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DEVELOPMENT OF A METHOD FOR OBTAINING SORBENT FROM BAGASSE OF SWEET SORGHUM FOR NEUTRALIZATION OF SOIL CONTAMINATION BY HEAVY METAL IONS

Об'єктом досліджень є промислові відходи рослинної сировини сорго цукрового (лігноцелюлозна багаса) та створений на її основі модифікований сорбент для очищення ґрунтів забруднених іонами важких металів. Традиційно модифікація рослинної сировини з отриманням сорбентів передбачає окиснення (гідроліз) рослинного матеріалу під дією сильних мінеральних кислот при високій температурі. Після кислотної обробки проводять лужне активування та багаторазове промивання сорбенту до нейтральної реакції промивних вод. При цьому вихід сорбенту становить 20–30 % від маси сировини. Крім того, утворюється значна кількість продуктів розкладання і великі об'єми шкідливих стічних вод. Тому в ході дослідження для отримання сорбенту використовували метод мерсеризації – обробка лігноцелюлозної багаси розчином лугу.

Головними складовими компонентами лігноцелюлозної багаси є лігнін і целюлоза, що зв'язані у біополімерні комплекси. В необробленому вигляді вони мають слабкі сорбційні властивості через фібрилярну будову та низький вміст в них вільних функціональних груп. За умов проведення процесу мерсеризації відбувається частково руйнація молекулярних зв'язків між волокон біополімерів і перехід у розчин низькомолекулярних фракцій полісахаридів. Структурний каркас лігноцелюлозної матриці при цьому зберігається, а його здатність до набрякання – зростає. Зберігається цілісність, рослинні волокна покращують свою структурно-порувату будову за рахунок збільшення внутрішньої адсорбційної поверхні. Оптимальними умовами процесу мерсеризації є гідромодуль 1:10 при початковій концентрації лугу 120 г/л. Максимальні величини сорбції іонів важких металів збільшуються у 2–3 рази. Вихід сорбенту досягає 60–80 %. Крім того, перевагами цього способу є відсутність шкідливих стічних вод.

Розроблений спосіб може застосовуватися для перероблянні відходів і іншої рослинної сировини (кукурудзи, соняшнику, цукрової тростини, тощо), а отримуваний сорбент – у агротехнологіях для знешкодження ґрунтів забруднених іонами важких металів.

Ключові слова: *мерсеризація відходу, сорбент для очищення ґрунтів, сорго цукрове, лігноцелюлозна багаса.*

1. Introduction

The key to successful development of the country is the preservation and restoration of the environment: soil, air, natural sources of water, flora and fauna. It is known that the accumulation of toxic heavy metals in soils and depressing their interaction with soil microflora negatively affects the productivity of land and the quality of agricultural production [1].

To neutralize contaminated soils using the following methods of cleaning [2–4]:

1. Mechanical – removing contaminated soil and transporting it to burial sites. The disadvantage of the method is that the pollutants simply move to another place where they should be controlled, and during removal and transportation it is possible that the contamination spreads by spraying the soil. In addition, this method is expensive.

2. Biological – growing on polluted soils of certain plants (phytoremediation). This method is much cheaper, but takes much more time. In addition, the plant biomass accumulating toxicant must be disposed of or buried.

3. Adsorption – adding chemicals to the soil, transfer soluble forms of pollutants to less dangerous insoluble materials. This method of fixing and stabilizing toxicants does not form waste and is the most environmentally acceptable. Therefore, recently, in some publications, the question of a possible improvement of the ecological condition and productivity of earth lands has been raised by using various types of sorbents in one form or another [5].

In the countries of Western Europe, they are manufactured in industrial volumes and have a number of sorbing materials of specific action, mainly on a mineral basis, for use in agriculture [6]. Known work on the use of mineral fertilizers and zeolites to reduce the transition of heavy metals in plants [7].

Previously, phyto-sorption methods were developed, which were based on the use of sorbents, microbiota and a number of plants to clean the soil from radionuclides [8]. Meanwhile, an integrated approach, which would include the use of chemically modified sorbents for separating heavy metals from their soil and storing them firmly in their pores (in particular, cadmium, lead and strontium), has never been used to date.

