

DETERMINATION OF THE COMPONENT COMPOSITION OF A SERIES OF CALCIUM-CONTAINING DEPOSITS AND TECHNOLOGICAL RECOMMENDATIONS FOR THEIR UTILIZATION

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Abstract. A comprehensive analysis of samples of calcium-containing mineral raw materials from deposits in Russia (Bryansk), Kazakhstan (Beineu, Shetpe, Zhanakorgan), and Uzbekistan (Fergana) was carried out to assess their suitability as precursors of calcium oxide used in technologies for the remediation of oil-contaminated soils. The study employed methods of X-ray phase analysis (XRD), elemental analysis, and infrared spectroscopy (FTIR). By the XRD method, the phase composition of the samples was established and their semi-quantitative evaluation was performed. Based on the obtained data, the samples were classified into three groups: 1) calcium oxide-hydroxide (Bryansk, Beineu, Fergana), characterized by a high content of CaO (76.1-92.5%) and Ca(OH)₂ (7.5–19.0%); 2) calcium hydroxide (Zhanakorgan), representing almost pure Ca(OH)₂ (99.6%); 3) carbonate (Shetpe), consisting of calcite CaCO₃ (>98%). FTIR results confirmed the presence of the identified phases. Elemental analysis showed high purity of most samples in terms of calcium (>52% CaO in normalized form). The sample from Bryansk contains an increased amount of silica (SiO₂ ≈ 4%). Technological recommendations for the use of the raw materials are proposed. The Beineu-1 and Beineu-2 samples, containing 92.5% and 81.0% CaO, respectively, were identified as the most promising raw materials for the energy-efficient production of active CaO.

Keywords: calcium-containing raw materials, X-ray diffraction, infrared spectroscopy, elemental analysis, classification, oil-contaminated soils

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Introduction

This work is devoted to the analysis of raw material sources of calcium-containing materials used for the production of calcium oxide, which is applied in technologies for the remediation of oil-contaminated soils. Calcium oxide (CaO, quicklime) is a highly reactive reagent widely used in metallurgy, construction, the chemical industry, and environmental applications [1, 2]. In particular, in remediation technologies for oil-contaminated soils, CaO is used for the stabilization of hydrocarbons, adjustment of the pH of the medium, and immobilization of heavy metals [3, 4]. The efficiency of these processes directly depends on the reactivity of CaO, which is determined by its purity, particle size (dispersion), and structure. These characteristics, in turn, are defined by the composition of the initial mineral raw materials and the conditions of their processing. Despite the abundance of deposits of carbonate and other calcium-containing rocks, their composition can vary significantly depending on geological conditions. The presence of associated phases (calcium hydroxide, dolomite, silicate and clay minerals) and chemical impurities (compounds of Mg, Fe, Si, and Al) significantly affects both the technology of calcium oxide production and its final properties [5–9]. Therefore, systematic research and comparative evaluation of the component composition of raw materials from various sources represent a relevant scientific and practical task.

Samples from Bryansk (Russian Federation), as well as from deposits in Kazakhstan (Beineu, Shetpe, Zhanakorgan) and Uzbekistan (Fergana), were investigated. The main research method is semi-quantitative X-ray phase analysis (XRD), supplemented by the results of elemental analysis and IR spectroscopy.

The aim of the work is to determine the component composition of the studied samples and to classify them based on the obtained data for further use in industrial purposes:

1. Bryansk, Russian Federation (sample 1)
2. Beineu, quarry 1 (sample 2)
3. Beineu, quarry 2 (sample 3)
4. Shetpe, quarry 1 (sample 4)
5. Shetpe, quarry 2 (sample 5)
6. Zhanakorgan (sample 6)
7. Fergana, Uzbekistan (sample 7)

Experimental part

Sampling and sample preparation were carried out in accordance with methods [10, 11].

1. X-ray phase analysis was carried out according to the standard method [12] using a powder diffractometer DW-XRD-27mini (Cu $\text{K}\alpha$ radiation, voltage — 40 kV, current — 40 mA, graphite monochromator). Scanning was performed in θ – 2θ mode in the angular range of 2θ from 5° to 120° .

