Physicochemical Laws and Optimal Conditions of The Process of Transferring Magnesium Ions into The Solution by Decomposing Dolomite Mineral with Nitric Acid

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Abstract

In this work, the physicochemical regularities of the heterogeneous decomposition process of dolomite from the Navbakhor deposit in a nitric acid environment were studied. In the study, complexometric titration and X-ray diffractometry methods were used, and the dependence of the reaction medium on concentration and temperature factors was analyzed. It was established that in the optimal process mode (120% stoichiometry, 35% HNO3), active dissolution of carbonate minerals is observed, and in the solid phase, mainly inert quartz and wollastonite phases are concentrated. X-ray diffraction analysis of the solid residue formed after acid decomposition showed that the main part of the carbonate minerals was dissolved during the reaction, and it was found that mainly quartz (73.6%), wollastonite (22.4%), and magnesium-aluminosilicate phases (4.0%) remained in the solid phase.

The obtained results have scientific and practical significance for improving the technology for obtaining magnesium-containing solutions and producing magnesium compounds based on the acid treatment of local dolomite raw materials.

Keywords: Dolomite, nitric acid, acid decomposition, magnesium extraction, stoichiometric norm, acid concentration.

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1. Introduction

The rapidly increasing demand for magnesium and its derivatives in the modern materials science and metallurgical industry necessitates the efficient use of the local mineral resource base. Dolomite reserves also exist in the territory of Uzbekistan, the effective use of which is of great importance from the point of view of the integrated processing of local mineral resources. Among the existing methods of processing magnesium-containing carbonate raw materials (thermal, hydrometallurgical), the process of acid destruction is one of the priority directions in terms of energy efficiency and product productivity. Chemical transformation of the mineral dolomite in a medium of mineral acids allows obtaining an enriched semi-finished product for the production of functional materials, forming active magnesium and calcium nitrates in the solution. Complex development of local dolomite deposits will not only ensure the raw material independence of the chemical industry, but also lay the foundation for the creation of highly efficient hydrometallurgical systems for processing mineral resources. The method of acid decomposition is relatively simple from a technological point of view and allows the transfer of magnesium and calcium ions from the mineral into the solution. In this process, the mineral dolomite decomposes under the influence of mineral acids, and magnesium and calcium nitrates or other salts are formed in the solution. Such solutions subsequently serve as a valuable source of raw materials for the extraction of magnesium compounds[1-2].

The issue of complex processing of calcium and magnesium-containing carbonate minerals, obtaining inorganic products with high added value, and developing waste-free technologies has been at the center of attention of the world scientific community in recent decades.

In particular, numerous studies have been conducted on the selective extraction of Mg and Ca compounds through chemical and thermochemical processing of the dolomite mineral, and the synthesis of sorbents, coagulants, fertilizers, and functional materials based on them [3-4].

In a number of studies, the simultaneous transfer of Ca and Mg ions into the solution during the direct decomposition of dolomite in acids has been noted. This situation complicates the subsequent stages of separation. Therefore, scientific work is mainly focused on optimizing process parameters[5-6].

Scientific research on acid decomposition is mainly devoted to the use of HCl, H2SO4, and HNO3 acids. Hydrochloric acid is characterized by its rapid dissolution of dolomite, however, subsequent separation of CaCl2 and MgCl2 solutions requires additional reagents.

When decomposing in sulfuric acid, a precipitate of CaSO4 is formed, which allows Mg2+ to remain in solution, however, the formation of gypsum complicates the filtration process. Nitric acid has a number of technological advantages in the decomposition of dolomite. According to scientific sources, HNO3 has a high degree of dissociation and is distinguished by its ability to completely dissolve carbonate minerals. The resulting Ca(NO3)2 and Mg(NO3)2 have high solubility and are stable in the solution phase. This facilitates the separation of Mg by selective precipitation or recrystallization. In a number of studies, the kinetics of dolomite decomposition in nitric acid were studied. The authors note that with an increase in acid concentration, the reaction rate increases, the intensity of CO2 release increases, and the complete decomposition of carbonate phases occurs. However, it has been noted that in high-concentration acids, excess acid remains in the reaction medium, and the crystallization properties of nitrate salts can change[7-10].

