of selective leaching has economic and environmental benefits, as it reduces the need for expensive and environmentally damaging mining and processing methods. Overall, the findings of this study suggest that selective leaching using acetic acid can be an effective and sustainable method for increasing the P_2O_5 content of nodular phosphorites.

Based on the results and limitations of this study, there are several potential directions for future research on nodular phosphorite concentration through selective leaching.

Firstly, more research could be conducted on the chemical and mineralogical changes that occur during the selective leaching process, with a focus on understanding the mechanisms behind the selectivity of the process and how it can be improved.

Secondly, there is a need for further investigation into the economic and environmental aspects of the selective leaching method, including the feasibility of scaling up the process for industrial applications and the potential environmental impacts of the process. This could provide valuable insights for the development of sustainable and cost-effective methods for the production of phosphorus-containing products.

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