2. The analysis of the content of major elements (Ca, Mg, Fe, Al, Si, K, Na, Ba, Mn in oxide forms) in solid samples was carried out using an inductively

coupled plasma atomic emission spectrometer (ICP-OES) Thermo Scientific iCAP PRO XP Duo (Germany), in accordance with the measurement methodology for solid objects.

3. IR spectra were recorded on a Fourier spectrometer Nicolet 5700 in the range of 400–4000 cm^{-1} . The spectra were recorded using OMNIC software. Samples were prepared in the form of pellets with KBr. Before analysis, the samples were dried at 110°C to remove adsorbed moisture.

Results and discussion

Figures 1–7 show the X-ray diffraction patterns of the studied samples. Table 1 presents the data on their component composition.

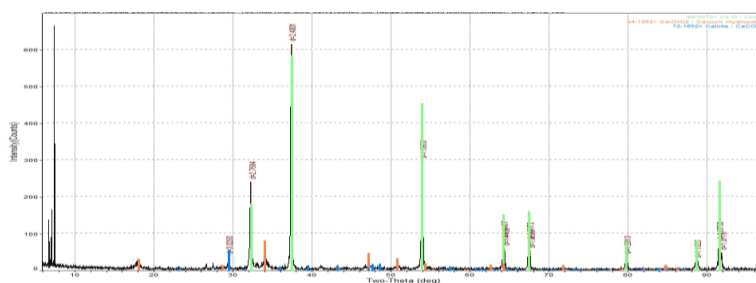


Figure 1 — X-ray diffraction pattern of sample 1

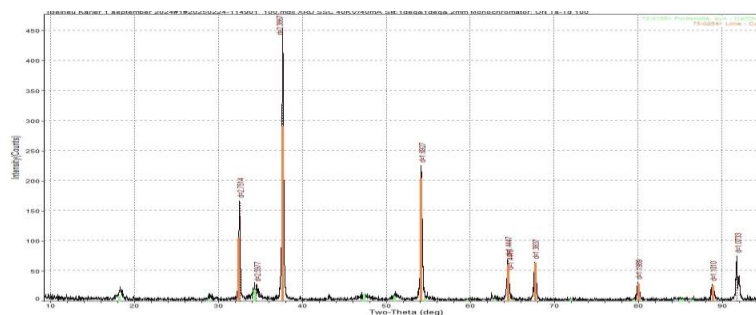


Figure 2 — X-ray diffraction pattern of sample 2

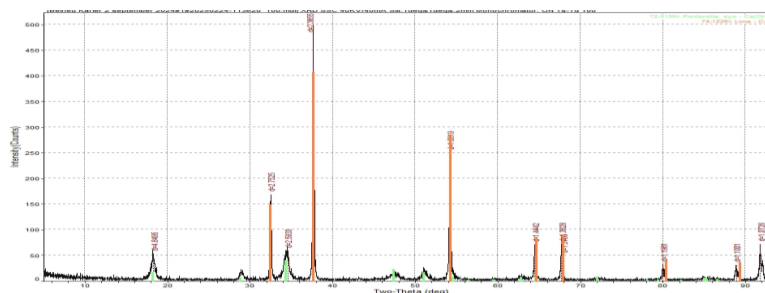


Figure 3 — X-ray diffraction pattern of sample 3

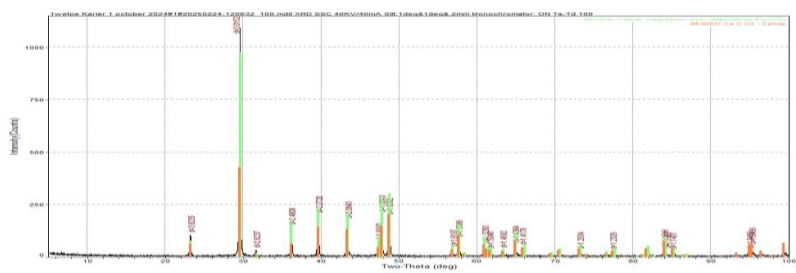


Figure 4 — X-ray diffraction pattern of sample 4

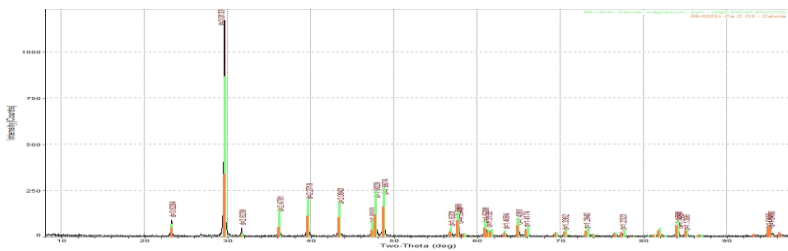


Figure 5 — X-ray diffraction pattern of sample 5

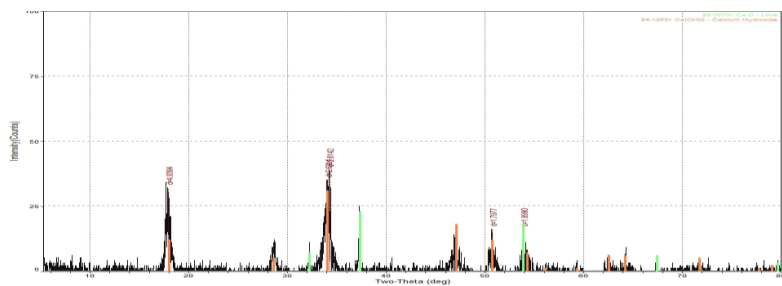


Figure 6 — X-ray diffraction pattern of sample 6

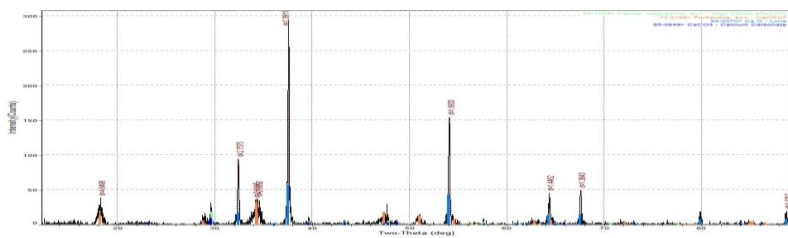


Figure 7 — X-ray diffraction pattern of sample 7

Table 1 — Data of semi-quantitative analysis by X-ray phase analysis (XRD) of the studied samples, %