Analysis of the literature shows that the issues of decomposition of dolomite in nitric acid at high concentrations, the dynamics of the solubility of Ca and Mg ions at various stoichiometric rates, the depth of decarbonization, and the effectiveness of selective separation have not been sufficiently studied.

Based on the foregoing, this research work is aimed at the decomposition of dolomite in nitric acid at various concentrations and stoichiometric norms, determining the general and water-soluble forms of Ca and Mg oxides, assessing the decarbonization process, and developing the scientific and technological foundations for the selective separation of magnesium.

As an object of research, local dolomite raw materials from the Navbakhor deposit of the Navoi region were used. The samples were first dried in laboratory conditions, ground in a ball mill, and sieved to a fraction of less than 0.25 mm. The chemical composition of dolomite was determined by classical chemical analysis methods, and the content of CaO-28.67%, MgO-18.42%, and CO2-42.57% was established, as well as SiO2-3.26%, Al2O3-1.12%, Fe2O3-1.24%, SO3-1.37% and H2O-1.90% as additional components. In the experiments, nitric acid (HNO3) of analytical purity, distilled water, and EDTA and corresponding indicator reagents were used for analysis. To study the process of acid decomposition of dolomite, nitric acid solutions with concentrations of 30, 35, 40, 45, 50, and 55% were prepared by diluting concentrated acid, and their concentration was controlled by the titrimetric method.

The process of nitric acid destruction of dolomite was carried out in glass thermostatic reactors equipped with a mechanical stirrer moving at a constant speed. During the experiments, the stoichiometric norm of the acid was changed in a wide range (from 50% to 140%, with a step of 10%), and its influence on the solubility of magnesium and calcium oxides was studied. The stability of the reaction medium was ensured for 30-40 minutes under isothermal conditions of 25-30°C. The concentration of cations in the obtained solutions was determined by the method of complexometric titration in accordance with GOST standards. In order to ensure the reliability of the results, all experiments were repeated in parallel, and the obtained values were processed using mathematical and statistical methods.

2. Results

X-ray structural analysis of the dolomite sample from the Navbakhor deposit in the Navoi region (Fig. 1) confirmed the high degree of crystallization of the object of research. The most intense reflections observed on the diffractogram (2θ ≈ 31.31°, 35.74°, 37.74°, 41.51°, and 45.32°) belong to the crystal lattice of the dolomite mineral of trigonal synonymy, the phase identification of which is fully consistent with the international ICDD database. The sharpness and intensity of these peaks indicate the stability of the dolomite phase in the sample and its stable structural state.

