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## The study of adsorptive capacity of Kulantau vermiculite.

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### ABSTRACT

The work is devoted to the study of the adsorption characteristic of the swelled vermiculite in relation to a number of matters: oil products and oxides of heavy metals. It was determined that principal cause of base-exchange capacity of minerals with developed structural cell is the hetero valent isomorphism in pattern. It is demonstrated that acidizing leads to sharp magnification of specific surface vermiculite adsorbent and volume of transitive holes.

**Keywords:** adsorption, vermiculite, a cellular structure, adsorber.

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## INTRODUCTION

Every year the circle of the scientific and technical problems linked to use of the adsorptive processes is widened.

Among possible technical mineral sorbents for wide usage, in particular, special attention is paid to adsorptive-active materials of natural mineral raw materials in processes of industrial discharge refining and receptions of pure matters. The perspective source of such raw materials are laminated silicates, first of all, vermiculite (Syrmanova & Kaldybekova, 2005; Greg, Syng, 1970, Batalova, 1982; Dullien, 1979.). High enough the adsorptive, catalytic and ion-exchange properties of minerals, appearance of effective methods regulation of their geometrical pattern and chemical nature of the surface, presence of large commercial deposits do economically expedient usage as vermiculite adsorbents. However because of insufficient level of research these materials have not found wide application in our country as adsorbents yet.

The questions of improvement of structural and sorption and strength characteristics, increases of selectivity to these or those substances, expansion of the range, simplification of manufacturing techniques and reduction in cost of production of industrial adsorbents are essential. However at present questions of practical application of the Kazakhstan vermiculites are insufficiently studied. Therefore, study of the adsorptive properties of vermiculites fields of the South Kazakhstan region for the purpose of definition of effective ranges of application and creation on this basis of adsorbents for catching of harmful admixtures and refining of products of chemical productions is actual problem.

### Methods

For study of the adsorptive characteristic of vermiculite were used solutions of inorganic and organic compounds with 10, 20, 30, 40, 50% concentration. The greatest practical interest represents Kulantau field in South Kazakhstan region (Syrmanova, Rivkina, Kaldybekova & Sakibayeva, 2011; Syrmanova, Kaldybekova, Sakibayeva and Brener, 2012; Ayzov, 1973; Aripov, 1970). The vermiculite maintenance in ore fluctuates largely - from the first unit to 25-35%. Volume weight of swelled vermiculite fluctuates from 75 to 200 kg/m<sup>3</sup>. Average volume weight for a field is 135 kg/m<sup>3</sup>. On fineness swelled vermiculite corresponds to fractions +0.15-5.0 mm. Thus to 80% vermiculite fineness is +0.5-5 mm. The index of grains brittleness is from 2 to 30-40%. The coefficient of swelled vermiculite fluctuates within 4.8-11, average coefficient - 6. On coefficient of swelling vermiculite belongs to hydro micas with average extent of hydration. The vermiculite reserves which have been counted up on categories C, with volume weight of 200 kg/m<sup>3</sup> compound 0.5 million t that is equivalent to 3.0-3.5 million m<sup>3</sup> of swelled vermiculite. Besides, by analyses of vermiculite prospects it is determined that ore contains and hydro biotitic volume weight which fluctuates from 200 to 460 kg/m<sup>3</sup>. After ore roasting hydro biotitic is well extracted by means of air classification and, undoubtedly can find application, both in building, and in agriculture. The quantity compounds 12-13% on field. With the account hydro biotitic hydro mica reserves compound 1.0 million t. The photos of samples of vermiculites reduction are given on fig. 1.



Expanded vermiculite (Irsu)(a)



Expanded vermiculite (Zhylandy) (b)



Expanded vermiculite (Kulantau) (c)

**Figure 1: Photos of samples of vermiculites fields of Irsu, Zhylandy and Kulantau**

Four plots of vermiculite appearance were revealed on the territory of northwest branches of Talas Alatau (Syrmanova & Kaldybekova, 2005; Greg & Syng, 1970; Batalova, 1982; Iskriksky, 1975; Syrmanova, Rivkina, Kaldybekova, Sakibayeva, 2011; Syrmanova, Kaldybekova, Sakibayeva & Brener, 2012; Ayvazov, 1973; Aripov, 1970)

The preliminary estimation received only the Chetvertay and partially Karatau zones, as the most accessible plots for small mine workings. On the field of Chetvertay mining zone vermiculite is traced on the plot by length to 1200 m at width from 200 to 400 m. The vermiculite maintenance fluctuates from 10 to 15%. The depth of vermiculite zones is not less than 20 m. The approximate reserves of vermiculite 1.2-1.5 million t is a slightly less than Karatau zone, where vermiculite is more leprous and its maintenance is within 5-10%. At conducting of directional operations on the fields bridged over conglomerates, vermiculite eliciting is possible in volume 5-6 million t. Total reserves will compound first ten millions tons.

