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Editor
Komarytskyy M.L.
Ph.D. in Economics, Associate Professor

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ТЕХНИЧЕСКИЕ НАУКИ

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PHOSPHORYLATION OF LIGNOCELLULOSE FOR THE PURPOSE OF WASTEWATER TREATMENT

Halysh Vita,
Trus Inna,
Deykun Iryna,
Benatov Daniel,
Ploskonos Victor,
PhD, Associate Professor
Gomelya Mykola
Doctor of Technical Sciences, Professor
Vozovych Anton,
Horianoi Volodymyr
Student
National Technical University of Ukraine
"Igor Sikorsky Kyiv Polytechnic Institute"
Kyiv, Ukraine

Introductions. The problem of pollution of water bodies with pollutants of various natures is now acute in many countries around the world. The rapid development of heavy and light industry requires the development of efficient energy-saving low-waste technologies [1-6]. The involvement of "green technologies" for the greening of production is also a priority. For this purpose, it is advisable to use secondary plant resources, namely waste and by-products of the agro-industrial complex in technologies to protect the environment from toxicants of inorganic and organic origin [7-8].

Millions of tons of plant waste and by-products of agriculture and food industry are generated annually in Ukraine and other countries, and only a small part of which is further used as raw material for other industries (microbiological, chemical, pharmaceutical, cosmetic, etc.); as fodder for cattle and poultry and as fertilizers [9]. The most common method of solid waste disposal is incineration, which prevents the production of new products and leads to pollution of the environment by combustion products. Therefore, there is a need to develop new effective ways to dispose of plant waste.

In recent years, scientists have performed a large amount of work to study the use as sorbents of various plant materials, based on wood waste or chemical products of wood [10], agricultural waste [11], shells of fruit and berry crops [12], to purify aquatic environments of various composition. Plant raw materials contain organic compounds of various chemical classes (aldehydes, ketones, saturated and unsaturated fatty acids, etc.), as well as inorganic substances, which determines the possibility of its use in sorption technologies. However, in the untreated state, such materials are characterized by low sorption capacity, which is due to the low-fibrillation structure, high density and low content of available active functional groups [13]. To increase the absorption properties, it is possible to carry out chemical modification of plant polymers at elevated temperatures using different reagents, which allows to give them new properties by increasing the specific surface area or by introducing additional active functional groups [14].

One of the methods of obtaining effective sorbents is phosphorylation. The production of such sorbents on the basis of vegetable waste consists either in phosphorylation of their carbon residue obtained under pyrolysis conditions, or in carbonization of lignocellulosic raw materials previously impregnated with phosphorylating reagents. As a result of such treatment, the final product acquires ion exchange properties due to the presence of phosphoric acid groups. The yield of the target product from natural raw materials during carbonization is quite low and is less than 30%.

Another way to obtain effective multi-purpose sorbents can be phosphorylation of plant waste without further treatment at high temperatures without access to oxygen. In this case, both functional groups of cellulose and lignin will be subjected to phosphorylation. In addition, depending on the conditions, there will be a partial destruction of low molecular weight polysaccharides, removal of extractives of different nature, which leads to the formation of a more developed porous structure of sorption materials.

Aim. The aim of the work is development of a method of chemical modification of walnut shells of different fractional composition using orthophosphate acid.

Materials and methods. For research as a starting material crushed walnut shells were used, the content of the main components in which was determined in accordance with generally accepted methods widely used in the field of chemical processing of wood and vegetable raw materials. The volume of adsorption pores was determined by adsorption of benzene vapor at a temperature of 18 oC in a desiccator.

The crushed raw materials were sorted into fractions and stored in a desiccator to maintain constant humidity and chemical composition. In the experiments used three fractions of the source material: 0.5 mm> fraction A> 1.0 mm; 1.0 mm> fraction B> 1.5 mm; 1.5 mm> fraction C > 2.0 mm.

