

## Activated carbons from vegetable waste

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**Abstract:** The paper substantiates the probability of solving a wide range of problems dealing with environmental protection from technogenic pollution with the help of activated carbons (ACs). One of the most important areas of protection against ecocide is detoxification of farmland soils from herbicide residues. It has been established that vegetable waste from agricultural crops, especially straw, is a valuable multi-tonnage raw material for the production of activated carbons. Activated carbons have been produced from a wide range of vegetable waste: straw (16 types), husks and sludge of coffee beans, husks of grains and sunflower stalks, rice husks, as well as nut shells and seeds of fruit trees. The adsorption properties (adsorption activity for iodine and methylene blue), porous structure parameters (total pore volume, mesopore volume, micropore size, total specific surface area, etc.) and other quality indicators of produced vegetable activated carbons (bulk density, mass fraction of ash, strength at abrasion) have been evaluated. Detoxification of soils from the remains of potent herbicides – Singer SP (active ingredient metsulfuron-methyl) has been studied. The fields of application of the produced vegetable ACs in various fields of adsorption technology have been proposed.

**Keywords:** vegetable raw materials; straw, activated carbons, porous structure; micropores; adsorption activity; soil detoxification; application of activated carbons.

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## Активные угли из растительных отходов

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**Аннотация:** Обоснована вероятность решения широкого круга проблем защиты окружающей среды от техногенных загрязнений с помощью активных углей (АУ). Показано, что одним из важнейших направлений защиты от экоцида является детоксикация почв сельхозугодий от остатков гербицидов. Установлено, что растительные отходы сельхозкультур, и, прежде всего, солома, являются ценным многотоннажным сырьем для получения активных углей. Получены активные угли из широкого спектра растительных отходов: соломы (16 видов), шелухи и шлама кофейных зерен, шелухи зерен и стеблей подсолнечника, рисовой шелухи, а также скорлупы орехов и косточек плодов фруктовых деревьев. Оценены адсорбционные свойства (адсорбционная активность по йоду и по метиленовому голубому), параметры пористой структуры (суммарный объем пор, объем мезопор, размер микропор, общая удельная поверхность и т.п.) и другие показатели качества (насыпная плотность, массовая доля золы, прочность при истирании) полученных растительных активных углей. Исследована детоксикация почв от остатков сильнодействующих гербицидов – Зингер СП (действующее вещество метсульфурон-метил). Предложены области применения получаемых растительных АУ в различных областях адсорбционной техники.

**Ключевые слова:** растительное сырье; солома; активные угли; пористая структура; микропоры; адсорбционная активность; детоксикация почв; применение активных углей.

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## 1. Introduction

Our planet is constantly facing various environmental problems. Most of them result from overexploitation of natural resources at such an accelerated rate that they cannot be restored at the same rate. All this entails the degradation of the environment as a result of human abuse. Human consumption is based on the acquisition of products that exceed our ability to regenerate [1].

Changing environmental risks resulted in 13.7 million deaths in 2016, representing 24 % of the world's total death. This means that almost one in four deaths worldwide was caused by environmental conditions. Disease agents and exposure pathways are numerous, and unhealthy environmental conditions are responsible for most categories of disease and injury [2].

Among all methods of protecting water resources, sorption is considered easier more economical to perform and manage. In addition to the main advantages, sorption does not cause secondary pollution from the formation of by-products. Minerals, organic and inorganic materials commonly used as adsorbents (e.g. clay, activated carbon (AC), industrial by-products, zeolite, polymeric materials, biofuels, agricultural waste, etc.) have a high adsorption capacity for removal various pollutants from wastewater [3–5].

In the light of the foregoing, special attention should be paid to the environmental safety of the agrarian complex, which provides the population with food, because agricultural soils on the planet is only 6 % of the total land area, and the number of inhabitants at the end of the 21st century will be 10 billion [6].

In particular, environmental technologies for the use of activated carbons cover all components of the biosphere: atmosphere, hydrosphere, lithosphere, and the man as the main object of the biosphere.

Activated carbons form a large class of porous solids that are widely used in industry. The porous structure of these materials allows them to be used in the adsorption of gases, vapors, and liquids [7, 8]. AC is a black amorphous solid containing a high percentage of carbon and other materials such as ash, water vapor and volatiles in a lower percentage. The large surface area of ACs provides a high ability to absorb chemicals from gases or liquids. This

property is related to the extensive internal pore structure that develops during the activation process.

Among the wide variety of initial carbon-containing materials for the production of ACs in recent years, various vegetable wastes as a constantly renewable source of raw materials are of particular interest.

The intensification of crop production in all advanced agro-industrial countries has led to a significant increase in straw the volume of cereals, oilseeds, industrial and other crops. So, only the straw volume of grain and cereal crops in Russia is over 80–100 million tons [9]. These straw volumes are currently disposed not quite rationally: they are plowed into the soil or burned. At the same time, crop straw is a promising raw material for obtaining activated carbons on its basis. On the other hand, the intensification of crop production causes the introduction of a large amount of potent herbicides into the soil. This approach makes it possible to protect cultivated plants from weeds, but significantly reduces the yield of crops cultivated in crop rotation (the so-called aftereffect of herbicide residues). Academician Yu.Ya. Spiridonov and his colleagues showed in their studies that the introduction of activated carbon into the soil at a dose of 50–100 kg per hectare can increase the yield of crops cultivated in crop rotation by 20–80 % and ensure the receipt of environmentally friendly crop production [10–13]. Therefore, the rationality of obtaining activated carbons from agricultural straw is due to the fact that such ACs will immediately be introduced into the soil at the place of the raw materials formation in the same areas.