Chemical analysis of varieties vermiculite ores of SKO fields is represented on tab. 2 from which follows, that even for most hydrated last variety, process of turning into vermiculite is not completed fully: transferring of iron from 2 valent forms to 3 valent also is not finished, i.e. micaceous minerals of fields do not contain some vermiculite in mineralogical value of this word, and "vermiculite" it is necessary to understand as the term most strongly hydrated micas of transitive number (vermiculites of Irsu and Zhylandy fields).

The distinctions in mineralogical composition of ores (Kulantau vermiculite) – the basic compounding - minerals phlogonite, and vermiculite of Irsu and Zhylandy – the basic phase – biotite and hydro biotite (tab. 1)

have determined also different approach to solution of the problem of ore dressing. For Kulantau ores the wet way of beneficiating with application of skimming machines is selected. For vermiculites of Irsu and Zhylandy fields the ore dressing is possible dry process at electromagnetic separation (Fedorov & Sevryugov, 1986; Dyachenko, Ostrovsky, Sharkina, Plastinina, Cokovsky, 1987; Polyakov & Klimenko, 1999; Gogol, Gruba, Boychuk, Dorofeev, Zaritsky, Lipovsky & Rebezo, 1985)

**Table 1: Chemical composition vermiculite ores of Irsu, Zhylandy and Kulantau fields**

Components, %	Vermiculite		
	Irsu	Zhylandy	Kulantau
K <sub>2</sub> O	7.5-10.4	5.8-11.5	-
MgO	6.8-11.6	0.4-27.4	15.5-22.8
Al <sub>2</sub> O <sub>3</sub>	10.2-16.3	9.5-30.6	10.2-12.8
SiO <sub>2</sub>	38.5-44.4	32.7-44.1	37.1-41.5
Fe <sub>2</sub> O <sub>3</sub>	0.01-0.03	0.14-21.2	6.4-17.2
H <sub>2</sub> O	0.6-5.5	0.85-4.4	8.3-17.5
FeO	-	2.4-30.0	1.4-2.8

The received results of qualitative characteristic of vermiculite of the Kulantau field are demonstrated on tab. 2

**Table 2: Qualitative characteristic of vermiculite Kulantau field**

Number sample	Dampness, %	Extent of hydration, %	Volume weight, kg/m <sup>3</sup>	The content of vermiculite, %
1	6.2	65	178	30
2	9.0	70	200	35
3	3.7	60	130	28
4	3.1	75	140	26

**Table 3. Chemical composition of swelled vermiculites of Irsu (1), Zhylandy (2) and Kulantau (3) fields**

Vermiculite	The content of reductants, %							
	SiO <sub>2</sub>	CaO	MgO	K <sub>2</sub> O + Na <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	H <sub>2</sub> O	The other
1	38.96	1.81	8.41	7.3	0.21	10.88	4.31	28.13
2	33.82	1.96	21.45	1.15	7.85	14.99	4.75	14.03
3	37.44	2.10	23.88	1.18	6.01	11.23	10.98	17.18

The microscopical analyses and results of X-ray structure analysis of test samples of vermiculite of the Kulantau field have demonstrated that the basic component is kochubite – (Mg, Fe, Al)<sub>6</sub>(Si, Cr)<sub>4</sub>O<sub>10</sub>(OH)<sub>8</sub>, a variation product of biotite with depressed maintenance K<sub>2</sub>O and the enhanced maintenance H<sub>2</sub>O, represented by scales of brown mica. As admixtures are observed: calcite, crystals of chlorite, quartz. The received results (tab. 3) basically do not contradict with the literary data (Syrmanova & Kaldybekova, 2012; Karasev et al., 2002); the object of studies in work was chosen Kulantau vermiculite.

