Synthesis and study of diatomite/alginate/Fe_3O_4 composite polymer material

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Introduction

Sorbents of various nature are widely used in various industries. Traditionally, the most common adsorbents are activated carbon (AC) and its derivatives, which have a high adsorption capacity for various pollutants, but require energy and are difficult to regenerate. A large number of works are devoted to the development and use of inexpensive and effective adsorbents. Among the various adsorbents, clays are substances that, due to their numerous advantages, such as high chemical stability in acidic media and adsorption capacity, high porosity and efficiency, have attracted the attention of researchers for many years.Diatomite not only has a high specific surface area and porosity, but is also natural and non-toxic. But diatomite cannot provide high ease of its separation from the solution and the possibility of repeated use. To solve this problem, the use of magnetic composite materials is promising.

Experimental

The original diatomite was obtained from a deposit in the Kharkiv region (Ukraine). In the process of performing the tasks in the experimental studies, the following raw materials and methods of experimental studies were used.The following reagents were used in the work: sodium alginate, diatomite (, ferric (III) sulfate, ferric (II) sulfate, methylene blue, ammonium hydroxide.

Application of nanosized Fe_3O_4 on diatomite was carried out as follows: a weight of diatomite was added to a solution of ferrum salts and a solution of ammonium hydroxide was added with intensive stirring. The obtained suspension was stirred for 30 min. The sediment was separated from the liquid by decantation until there were no sulfates in the filtrate. To prepare the suspension, a diatomite suspension with applied Fe_3O_4 was added to a 2% sodium alginate solution to obtain a 1% alginate solution and the corresponding solid phase content. Pellets of composite adsorbent were obtained at a laboratory facility.

The installation consists of a peristaltic pump, a tripod, a tube, and a replaceable

Results

The conducted microscopic analysis of diatomite and diatomite with alginate (Fig. 1a, b) showed a homogeneous and porous structure in comparison with the calcium alginate-diatomite- Fe_3O_4 composite. (Fig. 1c). Diatomite has large volume voids and a porous structure, which determines its choice as a potential sorbent for pollutants.The quantitative analysis is given in Table 1 and gives the weight ratios of the main elements in diatomite: O (50.78%), Mg (0.18%), Al (1.24%), Si (41.28%), K (0 .13%), Fe (1.18%) and Na (0.99%). The obtained results of the energy dispersion analysis indicate an increase in the carbon content in the calcium alginate-diatomite composite and an increase in the iron content in the structure of the diatomite-alginate- Fe_3O_4.

Table 1 Elemental composition of samples

Element	% mass	Error, %	Element	% mass	Error, %	Element	% mass	Error, %
Diatomite			Diatomite-alginate			Diatomite-alginate-Fe3O4		
Na_K	0.99	0.11	C_K	22.11	0.40	C_K	16.93	1.41
Mg_K	0.18	0.07	O_K	49.73	0.30	O_K	48.41	1.01
Al_K	1.24	0.10	Na_K	0.66	0.03	Na_K	0.95	0.13
Si_K	41.28	0.34	Mg_K	0.05	0.02	Mg_K	0.06	0.08
Cl_K	0.14	0.08	Al_K	0.40	0.02	Al_K	0.47	0.09
K_K	0.13	0.09	Si_K	25.13	0.17	Si_K	24.76	0.57
Ca_K	3.37	0.17	S_K	0.04	0.02	K_K	0.09	0.10
Fe_K	1.88	0.28	K_K	0.03	0.02	Ca_K	1.16	0.15
O	50.78	0.36	Ca_K	1.37	0.04	Fe_K	7.18	0.48
			Fe_K	0.48	0.06			

