Using wastes of buckwheat processing as sorption materials for the removal of pollutants from aqueous media: a review

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Abstract. Based on data from various literature sources, information is given on the chemical composition of straw and husk of buckwheat grains. The information on buckwheat processing waste as a source for obtaining various valuable components is provided. Literature data on the use of ground buckwheat straw as a sorption material for the extraction of heavy metal ions and oil products from natural and sewage are presented. The possibility of increasing sorption characteristics by buckwheat straw treatment with various chemical reagents is shown. Determined that the husk of buckwheat grains contains cellulose and lignin most of all. Information is given on the use of the fruit shells of buckwheat grains as sorption materials for the removal of metal ions, oil and oil products from aqueous media. It was shown that to increase the sorption characteristics of these pollutants is possible by treating the sorption material with acidic chemical reagents and high-frequency low-pressure plasma. The parameters of acid treatment or plasma exposure are determined at which the highest adsorption indicators.

Native straw and buckwheat husk are effective sorption materials for the removal of heavy metal ions and oil from aqueous media. It is possible to increase the sorption characteristics according to pollutants by a chemical or physical and chemical modification.

1. Introduction

In the world community, a new innovative direction in the field of environmental protection is currently developing rapidly such as the use of agricultural waste as reagents for the removal of pollutants from natural and sewage waters [1–10].

Now in the world, crops are grown most of all. Accordingly, when they are processed, a huge amount of various wastes such as straw, flour, flooring, grain husks, etc is formed. The latter are used in various industries as sorption materials for the extraction of harmful substances from aqueous media. Thus, previously published reviews showed that waste from the processing of wheat [11], barley [12] and oats [13] are effective sorption materials for the removal of metal ions, dyes, pesticides, oil and products of its processing from aqueous media.

The aim of this work is to analyze and summarize the literature on the use of waste from the processing of buckwheat as sorption materials for the removal of pollutants from aqueous media.
2. Materials and methods
The microstructure of the surface of the buckwheat husk was studied using a high-resolution scanning electron microscope TESCAN MIRA 3 LMU. To study the specific surface area and porosity of the buckwheat husk at the micro level, we used the method of gas adsorption using the SoftSorbi – II ver.1.0 device.

3. Results and discussion

3.1. Basic information about buckwheat
Buckwheat (*Latin Fagopyrum*) is a genus of herbaceous plants of the Buckwheat family (*Polygonaceae*) (Figure 1, a). It is annual or perennial herbaceous plant, 10 – 100 cm high. Stems are bare, upright, branched, and evenly leafy. The leaves are alternate, arrow-triangular or arrow-ovate, 2.5–9 cm long, the tip is elongated. The flowers are bisexual, dimorphic, heterostyle. The perianth is five-parted, yellowish-green, white or light pink, 1.3–4 mm long. Fruits are trihedral, dull nuts, 4.5 –6.5 mm long. In practical terms, the most important plant of the genus is seed buckwheat (*Fagopyrum esculentum*), a bread and honey plant [14].

![Figure 1. Buckwheat a), buckwheat straw b) and c) buckwheat husk.](image)

It has been found that seed buckwheat contains a large amount of nutrients – 63 % carbohydrates, 11.7 % protein, 2.4 % fat, 9.9 % fiber, 11 % water and 2 % minerals [15]. Buckwheat contains a lot of iron, as well as calcium, potassium, phosphorus, iodine, zinc, fluorine, molybdenum, cobalt, as well as vitamins B1, B2, B9 (folic acid), PP, E. The flowering aerial part of buckwheat contains rutin, phagopyrine, pyrocatechol, gallic, chlorogenic and caffeic acids. Seeds have starch, protein, sugar, fatty oil, organic acids (maleic, menolenic, oxalic, apple and lemon), riboflavin, thiamine, phosphorus, iron. By the content of lysine and methionine, buckwheat protein is superior to all cereals; it has high digestibility – up to 78 % [16].