### DISCUSSION OF RESULTS

On the basis of adsorptive-structural studies the classification schema of argillaceous sorbents, on features of cellular structure it is possible to divide into three basic types (Greg & Syng, 1970; Syrmanova, Kaldybekova, Sakibayeva and Brener, 2012; Karasev et al., 2002)

1. Fissile minerals with extended structural cell. The basic representatives are montmorillonite and vermiculite. In the course of adsorption of polar matters the grating of these minerals depending on the sort of base exchange, electronic and geometrical structure of adsorbate is extended on 3-12 Å, and in interpackage space is implanted one or several molecular layers of adsorbed matter. Thus, montmorillonite and vermiculite should be referred to micro porous sorbents with pore size modified in the course of adsorption.

Except the fundamental micro porosity caused by crystalline constitution, these minerals have secondary, basically the transitive holes formed by gaps between contacting fragments. Their radius, according to X-ray scattering under small angles and capillary condensation of steams, compounds 50-90 Å. As measuring of adsorption demonstrate non-polar matters which molecules are adsorbed only on an exterior surface of montmorillonite and vermiculite, the surface of secondary pores of these minerals and their volume is much less than surface and volume of fundamental holes. The data on tab. 10 provide guidance on parameters of micro transitive holes of the basic exponents of sorbents of this type.

2. Fissile-banded minerals. The basic exponents are palygorskite and sepiolite. The adsorptive properties palygorskite and sepiolite are determined, on the one hand, zeolite channels in structure with sizes 3.7x6.4 Å and 5.6x11.0 Å accordingly (fundamental holes), and on the other hand – porous space of size in which (secondary pore system) is aggregated acetous or fibrous parts of minerals. Zeolite channels palygorskite and sepiolite are accessible to water molecules, methanol, ammonia, methyl amine, nitrogen. In difference to montmorillonite and vermiculite the surface and volume of secondary pores of these minerals are great enough (tab. 4), as causes their high adsorptive properties in relation to hydrocarbons. The volume of secondary pores palygorskite can be increased considerably by extrusion of small wetpaste through spinnerets.

3. Fissile minerals with rigid grating. The basic exponents are talc, pyrophyllite, hydromica, and kaolinite. All these minerals are characterized by presence of only exterior adsorbing surface, and their porosity is caused by gaps between contacting fragments.

**Table 4: Varieties of adsorbent holes**

Adsorbent	V <sub>s</sub> , cm <sup>2</sup> /g	Varieties of holes						V <sub>mn</sub> /V <sub>s</sub>
		Transitive holes			Micropores			
		V <sub>tra</sub> , cm <sup>3</sup> /g	S <sub>tra</sub> , m <sup>2</sup> /g	r <sub>tra</sub> , Å	V · cm <sup>3</sup> /g		τ Å	
					Xray structure	adsorb		
Vermiculite Kulantau	0.18	0.03	12	90	0.19	0.15	5.6	0.83
Montmorillonite	0.37	0.05	39	45	0.36	0.32	9.6	0.86
Palygorskite	0.45	0.29	153	80	0.18	0.16	3.7x6.4	0.36

Note: V<sub>s</sub> – limiting adsorptive volume; V<sub>tra</sub> – volume of transitive holes; S<sub>tra</sub> – their specific surface; r<sub>tra</sub> – their radius; V – volume of micropores determined under the X-ray structure and adsorptive data; τ – thickness of micro pores.

The magnitude of specific surface and the size of transitive holes of fissile silicates with rigid grating are in many respects determined with dispersity of fragments which, in turn, depends on perfection of crystalline structure. In texture of high dispersed samples along with transitive holes it is possible to guess presence of a small amount of micro pores (r = 10-15 Å).

Clay minerals, possessing similar building blocks, at the same time differ from each other in the way of their junction in packages, the nature and bond energy between the last, nature settlement of tetrahedral and octahedral grids and variety of other more thin structural features. All it, undoubtedly, also causes a variety of colloidal-chemical properties of separate exponents of considered group of silicates observed in practice as properties of any dispersible material it is in many respects determined with pattern of composing it dispersible fragments.