Sample ID	CaO	Ca(OH) ₂	Magnesian calcite*	CaCO ₃
1. Bryansk, RF	78.8	16.9	-	4.3
2. Beineu, quarry 1	92.5	7.5	-	-
3. Beineu, quarry 2	81.0	19.0	-	-
4. Shetpe, quarry 1	-	-	99.9	0.1
5. Shetpe, quarry 2	-	-	98.7	1.3
6. Zhanakorgan	0.4	99.6	-	-
7. Fergana, Uzbekistan	76.1	19.1	1.5	3.3

* Mg_{0,03}Ca_{0,97}(CO₃)

Based on the analysis of the component composition of calcium-containing samples from various deposits in Russia, Kazakhstan, and Uzbekistan, three main groups can be identified:

1. Calcium oxide–hydroxide group. This group includes samples containing calcium oxide and calcium hydroxide (samples 1–3 and 7). It should be noted that samples 1 and 7 also contain carbonates and are generally similar in composition. In samples 2 and 3 (Beineu, quarries 1 and 2, respectively), only calcium oxide and calcium hydroxide are observed. At the same time, sample 2 shows the highest relative content of calcium oxide.

2. Calcium hydroxide group. The sample from Zhanakorgan is characterized by an exclusively high content of calcium hydroxide (Ca(OH)₂ — 99.6%).

3. Calcite group. The samples from Shetpe (quarries 1 and 2) consist entirely of calcite.

Table 2 presents the data on the elemental composition of the selected major elements (normalized compositions). Normalized compositions allow you to compare the relative content of elements to each other in different samples. The content of the remaining elements is below the limits of their detection.

