ТЕХНИЧЕСКИЕ НАУКИ И ТЕХНОЛОГИИ

МАТЕРИАЛТАНУ
МАТЕРИАЛОВЕДЕНИЕ
MATERIALS SCIENCE

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INVESTIGATING CHRYSOTILE ASBESTOS WASTE AS A POTENTIAL MATERIAL FOR SYNTHETIC ADSORBENTS

СИНТЕТИКАЛЫҚ АДСОРБЕНТ МАТЕРИАЛЫ РЕТІНДЕ ХРИЗОТИЛ АСБЕСТ ҚАЛДЫҚТАРЫН ҚОЛДАНУЫН ЗЕРТТЕУ

ИССЛЕДОВАНИЕ ОТХОДОВ ХРИЗОТИЛ-АСБЕСТА КАК ПОТЕНЦИАЛЬНОГО МАТЕРИАЛА ДЛЯ СИНТЕТИЧЕСКИХ АДСОРБЕНТОВ

Annotation. Technological methods have been developed to acquire an active component with a high SiO$_2$ content, which serves as a matrix foundation for synthetic adsorbents made from chrysotile asbestos production waste composite materials. Comparative analyses were conducted between the obtained adsorbent and a zeolite adsorbent of the NaX model. The physico-chemical analyses unveiled that the synthetic adsorbent obtained is not inferior in any aspect, and in certain instances, even outperforms in the demetallization process of heavy petroleum raw materials.

The demetallization process exhibits an optimal temperature range of 380-410 °C, with a contact time of 60 minutes and a pressure range of 8-10 atm. Concurrently, the concentration of vanadium in heavy crude oil decreases significantly from 540 to 28 micrograms per kilogram, while nickel content diminishes from 50 to 26 micrograms per kilogram. Moreover, iron content experiences a reduction from 50 to 12 micrograms per kilogram, and sulfur content decreases from 3.50 % to 2.39 %.

Keywords: Adsorbent; asbestos waste; demetallization; heavy oil feedstock; sulphur.

Аңдатпа. Хризотилді асбест өндірісінің қалдықтарын пайдалана отырып, композитті материалдар негізінде синтетикалық химиялық адсорбенттердің матрицалық негізін құру үшін жоғары SiO$_2$ құрамы бар белсенді компонентті алудың технологиялық режимдері әзірленді. Алынған химиялық адсорбенттің NaX үлгісіндегі цеолиттік химиялық адсорбентпен салыстырмалы талдаулары жүргізілді. Жүргізілген физика-химиялық талдаулар нәтижесінде алынған синтетикалық химиялық адсорбент ешбір жағдайда кем түспейтіні, ал кейбір жағдайларда ауыр мұнай шикізатын деметализациялау процесінде жақсырақ жұмыс істейтіні анықталды.

Деметализациялау үшін оңтайлы температура аймағы 380-410 °C, байланыс уақыты 60 минут, қысым 8-10 атм. Бұл ретте ауыр мұнай шикізатындағы ванадий мөлшері 540-тан 28 мкг/кг-ға дейін, никель мөлшері 50-ден 26 мкг/кг-ға дейін, темір 50-ден 12 мкг/кг-ға дейін, күкірт 3,50 %-дан 2,39 %-ға дейін төмендейді.

Түйін сөздер: Адсорбент; асбест қалдықтары; металлсыздандыру; ауыр мұнай; күкірт.

Аннотация. Разработаны технологические режимы получения активного компонента с высоким содержанием SiO₂ для создания матричной основы синтетических химадсорбентов на основе композиционных материалов с использованием отходов производства хризотил-асбеста. Были проведены сравнительные анализы полученного химадсорбента с цеолитным химадсорбентом модели NaX. В результате проведенных физико-химических анализов было выявлено что, полученный синтетический химадсорбент ничем не уступает, а в некоторых случаях даже лучше показывает себя в процессе деметаллизации тяжелого нефтяного сырья.

Оптимальной температурной зоной деметаллизации является 380-410 °C, время контакта - 60 минут, давление – 8-10 атм. При этом содержание ванадия в тяжелом нефтяном сырье снижается с 540 до 28 мкг/кг, содержание никеля с 50 до 26 мкг/кг, железа с 50 до 12 мкг/кг, а серы с 3,50% до 2,39%.