3.2. Sorption properties of buckwheat components

3.2.1. Straw

The most massive waste from the processing of buckwheat is straw (Figure 1, b). The composition of buckwheat straw is shown in Table 1 [17].

| Indicators                   | Value | Indicators       | Value |
|-----------------------------|-------|------------------|-------|
| Feed units                  | 0.34  | Calcium, g       | 9.3   |
| Dry matter, g               | 906   | Potassium, g     | 12.73 |
| Crude protein, g            | 45    | Phosphorus, g    | 1.3   |
| Digestible protein (cattle), g | 20.7 | Magnesium, g     | 1.91  |
| Lysine, g                   | 1.22  | Sodium, g        | 1.55  |
| Methionine + cystine, g     | 1.35  | Iron, mg         | 199.98|
The presence of proteins in buckwheat straw can contribute to the adsorption of heavy metal ions (HMI) (Table 1). However, contrary to what was expected, information on the adsorption of HMI by buckwheat straw was very limited. In particular, the removal of Cu$^{2+}$ and Cr$^{6+}$ ions by a sorption material from a mixture of buckwheat straw and coal from iron agraterata (Agerátina adenóphora) was studied [18 – 20]. The influence of adsorption parameters, such as pH, dosage of SM, adsorption time and initial concentration of HMI, was determined. It was found that the maximum sorption capacity of the composite material on Cr$^{6+}$ ions is from 46.2 to 55.2 mg / g in the temperature range 298 – 308 K under the following conditions: pH = 1, t = 4 h, 120 rpm of the mixer, – 45 mg / dm$^3$, dosage of sorption material – 0.4 g / dm$^3$. It was found that the adsorption isotherm of Cr$^{6+}$ ions is most adequately described by the Langmuir model. Certain thermodynamic parameters $\Delta G = -35.99$ kJ / mol, $\Delta H = 143.7$ kJ / mol and $\Delta S = 760.45$ J / mol · K indicate that the process is spontaneous and exothermic [21]. At the same time, it was determined that the adsorption isotherm on Cu$^{2+}$ ions is more accurately described by the Freundlich model, and the kinetics of the process obeys the pseudo-second order model. It was determined that the highest sorption characteristics of the studied SM on Cu$^{2+}$ ions are achieved at pH = 3 [22].

The high fiber content (~ 350 g / kg dry weight) in buckwheat straw (Table 1) can contribute to the adsorption of oil and its processed products [23]. Initially, certain physicochemical parameters of crushed straw were determined: buoyancy – 77.2 %, bulk density – 0.111 g / cm$^3$, total pore volume on water – 2.95 cm$^3$ / g. It was determined that the maximum sorption capacity of buckwheat straw in relation to Devonian oil under static conditions is 6.26 g / g, and the maximum water absorption is 8.45 g / g. It is indicated that treatment of buckwheat straw with a weakly concentrated H$_2$SO$_4$ solution (0.5 %) contributes to a certain increase in oil intensity and a decrease in water absorption when removing the oil film from the water surface.

### 3.2.2. Husk

Another large-tonnage waste from the production of buckwheat is fruit shells (husk, shuck) (Figure 1, с). In the industrial buckwheat processing, the husk accounts for 14 to 30 % of the total grain weight [24].

Figure 2 shows a highly developed microrelief of the surface of the buckwheat husk; micropores up to 0.1 μm in size are noted.

![Figure 2. The microstructure of the surface of the buckwheat husk.](image-url)
To study porosity at the microlevel and specific surface, we used the gas adsorption method. According to the results, it was found that the specific surface area of the husk by the BET method (SBET) was $21.5 \text{ m}^2/\text{g}$ (Table 2). The number of pores in the nanometric range from 1.7 to 300 nm is $0.63 \times 10^{-3} \text{ cm}^3/\text{g}$.

| Physical properties of buckwheat husk |
|---------------------------------------|
| $S_{\text{BET}}$ (m$^2$/g) | Total surface area of pores, $S_p$ (m$^2$/g) | The total volume of pores with a diameter of 1.7 up to 300 nm, $V_p \times 10^{-3}$ (cm$^3$/g) |
| 21.5 | 0.32 | 0.63 |

Buckwheat husk contains fiber – up to 50 %, lipids – 4–5 %, polysaccharides – 70 %, crude protein – 34 %, 0.2–0.3 % sugars, 9–10 % ash [24]. They contain such elements as potassium (3.1 g/kg), sodium (0.2 g/kg), copper (1.9 mg/kg), calcium (4.8 g/kg), magnesium (2.2 g/kg), zinc (5.4 mg/kg), iron (80.3 mg/kg), sulfur (1.7 g/kg), manganese (21.8 mg/kg) and others in their composition [25]. The amino acid content in the composition of the shells of buckwheat grains is determined and shown in table 3.

