

STUDY OF SURFACE PROPERTIES OF MAGNETIC SORBENTS

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Abstract

Ensuring environmental safety and creation of favorable environment is one of the directions of the National Strategy of Sustainable Social and Economic Development of the Republic of Belarus for the period up to 2030. One of the methods of rehabilitation of polluted and other ecologically destabilized territories is the use of sorbents. Magnetic sorbents, which combine high sorption and magnetic properties, have been recently considered as promising sorbents. The latter allow controlling the movement of the sorbent in the treated medium.

The paper presents the results of the study of surface properties of sorption materials (magnetic sorbents) obtained from iron-containing industrial wastes, composite magnetic sorbents obtained on the basis of activated carbon particles and magnetic sorbents, and initial activated carbon particles. The sizes of the obtained particles and elemental composition were determined by scanning electron microscopy. The integral acidity of the surface of the obtained samples of sorbents and initial particles of activated carbon was evaluated by pH-metry. Acid-base properties of the surface of the obtained samples were investigated by spectrophotometric method using Gammet indicators. Identification of surface centers of the investigated composites was carried out by comparing the values of pK_a constants corresponding to the peaks with the known ionization constants of functional groups. The amount of detected functional groups was determined by Boehm titration. It was found that CMC-105 has a higher adsorption activity to a wide range of organic compounds than the initial particles MS-105 and AC, which indicates a synergistic enhancement of the sorption properties of the obtained CMC. Based on the results obtained, the mechanism of sorption of organic compounds by the studied sorbents is given.

Keywords: iron-containing waste, raw materials, processing, method, recycling.

ИЗУЧЕНИЕ СВОЙСТВ ПОВЕРХНОСТИ МАГНИТНЫХ СОРБЕНТОВ

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Реферат

Обеспечение экологической безопасности и создание благоприятной окружающей среды является одним из направлений Национальной стратегии устойчивого социально-экономического развития Республики Беларусь на период до 2030 года. Одним из методов обеспечения реабилитации загрязненных и иных экологически дестабилизированных территорий является применение сорбентов. В качестве перспективных сорбентов в последнее время рассматривают магнитные сорбенты, объединяющие в себе высокие сорбционные и магнитные свойства. Последние позволяют контролировать перемещение сорбента в обрабатываемой среде.

В работе представлены результаты исследования свойств поверхности сорбционных материалов (магнитных сорбентов), полученных из железосодержащих отходов производства, композиционных магнитных сорбентов, полученных на основе частиц активированного угля и магнитных сорбентов, и исходных частиц активированного угля. Методом сканирующей электронной микроскопии определены размеры полученных частиц, а также элементный состав. Методом pH-метрии проведена оценка интегральной кислотности поверхности полученных образцов сорбентов, а также исходных частиц активированного угля. Исследованы кислотно-основные свойства поверхности полученных образцов спектрофотометрическим методом с помощью индикаторов Гаммета. Проведена идентификация поверхностных центров исследуемых материалов путем сопоставления значений констант pK_a , соответствующих пикам, с известными константами ионизации функциональных групп. Титрованием по методу Boehm определено количество обнаруженных функциональных групп. Установлено, что сорбент КМС-105 обладает более высокой адсорбционной активностью к широкому кругу органических соединений, в сравнении с материалами из которых данный сорбент был получен, что свидетельствует о синергетическом усилении сорбционных свойств полученного КМС-105. На основании полученных результатов приведен механизм сорбции органических соединений исследуемыми сорбентами, а также сделан вывод о типе загрязняющих веществ, адсорбция которых полученными материалами будет наиболее эффективной и целесообразной.

Ключевые слова: железосодержащий отход, сырье, переработка, метод, повторное использование.

Introduction

The development of new methods for processing industrial waste is a pressing and important task facing modern researchers. Of particular interest are finely dispersed iron-containing wastes characterized by a multicomponent composition. Currently, there are no registered uses for such wastes [1]. One method of processing such wastes is to extract iron (II, III) salts from them and then obtain the target product [2–6]. In this work, magnetic particles were obtained from the isolated iron (II, III) salts, which were considered as sorbents and magnetic cores for the production of composite magnetic sorbents (CMS) based on them [7].