Ключевые слова: Адсорбент; асбестовые отходы; деметаллизация; тяжелое нефтяное сырье; сера.

Introduction. Currently, the oil industry places considerable emphasis on technologies that enable the processing of heavy, high-viscosity, sulfur-based oil feedstocks and oil residues [1, 2].

It has been determined that heavy oils and bitumen contain significant quantities of rare metals, namely vanadium and nickel. Vanadium finds applications in diverse fields such as the military, metallurgical industries, animal husbandry, medicine, and others [3].

The interest in oils and natural bitumen as sources of vanadium, nickel, and other rare earth metals is driven by the observation that their metal concentrations are often comparable or even higher than those found in the most concentrated ores. However, the presence of metals and their oxides in crude oil poses challenges for its subsequent refining processes, particularly in deep refining. This is due to the fact that metals such as vanadium, iron, and nickel, which are present in the residue, act as "poisons" for catalysts during catalytic cracking. Consequently, they interfere with oil cracking and the utilization of cracking products [4].

To eliminate metals from oils, various techniques are employed, including extraction-sedimentation, adsorption, adsorption-catalytic, hydrogenation, thermal, chemical, and unconventional methods [5-9].

In numerous instances, the demetallization of oil and petroleum products is addressed through de-asphalting, hydrotreating, and hydrocracking. Methods that involve extensive feedstock degradation, such as coking and thermal contact cracking, show significant potential for substantially enhancing the production of fuel fractions and concentrating the majority of vanadium and nickel in coke [10].

The development of electrochemical techniques for metal removal, de-asphalting, and demetallization of crude oil or its fractions can be regarded as unconventional approaches. However, these methods possess certain drawbacks, such as the requirement for oil dilution with a solvent to decrease its viscosity, their inability to be implemented for in-line oil purification, and the necessity for supplementary purification or disposal of the reagents employed [11].

As the density of the oils increases, the concentration of resinous-asphaltene substances, metals, and sulfur within them also increases. These substances directly interact with the catalyst, resulting in swift poisoning, tarring, and carburization of its active surface [12].

At the same time, metals are valuable associated components, the concentration of which in oils and oil residues may be comparable to, and even higher than, their concentration in ore sources. Consequently, there is a growing interest in the demetallization of heavy petroleum raw materials, aiming to enhance efficiency, reduce processing costs, and generate metal concentrate

as a marketable product or intermediate [13-14].

The primary method for purifying hydrocarbon mixtures from sulfur and metal impurities is through hydrotreating using solid-phase catalysts. However, this process has several drawbacks, including heightened susceptibility of catalysts to poisons, excessive hydrogen consumption, and elevated pressure levels. Consequently, for smaller and medium-scale catalytic facilities, this technology proves to be unprofitable. This serves as one of the factors driving the exploration of alternative methods for purifying hydrocarbon feedstocks [15].

One unresolved approach is the utilization of diverse synthetic zeolites. The extensive range of chemical compositions inherent in zeolites allows for the manipulation of materials' physicochemical properties in a desired manner. The physicochemical properties of substances can undergo substantial alterations when their molecules are confined within pores of similar dimensions to the molecules themselves.

The distinctive structure of zeolites, with strategically positioned defects and active chemical sites, can serve as a platform for facilitating targeted chemical reactions. Zeolites are progressively being employed as catalysts [16-17].

Oil cracking in zeolites is one of the most crucial industrial applications of zeolites, defining their significance. The presence of nanoscale cavities, potential for an organized configuration of active centers, selectivity, and directed exposure to chemical groups of molecules make zeolites not only a tool of nanotechnology but also the subject of investigation.

In light of this, our research aimed to develop a refining method for oil in a continuous flow, guaranteeing the realization of a reagent-free technology while considering environmental requirements.

Materials and methods. Materials:

The following materials are used as starting materials for charge preparation and production of an active adsorbent:

– Asbestos waste (AW) obtained after hydrochloric acid leaching, provided by Kostanay Minerals JSC (Republic of Kazakhstan). The chemical composition of the AW is presented in Table 1. The material size is -2.0+1.0 mm.