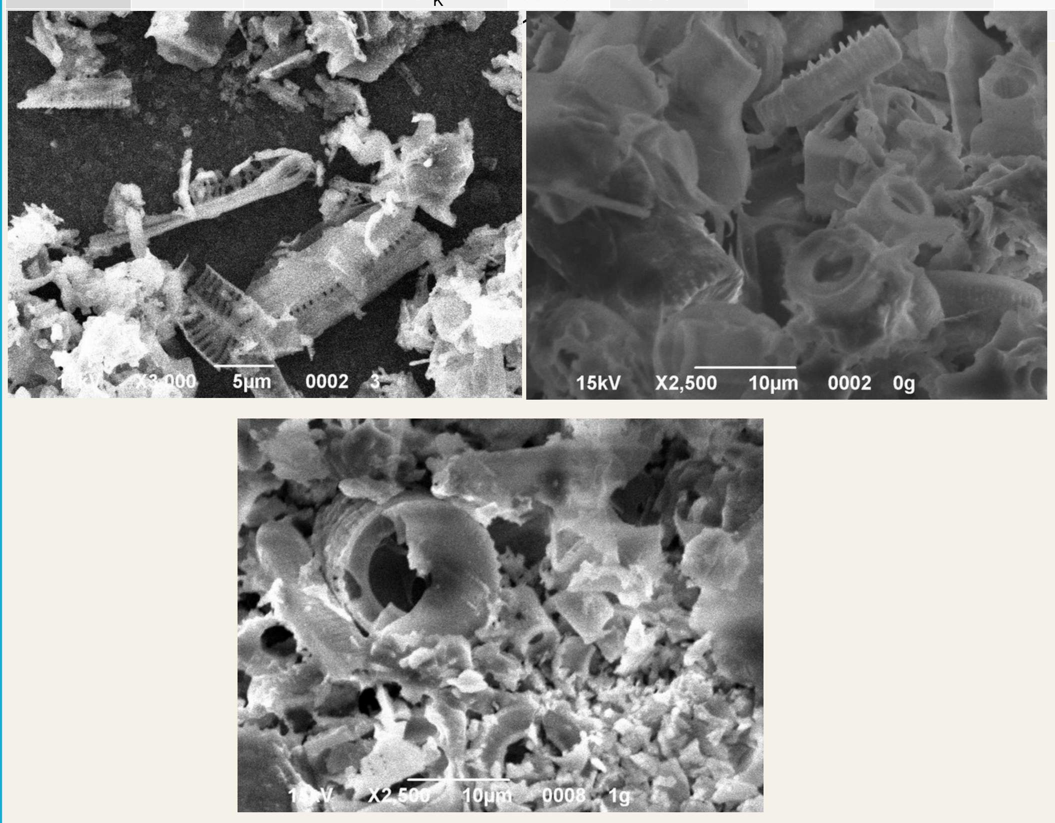

Fig. 1 Photomicrographs of samples a— diatomite, b- diatomite-alginate, c- diatomite-alginate- Fe_3O_4