When selecting a sorbent for use in certain technologies for cleaning contaminated environments, the characteristics of the sorbent surface play an important role. It is heterogeneous and consists of a set of Lewis and Brønsted adsorption centers that are quite strongly bound to the solid. On the surface of any sorbent, there are a number of active centers with different dissociation constants (K_a). One of the primary tasks

in studying the properties of the sorbent surface is the experimental study and determination of the type and number of these active centers. A complete description of the surface properties of solids involves not only determining the total acidity, but also quantitatively assessing the content of active centers and differentiating them by strength and type.

The aim of this work is to study the surface properties of sorbents obtained from finely dispersed iron-containing waste (FDIW) by chemical co-precipitation of iron (II, III) hydroxides.

Research methods and baseline data

One of the most common methods for obtaining magnetic sorbents is the Massart co-precipitation method [8]. This method was modified for the synthesis of magnetic sorbents (MS) and composite magnetic sorbents (CMS) from FDIW. Scale was used as FDIW. This waste is a mixture of mainly iron (II, III) oxides formed by the direct action of oxygen when metals are heated in air.

The essence of the modified Massart method for obtaining magnetic sorbents from scale was as follows: iron ions were pre-leached from the scale with a sulfuric acid solution. A hot KOH solution was added to the resulting solution to a pH of 8–10. The resulting suspension was allowed to settle for 30 minutes, after which it was filtered. The retained sediment was washed with distilled water to a pH 7. The sediment was then dried at 105 °C to a constant weight. CMS was obtained by co-precipitation of iron (II, III) hydroxides on the surface of activated carbon (AC). The resulting particles were then washed and dried at 105 °C. The resulting sorbent samples were designated MS-105 and CMS-105.

The surface structure, particle size, and elemental composition of the obtained MS-105 and CMS-105 samples were studied using scanning electron microscopy (SEM) on a JEOL JSM-5610 LV microscope.

The acid-base properties of the surface of the obtained samples were studied using the pH measurement method. The essence of the method was that electrodes were immersed in 30 cm³ of double-distilled water, and after the pH values stabilized, 0.3 g of the sample was added. Then, the change in pH values over time was recorded, and $pH = f(t)$ curves were plotted. Glass and silver-chloride electrodes were used. Measurements were taken at room temperature (20.9 °C).

The distribution of surface centers according to acid-base properties was studied by spectrometric method using Hammett indicators (16 indicators with dissociation constants (pK_a) ranging from -0.29 to +12.9) and determined according to the method described in [9–11].

For the MS-105 sorbent, indicator solutions with a concentration of 10^{-4} mol/dm³ were prepared, and for CMS-105 and AC, 10^{-3} mol/dm³. Next, a certain volume of indicator solution (V_{ind}) was added to graduated test tubes and brought to 5 cm³ with distilled water. After 30 min, the optical density of the resulting solution (D_0) was measured.

Next, 0.1 g of the sorbent sample was added to the resulting indicator solutions, mixed, and left for 120 min, after which the solution was separated from the sample (using a magnet for MS-105 and CMS-105, and decantation for AC) and its optical density (D_1) was measured.

A blank experiment was conducted in parallel. For this purpose, 0.1 g of sorbent was placed in 5 cm³ of distilled water, the suspension was stirred, after 120 min the decantate was brought to 5 cm³ and the optical density of the new solution (D_2) was measured.

The concentration of active centers (q , mmol/g) corresponding to specific pK_a values, equivalent to the amount of indicator adsorbed on the sample surface, was calculated using the following equation

$$q = \frac{C_{ind} \cdot V_{ind}}{D_0} \cdot \left(\frac{D_0 - D_1}{m_1} + \frac{D_0 - D_2}{m_2} \right), \quad (1)$$

where C_{ind} is the concentration of the indicator in the solution (mmol/dm³), V_{ind} is the volume of the indicator taken for the study (dm³), m_1 and m_2 are the masses of the test substance in the first and second samples (g), D_0 is the optical density of the initial indicator solution, D_1 and D_2 are the optical densities of the indicator solution after treatment with the sorbent sample and in a blank experiment.

The distribution curves of adsorption centers by acid strength on the surface of the samples were plotted in the coordinates $q(pK_a) = f(pK_a)$.

Table 1 – Elemental composition of the obtained MS-105 and CMS-105 samples

| Sample | Element content, wt. % | | | | | | |
|---------|------------------------|------|------|------|------|-------|-------|
| | Al | Si | S | K | Cr | Fe | O |
| MS-105 | 0,36 | 0,71 | 0,45 | 0,96 | 0,86 | 73,30 | 23,36 |
| CMS-105 | 1,16 | 2,31 | 0,26 | 1,68 | 0,75 | 69,10 | 24,74 |

As can be seen in Figure 2, when sample MS-105 (No. 1) interacts with deionized water, a smooth increase in the basicity of the medium is observed, which indicates the presence of already hydroxylated Brønsted centers.