Another potential vegetable material for the AC production is the large-tonnage waste of the food industry that processes vegetable materials. In this regard, multi-ton vegetable waste from the production of coffee, sunflower oil, as well as nut shells and fruit stones, which belong to the type of compacted vegetable raw materials, are of great promise [14–23].

Taking into account the structure peculiarities of each type of vegetable waste in each specific case, it is necessary to select the optimal modes of thermal treatment of raw materials at carbonization and activation stages in order to ensure the optimal development of the microporous structure and high strength (especially in the case of compacted plant

materials), and on the other hand, prevent ashing of the granules.

Thus, the aim of the study is to obtain new activated carbons from a wide range of large-tonnage vegetable waste, study the porous structure and adsorption properties, and predict the most rational areas for their application.

## 2. Materials and Methods

### 2.1. Preparing ACs from the straw of various crops by steam-gas activation method

As shown by the analysis of the straw composition of a number of agricultural crops (see Table 1), the carbon content in dry matter is 38–40 %, which predicts the possibility of its use to obtain activated carbons.

The procedure for producing ACs from the straw of various crops was as follows. The straw was crushed, loaded into a steel retort, which was closed with a lid with outlets, and placed in an electric furnace with nitrogen supplied to the retort to create an inert atmosphere. The retort was heated at a temperature rise rate of 5–7 °C per min to 450–500 °C and kept at the final carbonization temperature for 30–60 minutes. After completion of the carbonization process, the retort was switched to the steam activation mode at 850–870 °C. The activation time varied from 40 to 60 minutes. After completion of the activation process, the retort was cooled to room temperature and the resulting product was unloaded [24, 25].

The quality of activated carbons was determined according to the methods adopted in adsorption technology: total pore volume ( $V_{\Sigma}$ ,  $\text{cm}^3 \cdot \text{g}^{-1}$ ) according to Russian Standard 17219–71; bulk density ( $\Delta$ ,  $\text{g} \cdot \text{dm}^{-3}$ ) according to Russian Standard 16190–70; adsorption activity for iodine according to Russian Standard 6217–74 in %; adsorption activity for methylene blue (MB) according to Russian Standard 4453–74 in  $\text{mg} \cdot \text{g}^{-1}$ ; mass fraction of ash ( $A^c$ , %) according to Russian Standard 12596–67.

Studies of the microporous structure of activated carbons were carried out by measuring nitrogen adsorption isotherms at a temperature of  $-196$  °C (77.4 K) on an ASAP 2020 device (Micromeritics, USA) using Dubinin-Radushkevich theory of volumetric filling of micropores:  $W_s$  is the volume of the sorption space,  $\text{cm}^3 \cdot \text{g}^{-1}$ ;  $V_{\text{mic}}$  is the volume of micropores,  $\text{cm}^3 \cdot \text{g}^{-1}$ ;  $x$  is the size of micropores, nm;  $S_{\text{sp}}$  is the total specific surface area,  $\text{m}^2 \cdot \text{g}^{-1}$ ;  $E_{\text{ad}}$  is the characteristic energy of adsorption,  $\text{kJ} \cdot \text{mol}^{-1}$ ;

**Table 1.** Carbon content in straw dry matter

Straw	Straw composition, %	
	Dry matter	Carbon (C) in dry matter
Winter wheat		40
Winter rye	86	38
Barley		40
Oats		39
Rapeseed	85	38
Oil radish	84	39
Colza		40
Soy	86	37

$S_{\text{mic}}$  is the specific surface area of micropores,  $\text{m}^2 \cdot \text{g}^{-1}$ ;  $S_{\text{mes}}$  is the specific surface area of mesopores,  $\text{m}^2 \cdot \text{g}^{-1}$  [26–28].

### 2.2. Producing ACs from the straw of various agricultural crops by chemical activation method

Literary sources show that it is possible to significantly improve the quality of activated carbon produced from loose vegetable materials, such as straw, sawdust, lignins, cellolignins, powdered peat, and others, through the use of chemical activation methods, the essence of which consists in the impregnation of a carbon-containing base with alkalis or alkali salts followed by heat treatment [29, 30].

When producing high-quality activated carbons based on non-carbonized raw materials (peat, sawdust, crushed wood, technical lignins, etc.), it is important to choose a chemical activating agent (CAA) that, during the formation of carbon crystallites in carbonization and activation, would not only catalyze the reactions oxidation, but would also effectively be introduced into the interplanar distances of crystallites. Moreover, a correctly performed operation of preliminary drying of the initial vegetable material ensures high-quality impregnation of the CAA wood raw material, and the selection of carbonization and activation modes in the combined process forms the desired pore size. The CAA washing stage opens the formed microporous structure at a minimum consumption of washing liquids. Taking into account that powder forms of activated carbons are used to purify liquid media, the quality of the produced carbons are assessed by the efficiency of extracting iodine and MB dye substances from water, which are accepted as test substances in international practice.

To assess the effectiveness of chemical activation of agricultural straw, rapeseed and white mustard straw were chosen as the raw material.