There are two principal reasons causing capacitance of exchange base of clay minerals (Greg & Syng, 1970; Karasev et al., 2002):

1. Displacements in pattern of quadrivalent silicon tervalent aluminium in tetrahedral grids and tervalent aluminium ions of the lowest basicity, usually magnesium, in octahedral grids lead to appearance of negative not compensated charge of structural cell. It can be counterbalanced by an opposite charge on sign, arising, for example, at the expense of substitution of structural oxygen part by hydroxyl groups, or that is characteristic for dioctahedral fissile silicates, taking by bases of some vacant octahedral stands.

However more often arising negative charge of grating is countervailed by extra structural base exchange. The last are laid out basically on basal plains of fragments of clay minerals.

**Table 5: Boiling point of P<sub>boil.</sub><sup>0</sup>C at stress ≈ 760 mm hg, refraction index  $n_D^{20}$  and vapour pressure P, mm of hg refined adsorbents at 20<sup>0</sup>C**

Adsorbate	P <sub>boil.</sub> <sup>0</sup> C	$n_D^{20}$	P, mm. of hg
Water		1.3330	17.5
Benzol	80.1	1.5017	73.8
Xylene	144.41	1.5056	61.9
Toluene	110.6	1.4962	63.6
Phenol	182	1.5403	91.6
Benzine	100-150	1.23	53.2
Kerosene	200-250	1.31	49.3
Oil	to 220	1.19	72.1
CdCl <sub>2</sub>	968	1.67	29.5
Pb(NO <sub>3</sub> ) <sub>2</sub>	946	1.78	34.8

2. The breaking of chemical bonds in tetrahedral and octahedral grids of crystals of clay minerals results, in the final accounting, in appearance on their sides of hydroxyl group's hydrogen which under certain conditions can metathesis.

The hydroxyl groups, laid out on sides of crystals and linked to atoms of silicon, it is possible to consider on properties close to surface hydroxyl groups of silica. In acid environment at pH=2-5 on the silica surface already there is rather a small amount of proton gene active sites. The capacity of hydroxyl groups of silica base exchange sharply increases at pH = 7. At last, in alkaline condition silica is characterized by the maximum capacitance of base exchange which for its some varieties reaches several milligrams-equivalents on 1 g of sample.

Vermiculite is characterized by essentially big sizes of crystals and therefore quantity  $\equiv \text{Si} - \text{OH} -$  grouping on the exterior surface capable to exchange protons, counting on 1 g of sample will be lower. If to take into account that gravity of superfluous negative charge of grating of vermiculite in 1.3-1.5 times above, than montmorillonite, so share of the surface hydroxyl groups linked to atoms of silicon, in its capacitance of base exchange, undoubtedly, below 15 %.

Thus, principal reason of base-exchange capacity of minerals with extended structural cell is heterovalent isomorphism in pattern.

Adsorption of water was studied by weight method, ammonia, various hydrocarbons, variety of oxygen - and nitrogen containing organic compounds. In experiments was used bidistilled water. In tab. 6 some physical and chemical constants of water and organic adsorbents are given; they will be in keeping with available reference data.

For adsorptive capacity studying of swelled vermiculite was used device (fig. 2), consisting of separating funnel 1, a flat-bottom flask 2, adsorptive 3, adsorbent 4 and adsorbate 5. The quantity of the adsorptive columns only 9 pieces of proportion of volume of solid phase (swelled vermiculite) and fluid phase (solutions of organic and inorganic compounds, waters) varied with 1:2 to 1:5. The data of outflow time is resulted in tab. 6-7.

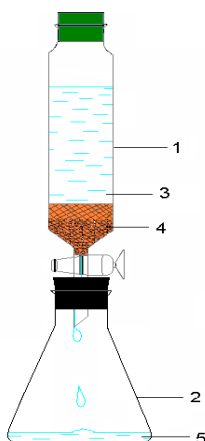


Figure 2: Device for adsorptive capacity studying of swelled vermiculite.

