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BIOSORBENTS – PROSPECTIVE MATERIALS FOR HEAVY METAL IONS EXTRACTION FROM WASTEWATER

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Abstract. The article deals with ecological safety, resource saving, economic efficienty in the technologies of wastewater purification from heavy metals ions. It is shown that modern technologies of wastewater purification from such substances need to be improved. A promising way to solve this problem is the production and wide use of carbon sorbents obtained from the waste of processing agricultural raw materials and waste generated at food enterprises. Scientific research is actively carried out in this area. This is due to the possibility of organizing the process of wastewater purification from heavy metal ions in a cheap and effective way. The development of biosorbent production is relevant for Ukraine, as the development of agriculture and food industry is of primary importance. The article presents the results of an analytical review of literary sources on various aspects of the scientific problem. In particular, possible sources of raw materials for the production of biosorbents, classical and modified technologies for their production, mechanisms of biosorption using raw materials with different chemical composition, sorption characteristics of biosorbents, and indicators of the economic efficiency of their production are considered.

Key words: wastewater, heavy metal ions, biosorption, technologies, mechanisms, efficiency indicators.

БІОСОРБЕНТИ – ПЕРСПЕКТИВНІ МАТЕРІАЛИ ДЛЯ ВИЛУЧЕННЯ ІОНІВ ВАЖКИХ МЕТАЛІВ ІЗ СТІЧНИХ ВОД

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Анотація. Статтю присвячено питанням екологічної безпеки, ресурсозбереження, ефективності, в тому числі й економічної, в технологіях очищення стічних вод від іонів важких металів. Показано, що сучасні технології очищення стічних вод від таких речовин потребують удосконалення, а перспективним шляхом вирішення цієї проблеми є налагодження виробництва та широке використання вуглецевих сорбентів на основі відходів переробки сільськогосподарської продукції та виробництв харчових продуктів. Наукові дослідження в даному напрямку активно проводяться. Це пов'язано з можливістю організації процесу очищення стічних вод від іонів важких металів дешевим і ефективним способом. Для України розвиток виробництва біосорбентів актуальний, оскільки розвиток сільського господарства та харчової промисловості є пріоритетним. У статті представлено результати аналітичного огляду літературних джерел за різними аспектами наукової проблеми. Зокрема розглянуто можливі джерела сировини для виробництва біосорбентів, технології їх отримання, механізми біосорбції, сорбційні характеристики біосорбентів та показники економічної ефективності їх виробництва.

Ключові слова: стічні води, іони важких металів, біосорбція, технології, механізми, показники ефективності.

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Introduction. Formulation of the problem

One of the topical problems of our time is complex processing of waste with the purpose of reducing the man-caused load on the environment. Wastewater, which contains heavy metals, like ions of cadmium, chromium, zinc, nickel, copper, is particularly dangerous. They have the properties of cumulative nature toxicants, have the mutagenic and carcinogenic effect on living organisms.

Electrochemical production is the main supplier of heavy metals to the environment. Such productions are in every enterprise of mechanical and instrument-making industries. Modern technologies of wastewater purification from heavy metal ions do not allow achieving the desired degree of purification. It is necessary to develop new technologies that will provide the efficient removing of pollutants from waste water and allow more rational and integrated use of water and other raw and energy resources.

Chemical precipitation, membrane technologies, ion exchange, flotation, electrochemical methods, coagulation, adsorption, distillation are used for wastewater purification from heavy metal ions. Most of these methods are expensive and complicated.

To purify large amounts of wastewater with relatively low content of heavy metal ions, sorption purification is widely used. Carbon and non-carbon sorbents are used for sorption of heavy metal ions from aqueous solutions. The required sorbents should be available, cheap, effective in removing contaminants, easy to utilize, environmentally friendly, and with the ability of regeneration.