Table 1. The chemical composition of the aqueous waste (AW) after leaching

Component	Chemical formula	Content, % by weight
Silicon oxide	$SiO_2 \cdot nH_2O$	90.0
Magnesium oxide	MgO	6.5
Iron oxide	Fe_2O_3	2.5
Impurities	–	1.0

– Calcium carbonate of the chemically pure «CP» brand is a product with a moisture mass fraction not exceeding 1.0%. It is a chemically pure product with a high content of calcium carbonate, where the mass fraction of calcium carbonate and magnesium expressed in terms of CaCO3 ranges from 99% to 99.9%. There are no impurities present in calcium carbonate. The mass fraction of the residue that is insoluble in hydrochloric acid is 0.22%, and the mass fraction of water-soluble substances is 0.014%.

– The sodium carbonate «CP» brand is a calcined preparation containing at least 99.8% sodium carbonate, with no more than 0.02% of insoluble substances in water.

Experimental technique. Adsorbents were synthesized by incorporating soluble nickel and molybdenum salts, specifically seven-way nickel sulfate and ammonium molybdate. Sodium lignosulfonate (2.0%) was utilized as a binder and plasticizer, maintaining the composition of

the resulting product even at elevated temperatures. The components listed in Table 2 were mixed and subjected to joint grinding, resulting in particle sizes below 100 microns. Subsequently, plastic molding was conducted with the addition of binding materials. The resulting molded materials were subjected to firing at a temperature of 650 °C for a duration of 48 hours.

Table 2. Composition of the active component for obtaining a catalyst by impregnation

Compound Name	Formula	Content, %
Serpentine after leaching	SiO_2	38,0
Calcium Carbonate	$CaCO_3$	44,0
Caustic soda	Na_2CO_3	5,0
Nickel Sulfate	$NiSO_4 \cdot 7H_2O$	3,0
Ammonium Molybdate	$(NH_4)2MoO_4$	10,0
Total		100,0

Methods of determination and used equipment for the study. To ascertain the mineralogical composition and structure of the formation, the synthetic adsorbent obtained, with the inclusion of various activator additives, was examined using the following equipment:

X-ray data were acquired using the BRUKER D8 ADVANCE instrument. X-ray fluorescence analysis was conducted on a Venus 200 Panalytical B.V. wave dispersion spectrometer located in Holland. Chemical analysis of the samples was carried out on an optical emission spectrometer with inductively coupled plasma, specifically the Optima 2000 DV by PerkinElmer in the USA. The elemental and phase composition of the samples was mapped using the JXA-8230 electron probe analyzer manufactured by JEOL in Japan. Thermal analysis was performed using the synchronous thermal analysis device STA 449 F3 Jupiter. The obtained results were processed utilizing the NETZSCH Proteus program.

Studies were conducted on the activity of samples using the electronic paramagnetic resonator (EPR) of the JECFA 200 spectrometer (Jeol, Japan).

To ascertain the total specific surface value of meso- and macroporous substances and materials, a SORBTOMETER-M device was employed utilizing the thermal desorption method of gas-adsorbate, specifically the BET method (based on the Brunaer-Emmett-Teller equation). The specific surface area measurement range spans from 0.3 to 2000 m2/g.

The microstructure and microanalysis of the adsorbents were investigated using the Quanta 3D 200i scanning electron microscope (SEM) from the United States. The SEM was operated at an accelerated voltage of 20 kilovolts (kV) and a pressure of 0.003 pascal (Pa).

Results and discussion. Characteristics of the synthetic adsorbents that were obtained
Microscopic analysis – SEM of the obtained adsorbents

Figure 1 displays electron micrographs of the synthetic adsorbent acquired at varying magnifications.

Based on the data presented in Figure 1, the synthetic adsorbent exhibits visible fine-needle fibers, which are composed of wollastonite-type needle fibers. The average length of these fibers ranges from 5 to 30 microns, while the average length of the needle phase is between 12 and 30 microns. Additionally, the average diameter of the needle phase measures 0.5 to 5 microns. This microscopic analysis confirms the formation of a wollastonite-type compound, which shares identical characteristics with natural wollastonite.
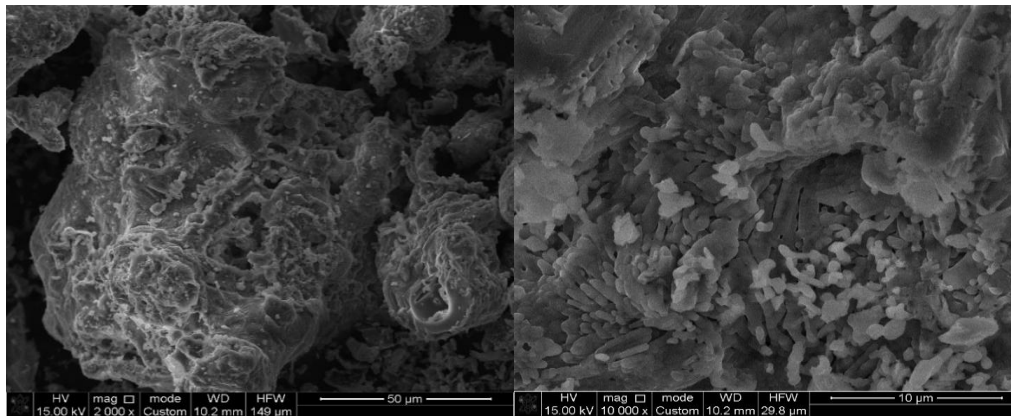
Figure 1. Electron micrography was conducted to visualize the synthesized adsorbent obtained