Table 6: Data on adsorptive capacity of the swelled vermiculite (1 of %-s' adsorptive) S:L=1:2

Nº	Adsorptive	Source concentration adsorptive, %	Time of passing of solution, minute	Concentration of adsorbate, %
1	Benzine	1	62	0.88
2	Benzol	1	64	0.74
3	Kerosene	1	61	0.93
4	Xylene	1	64	0.30
5	Oil	1	37	0.83
6	Toluene	1	67	0.60
7	Phenol	1	75	0.48
8	CdCl <sub>2</sub>	1	After days	-
9	Pb(NO <sub>3</sub> ) <sub>2</sub>	1	After days	-

Table 7: Data on adsorptive capacity of the swelled vermiculite (10 %-s' adsorptive) S:L=1:5

Nº	Adsorptive	Source concentration adsorptive, %	Time of passing of a mud, minute	Concentration of adsorbate, %
1	Benzine	10	4	0.22
2	Benzol	10	7	1.27
3	Kerosene	10	8	7.28
4	Xylene	10	11	8.33
5	Oil	10	6	-
6	Toluene	10	9	0.83
7	Phenol	10	8	-
8	CdCl <sub>2</sub>	10	14	8.03
9	Pb(NO <sub>3</sub> ) <sub>2</sub>	10	13	10.1

Table 8: Data on adsorptive capacity of the swelled vermiculite (20 %-s' adsorptive)

Nº	Adsorptive	Source concentration adsorptive, %	Time of passing of a mud, minute	Concentration of adsorbate, %
1	Benzine	20	6	0.65
2	Benzol	20	9	2.18
3	Kerosene	20	10	16.05
4	Xylene	20	25	16.4
5	Oil	20	7	-
6	Toluene	20	11	1.21
7	Phenol	20	16	-
8	CdCl <sub>2</sub>	20	15	13.02
9	Pb(NO <sub>3</sub> ) <sub>2</sub>	20	18	15.11

**Table 9: Data on adsorptive capacity of the swelled vermiculite (30 %-s' adsorptive)**

№	Adsorptive	Source concentration adsorptive, %	Time of passing of a mud, minute	Concentration of adsorbate, %
1	Benzine	30	9	0.84
2	Benzol	30	13	3.29
3	Kerosene	30	13	23.5
4	Xylene	30	37	26.3
5	Oil	30	9	-
6	Toluene	30	16	1.89
7	Phenol	30	23	-
8	CdCl <sub>2</sub>	30	22	16.92
9	Pb(NO <sub>3</sub> ) <sub>2</sub>	30	26	17.45

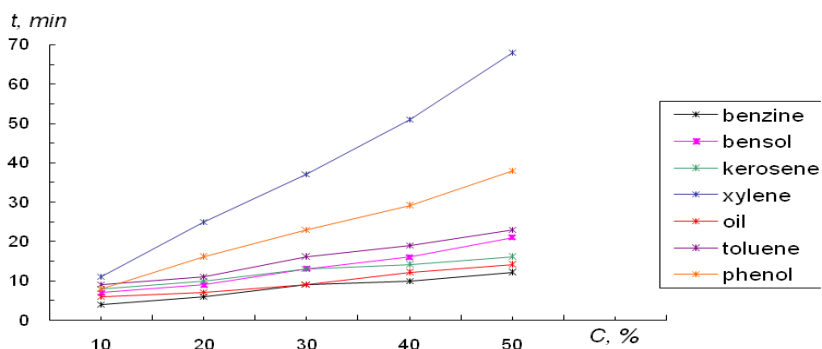
**Table 10: Data on adsorptive capacity вспученного vermiculite (40 %-s' adsorptive)**

№	Adsorptive	Source concentration adsorptive, %	Time of passing of a mud, minute	Concentration of adsorbate, %
1	Benzine	40	10	1.13
2	Benzol	40	16	4.18
3	Kerosene	40	14	36
4	Xylene	40	51	38
5	Oil	40	12	-
6	Toluene	40	19	2.42
7	Phenol	40	29	-
8	CdCl <sub>2</sub>	40	26	20.08
9	Pb(NO <sub>3</sub> ) <sub>2</sub>	40	32	18.3