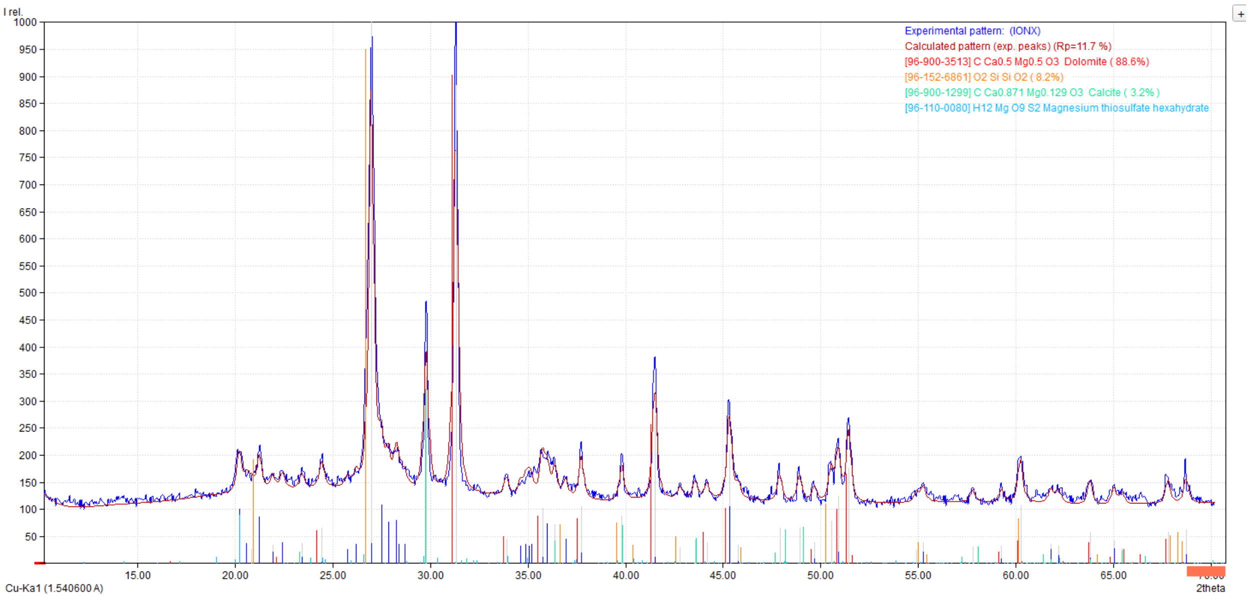

Figure 1. X-ray diffraction analysis of dolomite.

According to the results of phase analysis, the mineral dolomite in the sample is 88.6%, the chemical formula of this mineral is CaMg(CO3)2, which belongs to the trigonal crystal system. At the same time, the sample contained quartz (SiO2) in an amount of 8.2%, the main diffraction peaks of which were observed in the 2θ ≈ 26.99°, 39.82°, and 50.53° regions. Calcite (CaCO3) was also identified as an additional mineral phase, the share of which was 3.2%. Peaks belonging to the calcite phase were recorded in the regions of 2θ ≈ 23.43°, 29.79°, 43.57°, and 47.90°. The study of the phase composition of the dolomite of the Navbakhor deposit in the Navoi region showed that the mineralogical skeleton of the sample consists mainly of dolomite, accompanied by inclusions of quartz and calcite. This circumstance reflects the complex physicochemical nature of local raw materials and indicates the need to take into account the presence of calcium and silicon compounds at the stage of acid destruction.

The obtained results are important for assessing the kinetics of magnesium release into solution and optimizing the technology for obtaining high-purity magnesium salts.

According to the data of Fig. 2, in the case of an acid norm of 50-70%, the degree of transition of magnesium oxide to solution is relatively low, for example, in the presence of HNO3 at a concentration of 30%, the degree of transition of MgO to solution at a norm of 50 was 42.94%, and at a concentration of 55%, this indicator decreased to 31.88%. With an increase in the acid norm to 80-100%, the degree of transition of magnesium oxide to solution increases sharply, reaching 73.60-89.12% at a concentration of 30%, respectively, and in solutions with a concentration of 40-45% - around 67.94-81.95%.

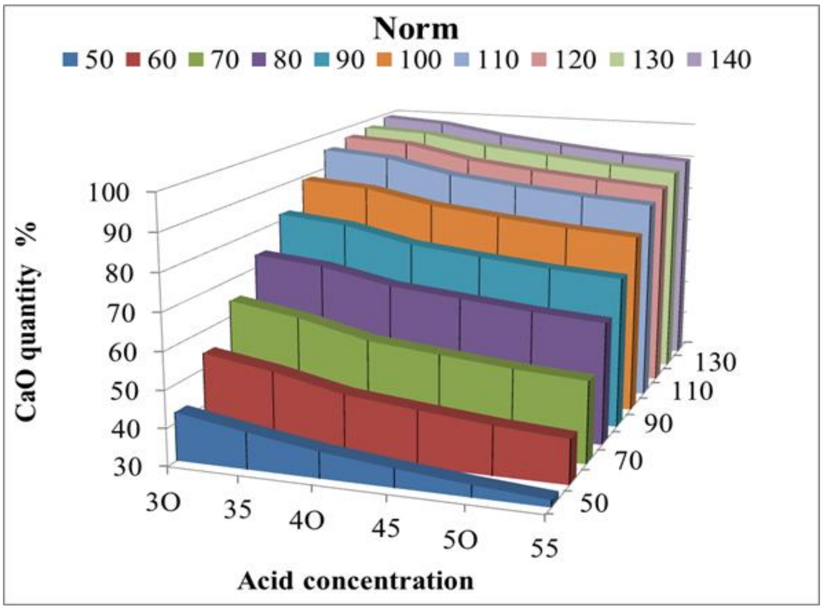

Figure 2. Dependence of magnesium oxide dissolution on acid concentration and its stoichiometric norm (%).