| Amino acid | Content (g/g) | Amino acid | Content (g/g) |
|------------|---------------|------------|---------------|
| Threonine  | 0.001         | Glutamine  | 0.0002        |
| Valine     | 0.002         | Glutamic acid | 0.003       |
| Isoleucine | 0.0009        | Alanine    | 0.003         |
| Leucine    | 0.008         | β-alanine  | 0.0005        |
| Lysine     | 0.0003        | tyrosine   | 0.0008        |
| Phenylalanine | 0.0008       | phosphoserine | 0.01        |
| Aspartic acid | 0.005        | Serine     | 0.005         |
| Asparagin  | 0.0004        | Taurine    | 0.0004        |

According to [28], the chemical composition of the shells of buckwheat grains in % of dry raw materials has alcohol-soluble substances – 4.9; total nitrogen – 0.63; ash – 1.9; easily hydrolyzable polysaccharides – 24.5; hardly hydrolyzable polysaccharides – 27.8. The remainder of the hardly hydrolyzable polysaccharides is mainly lignin – 30.7.

Considering the shuck composition of buckwheat grains, the latter is a promising raw material for the isolation of polysaccharides [29] and flavonoids [30, 31] from it. The fruit shells of buckwheat grains are more widely used in various sectors of the economy compared to buckwheat straw. In particular, it indicates the use of shuck of buckwheat grains as filler for pillows [32], concrete [33], composite materials [34, 35], as a feed additive [36], extracts as a dye [37, 38], etc.

The traditional scheme of the adsorption mechanism is to increase the concentration of substances on the phase interface (Figure 4).

![Figure 3. Scheme of the adsorption mechanism on a highly developed surface.](image)

However, as it was indicated earlier (Table 3), the presence of proteins and amino acids in the composition of buckwheat shuck can contribute to the adsorption of HMI from aqueous media.

The adsorption of Au (III) ions from gold-containing sewage was studied [39]. It was found that the largest sorption capacity (1 mmol/g) is observed at pH = 2.5. With increasing temperature from 15
°C to 35 °C and contact time, the sorption capacity increases. It is also possible to increase the maximum sorption capacity to 2.84 mmol/g by modifying buckwheat shuck with an orthophosphoric acid solution [40]. The possibility of regeneration of the sorption material using 0.05% eluent solutions of thiourea in 0.1 mmol/dm³ HCl solution is shown.

Buckwheat shuck was studied as a sorption material for the extraction of Cr⁶⁺ ions from model solutions with an initial concentration of 100 mg/dm³. It was determined that the sorption capacity increased with decreasing pH and amounted to 99.87% at pH = 2 and a dosage of sorption material of 5.0 g/dm³ [41]. It was determined that the Langmuir model describes the adsorption isotherm of Cr (VI) ions on buckwheat husk best of all, and the maximum adsorption capacity at 45 °C, determined from the Langmuir equation, is 63.61 mg/g. An analysis of the thermodynamic parameters (∆G°, ∆H°, and E) showed that the adsorption process was a spontaneous, endothermic, and chemisorption process. Besides, analysis of x-ray photoelectron spectroscopy showed that the adsorbed Cr (VI) ions are partially reduced to Cr (III) ions. IR Fourier spectra showed that carboxyl and amino groups are involved in the chemisorption process. It has been established that the most probable mechanisms of Cr (VI) removal using buckwheat husks are electrostatic attraction and chemical reduction [42].

Besides, buckwheat shuck was used to extract Hg²⁺ ions from aqueous solutions with an initial concentration of the last 802.36 mg/dm³. It was found that at pH = 4 and a buckwheat husk dosage of 2 g/dm³, the maximum sorption capacity was 110 mg/g [43]. The results showed that the kinetics of adsorption of buckwheat shuck by Hg (II) ions is well described by the pseudo-second order reaction model, and the calculated thermodynamic adsorption parameters ∆G, ∆H and ∆S were −5.83 kJ/mol at 35 °C, 73.1 kJ/mol and 256 J/mol·K, respectively. It has been determined that the adsorption isotherm is described by the Langmuir equation best of all, and the maximum adsorption capacity on Hg (II) ions is 243.90 mg/g at 35 °C. In addition, a study of adsorption selectivity showed that buckwheat husk exhibits a strong affinity for mercury ions in aqueous solutions and exhibits 100% selectivity on Hg (II) ions in the presence of Zn (II) and Cd (II) ions [44].