X-ray diffraction analysis of adsorbents
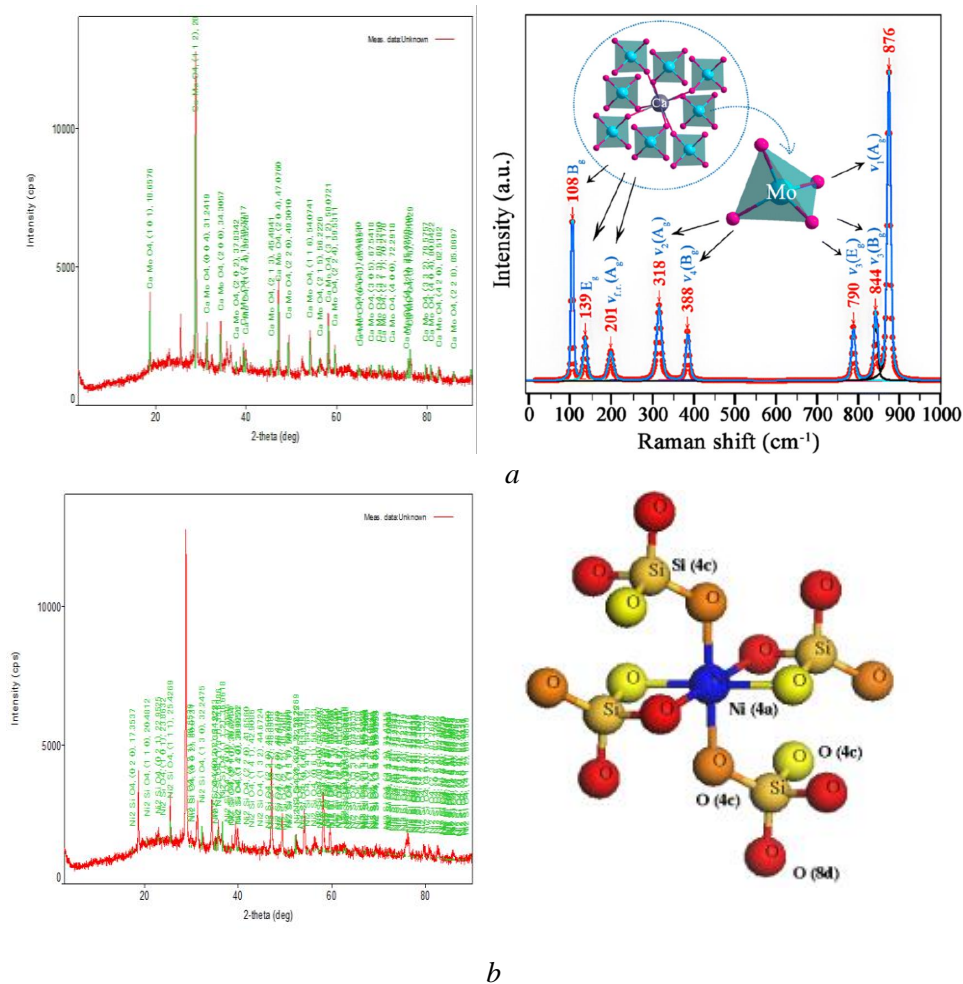The Figure 2 presents the X-ray diffraction analysis of the adsorbents.

a

b

Figure 2. Illustrates the X-ray diffraction analysis of the adsorbents, specifically showcasing (a) a molybdenum compound and (b) a nickel compound