The method was carried out as follows. The initial straw was crushed to a particle size of 1–8 mm. The crushed raw material was placed in a steel retort and dried at a temperature of 280–600 °C in an atmosphere of steam gases (water vapor and light volatile substances released during heating) with holding at the final temperature for 1–3 hours. After the drying process was completed, the product was placed in a mixer equipped with a stirrer, and a saturated solution of a CAA was added in portions with a density of 1.3–1.5 g·cm<sup>-3</sup> and a ratio of the dried raw material and agent equal to 1 : (1.75–2,5). NaOH or KOH was used as CAA. The impregnation was carried out at a temperature of 30–50 °C for 20–40 minutes with constant stirring of the dry crushed raw material and the CAA solution. The impregnated product was then sent to the combined carbonization-activation process. For this, a chamber or drum-type furnace made of special steel was used in an atmosphere of steam gases at a temperature of 550–800 °C with a rise rate of 26–35 °C per min, holding at a final temperature for 1–2 hours. Upon completion of the activation process, the carburized material was unloaded from the furnace, cooled to 70–90 °C, and CAA was washed first with water, then with 1.5–5.0 % HCl, and again with water to pH = 4–6. The resulting powder activated carbon (PAC) was separated from the moisture in a filter press and dried at a temperature of 100–120 °C to a residual moisture content of 3–8 wt.%. Then, for analysis, the resulting activated carbon was crushed to a particle size of less than 100 µm.

### ***2.3. Producing activated carbons from food industry waste***

In the food industry, activated carbons are often used to extract impurities and clarify solutions. The expansion of the range of food activated carbons can be achieved through the use of various types of vegetable materials and waste from the food industry itself [31–33].

#### *Coffee production waste*

One of the promising sources of carbon-containing raw materials is coffee sludge, a product of processing coffee beans into instant coffee by extracting caffeine with solvents. The authors used coffee sludge from Moscow Coffee House on Shares as a starting material for producing activated carbon.

Carbonization of the initial sludge (mass fraction of ash – 1.0–1.5 %; mass fraction of moisture – 6 %; carbon content – 20–24 %) was carried out in a rotary electric furnace with a retort diameter of 54 mm in an inert nitrogen atmosphere at a temperature of 450 °C. Then the carbonizate was subjected to steam-gas activation in a muffle electric furnace at a temperature of 850 °C for 90 minutes. The yield of the finished product was 6 wt. %.

Another vegetable waste from coffee production is the husk formed during the primary processing of coffee beans. The procedure for producing AC from this type of raw material was carried out according to the same technological modes as for coffee sludge.

#### *Activated carbons from sunflower seed husks*

A promising multi-tonnage vegetable waste is sunflower seed husks formed at the stage of sunflower oil production [34].

Carbonization of the initial husk was carried out in a rotary electric furnace with a retort diameter of 54 mm in an inert nitrogen atmosphere at a temperature of 450 °C. Then the husk carbonizate was subjected to steam-gas activation in a muffle electric furnace at a temperature of 850 °C for 90 minutes. The yield of the finished product was 8.2 wt. %.

#### *Producing activated carbons from sunflower stalks*

Sunflower stalks are the largest non-utilizable part of this crop in terms of weight and volume. The carbonization of this type of raw material was carried out at 800 °C with holding at the final temperature for 30 minutes. Steam-gas activation of carbonizate was carried out at 820 °C for 1 hour.

#### *Producing activated carbon from rice husks*

Rice husk (RH) is a large-tonnage waste generated during threshing of rice and can be considered as a promising raw material for the production of activated carbons. The production of activated carbon from this type of raw material was carried out according to the mode established for coffee bean husks and coffee sludge. Since the strength of activated carbon from RH was low, its properties were determined by the quality indicators used to evaluate powder activated carbons. The closest analogue of RH activated carbon is industrial powdered carbon based on raw charcoal – coal of OU-A grade produced by JSC “Sorbent” (Perm) according to Russian Standard 4453–74 “Activated clarifying coal” [35].

#### *Activated carbons from nut shells and fruit stones*

Nut shells (walnut, cedar, Manchurian, hazelnuts, etc.) and fruit stones (peach, apricot, plum, etc.) are a type of compacted vegetable material.

The total waste of this type of vegetable material in the Russian Federation is several tens of thousands of tons per year. Having initial high strength, they make it possible to produce activated carbons that are not inferior in strength properties to activated carbons from coconut shell.

Peach and apricot stones, walnut and pine nut shells were chosen for research. The carbonization of the product was carried out in a carbon dioxide environment. 1 liter of product with a certain bulk density was loaded into a metal retort. The carbonization temperature was 450 °C at a temperature rise rate of 5–8 °C per minute, holding at the final temperature for 20 minutes. The produced carbonizate was crushed and subjected to sieving with separation of a fraction of 1.0–3.0 mm. The product yield during carbonization was 25–29 %.

The product was activated in the same retort with water vapor at 870–900 °C to a bulk weight of about 400 g·dm<sup>-3</sup>. The yield of the active product from the weight of the original shell or stone was 12–15 %.

### 3. Results and Discussion

#### 3.1. Characteristics of ACs produced from straw by steam-gas activation method

The characteristics of the produced activated carbons, which have been divided by segments of use, are presented in Table 2.

All activated carbons are characterized by a developed total pore volume  $V_{\Sigma}$  from 2.5 to 4.1 cm<sup>3</sup>·g<sup>-1</sup>. Such a highly developed total pore volume makes it possible to carry out well the impregnation and modification of such AC with various chemical additives obtaining chemisorbents and catalysts.