When the acid norm was brought to 110-120%, the degree of magnesium transfer to the solution increased further, and at a concentration of 30% HNO3 it was 95.50-97.07%, at a concentration of 35% 94.00-95.89%, at a concentration of 40% 90.08-92.08%. When the norm was increased to 130-140%, the degree of MgO transfer to the solution reached 97.84-99.11% at a concentration of 30%, which indicates almost complete decomposition of dolomite. At the same time, with an increase in the acid concentration to 45-55%, a slight decrease in the degree of magnesium dissolution was observed, for example, at a norm of 140, at a concentration of 30%, it was 99.11%, and at a concentration of 55%, it was 89.96%. This is explained by the formation of saturated solutions of calcium nitrate and magnesium nitrate salts in the reaction medium in high-concentration acid, as well as the slowing of diffusion processes due to an increase in the ionic stress of the reaction medium.

With the change in the amount of calcium oxide in the reaction mixture formed during the decomposition of dolomite in nitric acid, depending on the acid concentration and its stoichiometric consumption rate (Fig. 3), an increase in the amount of CaO was observed at all stoichiometric norms with an increase in the acid concentration from 30% to 55%. For example, at a 50% stoichiometric norm, the CaO content was 10.01% at a 30% acid concentration, and at a 55% concentration, it increased to 14.45%, i.e., an increase of almost 1.44 times was noted. This is due to the fact that the amount of added acid decreases with increasing acid concentration. At the same time, as the acid consumption increases to the stoichiometric norm, the proportion of CaO in the reaction mixture decreases. With an increase in the concentration of 30% acid from the norm to 140%, CaO decreased from 10.01% to 4.55%. This situation leads to an increase in the amount of added acid with an increase in the norm and a decrease in dolomite components in the mixture.

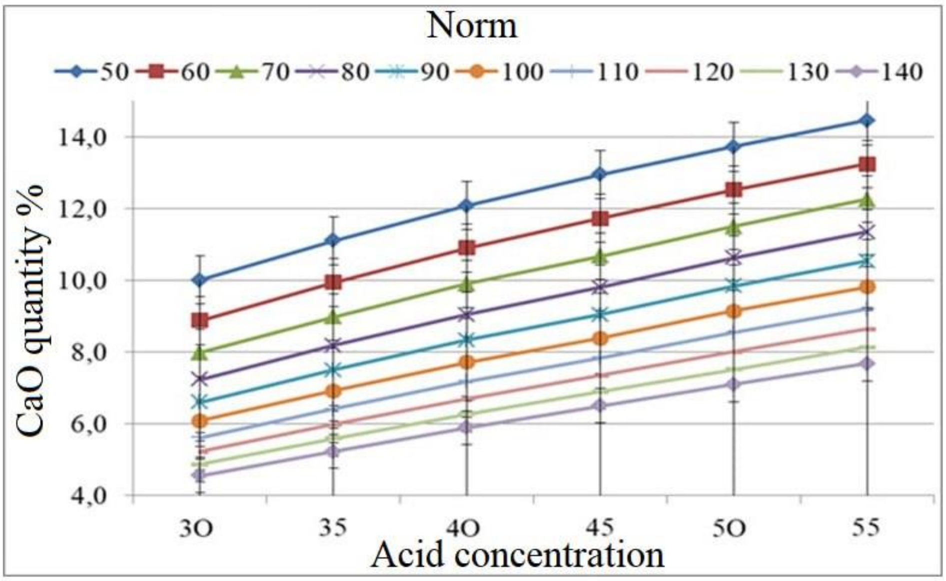

Figure 3. Dependence of the change in the total amount of calcium oxide in the reaction mixture formed during the decomposition of dolomite in nitric acid on the acid concentration and its stoichiometric norm.

Regular changes in the total amount of magnesium oxide (MgO) depending on the acid concentration and its stoichiometric consumption rate, with an increase in the acid concentration from 30% to 55%, an increase in the amount of MgO is observed at all stoichiometric norms. For example, at a 50% stoichiometric norm, the MgO content in 30% acid was 6.43%, and at a 55% concentration, it increased to 9.28% (Fig. 4). At the same time, it was established that the proportion of MgO in the reaction mixture decreases with increasing acid consumption relative to the stoichiometric norm. With an increase in the norm from 50% to 140% in acid with a concentration of 30%, MgO decreased from 6.43% to 2.93%. This is explained by the fact that with an increase in acid consumption, the proportion of acid in the reaction mass increases, and the relative content of mineral components of dolomite, including magnesium compounds, decreases.