Modification of the nut shell was performed by treatment with H_3PO_4 solutions in heat-resistant glass flasks in a water bath using reflux condensers to prevent loss of components of the modifying solution and to maintain a constant value of the hydromodule 5: 1. The duration of the treatment was from 60 to 180 min, the acid concentration was from 5 to 75%, the temperature of the reaction mixture was 95 °C. At the end of the treatment, the phosphorylated lignocellulosic product was separated from the solution by filtration, washed with distilled water to neutral pH values of the wash water, dehydrated and dried to a humidity of 5-6%.

The gravimetric method was adopted to determine the yield of biosorbents. The absorption capacity of the starting material and modified materials towards methylene blue was determined under static conditions using a model solution with a dye concentration of 100 mg/l. The process duration is 24 h. The initial and equilibrium concentrations of sorbate were determined by spectrophotometric method on a SPEKOL 11 spectrophotometer. The wavelength was 630 nm.

Studies of ion-exchange properties (static exchange capacity) of untreated and phosphorylated raw materials were determined by the amount of Na^+ sorption from 0.1 N NaOH solution during 24 h.

Mathematical processing of experimental and multicriteria optimization was performed using computer software. To reproduce the experimental data, a second-order polynomial was used, which for two independent variables has the form:

$$y_i = b_0 + b_1x_1 + b_2x_2 + b_3x_1x_2 + b_4x_1^2 + b_5x_2^2,$$

where y_i – indicators of phosphorylated lignocellulosic sorbents; b_0, b_1, b_2, b_3, b_4, b_5 – coefficients of the mathematical model; x_1 i x_2 – values of factors.

Variable functions (y_i) were determined by the following indicators of sorbents: y_1 - product yield,%; y_2 - sorption efficiency of methylene blue,%; y_3 - static exchange capacity for Na^+, mg-eq/g.

Results and discussion. The primary task of the research work was to determine the chemical composition of the starting material and to estimate the volume of adsorption pores. For this purpose, crushed walnut shells were used, namely fraction A. The results of the study are given in Table 1 and for comparison also the chemical composition of other representatives of solid plant waste of the agro-industrial complex is shown.

As can be seen from the data, the cellulose content in walnut shells is close to the content of the same component in the shells of cherry, peach, apricot and buckwheat husks, but much lower than in collapsed corn cobs. The content of lignin in the studied raw material, which usually limits the diffusion of reagents to the intercellular space, is much lower compared to other representatives of plant waste. The same can be said about the content of mineral components. The lower content of the polyaromatic component may indicate a faster course of sorption processes.

Substances extracted with water and 1% alkali solution include inorganic salts, starch, pectins, some low molecular weight polysaccharides, cyclic alcohols, dyes and tannins, etc., i.e. different classes of organic substances that contain different functional groups that can take part in the reactions of accession, substitution, complexation and other.

Table 1

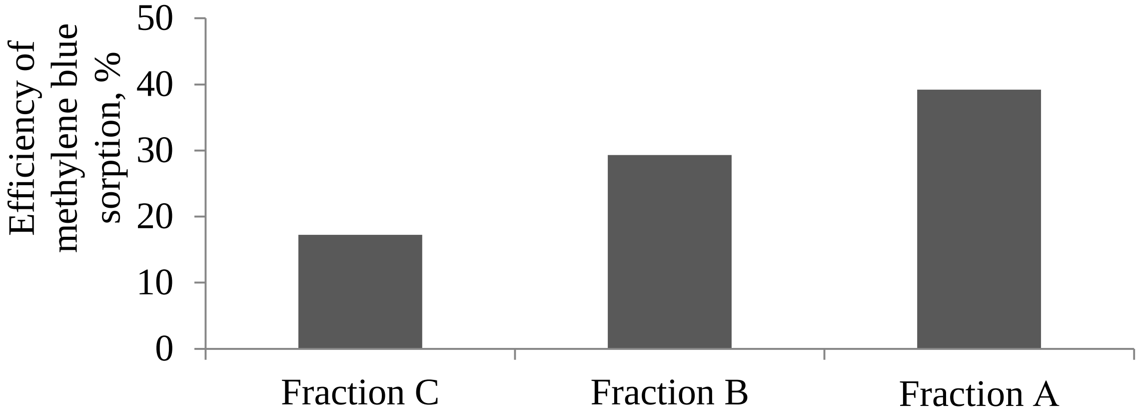
Characteristics of vegetable waste from the food industry