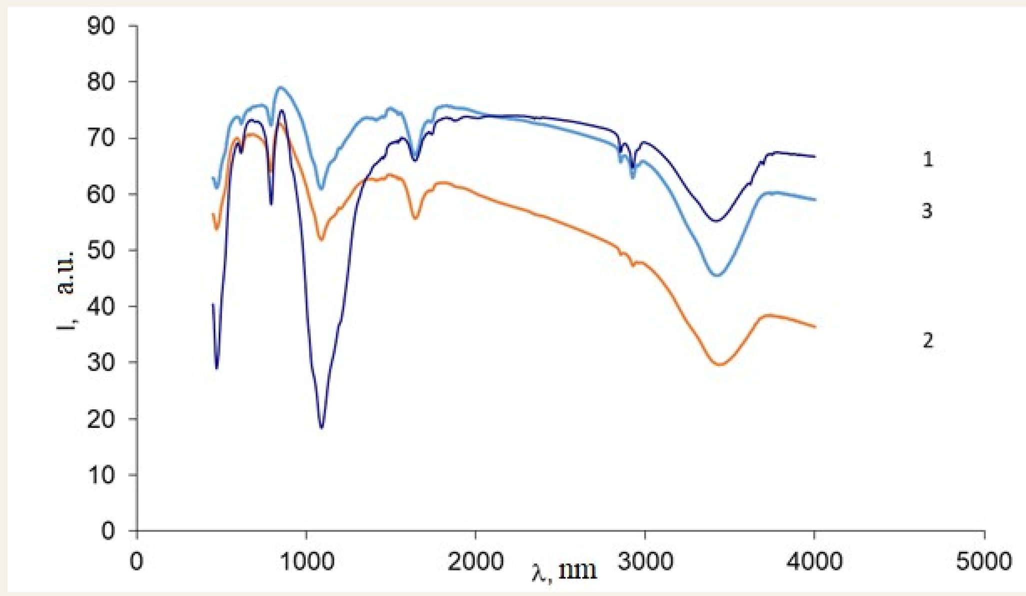

Fig. 2. IR spectra of samples: 1- diatomite, 2- diatomite-alginate, 3- diatomite-alginate- Fe_3O_4

IR spectroscopy was used to detect active functional groups. The presence of the Si–OH bond leads to the formation of a band at 3700 cm^{-1} and 796 cm^{-1}.As can be seen from Figure 2 (curves 1-3), the main bands, the intensity of which changes significantly for samples D, D-Al, D-Al- Fe_3O_4, correspond to the adsorption centers of diatomite and fall on the wave number 3433, 1047, 1086, 922, 794 and 614 cm^{-1}. The broad band at 3434 cm^{-1} in the spectrum is the area corresponding to interlayer molecules and framework hydroxyl groups. The wave number of 1090 cm^{-1} corresponds to the Si-O-Si bond.A comparison of the IR spectra of diatomite and alginate composites shows that the intensity of the Si-O-Si and Si-O-Al bands decreases with a decrease in the content of diatomite in the composites. A less intense broad peak at 1090 cm^{-1} is observed, which is associated with overlapping of C-C bonds of the alginate matrix and deformation of the Si-O-Si bond. For D-Al and D-Al- Fe_3O_4 samples, the peak located at 3434 cm^{-1} belongs to the –OH group, and the broad peak centered at 1646 cm^{-1} corresponds to the vibration of the COO− carboxyl group. The intensity of the 3434 cm^{-1} peak increases, respectively, for D-Al, D-Al- Fe_3O_4 composites.X-ray phase analysis data (Fig. 3) show the presence of three main phases: cristobalite, kaolinite and calcite. X-ray images show an intense narrow line corresponding to crystalline cristobalite (JCPDS No. 39-1425), which is the main component of diatomite. In the D-Al sample, the intensity of the peaks decreases, and in the D-Al-Fe_3O_4 sample, peaks corresponding to magnetite appear.