**Table 2.** Parameters of AC produced from straw of various crops

Sample	$V_{\Sigma}$ , cm <sup>3</sup> ·g <sup>-1</sup>	$W_s$ , cm <sup>3</sup> ·g <sup>-1</sup>	$\Delta$ , g·dm <sup>-3</sup>	Adsorption capacity according to		$A^c$ , %
				iodine, %	MB, mg·g <sup>-1</sup>	
<b>Oilseeds</b>						
Rapeseed	4.14	0.48	135	39	87	16.5
Camelina	2.43	0.69	140	43	82	15.6
Niger seed	2.45	0.27	106	44	58	33.6
<b>Mustards</b>						
Colza	2.28	0.57	135	62	73	22.1
White mustard	4.0	0.45	60.9	50	64	24.4
Sarepta mustard	2.81	0.57	111	56	67	27.3
<b>Grains</b>						
Barley	3.53	0.44	60.5	43	37	31.2
Wheat	3.61	0.73	66.5	64/20	52	12.2
Oats	3.97	0.44	72.5	50	44	28.2
Rye	3.42	0.62	70.0	52	49	14.7
<b>Food and fodder crops</b>						
Soy	2.27	0.51	108	49	61	30.8
Turnip	2.55	0.20	131	31	69	26.2
Amaranth	2.45	0.17	136	35	46	34.9
Jerusalem Artichoke	3.00	0.12	95.3	34	58	18.1
Clover	2.61	0.51	121	33	47	24.8
<b>Industrial crops</b>						
Flax	1.94	–	142	34	39	14.0

It is worth noting the well-developed volume of sorbing micro- and mesopores ( $W_s$ ) reaching 0.48–0.69  $\text{cm}^3 \cdot \text{g}^{-1}$  in oilseed rapeseed and camelina, and 0.44–0.73  $\text{cm}^3 \cdot \text{g}^{-1}$  in grains (wheat, rye, barley, and oats). This is obviously due to the good optimization of the carbonization and activation modes, which make it possible to achieve a good development of the volume of the sorption space even in such a fairly loose material. Thus, for example, for industrial powder ACs based on coal grades KAD-ground, UAF, UAM, it was 0.20–0.25  $\text{cm}^3 \cdot \text{g}^{-1}$ . Taking into account the fact that the adsorption activity for iodine and MB is 35–64 and 47–87 %, respectively, such carbons are quite applicable for wastewater treatment and even, possibly, for drinking water purification by carbonization (introduction of coal pulp into the reaction chamber) at waterworks.

Produced activated straw-based carbons in a granular form (particle size 0.5–5.0 mm), of course, had low strength, which cannot be determined by the method of Russian Standard 16188–70. However, they are sufficiently structured, which makes it easier to extract them from the liquid phase after the adsorption of impurities. That is, liquid-phase adsorption processes are much easier to carry out on them than on powder AC, due to the simplification of the filtration stage after purification.

The bulk density, as a rule, is in the range of 100–140  $\text{g} \cdot \text{dm}^{-3}$ , although low, but significantly higher than that of AC produced from rice husks, coffee bean husks and similar agricultural waste – 30–50  $\text{g} \cdot \text{dm}^{-3}$ . This also speaks in favor of their more convenient application in the processes of water purification and water treatment.

Oilseed-based activated carbons (rapeseed and camelina) had a higher density of 135–140  $\text{g} \cdot \text{dm}^{-3}$  and, consequently, higher strength. This is due to the fact that oils contained in them play the role of a binder and compact the frame of the finished ACs. As a result, during activation, a large volume of the sorption space  $W_s$  develops, which leads to high adsorption of iodine 39–44 % and MB 82–87  $\text{mg} \cdot \text{g}^{-1}$ .

All mustards (colza, white mustard, Sarepta mustard) had a developed volume of  $W_s$  and, therefore, good adsorption of iodine 44–50 % and MB 64–73  $\text{mg} \cdot \text{g}^{-1}$ .

Grain straw (barley, wheat, oats, rye) had perhaps the highest  $W_s$  – 0.44–0.73  $\text{cm}^3 \cdot \text{g}^{-1}$  and, therefore, good adsorption properties: 50–64 % iodine and 44–52  $\text{mg} \cdot \text{g}^{-1}$  by MB. This is obviously due to the denser structure of the original straw and,

hence, the greater formation of carbon crystallites at the carbonization stage.

It is necessary to single out the ACs from soybean straw, which had  $W_s$  0.51  $\text{cm}^3 \cdot \text{g}^{-1}$  and adsorption of iodine and MB 49 % and 61  $\text{mg} \cdot \text{g}^{-1}$ , respectively.

ACs based on radish, amaranth, Jerusalem artichoke, and clover straw also had good adsorption and physicochemical characteristics, but were inferior to AC groups from oilseeds, grains and mustard crops.

The ash content in all ACs was quite high and averaged 20–25 %. For industrial ACs, this figure was 10–15 %, and for good carbons based on coconut shells – it reached only 3–5 %. This means that it is not desirable to use ACs from straw for refining and clarifying solutions and liquids and obtaining highly pure substances.

It should be noted that the yield of the finished product was quite high – 8–12 % of the weight, which implies a good profitability of the industrial production of vegetable activated carbons (VACs).

The parameters of the microporous structure of vegetable active carbons are presented in Table 3.

The analysis of the parameters the AC microporous structure, shown in Table 3, allows us to draw the following conclusions. The volumes of micropores were quite well developed and lay in the range of 0.12–0.20  $\text{cm}^3 \cdot \text{g}^{-1}$  (with the exception of amaranth – 0.05  $\text{cm}^3 \cdot \text{g}^{-1}$ ). At the same time, the greatest development of micropores among oilseeds was observed in rapeseed – 0.160  $\text{cm}^3 \cdot \text{g}^{-1}$ , and among grains it was observed in wheat – 0.200  $\text{cm}^3 \cdot \text{g}^{-1}$ . Niger (0.185  $\text{cm}^3 \cdot \text{g}^{-1}$ ) and Jerusalem artichoke (0.180  $\text{cm}^3 \cdot \text{g}^{-1}$ ) should be noted among other straw types.