The kinetics of adsorption of Zn (II), Cd (II), Co (II), Cu (II), Ni (II) ions from model solutions with an initial concentration of 10–50 mg/dm³ of buckwheat shuck at a temperature of 25 °C in the pH range = 5 – 6 has been studied. Experiments determined that the lowest final concentrations are achieved with Cu²⁺ ions, and the highest ones with Co²⁺ ions (Figure 5) [45].

![Figure 4. Diagram dependences of the adsorption kinetics of Zn (II), Cd (II), Co (II), Cu (II), Ni (II) ions by buckwheat shuck at an initial concentration of metal ions of 20 mg/dm³ [45].](image)

Figure 4. Diagram dependences of the adsorption kinetics of Zn (II), Cd (II), Co (II), Cu (II), Ni (II) ions by buckwheat shuck at an initial concentration of metal ions of 20 mg/dm³ [45].

It has been determined that the adsorption kinetics of these metal ions is described by the equation with fractional derivatives, and adsorption is a physical process.

Sorption material was obtained by lignification of buckwheat shuck by successive hydrolysis of the latter with acids with a concentration of 0.5–10% at a temperature of 80–200 °C and a water module of 1:10 for 20 – 180 minutes. Then, the obtained lignin was activated with an aqueous solution of an alkali metal carbonate with a concentration of 0.5–1.5% at a temperature of 70–110 °C with a water module of 1:10 for 20–180 minutes. Then, the sorption material was neutralized [46]. The latter is an odorless dark brown powder, practically insoluble in water. The lignin content in the reagent was 79–88%. The obtained sorption material was studied to extract metal ions, dyes, and some other pollutants from model solutions with an initial concentration of 5 or 50 mg/dm³ (Table 4) [47].
Native buckwheat shuck and processed during the extraction of polysaccharides of water, oxalate and alkaline extraction were studied to extract diesel fuel and toluene from aqueous media [16, 48]. It is indicated that the maximum degree of purification of dissolved and emulsified NPs with buckwheat shuck, in particular, is small and does not exceed 70% [49]. It is proposed to use sorbent obtained on the basis of buckwheat husks and polyurethane foam [50–52] for the purification of water from oil and oil products. The advantages of the latter include hydrophobicity and oleophilicity, versatility, a high percentage of profitability, ultra-fast adsorption, low density, non-toxicity, the ability to squeeze out absorbed oil up to 10 times. It is indicated that the oil capacity increases with increasing content of buckwheat husk in the composite material, reaching 16 g / g. It is shown that the oil capacity of the sorption material, called "PPU-45 SHG", depends on the volume of crumb grain and the type of oil. With a decrease in grain size and an increase in oil viscosity, the absorption capacity of the reagent increases. Among the advantages, it should also be noted the possibility of releasing the reagent in any geometric form, unlimited shelf life and speed of production (2 – 10 minutes). The latter circumstance allows the creation of mobile mini-plants for the production of adsorbent directly at the place of oil spill [51].

### 3.2.3. Composites

A composition consisting of polyurethane foam and buckwheat shuck called “PUF-0ZG” was studied as a carrier for immobilizing oil-destroying microorganisms on its surface, which were used as a strain of *Rhodococcus erythropolis* O210, association V, consisting of a community of microorganisms (*Pseudomonas pickettii*, *Arthrobacter sp.*, *Aeromonas salmonicida*, *Vibrio sp.*), as well as activated silt, over silt fluid and the bacterial drug Destroyl, which is a powder consisting of microorganism cells isolated from a natural microbial culture [53]. It is indicated that it is possible to regenerate the absorbed oil products from saturated sorption material by squeezing or centrifugation [54]. The amount of regenerated oil is ~ 80–85 %.

### 3.2.4. Ash

In addition to native and modified buckwheat shuck samples used as sorption materials of heavy metal ions, dyes, and petroleum products, ash obtained by burning buckwheat husks also has sorption properties. The ash content in the buckwheat shuck varies from 1.5–2.3 %, in the straw – 2.8–11.1 % [55]. Nevertheless, multi-ton dumps of waste from buckwheat processing make it promising to use the latter as promising sorption materials after heat treatment of raw materials.