Thus, the development of new sorption technological measures and materials seems to be relevant from the point of view of environmental conservation and the development of efficient farming, and in fact, a strategic task, protecting the national interests of Ukraine.

Therefore, there is an urgent need to find and develop new innovative technologies for obtaining plant sorbents, the use of which in agricultural production will significantly reduce the anthropogenic load on the environment.

2. The object of research and its technological audit

The object of research is the industrial waste of plant raw materials of sweet sorghum (lignocellulosic bagasse) and a modified sorbent created on its basis for cleaning soils contaminated with heavy metal ions.

A promising raw material for sorbents is desulphurized lignocellulosic bagasse of sweet sorghum stems, which remains after the production of sugar-containing products and the output of which is about half of the total initial mass of stems. It is necessary to take into account the economic importance of the processing of cheap raw materials, which is a waste of production and in the future has no practical use. Therefore, in the conditions of complex processing of biomass sorghum in a closed cycle, it is possible to obtain highly profitable production with a significant reduction in consumables. Such production involves the production of sugar-containing products for the food industry from the juice of the stems, and from the production wastes – an environmental sorbent.

The study used desulphurized crushed *lignocellulosic bagasse* (feedstock) and *mercerized lignocellulosic bagasse*, which was obtained by chemical modification of the feedstock.

Technological parameters of *lignocellulosic bagasse*: average particle size of 5–8 mm, moisture content 4 %, cellulose 39.2 %, lignin 14.4 %.

One of the most problematic places in the technology of edible sugar-containing products from sweet sorghum is a significant amount of waste – desulphurized lignocellulosic bagasse. This care is now not used at all, or partially used as fuel. Another problem is significant soil contamination with heavy metal ions, which can migrate to plant products.

3. The aim and objectives of research

The aim of this research is development of a method for chemical processing of waste plant sorghum.

To achieve this aim it is necessary to perform the following tasks:

1. To investigate the conditions of the mercerization process of sweet sorghum lignocellulosic bagasse.
2. To determine the structural-porous and ion-exchange properties of the initial and mercerized bagasse, as well as to investigate the morphological and chemical structure of their surface.
3. To investigate the sorption-kinetic properties of the initial and mercerized bagasse with respect to the ions Cd^{2+}, Pb^{2+} and Sr^{2+}.

4. Research of existing solutions of the problem

It is known that sorption methods for cleaning soils are the most simple, affordable and effective, where cheap plant waste generated directly in agricultural production is used as sorbents: meal, husk, bran, sawdust [9].

Large-scale industrial waste of sorghum biomass can also be used to obtain materials on their basis with high sorption characteristics for binding toxic heavy metals in soils [10, 11].

Traditionally, the technology of obtaining sorbents from plant materials involves the oxidation (hydrolysis) of plant material under the action of strong mineral acids at high temperature. After acid treatment, alkaline activation and repeated washing to neutral reaction of wash water, etc., is carried out [12]. With this cultivation, the sorbent yield is 20–30 %, at the same time, a significant amount of decomposition products and large volumes of harmful wastewater are formed, which pollute the environment and require the use of expensive anticorrosion equipment.

With this in mind, there is a need to search for less costly and environmentally friendly technologies for processing plant biopolymers to obtain sorbents.

An alternative way to obtain sorbents can be the treatment of lignocellulosic plant biopolymers with alkali solution (mercerization) [13]. This method is rather actively used for processing cellulose fiber of coniferous woods, as well as cotton in the paper industry [14, 15].

When processing waste plant materials with alkali, partial destruction of molecular bonds between biopolymer fibers and transition of low molecular weight polysaccharide fractions is observed, the structural frame of the lignocellulosic matrix is preserved, and its ability to swell increases [16]. An important change from the point of view of sorbent production is that while preserving the integrity, the plant fibers improve their structurally porous structure by increasing the internal adsorption surface [17, 18].

In addition, the main advantages of this method of obtaining a sorbent are its simplicity in hardware design, low water consumption and the absence of harmful wastewater.

Summing up the above analysis of literary sources, let's believe that a promising direction of improving the technology of manufacturing plant sorbents is a method of obtaining a sorbent for toxic metal ions by mercerizing lignocellulose from sweet sorghum.