A sharp change in pH to the basic range during the first 40 seconds after immersion of the CMS-105 sample (No. 2) in deionized water indicates the presence of rapidly hydrated Lewis aprotic centers, which, when interacting with water, transform into Brønsted centers.

When sample AC (No. 3) interacts with deionized water, an increase in the acidity of the medium is observed during the first

To characterize the acid properties of the obtained sorbents, the surface acidity function was used, which was calculated using the equation

$$H_0 = \frac{\sum q(pK_a) \cdot pK_a}{\sum q(pK_a)}. \quad (2)$$

The number of functional groups was determined by titration using the Boehm method [12–13]. We placed 0.1 g of the sample into 20 cm³ conical flasks. We added 10 cm³ of 0.011 M of the corresponding solution to the samples: sodium bicarbonate solution to determine carboxyl groups; sodium carbonate solution to determine the sum of carboxyl and lactone groups; hydrochloric acid solution – to determine basic groups. The mixture was shaken and left for 24 hours to reach equilibrium at room temperature. After filtration, three 3 cm³ samples were taken from the equilibrium solutions, transferred to 20 cm³ conical flasks, and then titrated with hydrochloric acid and sodium hydroxide solutions with methyl orange and phenolphthalein indicators, respectively.

Results and discussion

The particle sizes of the samples obtained were determined using SEM; the data are presented as micrographs of the particles (Figure 1), from which their shape and size can be determined based on the resolution of the photographs.

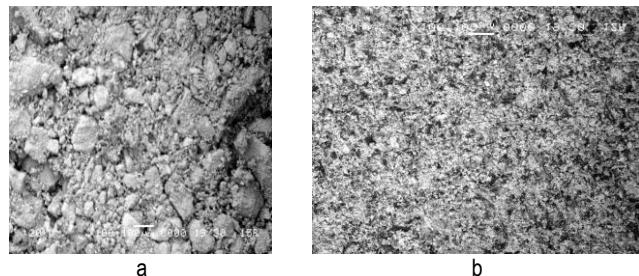


Figure 1 – Micrographs of samples MS-105 (a) and CMS-105 (b)

The size of the particles obtained ranges from 20 to 200 μ m for MS-105 and from 2 to 15 μ m for CMS-105.

The elemental composition of the materials obtained was determined. The results are presented in Table 1.

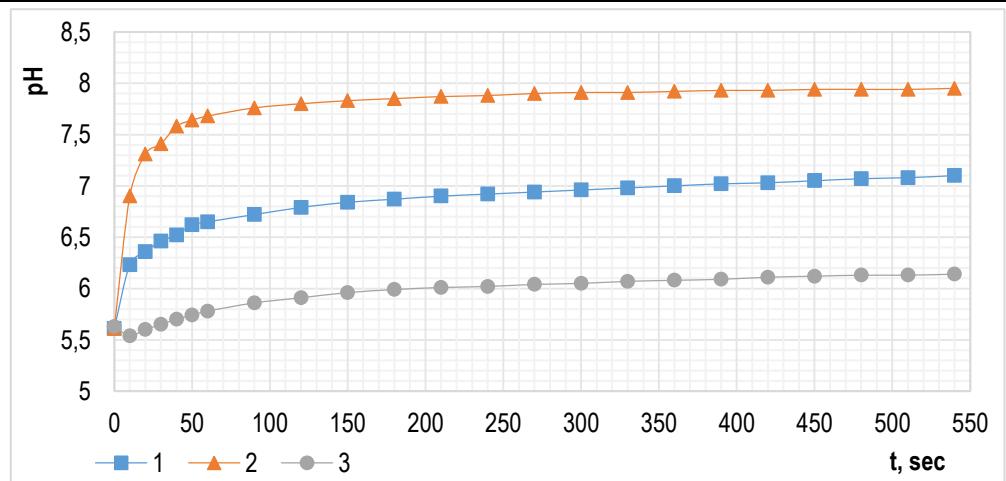
As can be seen from the table, the iron content in the obtained magnetic sorbents is higher than in CMS. The obtained samples also contain Al and Cr impurities, which, according to [14], may be present in scale. The S and K content is explained by the residual K_2SO_4 content (part of the stock solution) on the particle surface. The absence of carbon in the composition of CMS-105 is explained by the specifics of the sample analysis using the SEM method.