Table 2 — Elemental analysis of selected elements in oxide form, %

	Sample ID	Al ₂ O ₃	BaO	CaO	Fe ₂ O ₃	K ₂ O	MgO	MnO	Na ₂ O	SO ₄ ²⁻	SiO ₂
1	Bryansk, RF	1.2100	0.0071	70.8	1.22	0.676	0.207	0.0457	0.496	0.481	4.04
2	Beineu, quarry 1	<0.014	0.0017	77.2	0.124	0.585	0.087	0.00230	0.247	1.43	0.0863
3	Beineu, quarry 2	<0.014	0.0039	80.3	0.126	0.997	0.362	0.00322	0.430	1.61	<0.7
4	Shetpe, quarry 1	<0.014	0.0009	66.0	0.260	0.973	0.0263	0.0255	0.468	1.13	0.245
5	Shetpe, quarry 2	<0.014	0.0009	52.4	0.204	0.592	0.0425	0.0287	0.291	0.500	0.222
6	Zhanakorgan	0.36	0.007	66.4	0.12	0.035	0.65	0.013	<0.02	<0.4	2.2
7	Fergana, Uzbekistan	<0.014	0.0012	67.2	0.0403	0.650	0.294	0.00354	0.251	2.73	<0.5

Figure 8 shows the IR spectra of the studied samples. Table 3 presents the positions of the absorption bands in their IR spectra.

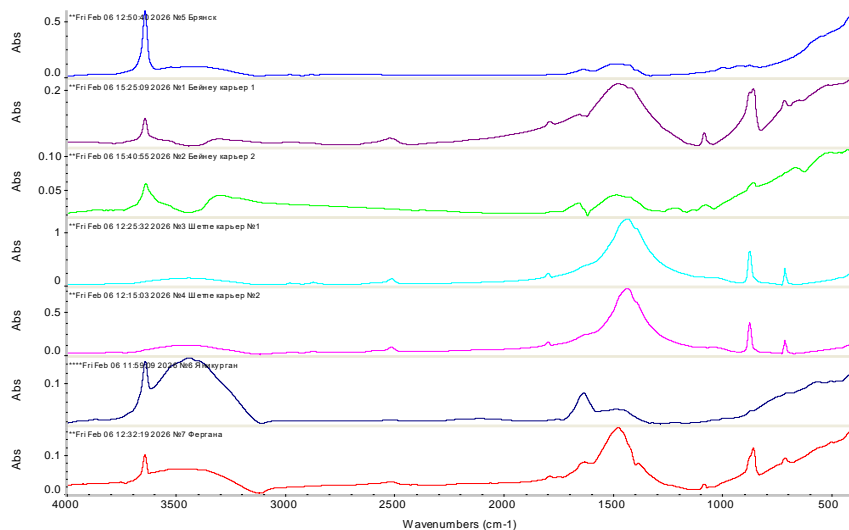


Figure 8 - IR spectra of the studied samples

Table 3 — Absorption frequencies of the studied samples

Sample ID						
Bryansk, RF	Beineu, quarry 1	Beineu, quarry 2	Shetpe, quarry 1	Shetpe, quarry 2	Zhanakorgan	Fergana, Uzbekistan
Wavenumbers, cm ⁻¹						
3642.7	3643.1	3639.3	-	-	3642.0	3643.4
3439.1	3303.4	3299.8	-	3448.5	3441.0	-
-	-	-	2981.2	2971.0	-	-
-	-	-	2873.0	2873.3	-	-
-	2521.5	-	2514.3	2514.5	-	-
-	1789.2	-	1798.0	1798.1	-	1786.2
1630.7	1652.1	1654.0	-	-	1633.9	1624.7
1480.0	1475.1	1480.3	-	-	1491.7	1476.0
-	-	-	1434.4	1434.0	-	-
-	-	-	-	-	1377.3	1380.9
-	-	1214.0	-	-	-	-
-	-	1137.0	-	-	-	1076.9
997.7	-	-	-	-	-	-
918.5	-	-	-	-	-	-
874.5	858.3	858.0	874.6	875.3	-	858.8
-	716.0	-	712.6	712.8	-	719.0
-	-	665.3	-	-	-	-
-	-	429.7	-	-	-	-

In samples 1–3 and 6–7, narrow and intense absorption bands in the 3640–3650 cm⁻¹ region are observed, corresponding to the stretching vibrations of

hydroxyl groups. These bands indicate the presence of free or weakly bonded OH groups, characteristic of pure calcium hydroxide ($\text{Ca}(\text{OH})_2$), and suggest the absence of hydrogen bonding. Deformation vibrations of OH groups are also present in the same samples (around 1640 cm^{-1}).