The X-ray diffraction analysis of the synthetic adsorbent (Figure 2) revealed the presence of newly formed minerals, specifically pollevite and liebenbergite. Pollevite crystallizes in a tetragonal syngony and exhibits dipyramidal and tabular crystal structures. It has a density range of 4.25-4.52 g/cm^3 and possesses an island structure. Moreover, it contains up to 72% $MoO2$ and 10% Mo. On the other hand, liebenbergite is characterized by small pyramidal crystals, and its grains crystallize in a rhombic syngony. It has a density range of 4.50-4.60 g/cm^3 and contains up to 56% nickel.

X-ray phase and X-ray analysis of samples

To ensure the reliability of the results regarding the formation of synthetic wollastonite, X-ray phase analyses were conducted on the acquired samples (Figure 3, Table 3).
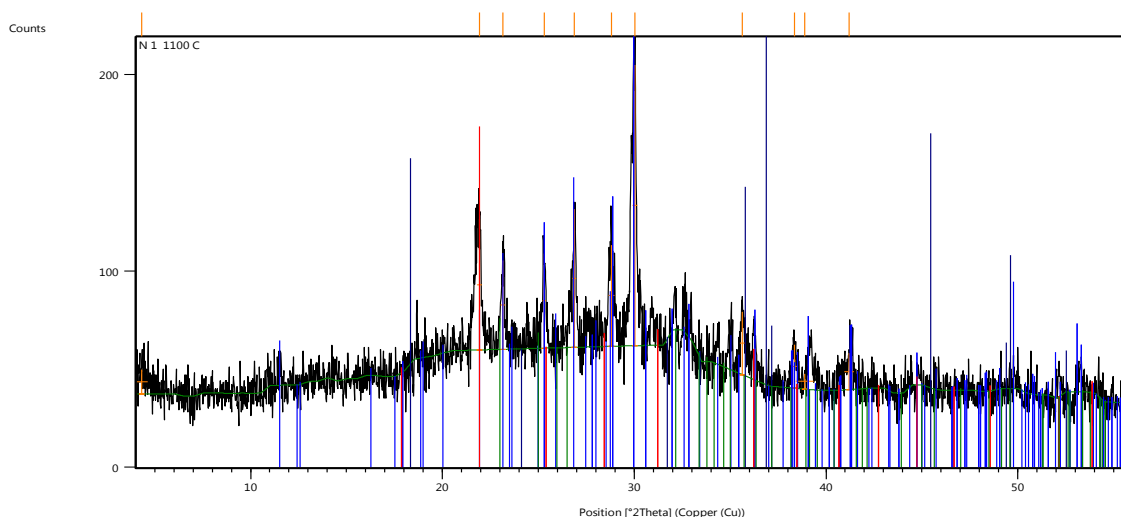

Figure 3. Diffractogram of the synthesized adsorbent

Table 3. Results of semi-quantitative analysis of crystal phases

Phase name	Formula	Content, %
Wollastonite	$CaSiO_3$	85,0
Powellite, syn	$CaMoO_4$	6,0
liebenbergite, high, syn	Ni_2SiO_4	2,0
Silicon Oxide	SiO_2	7,0

The data obtained from X-ray phase analysis confirms that a well-formulated synthetic material is obtained during high-temperature synthesis, with a content of the main mineral in the form of wollastonite $Ca(SiO3)$ at 80-85%. Additionally, the additives of activators in the form of ammonium molybdate and nickel sulfate contribute to the formation of new minerals, specifically vellite and liebenbergite. These minerals are completely synthesized based on free calcium and silicon compounds. The presence of these compounds does not deteriorate the physicochemical properties of the synthesized material; instead, it aids in the development of a porous structure due to their low density and specific surface area of minerals. Additionally, the resultant product comprises 7% of free amorphous silica in the form of cristoballite.

Comparison of several adsorbents for adsorption properties. EPR analysis

In order to determine and compare the activity of materials, the characteristics were examined through the utilization of Electron Paramagnetic Resonance (EPR).

The behavior of magnetic moments within a magnetic field is contingent upon several interactions involving unpaired electrons, both amongst themselves and with the surrounding environment. The most pivotal among these interactions include spin-spin and spin-orbital interactions, as well as interactions between unpaired electrons and the localized nuclei (hyperfine interactions). Additionally, interactions with the electrostatic potential generated by ions in the immediate environment at the unpaired electron's location, along with other interactions, also play a significant role.