A thermal installation has been developed and is being used for burning buckwheat shuck in order to obtain a sorbent [54] at temperatures up to 1000 °C with a productivity of 5 t/g. It was shown that the pyrolysis temperature of raw materials affects the yield of the target product and adsorption

| Pollutant       | Maximum sorption capacity (mg / g) | Efficiency extraction (%) |
|-----------------|------------------------------------|---------------------------|
| Ni (II) ions    | 2.3 / 6.7**                       | 76.9 / 83.2**             |
| Cu (II) ions    | 1.3 / 6.9                         | 85.5 / 51.8               |
| Sr (II) ions    | 4.4 / 7.8                         | 88.1 / 53.8               |
| Cd (II) ions    | 5.9 / 7.4                         | 91.9 / 56.5               |
| Pb (II) ions    | 36.4 / 42.5                       | 90.2 / 76.4               |
| Methylene blue  | 6.7 / 78.6                        | 90.5 / 75.2               |
| Congo Red       | 4.3 / 10.5                        | 42.2 / 29.0               |
| Uric acid       | 3.7 / 2.75                        | 37.8 / 20.3               |
| Ammonia nitrogen| 1.5 / 6.9                         | 81.6 / 18.1               |

* initial pollutant concentration of 5 mg / dm³; ** initial concentration of pollutant 50 mg / dm³
activity. The product of buckwheat husk pyrolysis (GS sorbent) is a black flake of size 1 – 5 mm, insoluble in water, not combustible and explosive \[54\], with a density of 0.08–0.12 g/cm\(^3\) \[55\]. It is also possible to obtain sorbents based on buckwheat shuck by processing raw materials using a plasma arc setup \[56\] or low-pressure plasma \[57\].

It is proposed to produce sorption materials in the form of ash from buckwheat shuck for the extraction of heavy and rare - earth elements from sewage \[58\].

The GS sorbent also showed a high adsorption capacity with respect to oil and oil products (5 –10 g/g) \[59\]. It was found that the degree of removal of diesel fuel from the water surface by the sorbent GS exceeds 90 %, oil - more than 99 %. To increase the quality indicators of sorbents obtained as a result of heat treatment of buckwheat shuck, it was proposed to process them in the presence of a number of substances, such as sulfur, halides in an oxygen-free medium \[60\].

It is possible to isolate up to 44 % of sorbed oil \[61\] in the case of a saturated reagent squeezing, but at the same time, its structure is destroyed \[62\]. The possibility of using the used reagent in the production of asphalt, fuel briquettes is shown. The research results showed that the obtained samples of fuel briquettes have strength of 15–20 MPa and relatively low ash content (10–12 %). It was found that the specific heat of combustion of the fuel is 5400–5800 kcal/kg \[67\].

3.2.5. Extracts

As it was mentioned earlier, buckwheat shuck include compounds of the flavonoid group (including rutin and quercetin), lipids, polysaccharides, coumarins, amino acids, tannins, and other substances of organic origin. This circumstance suggests the use, in particular, of an alkaline extract from buckwheat shuck (ELG), which is used to precipitate ITM from sewage along with or instead of conventional methods for the extraction of metal ions \[63\]. Concentrated ELG represents a viscous product of dark brown color with a density of 1230 kg/m\(^3\), pH = 11.5–12.3. The content of mineral and organic substances in the ELG is 137.84 g/dm\(^3\) and 123.4 g/dm\(^3\), respectively. The molar mass of organic substances contained in aqueous ELG is 300–600 g/mol.

The results showed that the sorption capacity of ELG has values, mEq / g: for Cu\(^{2+}\) ions – 7.56, Ni\(^{2+}\) – 10.05, Cd\(^{2+}\) – 5.25, Cr\(^{6+}\) – 6.53 and is at a level characteristic of cation exchange KS-1 or KB-4 resins.

A comparison of the results of experiments on the treatment of sewage from heavy metal ions using lime milk and ELG showed the expediency of using the latter due to the higher degree of purification. In addition, ELG, which is a solution, unlike lime milk, is not deposited on the walls of pipelines and does not clog shut-off valves.

4. Summary

Thus, the above data summarizes the literature on the use of waste from the processing of buckwheat as sorption materials for the extraction of pollutants from aqueous media. The chemical compounds present in the composition of straw and husks of buckwheat contribute to high chemical adsorption indicators on metal ions, oil and the products of the processing of the latter. It has been revealed that treatment of buckwheat shuck with chemical reagents and arc plasma, as well as with low-pressure plasma, contributes to an increase in the adsorption characteristics on the above pollutants.

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