The absorption bands observed in the $3000\text{--}3450\text{ cm}^{-1}$ region are attributed to O–H bonds associated with groups participating in hydrogen bonding. For the Shetpe quarry samples (4 and 5), absorption bands in the $2870\text{--}2980\text{ cm}^{-1}$ region are observed, which are characteristic of hydrocarbons, i.e., indicating the presence of organic impurities. In addition, the spectra of the Shetpe samples (4 and 5) are dominated by intense carbonate-ion bands: the asymmetric stretching vibration ν_3 in the $1420\text{--}1460\text{ cm}^{-1}$ region, as well as deformation out-of-plane ν_2 ($\sim 870\text{ cm}^{-1}$) and in-plane ν_4 ($\sim 710\text{ cm}^{-1}$) vibrations.

The IR spectroscopy data are actually fully consistent with the conclusions of XRD regarding the component composition:

- Bryansk (1): The IR spectrum shows the presence of OH groups (moderate), CO_3^{2-} (weak), and water. XRD confirms a mixed composition with a predominance of CaO.

- Beineu-1 (2) and Beineu-2 (3): The IR spectra exhibit strong OH group bands, while CO_3^{2-} bands are weak or absent.

- Shetpe-1 (4) and Shetpe-2 (5): The IR spectra are characterized by absorption bands of carbonate groups. XRD confirms an almost pure calcite composition.

- Zhanakorgan (6): The IR spectrum shows intense OH group bands characteristic of calcium hydroxide, with no CO_3^{2-} group bands detected.

- Fergana (7): The IR spectrum, similar to that of Bryansk, shows the presence of OH groups (moderate), carbonate groups (moderate), and water, confirming the XRD data indicating a mixed composition containing CaO, $\text{Ca}(\text{OH})_2$, and calcite.

Technological aspects (recommendations) of raw material utilization

The performed analysis made it possible to classify the investigated calcium-containing deposits according to their component composition and to determine their suitability for the production of calcium oxide.

1. Raw materials of the Calcium oxide–hydroxide group are the most preferable for obtaining highly active CaO. Samples Beineu-1 and Beineu-2 are particularly notable, requiring minimal energy input for activation (dehydroxylation at $\sim 500\text{--}600\text{ }^\circ\text{C}$). Samples Bryansk and Fergana are also promising; however, the presence of impurities (SiO_2 , Fe_2O_3 , Mg) requires control of their influence on the process and the properties of the final product.

2. Raw materials of the calcium hydroxide group (Zhanakorgan) are easily converted to CaO under low-temperature calcination. The key risk is a high tendency toward recarbonation during storage, which necessitates airtight packaging.

3. Raw materials of the carbonate group (Shetpe) represent a classical but the most energy-intensive feedstock for lime production, requiring high-temperature decarbonation (850–1000 °C).

Overall, the results of the study provide valuable information for selecting optimal raw materials and developing technological regimes for calcium oxide production for various industrial applications, including the treatment of oil-contaminated soils. Further research may include determining the purity of the obtained CaO and its reactivity.

Conclusion

1. A comparative study of calcium-containing mineral raw materials from seven deposits was carried out using a combination of methods (XRD, IR spectroscopy, and elemental analysis). Fundamental differences in their phase composition were established.

2. A classification of the raw materials into three groups was proposed: calcium oxide–hydroxide, calcium hydroxide, and carbonate groups.

3. The most promising raw material for the energy-efficient production of highly active calcium oxide is limestone from the Beineu deposit (Quarry 1 and Quarry 2), which is characterized by the highest CaO contents (92.5% and 81.0%, respectively) and the lowest concentrations of inert impurities.

4. The raw material from the Zhanakorgan deposit (almost pure Ca(OH)₂) requires low-temperature treatment but special storage conditions.

5. The sample from Bryansk, despite its high CaO content, contains significant amounts of SiO₂ and Fe₂O₃, which must be taken into account when developing its processing technology.

6. The obtained results provide a scientific basis for selecting optimal raw materials and thermal activation regimes for the production of calcium oxide for environmental remediation technologies.