Material	Component content, %						Adsorption pore volume, cm^3/g
	Cellulose	Lignin	Ash	Extractives			
				Alcohol-benzene mixture	Hot water	1% solution ofNaOH	
Walnut shells	41,2	37,5	2,3	5,2	10,4	25,0	0,03
Cherry seed shells	41,2	51,3	3,8	-	-	-	0,01
Apricot seed shells	34,6	48,1	2,5	-	-	-	0,03
Peach seed shells	38,3	50,3	5,1	-	-	-	0,02
Buckwheat husk	46,7	52,4	3,5	-	-	-	0,03
Corn cob	70,7	28,6	4,3	-	-	-	0,16

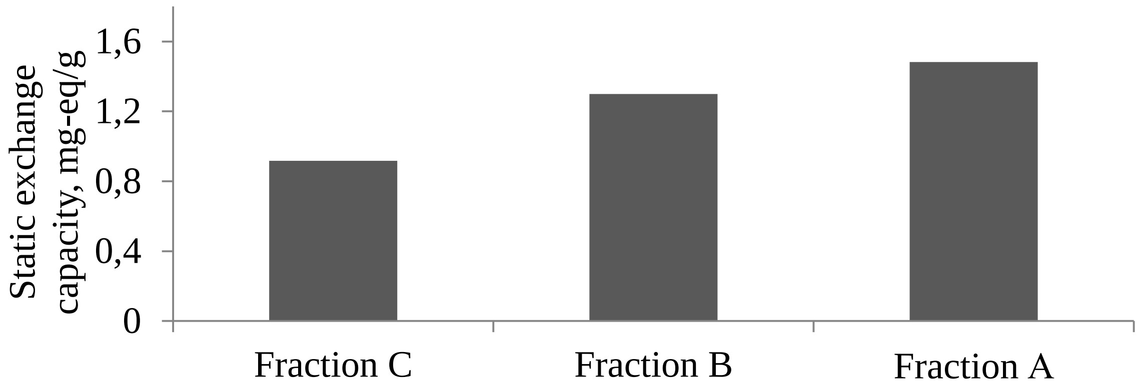
In terms of the volume of adsorption pores, the walnut shell is not inferior to other representatives of plant waste, but it is significantly inferior to corn cobs. The sorption properties of plant materials can be increased by machenical tretment, which consists in grinding the raw material, which increases the availability of active functional groups, as well as the specific surface area of the material, the volume of adsorption pores.

Fig. 1 shows the results of the study of sorption properties, namely the efficiency of removal of methylene blue from aqueous solution and static exchange capacity for Na^+ onto starting material of different fractional composition. The results of the study showed that the fractional composition of the material significantly affects its absorption properties. Reducing the size of the natural raw material leads to an increase in the efficiency of extraction of methylene blue from 17.2% for fraction

B with dimensions of 1.5-2.0 mm to 39.20% for fraction A with dimensions of 0.5-1.0 mm The static exchange capacity increases by an average of 30% for each subsequent fraction.