Registration conditions: Frequency of 9.445 gigahertz (GHz) within a magnetic field of Fc = 340 millitesla (mT). Scanning range of 7.50 mT. Frequency and amplitude of modulation Fr = 100 kilohertz (kHz). Width of 0.6 mT. EPR spectra were recorded at room temperature using a JASCO 200 spectrometer (Jeol, Japan) while employing the Mn(2+) standard in MgO.

The initial samples were examined, as well as after undergoing treatment (annealing) in a hydrogen plasma for 30 minutes.
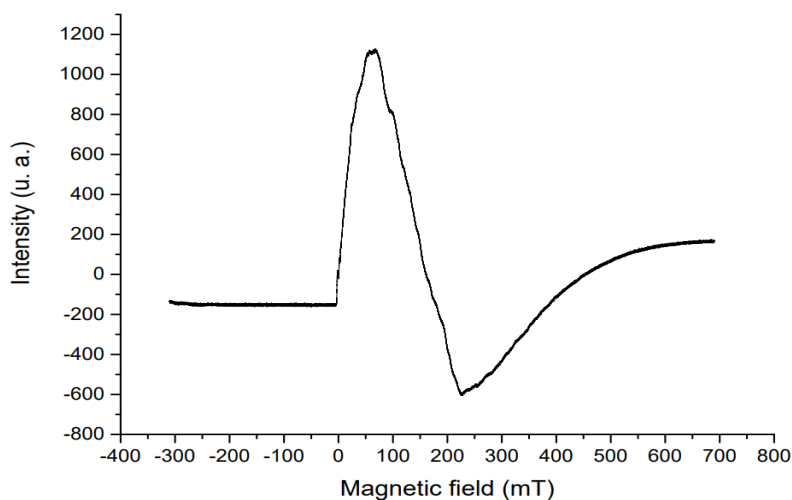

Figure 4. Initial asbestos wastes

An EPR signal of low intensity was detected in sample 1 (initial AW). Particles with unbalanced charge could potentially form due to the crushing and grinding of serpentinite ores. The composition of the studied samples comprises numerous chemical elements that demonstrate significant magnetic activity, including cobalt, chromium, manganese, sodium, potassium, titanium, and others.

The second sample (AW after leaching) exhibits a symmetrical spectrum, suggesting a homogeneous distribution of paramagnetic centers within the sample.

Sample 2 exhibits a comparatively lower intensity of the intense signal when compared to sample 3. Following the annealing process in hydrogen plasma, the intensity experiences a decrease over a duration of 30 minutes. The treatment in hydrogen plasma effectively passivates the uncompensated charge (paramagnetic centers) present in the sample.
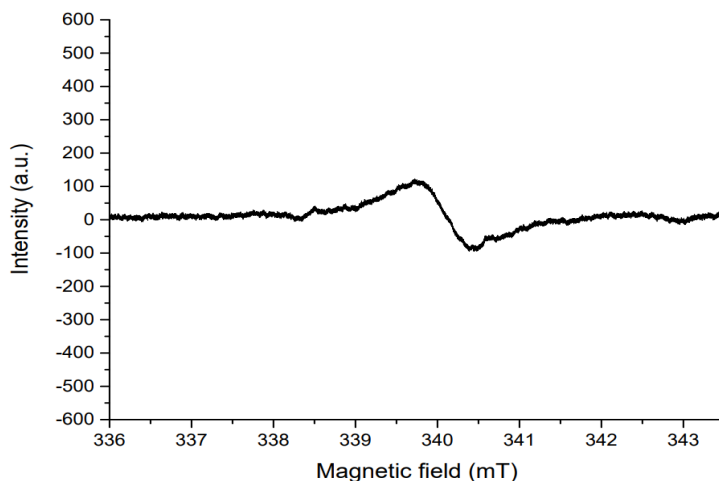
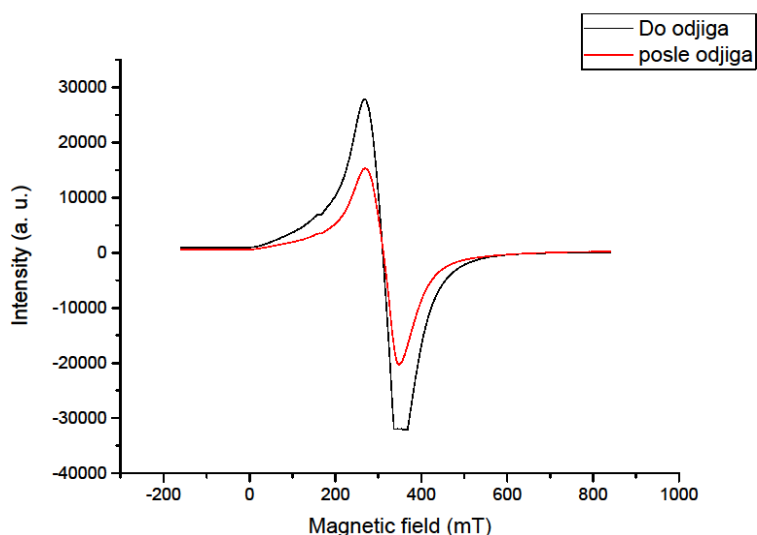**Figure 5.** AW after leaching