5. Methods of research

As a source of raw materials, desulphurized air-dried ground lignocellulosic bagasse of sweet sorghum (*Sorghum Saccharatum (L.) Moench*) with an average particle size of 5–8 mm and a moisture content of 4 % was used. The pulp content in the feedstock sorghum was 39.2 %, and the lignin content was 14.4 %. Mercerized lignocellulosic bagasse product was obtained by chemically modifying the raw material with an aqueous solution of sodium hydroxide with a concentration of 120 g/l at a temperature of 10–15 °C for 60 minutes in different ratios of the solid phase in a liquid (hydromodule).

In the study of the structural-porous, sorption, and ion-exchange properties of the initial and modified lignocellulosic bagasse, both standard and special methods were used. Thus, the values of the static exchange capacity (SEC, mg-eq/g) were determined according to the standard by the sorption value of 0.1 *n*HCl solution and 0.1 *n*NaOH solution [19]. The morphological structure of the initial and mercerized lignocellulosic bagasse samples was studied using the SEM (Scanning Electron Microscopy) method under the condition that the samples were prepared according to the procedure [20]. Micrographs of the studied samples were taken on a JEOL electron microscope model JSM-5500LV (Japan) with a dispersion energy analyzer EDS/EDX/EDAX and ESEM.

The chemical structure of the surface of mercerized bagasse and the determination of functional groups on its surface was investigated by infrared (IR) spectroscopy on a Nicolet NEXUSFT-IR spectrometer (USA) with a built-in diffuse reflectance device SMART Collector.

Sorption experiments were carried out under static conditions from model solutions of ground washings contaminated with heavy metal ions according to [21, 22].

The concentration of heavy metal ions was determined on an atomic adsorption spectrometer KAS-1 (SELMA) (Ukraine) after atomization of the sample in an air-acetylene flame. The method is based on the property of atoms in the ground state to absorb light of specific and specific for each type of atom wavelength.

6. Research results

Secondary raw material is used – lignocellulosic desugared bagasse, which remains after the production of sugar-containing products. The main components of bagasse are cellulose and lignin, connected to biopolymer complexes, which in their unprocessed form have weak sorption properties through the fibrillar structure and low content of free functional groups in them. Under the conditions of the mercerization process of these materials, their morphological structure and physico-chemical characteristics are improved.

So, Table 1 shows the data characterizing the structural-porous and ion-exchange properties of the initial and mercerized alkali (concentration of 120 g/l) bagasse (at different hydromodule processing).

Table 1

Characteristics of the properties of the original and mercerized bagasse

Hydromodule	Specific bulk density, g/cm^3	Swelling degree in water, g/g	Static exchange capacity for Na^+, mg-eq/g	Static exchange capacity for Cl^-, mg-eq/g	Sorbent yield, %
Original bagasse	0.36	4.8	0.45	0.12	–
1:5	0.26	5.9	1.38	0.45	87
1:10	0.16	6.8	1.44	0.38	64
1:20	0.12	12.5	1.65	0.41	59

From the obtained results it can be seen that in the process of mercerizing the raw material part of the material goes into solution, the specific bulk density decreases 1.5–2.5 times. The yield of the finished sorbent is 60–80 % of the initial amount of raw materials and is reduced only in the case of using the maximum hydraulic module.

The results of titration of sorbents with mercerized bagasse of 0.1 *n*HCl and 0.1 *n*NaOH indicate a fairly high value of the static exchange capacity for both alkali metal cations and chlorine anions. The static exchange capacity (SEC) for Na^+ cations is 0.45 mg-eq/g for untreated bagasse. This indicates the presence of protogenic groups in the raw material, most likely of alcohol type. As a result of bagasse mercerization, SEC of the material increases both in Cl^- anions and in Na^+ cations. This creates prerequisites for enhancing its sorption activity in ion-exchange and complex-forming sorption of multiply charged transition and heavy metals, including toxic Cd^{2+}, Pb^{2+}, Sr^{2+}.

Confirmation of these results is studies that characterize the morphological structure of the surface of the original and mercerized bagasse (Fig. 1, 2).