**Table 11: Data on adsorptive capacity of the swelled vermiculite (50 %-s' adsorptive)**

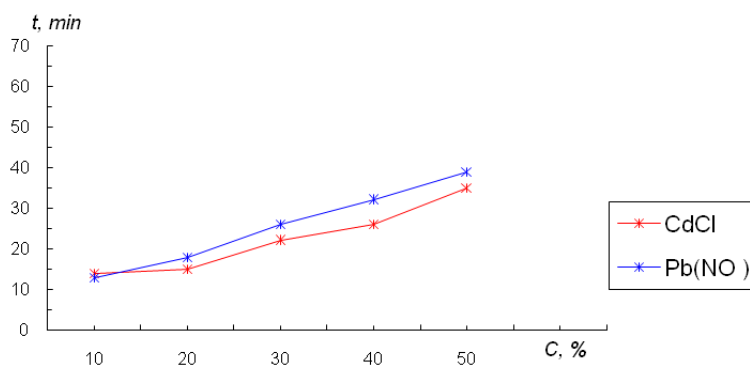
№	Adsorptive	Source concentration adsorptive, %	Time of passing of a mud, minute	Concentration of adsorbate, %
1	Benzine	50	12	1.26
2	Benzol	50	21	4.93
3	Kerosene	50	16	47.81
4	Xylene	50	68	47.26
5	Oil	50	14	-
6	Toluene	50	23	2.74
7	Phenol	50	38	-
8	CdCl <sub>2</sub>	50	35	22.5
9	Pb(NO <sub>3</sub> ) <sub>2</sub>	50	39	21.06

From experimental data it is visible that optimum for experiment conducting the proportion solid and fluid phase 1:5 is, in connection, with all further experiments on studying adsorptive characteristics of solutions of organic and inorganic compounds have been continued at a proportion of solid and fluid phase 1:5.



**Figure 3. Dependence of time outflow of organic adsorptives from initial concentration**





**Figure 4. Dependence on time outflow of inorganic adsorptives from initial concentration**

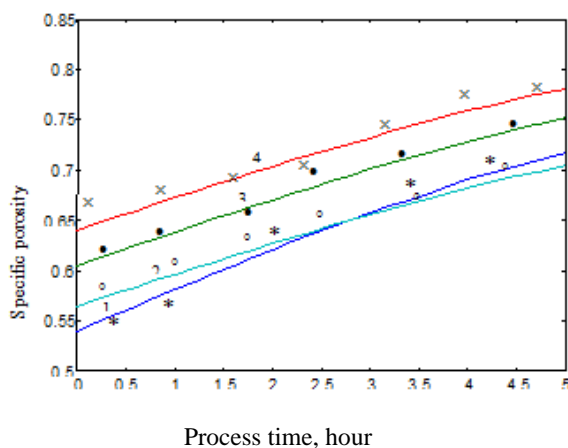
For studying adsorptive characteristics of vermiculite were used solutions of inorganic and organic compounds with 10, 20, 30, 40, %-s' 50 concentration. The data depending on time of the outflow from concentration of solutions of organic (fig. 3) and inorganic (fig. 4) compounds are resulted in drawings.

It is necessary to note that with magnification of solutions concentration of both organic and inorganic compounds proportional magnification of time outflow through adsorptive column, containing vermiculite is observed.

The results of comparison of calculated values of specific porosity of the swelled vermiculite and extent of catching of various substances and the corresponding sizes received during pilot studies, and also experimentally certain resources of swelling are given in figures 7,8.

The experiments have demonstrated that optimum for experiment conducting proportion of solid and fluid phase is 1:5. From diagram it is visible that at correctly chosen resource of porosity mathematical model developed in the second partition describes process of swelling with enough high accuracy. The error does not exceed 5 %. It is demonstrated that acidizing leads to sharp magnification of their specific surface and volume of transitive holes (Karasev et al, 2002; Syrmanova, Kaldybekova, Botabayev, Beloborodova, Tuleyov & Sakibaeva, 2013; Iskritsky, 2010). At first adsorbent contacts with arriving concentrated solution. As a result of filtering through adsorbent layer almost all permeate is removed from solution. The uppermost part of the layer is saturated, and the basic adsorption originates in rather narrow part of the layer of sorbent where concentration is quickly modified. When this field reaches in advance set limiting value, it is considered that there was a breakthrough.

In figures 5 and 6 results of processing of the received data on breakthrough of various contaminants through the layer of the swelled vermiculite are demonstrated.



Original porosity: 1- 0.25; 2 -0.32; 3 - 0.38; 4-0.42. Dots demonstrate experimental data.