To characterize the surface of the obtained samples and establish the belonging of surface centers to the Brønsted or Lewis type, the integral acidity of their surface was evaluated using pH-metry.

Figure 2 shows the curves of changes in the pH values of aqueous suspensions of sorbent samples over time.

20 seconds, which may indicate the presence of Lewis acid centers on the sorbent surface. A further increase in pH indicates that OH-groups are associated with the sorbent surface, which exhibit weak acidic properties and donate H^+ protons. However, the acidity of such centers is weak, and with further adsorption of water molecules, an increase in pH values is observed, which indicates the weakly basic properties of the surface [15].

To determine the number of adsorption centers, adsorption center distribution curves (ACDC) were constructed on the surface of the sorbents (Figures 3, 4).



1 – aqueous suspension of sorbent MS-105; 2 – aqueous suspension of sorbent CMS-105; 3 – aqueous suspension of sorbent AC
Figure 2 – Curves showing changes in the pH values of aqueous suspensions of sorbent samples over time

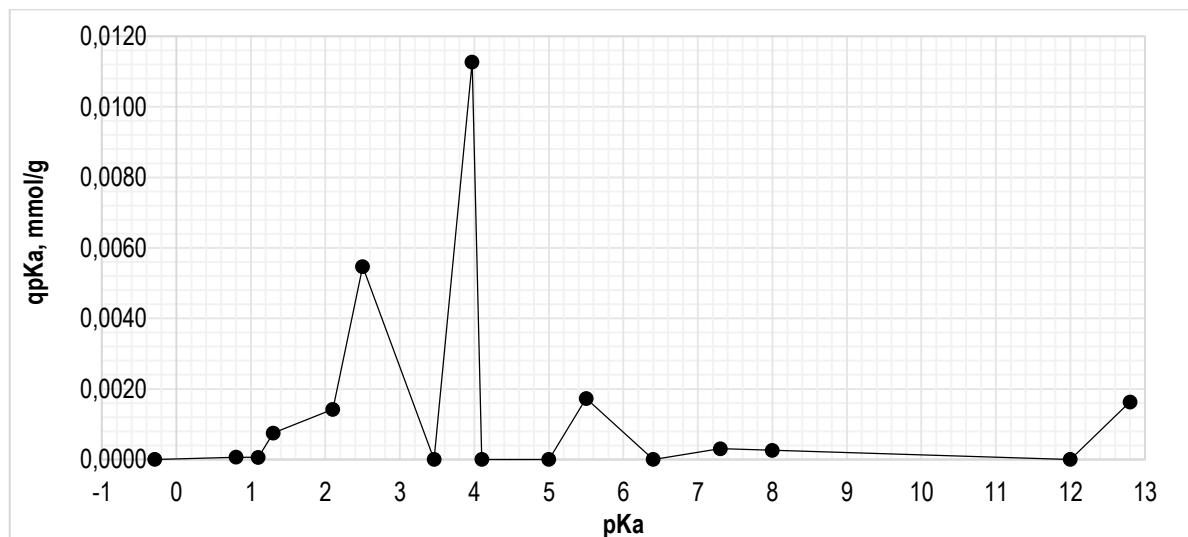


Figure 3 – Distribution of adsorption centers on the surface of MS-105

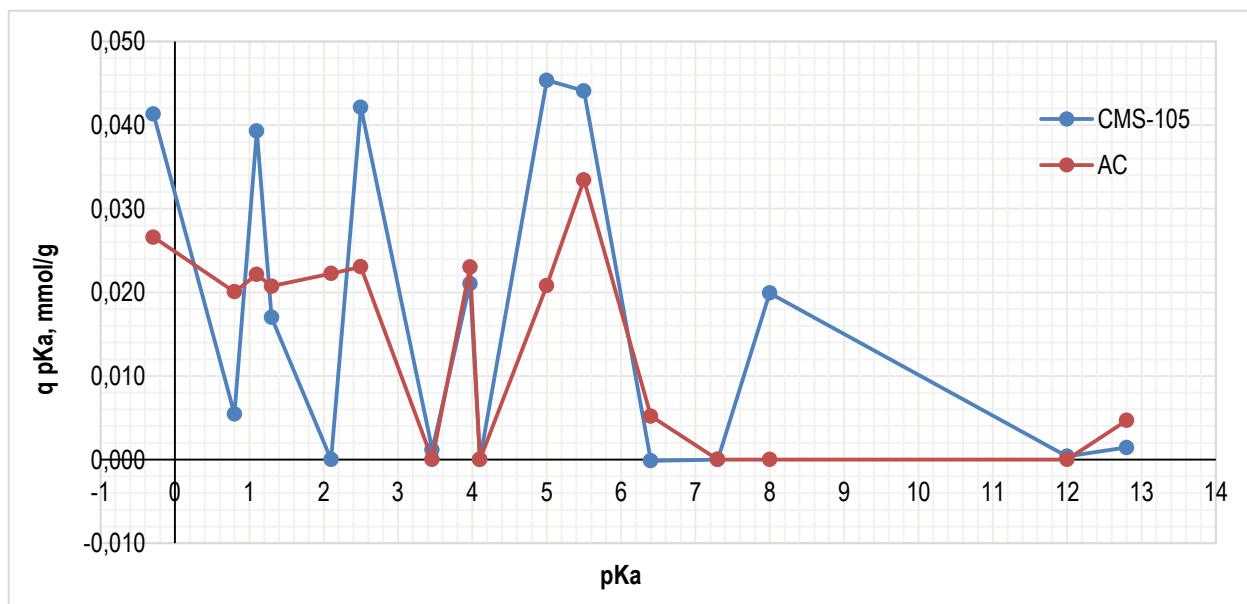


Figure 4 – Distribution of adsorption centers on the surface of CMS-105 and AC

The distribution of active centers across a wide energy range of the controlled parameter (pK_a) allows us to determine the presence or absence of certain groups of adsorption centers.

Analysis of the obtained ACDC curves shows that in the presence of water, the surface of MS-105 is dominated by Brønsted acid centers ($pK_a = 1.2 - 5.5$), which are hydroxyl groups of the acid type, and neutral Brønsted centers ($pK_a = 7.3$).