Figure 6. Synthetic adsorbent

The third sample (Synthetic adsorbent) exhibits a highly pronounced signal. Following exposure to hydrogen plasma treatment (annealing), the intensity exhibits a gradual decrease over a period of 30 minutes. The hydrogen plasma treatment effectively passivates the uncompensated charge (paramagnetic centers - PMCs) present in the sample. Hydrogen particles selectively bind to low-energy uncompensated charges, while the stable PMCs retain their activity.

The EPR signal is observed in sample 4 (Factory zeolite). The EPR signal is influenced by multiple paramagnetic centers. The fourth sample exhibits a symmetrical spectrum, suggesting a homogeneous dispersion of paramagnetic centers within the sample.

There may be multiple explanations for the emergence of an electron paramagnetic resonance (EPR) signal in these samples, including the presence of unpaired electrons within the Sao or Cao-Ca or Ca-Ca chains. The application of high-temperature treatment during the synthesis process promotes the generation of particles with an unbalanced charge. Furthermore, the intensity of the EPR signal influences the grinding process of the substances.
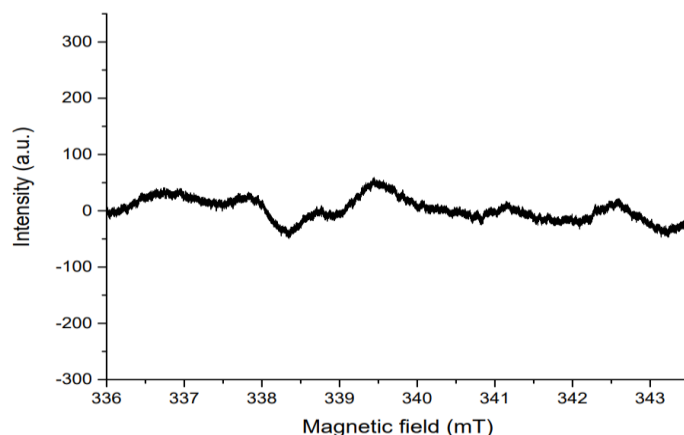
Figure 7. Zeolite from factory

Based on the analyses conducted, it can be inferred that the synthetic adsorbent possesses a significant concentration of paramagnetic centers, signifying exceptional adsorption characteristics even under elevated temperatures in a hydrogen plasma environment.

The BET method. The selection of an adsorbent for a specific practical problem is primarily determined by information regarding its porous structure. To appropriately choose the appropriate sorbent, it is necessary to ascertain whether the pore distribution in terms of radii aligns well with the molecular sizes of the specified sorbates, ensuring their effective penetration into the granules of the sorbent.

The adsorption potential is most pronounced in micropores due to the combined influence of adjacent wall adsorption potentials. Consequently, the affinity for adsorption of molecules capable of entering micropores is significantly greater compared to molecules confined within meso- or macropores.

In order to ascertain the specific surface area and pore distribution across the radii of porous bodies, the BET theory method was employed (Table 4). The measurement was conducted using a SORBTOMETER-M device.

To compare the specific pore surface, the following materials were subjected to analysis: initial asbestos waste, asbestos waste after the leaching process, pure zeolite, and the adsorbent synthesized by our team. The measurement was performed using a SORBTOMETER-M device.