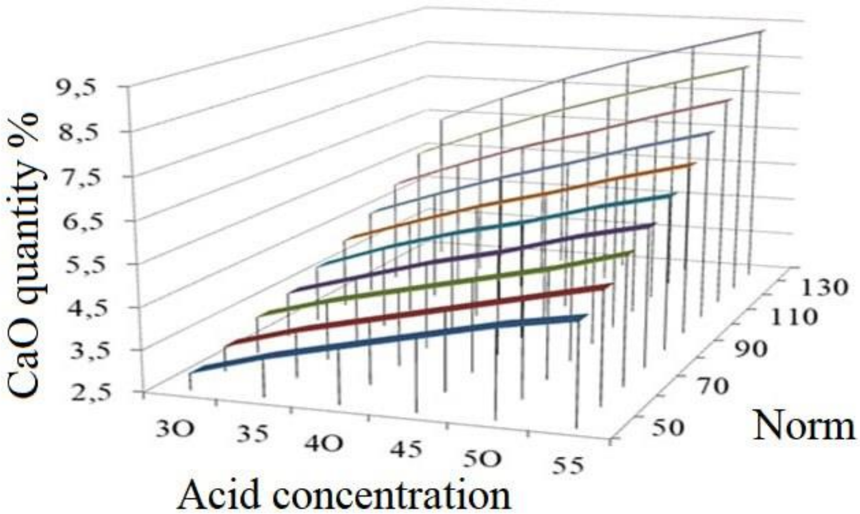

Figure 4. Dependence of the change in the total amount of magnesium oxide in the reaction mixture formed during the decomposition of dolomite in nitric acid on the acid concentration and its stoichiometric norm.

X-ray diffraction analysis of the solid residue formed after the nitric acid decomposition of dolomite showed significant changes in its mineralogical composition. According to the results of phase analysis, quartz (SiO2) prevailed as the main mineral phase in the solid residue, and its share was 73.6%. Also, a calcium silicate mineral - wollastonite (CaSiO3) was detected in the amount of 22.4%, and a small amount of magnesium-aluminosilicate phase of the type (MgAl2Si3O10) was found in the amount of 4.0%. Analysis of the experimental results confirms the high solubility of carbonate phases and the intensification of magnesium and calcium cations in the reaction medium during the nitric acid destruction of dolomite. At the same time, the concentration of acid-resistant siliceous minerals and complex silicate compounds is observed in the solid phase of the system. This indicates the selectivity of the technological process and determines the high prospects for using the resulting enriched silicon residue as a secondary raw material in the production of silicate materials or fillers for the construction industry.

3. Conclusion

The fundamental physicochemical regularities of the process of nitric acid destruction of local dolomite raw materials from the Navbakhor deposit of the Navoi region were comprehensively studied. Based on experimental studies, the mechanisms of interfacial interaction in heterogeneous systems were analyzed, and an optimal technological regulation for the extraction of magnesium cations into the liquid phase was developed. It has been established that the acid concentration in the reaction medium and its norm relative to the stoichiometer are the main factors determining the kinetics of decomposition of carbonate minerals, which allows controlling the yield of magnesium oxide in solution.

Analysis of the obtained data showed that with an increase in the acid norm to 110-120%, the degree of transition of magnesium oxide to solution reaches 90-97%, ensuring the highest efficiency of the process. At an acid concentration of 30-35%, the degree of decarbonization of dolomite reaches 97-99%, which indicates almost complete decomposition of the mineral. The results of X-ray diffraction analysis showed that during nitric acid processing of dolomite, the main part of the carbonate minerals passes into the solution, and the solid phase is mainly composed of quartz (73.6%), wollastonite (22.4%), and magnesium-aluminosilicate phases (4.0%). The results of experimental studies make it possible to form an optimal technological scheme for obtaining magnesium-containing finished products based on Navbakhor dolomite. The developed acid decomposition paradigm fully meets the modern requirements of chemical and environmental engineering, ensuring the economic efficiency of industrial processing of local raw materials. These scientific developments are of great importance in expanding the production capacity of magnesium compounds and increasing the range of import-substituting chemical products.

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