Activated carbon, based on wood or coal, has been widely used as a sorbent. The process of its production is energy-consuming, and the product is expensive. It is practical to regenerate such sorbents. But the regeneration of carbon sorbents is also complicated and energy-consuming. Using sorbents obtained from agricultural and food waste processing is one of the ways of reducing the cost of technology for sorption wastewater purification from heavy metal. Such sorbents, being non-living biomass, are called biosorbents. Biosorbents have been actively developed in the world lately. Their industrial production, though, is not yet well established [1–3].

There are many food and agricultural enterprises in Ukraine. In their activity, a lot of solid wastes of plant and animal origin are formed. Some of these wastes are recycled to biofuels, some are used to feed animals. But the significant part of such wastes is not utilized. They are exported to landfill sites and create environmental problems. Using sorbents based on secondary raw materials from food and agricultural enterprises will allow enterprises to increase the economic efficiency of production due to the reduction of environmental charges, the cost of water supply and sanitation by reusing purified wastewater in production. Thus, it is possible to solve vital problems, such as resource saving and keeping the natural balance in the environment [4–5].

It should also be noted that using local raw materials as sorbents will reduce the cost of wastewater treatment. Since Ukraine is an agrarian country, there will always be enough raw material for the production of sorbents. Besides, the issue of sorbents regeneration will become less relevant, because sorbents spent can be utilized, for example, as biofuels [2].

Purpose and objectives of the research. To launch in Ukraine the production of sorbents based on the waste from processing plant and animal raw materials, it is necessary to study first how such sorbents are developed in the world.

To do this, it is necessary to solve a number of problems:

– to analyze which wastes from processing of agricultural and food products are a promising raw material for biosorbents production;

– to study the technologies of obtaining these biosorbents;

– to study biosorbents' sorption characteristics in relation to heavy metal ions, and mechanisms of biosorption processes during wastewater treatment;

– to analyze the economic efficiency of biosorbents production.

State of the biosorbents development issue

Sources of raw materials for the biosorbents production. It is known from literary sources that promising natural carbon sorbents for sewage purification, are sorbtion materials obtained from bean shells, corncobs [6], walnut [7], sugar cane [8], carrot waste [9], sugar beet pulp [10], cottonseed pulp [11], rice husk [12, 13], sunflower seed husk [14], soybean meal and pulp of sunflower seeds, wheat straw [15] , skins of oranges and bananas [16], waste processing of molluscs and crustaceans [17], wastes from poultry farms [18], cherry stones [19], shells of pine nuts [20], hazelnuts [21] and peanuts [22], cork [23], tea processing waste [24-26], buckwheat husk [27], waste processing of pecan nut [28] and almonds [29]. It is also noted that raw materials for biosorbents may include nucleus of apricots and plums, peaches and olives, seeds of apples and grapes, others.

Waste from processing agricultural and food products have high carbon and low ash content, but, at the same time, they have low sorption capacity. For example, according to the data of the authors [27], the sorption capacity of buckwheat raw husk for lead ions is 1.4 mg/g, and that of the husk of sunflower seeds is 0.5 mg/g. That is why raw materials like these must undergo special treatment.

Classical technology for obtaining carbon sorbents based on wood. The choice of technology for obtaining biosorbents based on the second raw materials depends on its chemical composition, physical and chemical properties, and the chemical properties of the substance adsorbed [30]. For example, the classical technology of obtaining wood based porous carbon sorbents involves thermal processing of raw materials without oxygen access. At the same time, the moisture and, partly, resins are removed from the raw material. The structure of the coal obtained after this treatment is coarse-porous, and it is not an effective sorption material yet. Methods of gas (or steam) oxidation and treatment with chemical reagents are used to provide the microporous structure of the semifinished product. Carbon dioxide (at 900°C) and water vapor (at 850°C) are used in steam-gas activation. A portion of coal burns out in gas activation. The degree of charring determines the type of pores (micropores prevail if its value is 50%, and macropores prevail at 75%). Oxides and carbonates of alkali metals, as well as iron and copper compounds, can be used as catalysts for the steam coal activation reaction. During chemical activation of coal, the semifinished product is treated with salts (carbonates, sulfates, nitrates) that give up an activation gas at a high temperature, and acids (nitric, hydrochloric, sulfuric, phosphoric, etc.), or concentrated solutions of salts (zinc chlorides, magnesium, iron, ammonium, thiocyanates of potassium, sodium carbonate and others). In particular, when the latter are used, the cellulose of the raw material goes into a solution. When the solution temperature rises, highly dispersed carbon amorphous is released. This carbon forms the microporous sorbent structure. Chemical activation is carried out at temperatures 200 to 650°C [30].