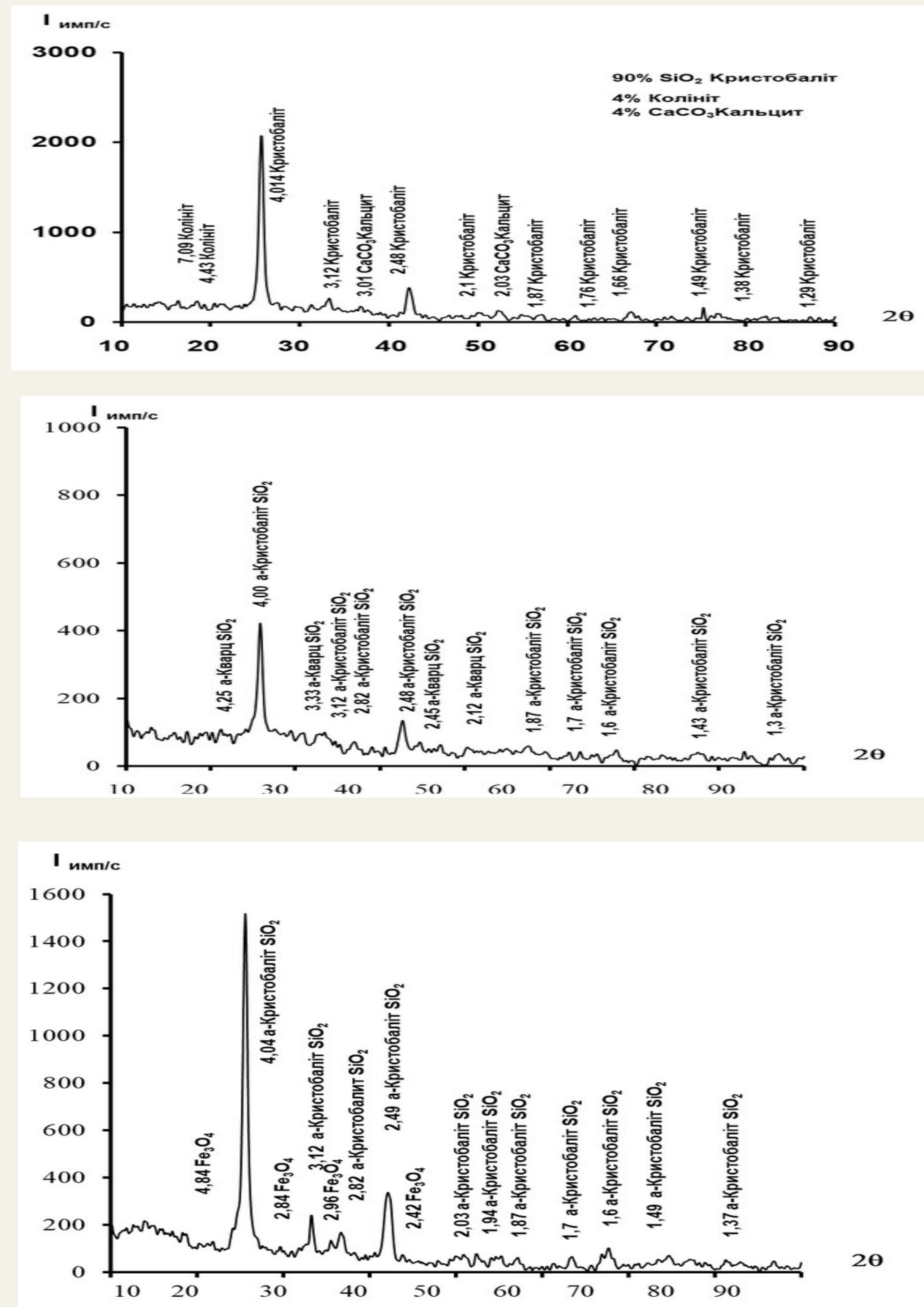

Fig. 3 X-ray diffraction patterns of samples a- diatomite, b- diatomite-alginate, c - diatomite-alginate- Fe_3O_4

Natural diatomite, which has a developed surface and can influence both the adsorption characteristics of the adsorbent and its mechanical characteristics, was chosen as a filler. A directly proportional dependence of the diameter of the air-dried alginate-diatomite balls on the diameter of the nozzle and the diatomite content is observed.

% solid phase	Nozzle diameter, mm	Diameter of "wet" granules, m	Diameter of "dry" granules, mm
1,5	1,5	1,5	1,0
	3,0	2,0	1,5
	3,5	2,5	2,0
3	1,5	1,5	1,5
	3,0	3,0	2,5
	3,5	3,0	2,5
5	1,5	2,5	2,0
	3,0	3,0	2,5
	3,5	3,0	2,5
7,5	1,5	3,0	2,5
	3,0	3,5	3,0
	3,5	3,5	3,0
10	1,5	3,0	2,5
	3,0	4,0	3,5
	3,5	4,0	3,5
20	1,5	3,5	3,0
	3,0	5,0	4,5
	3,5	5,0	4,5

An increase in the diatomite content leads to a natural increase in the size of the granules. Diatomite content of more than 20% did not allow high-quality granulation at the experimental plant. The optimal size of the adsorbent granules is 1.0-3.0 mm, since internal diffusion becomes more difficult when the size increases.

Conclusion

The conditions for the formation of spherical granules of diatomite in the presence of sodium alginate C[CaCl2]= 0.024 mol/l, ω(diatomite) = 5 – 20% have been established. It was established that with an increase in the content of the solid phase in the polymer composite material D-Al-Fe3O4 from 14% to 18%, the strength of the granules changes. And the strength of granules with a larger diameter decreases with an increase in the content of the solid phase, which is associated with the complication of the diffusion of Ca2+ ions.

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