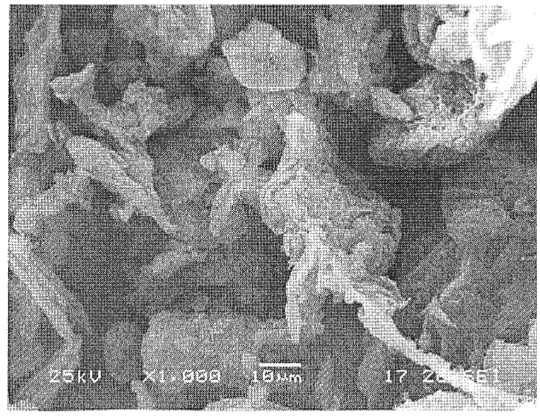

Fig. 1. Microphotos of the original bagasse surface

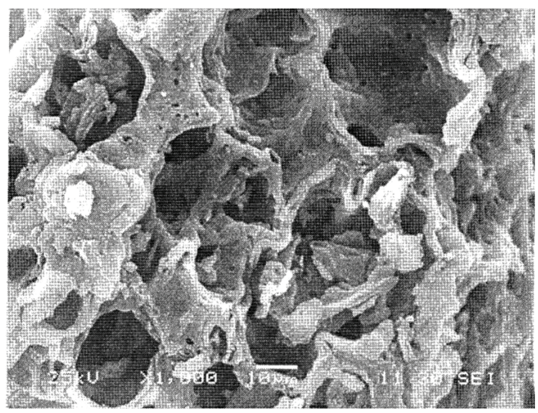

Fig. 2. Microphotos of the mercerized bagasse surface

So, in Fig. 1, the cellular structure of the material is not observed, probably because the bagasse has undergone pre-pressing and drying. The micrograph shows filaments of cellulose and lignin globules, which form a rather dense porosity, which prevents access of the sorbate to the surface formations. Fig. 2 clearly shows the relief of the bagasse surface after mercerization, which indicates the effectiveness of such processing. It can be seen that the pores have a shape that is close to cylindrical and spherical, but this feature is not observed on the entire surface of the sample. The preferred pore size is 10–18 nm, which, according to the international classification, makes it possible to classify the obtained sorbents into the meso- and micro-porous class [23].

Thus, comparative microscopic studies of the surface of the original and mercerized bagasse allow to see the changes in the surface of the mercerized bagasse, indicating the effectiveness of the bagasse modification by means of alkaline treatment. An increase in porosity and an effective contact surface should contribute to an increase in the adsorption characteristics of the obtained sorbents.

The chemical structure of the surface of mercerized bagasse and the determination of functional groups on its surface was investigated by IR spectroscopy. A sample of the sorbent was weighed, mixed with KBr powder in a mass ratio of 1:20, and ground in a mortar until homogeneous. The spectra were recorded in the range from 400 to 4000 cm^{-1} and were decoded according to the method of IR-spectroscopy for the analysis of plant materials [24]. Absorption bands characterizing its total chemical composition were detected in all spectra of the investigated sample.

It should be noted that the complex structure of plant material makes it difficult to analyze and only makes it possible to determine the main components of the surface of the mercerized bagasse, since the areas of reflection and absorption can overlap or shift. Fig. 3 represents the dependence of the reflection of electrons (%) on the magnitude inverse of the wavelength 1/λ (wave number).

Special attention is attracted by the region of deformation vibrations 1450–1650 cm^{-1}, where there are four peaks, indicating the presence of aromatic structures on the surface. They are formed by vibrations of sesquialteral carbon ties. If consider in more detail the first peak in the region of 1600 cm^{-1}, then it is possible to assume that the atoms of the aromatic ring are replaced. In this case, it can be seen that the peak is shifted to the region of 1650–1585 cm^{-1}, which indicates a three asymmetrical substitute. This is also confirmed by a peak in the region of 1525–1475 cm^{-1}. A complex pattern takes place in an area below 700 cm^{-1}. However, the reflection band near 750 cm^{-1} confirms the presence of a substitute at position 1, 2, 4 with another unsubstituted hydrogen in position 3. The reflection in the region of 1110–1070 cm^{-1} indicates replacement in positions 1, 2, 3 and 1, 3, 5.

In the region of 650–810 cm^{-1}, the presence of a field of cyclic compounds can be determined; however, this interpretation will not be entirely accurate due to the presence of overlapping spectra.