Using untreated wastes can cause secondary pollution of water, that is why a special treatment of secondary plant and animal raw materials is still necessary. A significant content of organic substances in these raw materials will be reflected in the increase of the COD and BOD values due to the increase of water-soluble carbon compounds concentration in wastewater.

Influence of the raw materials processing technology on the sorption characteristics of biosorbents. By different technological processing of the secondary raw material, it is possible to obtain a sorbent that will be different in the morphology of the porous structure, pore diameter, granulometric composition of particles, specific area of phase contact (Table 1).

Table 1. Surface characteristics of rice husking before and after special treatment [13]

Characteristic	Rice husking			
	initial	acid treatment	alkali treatment	burning with oxygen access
Specific surface area of phase contact, m^2/g	170	196–200	196–200	300–400
Pore diameter, nm	not indicated	1.0–4.4	1.0–29.0	8.5–67.0

Peculiarities of technological treatment affect the content of carbon and silicon (Table 2), other impurities (in particular, calcium, magnesium, aluminum, iron, and manganese), the presence of functional groups that can enter the process of ion exchange in the sorbent. This affects the structure of the sorbent and its ability to remove selectively the heavy metal ions from wastewater, and also determines the application area of the sorbent.

The adsorption properties of the prepared material are improved by the formation of more advanced pore structure, increase of the surface area of sorption, increase in the content of individual components or functional groups in the sorbent. The review of literary sources allowed us to summarize information about the influence of the biosorbents obtaining technologies on the biosorbents sorption capacity for a kind of heavy metal ions typical of wastewater – copper ions (Table 3). In particular, we were interested in the indicator of the static exchange capacity of sorbents (*SEC*, *mg /g* or *mmol /g*). This indicator reflects the amount of metal adsorbed (in *mg* or *mmol*) by one gram of the sorbent [4–5]. It is calculated by the equation:

$$SEC = (C_s - Cp) \cdot V/m,$$

where C_s is the concentration of a pollutant in the source water; C_{eq} – equilibrium concentration of a pollutant in the purified water; V – volume of water added to the sorbent for purifying; m – the mass of the sorbent used for sorption.

Table 2. Influence of the rice husk processing technology on the content of carbon and silicon in biosorbents [13]

№	Processing technology	Content of components,%	
		C	SiO_2
1	Rice husk is washed with water and dried at 180°C	81.0	12.0
2	Carbonization of raw materials with oxygen at 300°C	70.0	22.9
3	Two-stage carbonization of raw materials with oxygen at 300 and 500°C	39.0	53.9
4	Two-stage carbonization of raw materials with oxygen at 300 and 600°C	2.0	95.0
5	Husk leaching with 0.1 n HCl solution	72.0	15.0
6	Husk leaching with 0.1 n HCl solution followed by two-stage carbonization at 300 and 600°C with oxygen	< 0.01	99.9
7	Hydrolysis of husk with alkali	83.0	0.05
8	Hydrolysis of husk with alkali followed by silica deposition of acid	not found	99.4
9	Husk leaching with 0.1 n HCl solution followed by raw material pyrolysis at 750°C	52.0	47.5

For a group of biosorbents (in particular, tea processing waste, soybean meal, and pulp of sunflower seeds), there is no *SEC* information in literary sources. However, there is information about the degree of copper ions extraction from wastewater, as the percentage of its initial concentration *(E,%)*. The degree of pollutant removal from the water is calculated by the formula:

$$E = (C_s - Cp) \cdot 100/C_s.$$

The information about this index was included in Table 3.