Regardless of the straw type, the sizes of micropores (full width) were almost the same and amounted to 1.51–1.58 nm (except for camelina – it had larger micropores of 1.66 nm), which predetermines the same adsorption energy  $E_{ad}$  – 21.0–24.0  $\text{kJ} \cdot \text{mol}^{-1}$  (with a small exception of camelina – 19.8  $\text{kJ} \cdot \text{mol}^{-1}$ ); it was slightly higher in Niger and Jerusalem artichoke, which indicates thinner micropores in AC from the straw of these crops. The total specific pore surface area ( $S_{sp}$ ) for all ACs was in the range of 400–520  $\text{m}^2 \cdot \text{g}^{-1}$  (except camelina, colza and turnip, in which it lay in the range of 320–380  $\text{m}^2 \cdot \text{g}^{-1}$ ). Narrower micropores (1.51–1.53 nm) will better absorb low molecular weight substances such as phenol. At the same time, on the contrary, larger micropores (1.60–1.66 nm) will better adsorb medium-molecular substances such as organ chlorines.

**Table 3.** Volume and sizes of micropores according to adsorption-desorption isotherms

Sample	$V_{mic}$ , $\text{cm}^3 \cdot \text{g}^{-1}$	$x$ , nm	$S_{sp}$ , $\text{m}^2 \cdot \text{g}^{-1}$	$E_{ad}$ , $\text{kJ} \cdot \text{mol}^{-1}$
<b>Oilseeds</b>				
Rapeseed	0.161	1.58	454	22.7
Camelina	0.135	1.66	380	19.8
Niger seed	0.185	1.51	521	25.5
<b>Mustards</b>				
Colza	0.124	1.53	350	24.9
White mustard	0.161	1.57	454	23.2
Sarepta mustard	0.163	1.50	454	26.2
<b>Grains</b>				
Barley	0.126	1.55	354	24.3
Wheat	0.200	1.54	564	24.4
Oats	0.162	1.54	456	24.6
Rye	0.160	1.54	450	24.3
<b>Food and fodder crops</b>				
Soy	0.160	1.56	451	23.9
Turnip	0.116	1.62	328	21.0
Amaranth	0.0458	1.57	129	21.0
Jerusalem Artichoke	0.180	1.53	506	25.1
Clover	0.144	1.60	405	22.1
<b>Industrial crops</b>				
Flax	0.13	1.61	321	21.5

Note:  $V_{mic}$  is the volume of micropores,  $\text{cm}^3 \cdot \text{g}^{-1}$ ;  $x$  is the width of micropores, nm;  $S_{sp}$  is the specific surface area,  $\text{m}^2 \cdot \text{g}^{-1}$ ;  $E_{ad}$  is characteristic energy of adsorption,  $\text{kJ} \cdot \text{mol}^{-1}$ .

The high development of meso- and micropores (which is expressed by the sorption pore volume  $W_s$ ) in all ACs will contribute to good adsorption kinetics and high adsorption activity when extracting high-molecular toxicants such as herbicides, dioxins, carcinogens, oil products, etc. from water and soil solutions.

### 3.2. Characteristics of AC from straw of various agricultural crops by chemical activation method

Table 4 shows the data on the adsorption activity of AC produced from rapeseed and white mustard straw by chemical activation method.

**Table 4.** Adsorption activity of AC from rapeseed and white mustard straw

AC sample	CAA, value	Ash, content, %	Adsorption capacity	
			by iodine, %	by MB, $\text{mg} \cdot \text{g}^{-1}$
Rapeseed straw	2.0 NaOH	2.3	240	500
White mustard straw	1.75 NaOH	3.8	150	350
OU-A (Russian Standard 4453–74)	–	–	70	225

The results presented in Table 4 show that the adsorption capacity for AC test substances from rapeseed and white mustard straw, produced by chemical activation, reached very high values: 1.5–3.0 times higher than that of industrial AC grade OU-A based on birch wood.

If we compare the AC quality given in Table 4 with the results of the adsorption capacity of AC from rapeseed and white mustard straw, produced by steam-gas activation method (see Table 2), it was almost 3–5 times higher.

The parameters of the porous structure calculated according to the theory of volumetric filling of micropores are shown in Table 5.

The results given in Table 5 show that chemical activation makes it possible to increase the volume of micropores bringing it to  $0.79 \text{ cm}^3 \cdot \text{g}^{-1}$  for AC from rapeseed straw and  $0.81 \text{ cm}^3 \cdot \text{g}^{-1}$  for white mustard straw. The share of micropores in the total pore volume was 71–86 %. Such a development of the microporous structure in activated carbons from straw produced by chemical activation made it possible to increase adsorption properties for the test substances, i.e. iodine and blue methylene, by several times.

However, it should be noted that the chemical activation method is much more complicated than the steam-gas activation method and it is difficult to implement it even in a large agricultural holding.

Therefore, having shown the possibilities of the chemical activation method, the authors abandoned it in further studies.

### 3.3. Characteristics of AC produced from food industry waste

#### 3.3.1. Characteristics of AC produced from coffee grounds

The results of determining the properties of AC are given in Table 6.