Deformational vibrations in the region of 750–650 cm^{-1} result from the presence of bound groups (–OH) and give very wide bands. Reflection in the area of 1000–1075 cm^{-1} indicate a substitution in the group (–OH). The reflection in the region of 1180 cm^{-1} usually corresponds to deformation oscillations (–OH).

The presence of carboxyl groups (–COOH) on the surface was established by a peak in the region of 950 cm^{-1}, which corresponds to the deformation vibrations (–OH) of the carboxyl groups. In the region of 1400–1440 cm^{-1}, stretching vibrations (C–O) are observed, which corresponds to carboxyl groups. Also, a peak corresponding to cyclic anhydride is observed in the vicinity of 1850 cm^{-1}; it is associated with the vibration of carbonyl groups (C=O). Absorption in the region of 1380 cm^{-1} corresponds to the aldehyde-type radical (–RCOH). A radical in this case may be a cyclic connection. The presence of aldehyde groups is also confirmed by vibrations (C–H) with two bands in the region of 2860–2900 cm^{-1}. The absorption peak in the 1760 cm^{-1} region corresponds to the vibrations of a group of atoms (–COOH) in dimeric molecules.

A complex picture is observed in an area of approximately 3000 cm^{-1}. This band gives three or more bands for cyclic compounds and their characteristic vibrations (=C–H). The presence of a large number of excitations in the region of 3300–3650 cm^{-1} complicates the interpretation of this region, but it can be said that almost all of it correspond to the fluctuations of amines (RNH_2).

Thus, the analysis of micrographs of the source and mercerized bagasse showed that as a result of chemical modification of the raw material, a change in the structural-porous structure of the material occurs. At the same time, due to partial hydrolysis and «loosening» of plant fibers of bagasse and improving access to functional groups, the ability of the material to the addition, complexation and ion exchange reactions increases. At the same time, the IR spectral analysis made it possible to establish the presence of hydrophilic functional groups of the alcohol and acid types substituted by metal ions (mainly sodium). The material also found amino groups, which indicates the presence of amino acids in mercerized bagasse.

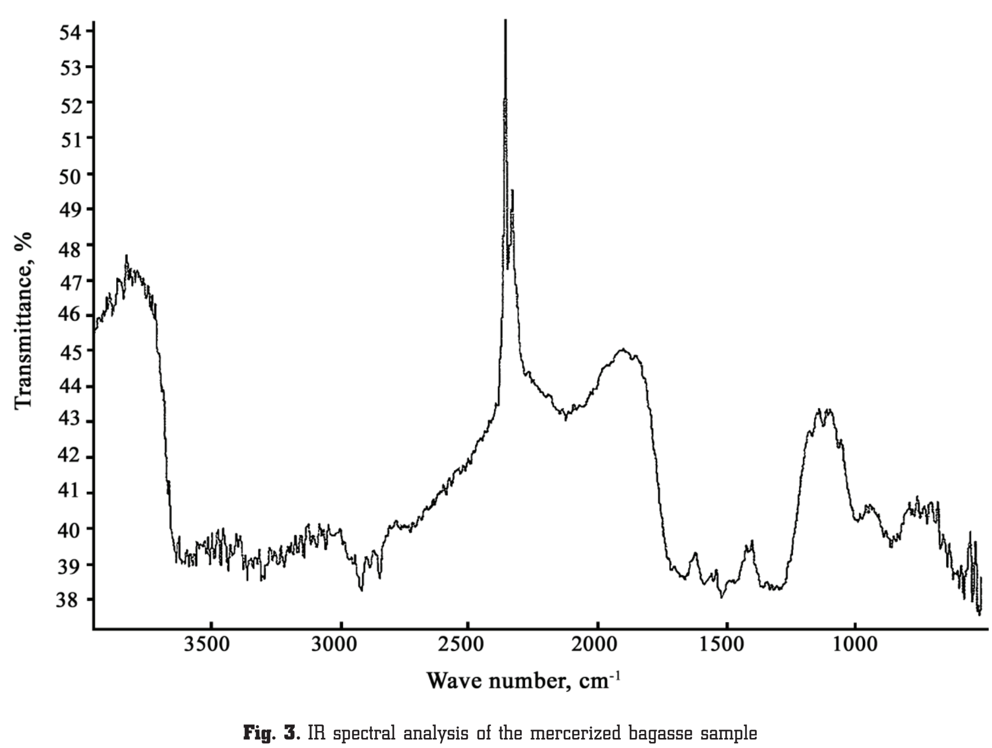

Fig. 3. IR spectral analysis of the mercerized bagasse sample

At the next stage, the sorption activity of mercerized bagasse with respect to Cd^{2+}, Pb^{2+} and Sr^{2+} ions was investigated.