The analys of the data given in Table 3 shows us that simple technologies (mechanical grinding) and more complex ones (carbonization, chemical modification) can be used to produce biosorbents. The temperature of the process, presence of oxygen and of a pH medium, the chemical composition of the raw material, the type and concentration of acid or alkali in the solution for chemical modification of the raw material, the rate of addition of this solution, the granulometric composition of a sorbent are the most important technological parameters that affect the efficiency of the sorption process. It is interesting to note that practically the same value of the sorption exchange capacity of the biosorbent can be achieved in one case (for example, with sunflower husk) only by grinding the raw material, and in another case (for example, with activated charcoal from a coconut shell) only after dehydration, pyrolysis, activation by steam and acid solution. There is a wide array of choices of acids or alkalis for processing raw materials. For example, HCl, H_2SO_4, HNO3, CH_3COOH, H_3PO_4, $H_2C_2O_4 \cdot H_2O$, HC_6O_4 are used as acids.

Table 3. Influence of raw materials processing technology on the sorption characteristics of biosorbents

Raw	Technologies for obtaining biosorbents	SEC, mg/g	literature
Sugar cane	Chemical modification: – sodium bicarbonate;	114.0	[8]
	– ethylenediamine;	139.0	
	– triethylentetramine.	133.0	
Carrot waste	Chemical modification by hydrochloric acid	32.74	[9]
Sugar beet cellulose	Chemical modification by hydrochloric acid	0.15	[10]
Cotton cellulose	Treatment by polyvinylpyrrolidon with concentration (40–100) g / l	41.3–46.07	[11]
Husk of rice	Washed by water and dried at 180°C	6.76	[13]
	Carbonization with oxygen at 300°C	6.08	[13]
	Two-stage carbonization with oxygen at 300 and 500°C	19.61	[13], [26]
	Two-stage carbonization with oxygen at 300 and 600°C	9.11	[13]
	Hardening of husk by 0.1 n solution of HCl	6.13	[13]
	Chemical modification by tartaric acid	29.0	[12]
Sunflower seeds husk	Mechanical grinding	14.0	[14]
	After removal of melanin treatment by 0.25M NaOH solution (pH = 9.0)	25.0	
	Chemical modification by acid	18.0	
Soybean meal and pulp of sunflower seeds	Chemical modification by solution of sodium monochloroacetic acid in an amount (1–10)% of the mass of the sorbent	*E* = 99.4–99.8%	[15]
Wheat straw	The crushed pieces were treated by 10% HNO_3 solution, washed by distilled water and neutralized by 1M NaOH solution, washed to pH = 7 and dried.	5.72	[15]
Banana skin	Chemical modification by HNO_3 solution, followed by neutralization with NaOH solution and washing with water.	4.75	[16]
Orange skin	Chemical modification by HNO_3 solution, followed by neutralization with NaOH solution and washing with water.	3.65	[16]
Chitosan	Algal hydrolysis, deacetylation of chitin exoskeleton of molluscs and crustaceans (at pH = 3.2)	40.03	[17]
Waste poultry farms	Dehydration at 170 ° C for 1 s, pyrolysis at 700 °C for τ = 45 min in nitrogen atmosphere and steam activation for 45 minutes, rinsing by 0.1 M HCl solution, then washing and drying at 105 °C.	12.71	[18]
Bones of cherries	Pyrolysis of raw material by subsequent oxidation (air, ozone, HNO_3, hydrogen peroxide)	15…28	[19]
Shells of cedar nuts	Preheating of the shell by access to oxygen at a temperature (290–300) C, oxidation with a solution of HNO_3 and hydrogen peroxide	7.94	[20]
Shells of walnut	Carbonization by subsequent activation of water vapor	99.76	[21]
	Similarly + the effect of ultrasound	119.77	
Shells of peanuts	Chemical modification by solutions of citric acid and NaOH	7.60	[22]
Cork powder	Chemical modification: – calcium chloride;	15.6	[23]
	– sodium chloride;	19.5	
	– sodium hydroxide;	18.8	
	– hypochlorite of sodium;	18.0	
	– sodium iodate.	19.0	
Tea production: – waste tea production	Drying of raw materials at a temperature of 25–26 °C and shredding	*E*=76%	[3]
– green tea leaves		*E*=62%	
– tea bush seeds		*E*=40%	
– waste tea production		8.64 ± 0.51	[24]
For comparison: Industrial sample of granular activated carbon from coconut shell	Dehydration at 170 °C for 1 s, pyrolysis at 700 °C for τ = 45 min in nitrogen atmosphere and steam activation for 45 minutes, rinsing by 0.1 M HCl solution, then washing and drying at 105 °C.	14.93	[25]