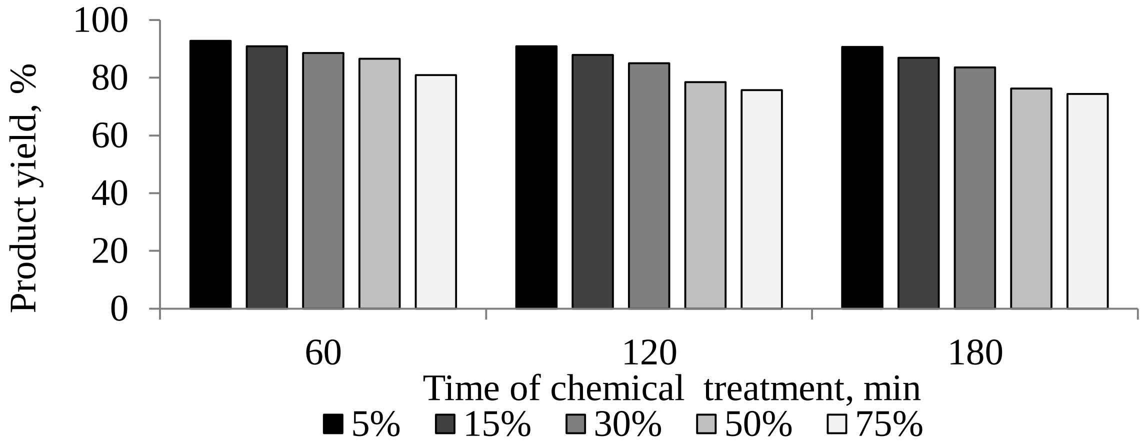
a



b

Fig. 1. Sorption properties of walnut shells of different fractional composition: a - efficiency of removal of methylene blue from aqueous solution, b - static exchange capacity for Na^+

To give the starting material additional sorption properties along with machenical treatment, it is advisable to use chemical modification. Fraction B of crushed walnut shells was used to study the effect of H_3PO_4 concentration and duration of treatment on the properties of phosphorylated lignocellulosic materials. The research results are presented in Fig. 2.



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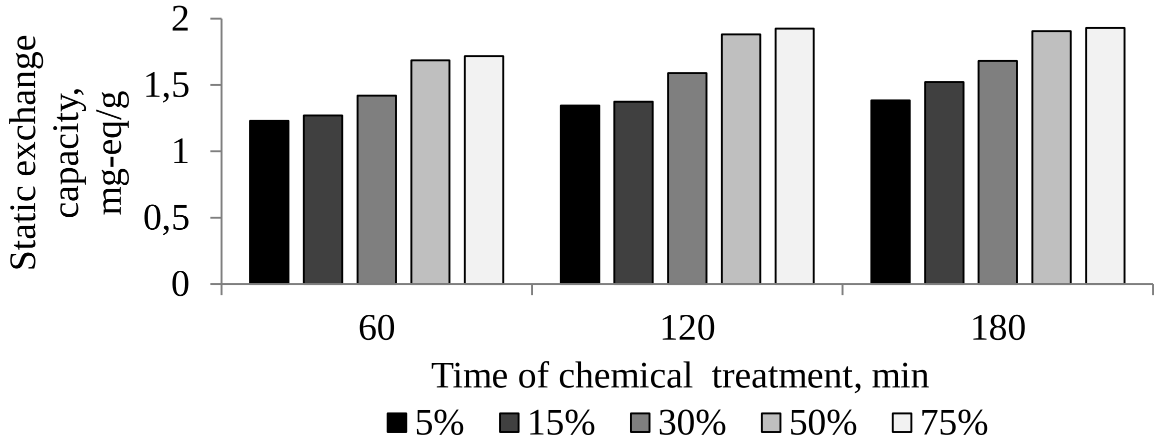

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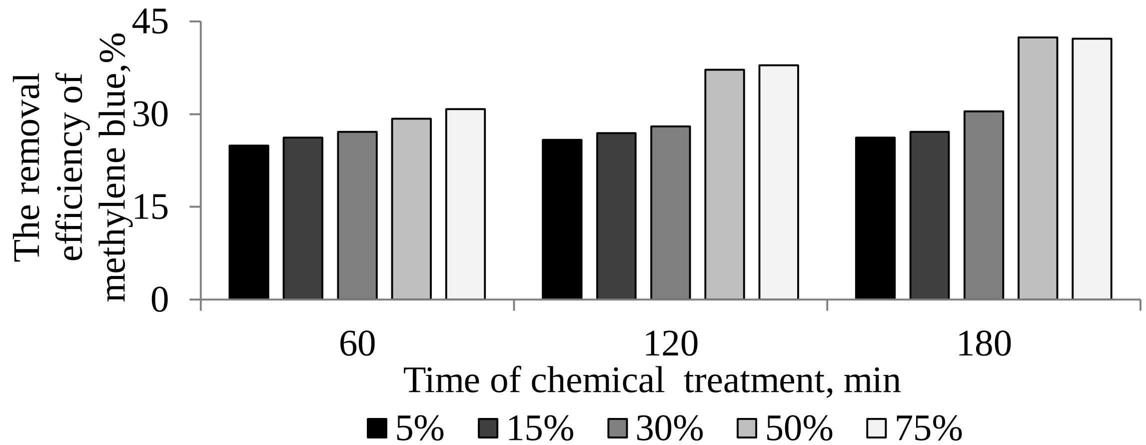