On the surface of KMS-105 and AC in the presence of water, Lewis basic centers ($pK_a = -0.29$) and Brønsted acidic centers ($pK_a = 1.1 - 5.5$) predominate, which indicates the presence of acidic centers on the surface of the studied samples in the presence of water, which tend to donate a free hydrogen proton. It is also worth noting that the number of adsorption centers detected on the surface of KMS-105 exceeds the number of these centers on the surface of MS-105 and AC. However, the surface of AC is characterized by a large number of types of active centers.

Having determined the acidity function H_0 for MS-105, CMS-105, and AC, which amounted to 1.92, 1.91, and 1.67, respectively, it was concluded that the surface of the obtained sorbents has weak acidic properties, although they are more pronounced in AC.

The nature of the active centers on the surface of the obtained sorbents was determined by comparing the pK_a values corresponding to the peaks shown in Figures 3 and 4 with the known ionization constants of the active centers. The data obtained indicate that the surface of the obtained sorbents mainly contains various acidic centers (Table 2).

The content of functional groups in the sample under study is shown in Table 3, the data in which indicate the presence of different types of acid Brønsted centers on the surface of the sorbent. The pK_a range of 3.6–4.9 in the spectrum corresponds to easily dissociating carboxyl groups. The presence of bands in the pK_a 5.0–6.4 region is associated with the presence of weaker carboxyl groups in the sorbent structure that do not have acceptor substituents in their immediate vicinity. The chemical environment of these Brønsted centers is probably represented by variously substituted aromatic rings, carbohydrate, and aliphatic fragments.

In a study of the surface active centers of iron(III) oxide, the authors [19] also found predominantly weak acid Brønsted centers.

The data in Table 3 show that the surface of the samples under study is characterized by the presence of a larger number of basic functional groups. Based on this, there is a need for a more detailed study of the basic Lewis-type centers.

It is also worth noting that CMS-105, obtained from materials of different nature, is characterized by a pronounced synergistic effect of the composite material.