The study of the sorption process of toxic heavy metal ions, native and mercerized rich, was carried out under static conditions from model water washouts of soils contaminated with lead, cadmium and strontium ions. The method of sorption experiments envisaged the addition of 0.5 g of sorbents to the model solutions (with an initial concentration of heavy metals in them 5 mmol/l) and content on a shaker for a certain time at a temperature of 20 °C. During sorption every 20 min the suspension was filtered, the content of heavy metal ions in the filtrate was determined by atomic adsorption spectrometry. The number of sorptionally withdrawn heavy metals (A, mmol/g) was calculated by the formula:

$$A=\frac{\left(C_o-C_p\right)\cdot V}{m\cdot 1000}, \quad (1)$$

where C_o, C_p – the concentration of heavy metal ions (initial and equilibrium), mmol/l; V – the volume of solution, ml; m – the sorbent sample weight.

Fig. 4, 5 show the kinetic characteristics of the sorption extraction of heavy metal ions of the original and mercerized bagasse.

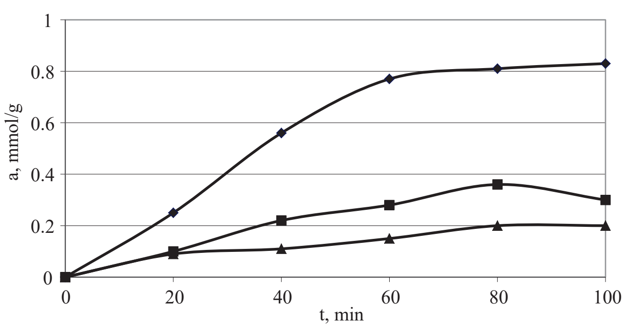

Fig. 4. Sorption kinetics of ions of heavy metals of the original bagasse: ♦ – Pb^{2+}; ■ – Cd^{2+}; ▲ – Sr^{2+}

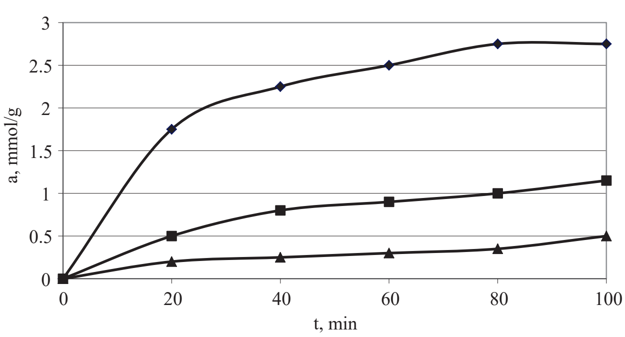

Fig. 5. Sorption kinetics of ions of heavy metals of the mercerized bagasse: ♦ – Pb^{2+}; ■ – Cd^{2+}; ▲ – Sr^{2+}

From the obtained kinetic curves for the initial bagasse (Fig. 4), it can be seen that the sorption equilibrium in the system occurs within 60–80 minutes. The maximum sorption capacity for Pb^{2+} is about 0.85 mmol/g, for Cd^{2+} – 0.37 mmol/g, and for Sr^{2+} – 0.20 mmol/h. These data indicate that the native bagasse has low sorption-kinetic properties.

The study of the kinetic properties of mercerized bagasse during the sorption of heavy metal ions from groundwater washings (Fig. 5) showed that sorption equilibrium occurs approximately twice as fast, and the maximum sorption values increase by a factor of 2–3. The results of these experiments indicate that mercerizing the bagasse significantly changes the composition of the functional groups and the ion-exchange properties of the surface of the obtained samples, thus affecting their sorption capacity. Sorbents obtained by mercerizing bagasse have a higher sorption capacity with respect to heavy metal ions as compared to untreated bagasse.