Mechanisms of biosorption processes. Mechanisms of metal ions biosorption are quite complex. They include physical adsorption, electrostatic gravity, deposition, chemical interaction with functional groups, and the interaction of metal ions with surface structures of microorganisms, their metabolites and exopolymers. The mechanism of sorption is individual. It is determined by the chemical composition of a raw material, by the technology of obtaining the sorbent and by its sorption properties, by the chemical structure of the adsorbed substance and its state in the solution.

For example, when sunflower husk containing cellulose, lignin, and hemicellulose is treated with an alkali solution, it increases the solubility of the melanoid component and leads to the splitting of the insoluble complex of biopolymers, with the release of water-soluble polysaccharides. Removing water and alkaline substances from the raw material allows functional groups of lignin to become more accessible for heavy metal ions. The result is the intensification of the chemisorption process. The alkaline medium promotes the formation of hydroxides of metals, which remain in the pores of the adsorbent and do not require extraction. Acid treatment of plant material waste results in partial hydrolysis of lignin. This causes the deterioration of the sorption characteristics of the biosorbent. The deterioration of the biosorbent's sorption properties, in comparison with the previous two methods of treatment, impede the process of chemisorption due to a significant amount of ballast substances [14].

Economic efficiency of biosorbents production. The indicators of economic efficiency of production should also be taken into account during the transition to the industrial production of biosorbents. After all, high costs can negate all the benefits of natural sorbents. The article [4] determines the economic efficiency of biosorbents. According to the authors, the cost of 1 ton of biosorbents (prices of 2015) is: biosorbent obtained by acid and alkaline (HCl and NaOH) treatment of sunflower husk – 1944 UAH/t; biosorbent obtained by low-temperature treatment of sunflower husk – 1303.2 UAH/t. To compare, the cost of medical activated carbon is 2685.6 UAH/t. These data demonstrate the economic attractiveness of the sorbents production based on plant raw material. Such biosorbents, in addition to effectively removing copper ions from wastewater, are also cheaper than traditional wood activated carbon.

Thus, an overview of the state of technologies of sorbents based on waste from secondary plant and animal raw materials has shown that scientific research in this direction is being actively carried out. A reason for this is the possibility to organize the process of wastewater purification from heavy metal ions more effectively and in a cheaper way. As the development of agriculture and the food industry is a priority task, the development of biosorbent production will be relevant for Ukraine. That is why further research will focus on analyzing Ukraine's raw material base in order to select objects for biosorbents production, on developing technologies for obtaining new sorption materials, and on the experimental study of the kinetics of the processes of wastewater sorption purification from heavy metal ions.

Conclusions

1. As a result of the literary review, it has been determined that raw materials can be used as effective biosorbents having in their chemical composition a high content of cellulose, lignin, hemicellulose, pectin and polyphenolic substances.

2. In order to improve the sorption properties of biosorbents based on the waste from processing plant and animal raw materials, their special treatment is required. It depends on the characteristics of the raw material's chemical composition and may include such processes as mechanical grinding, carbonization, pyrolysis, acid and alkaline hydrolysis.

3. Mechanisms of heavy metal ions biosorption from wastewater are individual for each filter material and based on a physical, physico-chemical, or biological process, or on a complex of such processes.

4. The sorption exchange capacity of most biosorbents is the same as that of wood activated carbon traditionally used in water treatment, and in some cases, is even better. The cost of carbon biosorbents is 1.3–2.0 times lower than that of industrially produced wood activated carbon.

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