The data in Table 6 indicates that the adsorbent UKSH-D from coffee sludge meets the requirements for carbons used in the food industry.

The qualitative characteristics of activated carbon produced from coffee husks are given below:

- bulk density –  $93 \text{ g} \cdot \text{dm}^{-3}$ ;
- ash content 35 wt. %;
- moisture content – 2.2 wt. %;
- total pore volume –  $0.52 \text{ cm}^3 \cdot \text{g}^{-1}$ ;
- volume of the sorption space by benzole –  $0.38 \text{ cm}^3 \cdot \text{g}^{-1}$ ;
- adsorption capacity by iodine – 55 %;
- adsorption capacity by MB –  $112 \text{ mg} \cdot \text{g}^{-1}$ .

Thus, in terms of its quality characteristics, the experimental sample of AC from coffee bean husks is at the level of industrial powder activated carbon of the UAF brand (TC 6-16-2409–80).

**Table 5.** Parameters of the porous structure of AC from rapeseed and white mustard straw produced by chemical activation method

Source straw	$V_{\Sigma}, \text{ cm}^3 \cdot \text{g}^{-1}$	$V_{\text{mic}}, \text{ cm}^3 \cdot \text{g}^{-1}$	$x, \text{ nm}$	$S_{\text{sp}}, \text{ m}^2 \cdot \text{g}^{-1}$	$S_{\text{mic}}, \text{ m}^2 \cdot \text{g}^{-1}$	$S_{\text{mes}}, \text{ m}^2 \cdot \text{g}^{-1}$
Rapeseed	1,10	0,79	1,15	1933	1498	183
White mustard	0,94	0,81	1,15	1705	1539	77,9

Note:  $V_{\Sigma}$  is the total pore volume,  $\text{cm}^3 \cdot \text{g}^{-1}$ ;  $V_{\text{mic}}$  is the volume of micropores,  $\text{cm}^3 \cdot \text{g}^{-1}$ ;  $x$  is the average pore width, nm;  $S_{\text{sp}}$  is the total specific surface area,  $\text{m}^2 \cdot \text{g}^{-1}$ ;  $S_{\text{mic}}$  is the specific surface area of micropores,  $\text{m}^2 \cdot \text{g}^{-1}$ ;  $S_{\text{mes}}$  is the specific surface area of mesopores,  $\text{m}^2 \cdot \text{g}^{-1}$ .

**Table 6.** Characterization of activated carbon from coffee sludge

Activated carbon/raw material	$\Delta, \text{ g} \cdot \text{dm}^{-3}$	Pore volume, $\text{cm}^3 \cdot \text{g}^{-1}$		$A^c, \%$	Adsorption capacity by iodine, %
		$V_{\text{mic}}$	$V_{\Sigma}$		
UKSH-D coffee sludge	380	0.26	1.10	6.0	82.0
BAU-A (Russian Standard 6217–74) charcoal	230	0.23	1.69	6.0	70.0

Potential applications of AC from coffee husks are detoxification of soils from pesticide residues, detoxification of feed and animal feed, purification of drinking and wastewater, organic liquids and other liquid-phase media.

*3.3.2. Characteristics of AC produced from sunflower seed hulls*

AC properties were determined by the following indicators:

- bulk density: 94.6–116 g·dm<sup>-3</sup>;
- ash content: 25–31.2 wt. %;
- moisture content: 2.3–2.8 wt. %;
- total pore volume: 1.35–2.1 cm<sup>3</sup>·g<sup>-1</sup>;
- volume of micropores: 0.09 cm<sup>3</sup>·g<sup>-1</sup>;
- volume of the sorption space by benzole: 0.44 cm<sup>3</sup>·g<sup>-1</sup>;
- adsorption capacity by iodine: 44–58 %;
- adsorption capacity by MB: 37–87 mg·g<sup>-1</sup>.

The calculation of the porous structure parameters showed the presence of a micropore volume of 0.19 cm<sup>3</sup>·g<sup>-1</sup> and a micropore width of 1.50 nm, the volume of the sorption space was 0.22 cm<sup>3</sup>·g<sup>-1</sup>, and the specific surface was 527 mg·g<sup>-1</sup>.

Thus, in terms of its quality characteristics, the prototype activated carbon is at the level of industrial powder activated carbon of the UAF brand (TC 6-16-2409–80).

Potential areas of application of the new activated carbon are detoxification of soils from pesticide residues, detoxification of feed and animal feed, purification of drinking and wastewater, organic liquids and other liquid-phase media.

*3.3.3. Characteristics of AC produced from sunflower stalks*

AC produced from sunflower stalks has the following parameters:

- bulk density: 130 g·dm<sup>-3</sup>;
- total pore volume: 1.8 cm<sup>3</sup>·g<sup>-1</sup>;
- ash content: 16.9 %;
- capacity by iodine: 41 %.

The resulting ACs had a bulk density of 85 g·dm<sup>-3</sup>, a total pore volume of 3.5 cm<sup>3</sup>·g<sup>-1</sup>, and capacity by iodine of 45.7 %. The total yield of activated carbon from the feedstock was 5.8 %.

Activated carbon from sunflower stalks has low adsorption characteristics, inferior to industrial grades of AC; however, it can be used for soil detoxification and wastewater treatment of agricultural processing enterprises.

*3.3.4. Characteristics of AC produced from rice hulls*

The research results of the RSH activated carbon are shown in Table 7 in comparison with the OU-A activated carbon.