Studies have shown that the existing problem of contaminated soils in Ukraine exists. Chemical pollutants can persist in the soil for many years and, including in ecological chains, cause long-term effects of toxicants. According to the National Science Center «Institute of Agriculture of the NAAS» (village Chabany, Kyiv region, Ukraine), at present, about 20 % of the territory of Ukraine is contaminated with heavy metals [25].

This problem can be partially solved by using sorbents for soil neutralization. The results of experimental laboratory tests of a sorbent created by the mercerization of lignocellulosic bagasse sweet sorghum indicate that its kinetic properties increase by 2–3 times compared with the original bagasse. In addition, quantitative characteristics of sorption of Cd^{2+}, Pb^{2+} and Sr^{2+} ions from complex ground extracts create conditions. It also opens up new possibilities for the targeted use of mercerized bagasse for sorption binding and strong fixation of heavy metal ions in its pores.

7. SWOT analysis of research results

Strengths. The positive effect of the research is development of an effective and economically viable technological process for the preparation of a sorbent by chemical modification of lignocellulosic waste (bagasse). The obtained result compares favorably with analogs in structural-porous characteristics, the number of surface functional groups and provides a high yield of the finished product.

Weaknesses. The weak points of this study are related to the fact that the proposed solutions were tested only on model solutions of soils contaminated with lead, cadmium and strontium ions. In terms of application of the developed sorbent on an industrial scale, its sorption properties may vary. Since, in the case of the use of sorbent in industrial conditions, the influence of additional factors on the sorption process, namely, the soil and climatic conditions of the process of soil neutralization, costs and conditions for the application of the sorbed element, etc. Therefore, all these factors require further practical research in the agricultural industry, for neutralization of soils from ions of heavy metals, improvement of their productivity and development of effective agriculture.

Opportunities. The obtained positive results of the action of mercerized bagasse to form insoluble complex compounds with heavy metal ions give grounds for their further practical use as a sorbent for neutralizing soils from pollutants.

Further research in this area should be directed to the study of the effectiveness of the sorbent under different conditions and costs in industrial process conditions in areas of contaminated soils and also on the development of regulatory documentation on the technology of the sorbent production with lignocellulose and sweet sorghum and its application in agricultural technologies.

Threats. Under the modern conditions, the country has sufficient technical and technological potential for the production of a wide range of industrial, environmental and medical sorbents from lignocellulosic waste. This could not only fill its domestic market, but also export sorption materials abroad.

However, the production of sorption material from lignocellulosic waste of sweet sorghum is now complicated because of the lack of proper domestic equipment to produce this product. Existing foreign equipment samples are notable for their high cost, and, accordingly, which will negatively affect the growth of the cost component of the final product.

Therefore, further research will be focused on the selection and, if possible, the development of process equipment for the line for processing lignocellulosic bagasse from sweet sorghum to produce a modified sorbent for cleaning soil from heavy metal ions.

8. Conclusions

1. The sorbent for the neutralization of soils from heavy metal ions is obtained and the optimal conditions for the process of its manufacture is established. These conditions consist in the treatment of lignocellulosic plant waste with sorghum with a sodium hydroxide solution with a concentration of 120 g/l of alkali at a temperature of 10–15 °C and a solid phase ratio in a liquid (hydromodule) of 1:10.

2. As a result of studies of microphotos of the original and mercerized bagasse, it is found that chemical modification of the raw material leads to a change in the structural-porous structure of the material. This is due to the partial hydrolysis and «loosening» of plant fibers of the bagasse and improving access to functional groups that are capable of addition, complexation and ion exchange reactions. Based on IR spectral analysis, the presence of hydrophilic functional groups of the alcohol and acid types substituted by metal ions (mainly sodium) is established.

3. According to the research results of the original and mercerized bagasse, it is established that the sorption-kinetic properties of heavy metal ions – Cd^{2+}, Pb^{2+} and Sr^{2+} into mercerized bagasse – are increased 2–3 times in comparison with the untreated bagasse.

The results obtained, taking into account the availability of the raw material resource and for its complex processing, will make it possible to reasonably approach the solution of specific practical problems associated with the development of modified sorbents. As well as their use as soil additives for the disposal of polluted soils from toxic heavy metals and to prevent further migration of heavy metals into plant products.

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