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Carbonation of natural minerals and their use in various fields

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Abstract

The economic benefits of using a natural mineral sorbent, in adsorption purification technology come with the challenge of waste disposal (mineral sorbent with adsorbed matter). Since regenerating natural sorbent using various solvents is economically impractical, and direct waste disposal leads to environmental pollution, the possibility of producing new materials from waste from the adsorption purification process was explored. The studies were conducted using a model system consisting of a natural mineral sorbent (bentonite clay, zeolite, diatomite) in a mixture with organic sorbates - petroleum product (fraction of diesel oil) and molasses (complex mixture of natural carbohydrates a by-product from the crystallization of sugar from sugar beet or cane).

It was found that during the heat treatment (400-450°C) of this model compounds, the surface of the mineral is firmly coated with thermal decomposition products of adsorbed organic matters, partially with coke-like substances. Using the elemental analysis method, the average formula for the C:H, which is within the general formula for the composition of coke.

Carbonation alters the pore structure particles of mineral. Carbonated samples are dominated by meso- and macropores with equivalent radii ranging from 80 to 20,000 nm. The total pore volume increases, while the total specific surface area decreases.

Studies conducted to determine the scope of practical applications of carbonated mineral samples adsorbed by petroleum products and molasses (a by-product of sugar production) have shown their potential application as active fillers for plastics, rubber and sealants, as well as adsorbents for medical purposes.

Keywords: Clinoptilolite; Diatomite; Montmorillonite Clay; Carbonization; Sorbent; Organophilization;

1. Introductions

The development of modern chemical technology, the processing of petroleum products, the production of pharmaceuticals and food products, as well as the purification of industrial and waste water are unthinkable without the use of various types of adsorbents (activated carbon, silica gels, synthetic and natural aluminosilicates, etc.) The growing interest of researchers and technologists in the use of natural mineral sorbents (bentonite clays of montmorillonite composition, diatomites, clinoptilolite-mordenite zeolites, etc.) in adsorption processes is due to their low cost and large reserves.

The economic benefit gained from using inexpensive natural raw materials is accompanied by the problem of waste disposal (mineral sorbent with adsorbed organics). Since regenerating natural sorbents with various solvents is economically impractical, and direct disposal of waste leads to environmental pollution, the possibility of producing new materials from waste from the adsorption purification process was studied.

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Study of a natural mineral - Askan clay with adsorbed petroleum products (waste from the process of clarification of used oils) showed that materials obtained after heat treatment (at 250–300°C for 1–2 hours) can be used as fillers for rubber and drilling fluids. Samples obtained after heat treatment (carbonization) of a model mixture of Ascanian clay with diesel oil were tested as fillers for technical rubber instead of carbon black [1]. Replacing carbon black in the compound, even partially (1:1), with carbonized mineral significantly improves not only the performance characteristics but also the plasticity and ductility of the rubber compound [2,3].

Testing mixture of a carbonized enriched Ascanian clay earth sample with diesel oil as a filler in construction sealants yielded positive results. Replacing expensive aerosol with this carbonized product resulted in increased material strength, a 50% increase in elasticity, and a reduction in product costs [4].

A practical clinical study (conducted at the Institute of Urology) of a carbonized sample of a mixture of enriched montmorillonite Askan clay "Askangel" mixed with varying amounts (5–30%) diesel fraction of oilm, at different temperatures (100–300°C) demonstrated positive results in the detoxification of uremic diseases in urology [5].

The multi-purpose application of the product obtained as a result of heat treatment of minerals sorbed by various organic petroleum products under different temperature conditions indicates the applicability of carbonization as a modification of minerals and the possibility of obtaining materials with the desired properties using carbonization. Hence, the study of the carbonization process of natural minerals, mixed with various organic compounds, is a pressing issue.

2. Materials and Methods

The objects chosen for the study were: layered montmorillonite clay with a layered structure (Askana deposit); framework-structured zeolite clinoptilolite with a framework structure (Khekordzula deposit); and dispersed silicate diatomite (Qisatibi deposit). The process of carbonization of a mixture of these minerals from the Georgian deposit (of various compositions and structures) with the following organic compounds was studied: with diesel oil (fraction t=400–450 °C of crude oil from the Norio deposit, which is rich in high-molecular aromatic hydrocarbons) and molasses (a waste product from sugar production, a complex mixture of natural) [6].