Table 7 shows the quality indicators of the adsorption capacity of AC from rice husks are low, which is explained by a large proportion of ash (72.3 %), which is not an activated component. Nevertheless, such AC can be used to treat wastewater from organic contaminants and oil products, although they are inferior in their adsorption properties to widely used industrial coal grade OU-A (Russian Standard 4453–74).

**Table 7.** Research results of RSH activated carbon and OU-A activated carbon

Indicator title	Values	
	Activated carbon RSH	OU-A
Bulk density, g·dm <sup>-3</sup>	243	–
Mass fraction of water, %	3.76	< 10.0
Mass fraction of ash, %	72.3	< 10.0
Total pore volume, cm <sup>3</sup> ·g <sup>-1</sup>	0.915	0.70–0.80
Adsorption capacity:		
– by iodine, %	8.7	> 80
– by blue ethylene, mg·g <sup>-1</sup>	32	> 225
Content of volatile substances, %	10.4	–
PH of water extract	9.33	–

Note: fraction composition is 0.1–0.7 mm.

### 3.3.5. Characteristics of AC produced on the basis of stone raw materials from nut shells

The properties of activated carbons produced on the basis of stone raw materials from nutshells are shown in Table 8 [36].

Table 8 shows that activated carbons from fruit stones are on the same level with activated carbons based on coconut shell – compacted vegetable raw materials widely used in the world in various adsorption processes.

Activated carbons from walnut shells are also not inferior in their strength and adsorption characteristics to AC from coconut shells. Activated carbons from pine nut shells are inferior (albeit slightly) to AC from coconut shells, but are at the level of AC grade AG-3 (Russian Standard 20464–75) widely used in Russia in various adsorption processes.

Consequently, ACs from domestic compacted vegetable raw materials have a wide range of applications in gas-phase and liquid-phase adsorption processes in industry, ecology, and agro-industrial complexes for soil and feed detoxification.

Summarizing the above, it should be noted that the main goal of this study was to show the possibility of various vegetable wastes as raw materials for the production of activated carbons and their use directly in the practice of crop production for soil detoxification from herbicide residues.

However, the existing serious shortage of domestic ACs makes it necessary to recommend the use of this AC type in other areas of agriculture, industry and ecology [37–40].

In general, ACs from vegetable residues, produced by steam-gas activation, have fairly good

adsorption properties for test substances: iodine 40–45 % and MB 40–80 mg·g<sup>-1</sup>. However, due to the fact that they have low strength and low bulk weight, i.e. belong to the PACs type, they can be used mainly in liquid-phase adsorption processes, such as:

- wastewater treatment from organic pollution, primarily wastewater from agricultural enterprises;
- some cleaning processes of chemical-pharmaceuticals;
- flotation of non-ferrous metal ores.

An important area of environmental protection, where the developed PACs can be used, is purification of gas emissions from waste incineration plants, where a powdered charge is supplied to the gas cleaning system: 90 % CaOH and 10 % PACs. The need for such AC is estimated at 4 thousand tons per year.

ACs produced from straw by chemical activation (in this study – based on white mustard and rapeseed straw) are of particular interest. Due to the peculiarities of this technology, the ash content in such AC is reduced to 2.3–3.8 %, and the specific surface area reaches 1700–1900 m<sup>2</sup>·g<sup>-1</sup> (moreover, the important specific surface area of mesopores reaches up to 183 m<sup>2</sup>·g<sup>-1</sup>). These qualities make it possible to use such PACs in any liquid-phase adsorption technologies (purification of medicines, food products (starch, molasses, and food acids), purification of drinking water by carbonization, and many others. It is important to mention the possibility of effective use of such PACs in the production of supercapacitors, without which it is impossible to operate the cars of the future – electric vehicles.

**Table 8.** Characteristics of activated carbons from nut shell and fruit seeds

Stone (fruit tree shell – raw material)	$\Delta$ , g·dm <sup>-3</sup>	Indicators of AC					
		Pore volume, cm <sup>3</sup> ·g <sup>-1</sup>		$R$ , %	Adsorption capacity by		$A^c$ , %
		$V_{mic}$	$V_{\Sigma}$		iodine, %	MB, mg·g <sup>-1</sup>	
Apricot	395	0.515	0.89	94.0	110.5	290	2–4
Peach	394	0.500	0.90	93.6	110.0	290	1–2
Walnut	400	0.395	0.98	90.0	98.5	261	2–4
Pine nut	165	0.35	1.81	87.0	78.0	134	3.6
Coconut (GCN830, Norit, The Netherlands)	415	0.427	0.96	92.5	99.9	275	1–2

Note:  $R$  – abrasion resistance, %.

The production of activated carbons by chemical activation has (as always) the finishing operations of washing first with hydrochloric acid and then with water to a neutral pH. This is what makes it possible to achieve a significant development of micropores and, of course, and low ash content.

Activated carbons from compacted vegetable raw materials (nut shells and fruit seeds) have high strength characteristics (90–94 % according to Russian Standard 16188–70), which are not inferior to the best world analogues, and, therefore, have a wide range of applications as in any liquid-phase (see above) and gas adsorption technologies (solvent recovery, gas sanitation, gas protection equipment, oil and gas processing, catalyst carriers, and many others).

Consequently, these types of vegetable wastes make it possible to obtain a wide range of AC for solving sorption problems in industry and environmental safety, and will also allow solving many issues of import substitution in the field of activated carbons and sorption materials based on them.