Table 4 – Active centers located on the surface of the studied sorbents

| Sorbent | MS-105 | CMS-105 | AC |
|------------------------|---|---|--|
| Functional groups (FG) | FeO-H ⁺ , Fe-O-H ⁶⁰ , Si-OH ^{δ-} | AlO; FeO-H ⁺ , R-CH-(CH ₂) _n -C=O, Fe-O-H ⁶⁰ , Si-OH ^{δ-} | Ar-OH, R-CH-(CH ₂) _n -C=O, -COOH, Si-OH ^{δ-} |
| pKa | 0-7, 7, 7-14 | -1,7-0, 0-7, 7-14 | -1,7-0, 0-7, 7-14 |
| Acid-base centers | Strong acid, weak acid, weak base Brønsted centers | Lewis bases, strong acids, weak acids, weak bases, Brønsted centers | Lewis bases, strong acids, weak acids, weak bases, Brønsted centers |

Table 5 – Sorption properties of the sorbent samples studied

| Sorbent | Sorption capacity value relative (mg/g) to | | | |
|---------|--|------------------|------------------|--------------------|
| | CSA | Fe ³⁺ | Cu ²⁺ | petroleum products |
| MS-105 | 167,02 | 0,70 | 30,60 | 1110,00 |
| CMS-105 | 185,00 | 1,76 | 46,70 | 1450,00 |
| AC | 81,10 | 2,09 | 21,80 | 2430,00 |

Studies have shown that magnetic sorbents (MS-105) and composite magnetic sorbents (CMS-105) obtained from finely dispersed iron-containing waste have pronounced sorption properties.

Analysis of the data presented in Table 5 confirms the direct dependence of the sorption capacity of materials on their acid-base properties. The KMS-105 sample, which has the largest number of basic and acidic centers, demonstrated the highest efficiency in relation to cationic surfactants (CSA), Fe³⁺ and Cu²⁺ ions, and petroleum products, exceeding the performance of MS-105 and AC (activated carbon) in most parameters.

AC showed the highest result in relation to petroleum products (2430.00 mg/g), while KMS-105 demonstrated a sorption capacity of 1450.00 mg/g. Despite this, KMS-105 proved to be more effective for the sorption of heavy metal ions (Fe³⁺ and Cu²⁺) and surfactants. These results confirm that the creation of a composite material based on magnetic

Table 2 – Types of active centers on the surface of MS-105, CMS-105, and AC sorbents

| Sorbent | pKa | Functional groups |
|---------|---------------|---|
| MS-105 | 1,2, 2,1, 2,5 | Strong Brønsted acid sites (hydroxyl groups) [16] |
| | 4,0 5,5 | Weak Brønsted acid sites [18] |
| | 7,3 | Neutral centers [17] |
| | 12,8 | Weak Brønsted bases [20] |
| CMS-105 | -0,29-0 | Lewis's weak foundation [17] |
| | 0,8-3,97 | Strong Brønsted acid sites (hydroxyl groups) [16] |
| | 5 | Weak carboxyl groups [18] |
| | 8 | Lactone groups [18] |
| AC | 12,8 | Weak silanol groups [18] |
| | -0,29-0 | Lewis's weak foundation [17] |
| | 0-2,5, 3,97 | Strong Brønsted acid sites (hydroxyl groups) [16] |
| | 5-5,5 | Weak Brønsted acid sites [18] |
| | 12,8 | Weak Brønsted bases [18] |

Table 3 – Titration results using the Boehm method

| Sorbent | Number of functional groups, mmol/g | | |
|---------|-------------------------------------|----------|-------|
| | Carboxylic | Lactones | Basic |
| MS-105 | 0,00 | 0,00 | 1,017 |
| CMS-105 | 0,05 | 0,07 | 1,667 |
| AC | 0,08 | 0,10 | 0,683 |

Based on the results of this work, as well as on the basis of literature data [20], the surface centers of the obtained materials were identified (Table 4).

According to [20], based on the research results obtained, it is more expedient to use sorption materials MS-105 and KMS-105 for collecting basic compounds, petroleum products, and heavy metal ions from the surface of contaminated environments.

Thus, the sorption properties of the obtained materials in relation to these pollutants were studied (Table 5).

particles and AC leads to a synergistic effect, which allows obtaining a material with improved sorption characteristics for a wide range of pollutants.

Thus, the proposed method for processing iron-containing waste to obtain composite magnetic sorbents is promising for the creation of effective materials capable of cleaning contaminated environments.

Conclusion

The studies conducted allowed us to draw the following conclusions:

1. The diversity of Brønsted centers on the surface of the obtained KMS-105, and their greater number, suggests its high adsorption activity to a wide range of organic compounds than that of the initial MS-105 and AC particles.
2. The studies indicate the possibility of applying iron-based hydroxide-oxide systems to the surface of activated carbon in the process of co-precipitation of iron (II, III) hydroxides, with the subsequent use of the obtained materials as sorbents. In this case, the co-precipitated hydroxides to some extent change the distribution of the acid-base activity of the activated carbon particle surface and, consequently, their sorption activity with respect to various pollutants.

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