To study the process of carbonization of a mixture of minerals with orgamic modifiers, the following research methods were used: method of elemental analysis, high-precision gravimetric method, X-ray, IR spectral and electron diffraction method studies [7].

3. Results and Discussion

It was found that during the process of heat treatment of a mixture of natural minerals with these organic compounds, the surface of the mineral becomes firmly coated with products of thermal decomposition of adsorbed organic substances, partly coke-like substances. Presumably, in this case, dehydrogenation, cyclization, and polymerization reactions result in the formation of hydrogen-depleted, partially coke-like substances. Elemental analysis revealed that the average C:H ratio in the thermal decomposition products is ($CH_{1.23}$ - $CH_{1.68}$), which is within the general compositional range of coke and corresponds to a carbon content of 8.82-11.82%.

Since, during the thermal treatment of a mixture of natural minerals with these organic compounds, the surface of the mineral is firmly coated with the products of thermal decomposition of organic substances, thereby causing organophilization of the surface of the mineral particles, these organic compound of mixtures can be considered a mineral modifier [8].

To determine the optimal conditions for carbonization of the study objects, various percentages (from 5 to 60%) of modifier (organic compound) were mixed with ground-crushed minerals (to a particle size of 0.16-0.30 μm) and the mixture was heat treated in the temperature range of 200 ° - 500 °C.

The optimal condition for carbonization of the object is considered to be complete hydrophobicity (not wetted and non-swelling in water). This optimal condition is: - in the case of modification of Ascan clay with glucose and molasses - heat treatment at a temperature of 500 ° C, modifier glucose 40%, modifier molasses 60%; in the case of zeolite clinoptilolite and diatomite - heat treatment at a temperature of 400 ° C, the same percentage of glucose and molasses. In the case of using petroleum products as modifiers, carbonization occurs in the temperature range of 200-250 ° C with a modifier content of 30%.

Since organic compounds are practically not extracted from carbonized samples by solvents, the content of organic substances in carbonized samples obtained by thermal treatment of a mixture of the studied minerals and an organic compound - diesel oil (Table 1) was determined by a high-precision gravimetric method.

Table 1 Content of organic compounds in carbonated samples of the studied minerals.

Sample	Ratio: diesel oil/mineral	Organic compound content %		
Askana Clay	3:1	28.75	9.58	8.85
Khekordzula Clinoptilolite	3:1	31.55	10.52	9.52
Qisatibi Diatomite	3:1	38.83	18.31	17.32

The fact that carbonization leads to hydrophobization of the objects under study is confirmed by the data on the adsorption of water vapor by natural and carbonized minerals (Table 2).

Table 2 Adsorption of water vapor by natural and carbonate minerals at different P/Ps values.

Sample	P/Ps mmol/g		
	0.15	0,44	0.96
Natural Ascana Clay	0.980	3.470	16.560
Carbonized Ascana Clay	0.720	1.560	11.360
Natural Clinoptilolite	0.630	0.960	5.233
Carbonized Clinoptilolite	0.021	0.092	3.625
Natural Diatomite	0.011	0.930	6.890
Carbonized Diatomite	0.004	0.190	4.420

According to the results of elemental analysis of carbonized samples of the studied minerals, the percentage of carbon in the carbonized samples is higher during the first 30 minutes of the carbonization process than during the subsequent period. We hypothesize that water evaporates during the first period, resulting in the surface of the mineral particles being coated with an organic layer. This layer subsequently (due to the thermal processes described above) forms a resinous, coke-like substance depleted of hydrogen. X-ray, IR spectral and electron diffraction studies confirm that the entire surface of the studied mineral is covered with thermal decomposition of modifier products [9]. No changes in the mineral's internal crystal lattice are observed. The appearance of new absorption lines in the spectrogram of carbonized mineral samples must be due to the presence of organic radicals formed during the thermal decomposition of the modifier. Since absorption lines characteristic of Si-C and Si-O-C chemical bonds are not observed in the spectra of carbonized mineral samples, chemisorption processes are likely not observed. Furthermore, organic compounds are not extracted from the mineral surface by solvents (benzene, hexane). Therefore, the process occurring on the surface of the mineral particles is physical in nature [10].