**Figure 5: Dependence of specific porosity on process time**

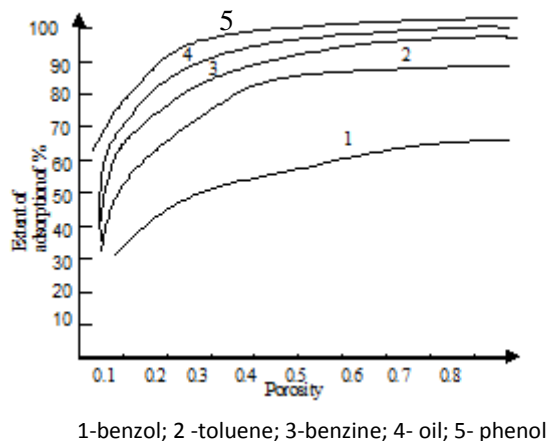


Figure 6: The extent of catching matters depending on sorbent porosity

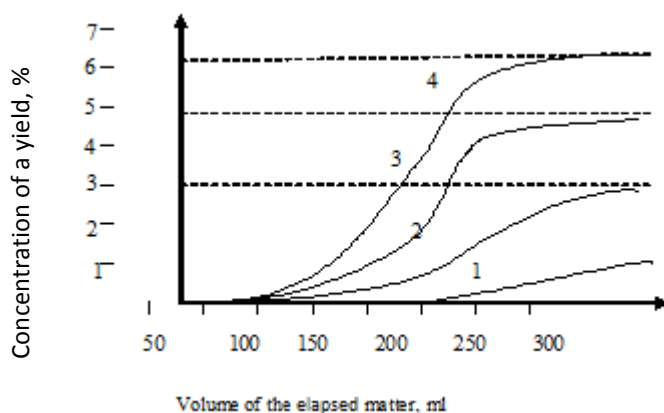


Figure 7: Breakthrough of contaminants through the layer of the swelled vermiculite

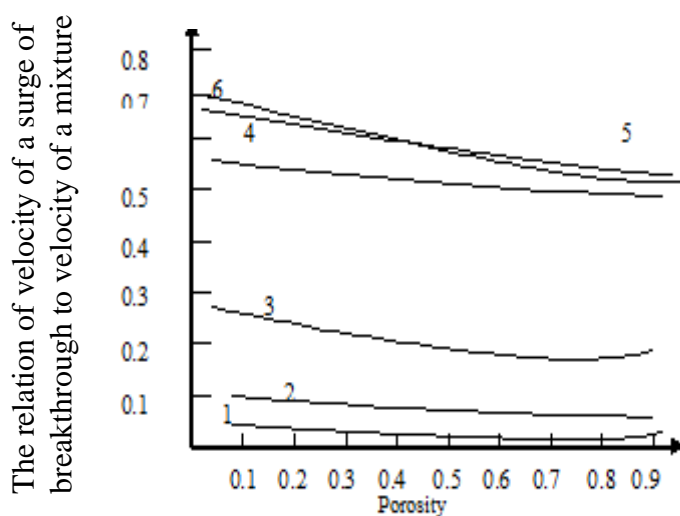


Figure 8: Dependence of relative wavevelocity of breakthrough on the layerporosity

The calculation of adsorber of periodic operating with motionless adsorbent is executed in following order.

1. Equilibrium concentration of contaminant in solid phase is determined. Under the known sorption isotherm is determined quantity of contaminant which can adsorb adsorbent at the produced temperature if process is continued to equilibrium state.
2. Type of adsorber and its design data are chosen. The fictitious velocity of mixture in an adsorber  $w = 0.05 \dots 0.1$  km/s is accepted, under the set discharge rate count up diameter of the device and pick up proximate standard size of adsorber of the chosen construction. Under design characteristics of the device and wave velocity of breakthrough pick up comprehensible altitude of adsorbent layer.
3. The mass-transfer coefficient is determined.
4. Checking of convergence of the found parameters, compounding the mass balance is carried out.
5. The duration of supportive stages of mixture treating process is determined.

The duration of other phases of cycle (denudation, drying and adsorbent cooling) count, as a rule, on the basis of experimental data or on the empirical equations (in view of complication of the mathematical specification statement of the applicable processes, caused mainly internal porosity of adsorbent).

### CONCLUSIONS

The adsorptive capacity of the layer of the swelled vermiculite is experimentally determined at catching of liquid contaminants: various organic matters and solutions of oxides of metals optimum phase relations S:L also are determined at adsorption of various contaminants.

Resources of swelling depending on the original porosity are determined.

Complex data on breakthrough of contaminants and wave velocity of breakthrough is obtained at filtering of various matters depending on porosity of the swelled vermiculite.

The procedure of calculation of adsorber of periodic operating with motionless adsorbent is resulted.

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