c

Fig. 2. The effect of H_3PO_4 and time of treatment on the yield (a) of product, static exchange capacity Na^+ (b) and methylene blue removal efficiency (c)

With an increase in the concentration of inorganic acid from 5 to 75%, there is a decrease in the yield of the final product for the entire studied range of process duration is observed (Fig. 2a). Moreover, the first 60 minutes of processing corresponds to the maximum reduction in product weight. This, in turn, may indicate the dissolution of extractives and low molecular weight fraction of polysaccharide. The subsequent increase in the duration of processing to 180 min has little effect on the yield of phosphorylated lignocellulosic product. With increasing concentration of inorganic acid, there is an increase in static exchange capacity is observed (Fig. 2b). Further processing does not lead to an increase in this indicator. In the process of phosphorylation of raw materials with increasing process parameters also increases the absorption efficiency (Fig. 2c), and its value increases with increasing concentration of H_3PO_4 to 75%, and with increasing time to 180 min. This can be explained by the fact that in the process of modification there is a removal from plant raw materials of different components takes place. The absorption of the cationic dye on such sorbents occurs both due to physical adsorption and by the mechanisms of chemisorption with the participation of functional groups of the modified plant material.

According to the results of the experiment, the regression equations of the process of phosphorylation of walnut shells were calculated, which have the following form:

$$y_1 = 98,084 – 0,0377x_1 – 0,101x_2 – 0,0013x_1x_2 – 0,0005x_1^2 + 0,0003x_2^2;$$

$$y_2 = 25,25 – 0,1589x_1 + 0,0128x_2 + 0,0021x_1x_2 + 0,0012x_1^2 – 0,00069x_2^2;$$

$$y_3 = 0,937 + 0,0051x_1 + 0,0051x_2 + 0,000077x_1x_2 + 0,00023x_1^2 – 0,00014x_2^2.$$

The values of the correlation coefficients for the above equations is close to 1, which indicates an adequate description of the process of obtaining phosphorylated lignocellulosic sorbents.

Conclusions. The chemical composition of walnut shells was studied and the volume of adsorption pores was estimated. It was found that the raw material consists of 41.2% cellulose, 37.5% lignin, also contains ash in the amount of 2.3%, substances extracted with an alcohol-benzene mixture, hot water and 1% alkali solution - 5 , 2, 10.4 and 25.0%, respectively, the volume of adsorption pores is 0.03 cm^3/g.

The influence of the fractional composition of the raw material on the sorption capacity, namely on the static exchange capacity for Na^+ and the efficiency of methylene blue removal was studied. It is shown that reducing the size of fractions from 1.5-2.0 mm to 0.5-1.0 mm leads to an increase in the efficiency of extraction of organic dye by 22% and to an increase in static Na^+ exchange capacity.

The influence of the main conditions of the process of phosphorylation of the raw material on the properties of the final product is investigated. It was determined that with an increase in the acid concentration to 75% and the duration of modification to 120 min, the static exchange capacity for Na^+ reaches the highest value, which is 1.9 mg-eq/g. The maximum value of the sorption efficiency of methylene blue is achieved at a concentration of the modifier in solution of 50% and a duration of 180 min and is 42.4%.

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