### 3.4. The application of ACs from vegetable waste of the agro-industrial complex for soil detoxification

It was logical to determine the effectiveness of ACs produced directly during soil detoxification from the residues of the applied herbicides. The experiments were carried out in the laboratory at the All-Russian Research Institute of Phytopathology of the Russian Academy of Sciences (Golitsino, Moscow region). To sow the sunflower test culture, we used pots with a capacity of 600 g of soil, which was contaminated with Singer herbicide at a dose corresponding to 5 g per ha, and a dose of AC was introduced at the rate of 100 g per 1 ha.

The test plants were grown under controlled conditions of the artificial climate laboratory (LIC) in Fetch chambers (Germany): air humidity in the chamber was 70%, daylight hours were 16 hours, nights were 8 hours, illumination during the day was 20 thousand lux, air temperature during the day was 25 °C and at night 16 °C. Soil moisture was maintained at the level of 60 % of the field moisture capacity by daily watering by weight of each vegetative vessel with demineralized tap water. After 30 days, the above-ground mass of test plants was cut and weighed. The level of antidote efficacy of experimental samples of activated carbons was

judged by the decrease in the mass of aboveground organs of test plants in comparison with the control variant without herbicide and activated carbon [37].

The calculation was carried out according to the formula

$$B = 100 - (A/K \cdot 100), \%$$

where  $B$  is the decrease in the mass of test plants relative to the control;  $A$  is the mass of test plants in the experimental variant;  $K$  is the mass of test plants in the control variant.

The data produced are shown in Table 9.

Table 9 shows that the suppression of growth in relation to pure control on herbicide-contaminated soils (using the example of Singer) with the use of AC from wheat and oat straw is only 4.9 %, while in the globally recognized activated soil-grade activated carbon of the Grosafe brand it reaches 12.2 %. This suggests that the effectiveness of AC produced from straw is 2.5 times higher than the imported preparation used for soil detoxification.

In another series of experiments, we evaluated the efficiency of detoxification of soils contaminated with the Singer SP herbicide on the most promising AC from vegetable waste. The results of experiments carried out according to the above methodology are shown in Table 10.

The results presented in Table 10 show that the introduction of selected types of AC into the soil almost doubles the preservation of the aboveground mass of the plant, which, if this technology is applied, will adequately increase the yield of cultivated crops.

**Table 9.** Effect of activated carbons on the phytotoxicity of metsulfuron-methyl (Singer, SP) on the example of sunflower plants

Option	Average weight, g	% of control
Singer, SP	1.1	73.2
Singer, SP + AU from oat straw	3.9	4.9
Singer, SP + AU from wheat straw	3.9	4.9
Singer, SP + AU from rapeseed straw	3.2	21.9
Singer, SP + Grosafe	3.6	12.2
Control (with out herbicides)	4.1	–

**Table 10.** Effect of activated carbons on the phytotoxicity of metsulfuron-methyl (Singer SP) on the example of a rapeseed plant

No	Option	Singer SP, g·ha <sup>-1</sup>	Above ground weight of test plants, g					average	Decrease in above ground weight of test plants, % of control
			by repetition						
			1	2	3	4	5		
1	VACs from sunflower husks, 100 kg·ha <sup>-1</sup>		3.8	3.6	3.9	3.6	3.7	3.7	33.9
2	VACs from rapeseed straw, 100 kg ha <sup>-1</sup>		4.5	4.1	4.5	4.9	4.0	4.4	21.4
3	VACs from coconut shell, 100 kg·ha <sup>-1</sup>	0.5	4.1	3.8	4.7	3.4	4.0	4.0	28.6
4	VACs from apricot stones, 100 kg·ha <sup>-1</sup>		4.4	4.2	3.8	4.4	4.0	4.2	25.0
K <sub>3</sub>	Control (Singer)		1.4	1.5	2.2	1.9	1.4	1.7	69.6
K	Control (soddy-podzolic soils)	–	5.8	5.5	5.4	5.8	5.4	5.6	0

#### 4. Conclusions

The studies showed that vegetable wastes make it possible to obtain a wide range of activated carbons of powder and granular types. Activated carbons from the straw of various agricultural crops: grain, oilseed, mustard, food, fodder, and technical crops have adsorption properties that allow them to be used directly in places of formation for detoxification of agricultural soils from herbicide residues, which makes it possible to increase the yield of agricultural crops cultivated in 20–80 % crop rotation.

PACs produced from agricultural straw by chemical activation deserve special attention, where it is possible to achieve the development of a micropore volume of 0.79–0.81 cm<sup>3</sup>·g<sup>-1</sup> and a total specific surface area of up to 1933 m<sup>2</sup>·g<sup>-1</sup>, which ensures their adsorption activity for iodine of 150–240 % and MB 350–500 mg·g<sup>-1</sup>, which is significantly higher than that of domestic and imported analogues.

Activated carbons from compacted vegetable raw materials (walnut and pine nuts shells and apricot and peach fruit stones) have high strength properties of 90–94 % according to Russian Standard 16188–70 and fairly high adsorption characteristics: for iodine – 98–110 % and for MB – 260–290 mg·g<sup>-1</sup>, which makes it possible to use them in any adsorption processes, including increased intensity (boiling and moving layers).

For the first time, ACs were produced and studied from a wide range of vegetable raw materials used in the food industry: coffee bean husks and sludge, sunflower seed husks and stalks, rice husks.

Based on the fact that these types of renewable vegetable wastes amount to hundreds of millions of tons in Russia as a whole, it can be predicted that their involvement in the production of activated carbons required in our country in the amount of 70 thousand tons per year will fully supply the market with them and solve the issue of import substitution.

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#### 6. Conflict of interests

The authors declare no conflict of interest.

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