During the carbonation process, minerals' hydrophobic properties are enhanced and active sites are destroyed, which is explained by non-stoichiometric, isomorphic substitutions in the crystal lattice. As a result of carbonation, the ζ-potential (zeta potential) and other electrical conductivity parameters change depending on the nature and degree of dispersion of the mineral. In the case of carbonation of minerals modified with molasses, the zeta potential decreases from 110 to 90, while in the case of minerals modified with glucose, the zeta potential decreases to 80.

Based on available data, the use of carbonized minerals as fillers in polymer composites is a priority. In this case, ensures high-quality interaction between the carbonized mineral (with an organophilic, high-specific surface area) and the polymer, which is essential for producing composite materials with the required strength [11].

Based on the above, the colloidal-chemical and structural-mechanical properties of carbonized samples of the studied minerals were studied. The results are presented in Table 3. For comparison, the same table shows the characteristics of one of the active fillers - carbon black (soot), grade PM-70 [12].

Table 3 Physicochemical properties and classification of Carbonized mineral samples

Sample	True specific gravity, g/cm3	Total porosity %	Specific surface area m^2/g	PH of the aqueous suspension	Specific electrical conductivity sim.m^{-1}	Elemental composition		Classification
						C	H	
Carbonized Ascana Clay	1.83	47.54	60.0	5.0	9.1	9.88	1.01	Moderately active
Carbonized Clinoptilolite	1.74	44.83	59.1	5.5	8.9	-	-	Moderately active
Carbonized Diatomite	1.48	70.70	30.0	4.8	3.2	-	-	Semi-active
Soot PM-70A	1.8-2.0	-	85-78	6.5-8.5	-	97.2-97.5	0.3	Active

The electrical conductivity in the case of carbonation of minerals modified with molasses is 5.15×10^{-2} sym^{-1}, while in the case of minerals modified with glucose, it is 4.99×10^{-2} sym^{-1}. The zeta potential and electrical conductivity of zeolite and diatomite differ slightly. The decrease in these values is due to a decrease in the degree of surface ionization during carbonation.

According to the data in Table 3, carbonized samples are classified as weakly acidic fillers; their surface is rough and coated with oxygen-containing compounds formed during carbonization. Further heating of the samples removes the oxygen-containing compounds, resulting in the carbonized filler acquiring alkaline properties.

The preservation of ion-exchange properties in carbonated samples shows that, although the degree of ionization of the clay particle surface decreases during carbonation and a sharp compression of the diffusion layer is also observed, X-ray and electron diffraction studies confirm that this process occurs on the surface of the mineral particles. No changes in the internal crystal structure of the minerals are observed.

Carbonation also alters the pore structure of mineral particles. Meso- and macropores with equivalent radii in the range of 80–20,000 nm predominates. The total pore volume increases, while the total specific surface area decreases. Ascana clay is an exception, as its specific surface area remains virtually unchanged.

4. Conclusion

Thus, the study of the carbonization process of the studied minerals modified by various organic compounds demonstrated the possibility of providing targeted changes in the surface structure of dispersed mineral particles depending on the area of their application.[13]

Practical testing of carbonized mineral samples treated with petroleum products (under semi-industrial conditions at the Kutaisi Rubber Products Plant) showed that using carbonized samples of Askan clay and clinoptilolite in place of carbon black in the production of rubber products (even partially in a 1:1 ratio) significantly improves not only working conditions but also the plastic properties of the rubber. In relation to standard parameters, the following parameters increase: tensile strength by 12%, relative elongation by 60%, residual elongation by 43%, hardness by 8%, and plasticity by 130%.

Practical trials using carbonized diatomite samples modified with petroleum products in the production of sealed building materials instead of expensive Aerosil demonstrated a significant improvement in the performance of the resulting sealant. Compared to established standards, the material's strength increased by 117%, elasticity by 50%, and flowability by 75%.

Practical clinical trials of carbonized Ascanian clay samples modified with glucose demonstrated positive results in urology for the detoxification of uremic patients.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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