Table 4. Specific surface area of materials

Sample	Surface area, m^2/g
Asbestos wastes	8,0-9,0
Asbestos waste after leaching	10,0-13,0
Synthesized adsorbent	0,15-0,30
Zeolite	90,0-95,0

As depicted in the table, the specific pore surface values of the resultant active component are relatively low. Micropores play a crucial role in both the material's activity and demetallization process, with their diameters (up to 2 nm) aligning with the sizes of adsorbed molecules. The development of the primary portion of the active component's inner surface is ensured by these micropores, and this characteristic will positively influence the properties of the synthetic adsorbent.

The removal of metal impurities from heavy oil raw materials through the process of cleaning. The thermocontact demetallization process involves the short-term interaction of crude oil with an adsorbent in a two-section reactor, which is heated to temperatures ranging from 300 to 450 °C. This process leads to the formation of hydrocarbon vapors when the raw materials come into contact with the heated adsorbent. These vapors, along with water vapor, are then transported through the gas outlet line.

The tar obtained from the slow coking unit of Pavlodar Petrochemical Plant LLP is a viscous immobile liquid. It contains water in a mass fraction of up to 0.1 wt.%, with a metal content of vanadium ranging from 400 to 600.0 mcg/kg, nickel ranging from 50 to 70.0 mcg/kg, and iron ranging from 30 to 70.0 mcg/kg. The mass fraction of sulfur is between 2.5% and 3.5%, with an ash content of -0.02 wt.%. The coking ability is 18 wt.%, and the density at 20 °C is 1000.0 kg/m3. The boiling point of the tar is 380 °C.

Table 7. Metal and Sulfur Content in Tar Composition Pre- and Post-Test, Based on Process Temperature

Raw material and type of adsorbent	T, °C	The content of the elements			
		V, mkg/kg	Ni, mkg/kg	Fe, mkg/kg	S, %
Initial tar	–	540,0	50,0	50,0	3,50
Modified adsorbent	320	50,0	50,0	50,0	2,65
	340	50,0	50,0	50,0	2,61
	360	45,0	43,0	40,0	2,58
	380	33,0	35,0	33,0	2,53
	400	30,0	31,0	21,0	2,45
	410	28,0	26,0	12,0	2,39
	420	23,0	22,0	7,0	1,97

As evidenced by the table, the demetallization of heavy oil raw materials exhibits an optimal temperature range of 380-410 °C. Simultaneously, the tar's vanadium content decreases from 50 to 28 micrograms per kilogram, the nickel content decreases from 50 to 26 micrograms per kilogram, iron decreases from 50 to 12 micrograms per kilogram, and sulfur decreases from 2.73% to 2.39%. Under these conditions, the maximum amount of metal recovery is achieved, ranging from 40-55%.

With a temperature exceeding 410 °C, the tar initiates boiling and coking reactions, resulting in detrimental consequences for the demetallization process. Hence, it is recommended to conduct the thermal adsorption process at a temperature not exceeding 410-420 °C.

Conclusion. Thus, methods of dehydration and active component preparation were devised to establish a matrix foundation for synthetic adsorbents using chrysotile asbestos production waste as composite materials. The physicochemical characteristics of the resulting adsorbents were assessed. Through the high-temperature synthesis of adsorbents and the inclusion of molybdenum oxychloride and nickel sulfate as activators, novel minerals in the form of vellite and liebenbergite were generated. These minerals were fully synthesized from available calcium and silicon compounds.

The presence of such compounds contributes to the formation of a porous structure due to their low density and specific surface area of minerals. In order to confirm the analyses, the adsorption properties of the composite catalysts obtained were practically investigated. The optimal temperature range for demetallization of heavy petroleum raw materials is 380-410 °C, with a contact time of 60 minutes and a pressure of 8-10 atm. Simultaneously, the vanadium

content in tar decreases from 540 to 52 micrograms/kg, the nickel content from 50 to 26 micrograms/kg, iron from 50 to 12 micrograms/kg, and sulfur from 3.50% to 2.39%.

An adsorbent, in the form of synthetic wollastonite derived from asbestos chrysotile waste, incorporates active nickel and molybdenum components. This adsorbent facilitates the purification of heavy oil raw materials, resulting in enhanced quality of the final oil products. Additionally, it enables the extraction of valuable metals like vanadium, nickel, and iron, which can be obtained as marketable products.

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