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КАЛЬЦИЙҚҰРАМДЫ БІРҚАТАР КЕН ОРЫНДАРЫНЫҢ КОМПОНЕНТТІК ҚҰРАМЫН АНЫҚТАУ ЖӘНЕ ОЛАРДЫ ПАЙДАЛАНУ БОЙЫНША ТЕХНОЛОГИЯЛЫҚ ҰСЫНЫСТАР

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Түйіндеме. Мұнаймен ластанған топырақтарды ремедиациялау технологияларында қолданылатын кальций оксидінің прекурсорлары ретінде кальцийқұрамды минералдық шикізаттың жарамдылығын бағалау мақсатында Ресей (Брянск), Қазақстан (Бейнеу, Шетпе, Жаңақорған) және

Өзбекстан (Фергана) кен орындарының үлгілеріне кешенді зерттеу жүргізілді. Зерттеу әдістері ретінде рентгенфазалық талдау (РФА), элементтік талдау және инфрақызыл Фурье-спектроскопия қолданылды. РФА әдісі арқылы үлгілердің доминантты фазалық құрамы анықталып, жартылай сандық бағалау жүргізілді. Алынған нәтижелер негізінде үлгілер үш топқа жіктелді: 1) кальций-оксид-гидроксидтік топ (Брянск, Бейнеу, Фергана), СаО мөлшері 76.1-92.5% және Са(ОН)₂ 7.5–19.0% құрайтын үлгілер; 2) кальций-гидроксидтік топ (Жанакорған), іс жүзінде таза Са(ОН)₂ (99.6%); 3) карбонаттық топ (Шетпе), негізінен кальциттен СаСО₃ (>98%) тұратын үлгілер. ИК-спектроскопия нәтижелері анықталған фазаларды растады. Элементтік талдау үлгілердің басым бөлігінде кальцийдің жоғары тазалығын көрсетті (>95% СаО, нормаланған). Брянск үлгісінде SiO₂ мөлшерінің жоғары екені анықталды (~4%). Зерттеу нәтижелері негізінде шикізатты тиімді пайдалану бойынша технологиялық ұсынымдар берілді. Энергия тиімділігі тұрғысынан белсенді СаО алу үшін Бейнеу-1 және Бейнеу-2 үлгілері (сәйкесінше 92.5 және 81.0 % СаО) ең перспективті деп танылды.

Түйінді сөздер: кальцийқұрамды шикізат, кальций оксиді, рентгенофазалық талдау, ИК-спектроскопия, элементтік талдау, жіктеу, мұнаймен ластанған топырақтар

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ОПРЕДЕЛЕНИЕ КОМПОНЕНТНОГО СОСТАВА РЯДА КАЛЬЦИЙ СОДЕРЖАЩИХ МЕСТОРОЖДЕНИЙ И ТЕХНОЛОГИЧЕСКИЕ РЕКОМЕНДАЦИИ ПО ИХ ИСПОЛЬЗОВАНИЮ

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Аннотация. Проведен комплексный анализ образцов кальцийсодержащего минерального сырья из месторождений России (Брянск), Казахстана (Бейнеу, Шетпе, Жанакорған) и Узбекистана (Фергана) для оценки их пригодности в качестве прекурсоров оксида кальция, используемого в технологиях ремедиации нефтезагрязненных грунтов. В работе применены методы рентгенофазового (РФА), элементного анализа и инфракрасной спектроскопии (ИК-Фурье). Методом РФА установлен фазовый состав образцов и выполнена их полуколичественная оценка. На основе полученных данных предложена классификация образцов на три группы: 1) кальциево-оксидно-гидроксидная (Брянск, Бейнеу, Фергана), характеризующаяся высоким содержанием СаО (76.1-92.5%) и Са(ОН)₂ (7.5–19.0%); 2) кальциево-гидроксидная (Жанакорған), представляющая собой практически чистый Са(ОН)₂ (99.6%); 3) карбонатная (Шетпе), состоящая из кальцита СаСО₃ (>98%). Данные ИК-спектроскопии подтвердили наличие идентифицированных фаз. Элементный анализ показал высокую чистоту большинства образцов по кальцию (>95% СаО в нормированном виде). Образец из Брянска содержит повышенное количество кремнезема (SiO₂ ≈ 4%). Предложены технологические рекомендации по использованию сырья. Наиболее перспективным для энергоэффективного получения активного СаО признан образец Бейнеу-1 и Бейнеу-2 (92.5 и 81.0 % СаО соответственно).

Ключевые слова: кальцийсодержащее сырье, оксид кальция, рентгенофазовый анализ, ИК-спектроскопия, элементный анализ, классификация, нефтезагрязненные грунты

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