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POTENTIAL FOR SORPTION-BASED EXTRACTION OF HEAVY METAL IONS FROM WATERS CONTAMINATED AS A RESULT OF MILITARY ACTIONS USING CARBONACEOUS MATERIALS OBTAINED FROM SECONDARY WASTE

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Abstract: The aim of the research was to study the adsorption behavior of heavy metal ions (Pb^{2+}, Cd^{2+}, Co^{2+}, Cu^{2+}, Fe^{2+}) in multicomponent aqueous solutions using natural carbonaceous materials. Hazelnut and walnut shell carbonaceous materials and activated carbon were chosen as adsorbents. The study was conducted in both single-component and multicomponent systems, under standard conditions (m_s = 1 g, V_x = 100 ml, t = 25 °C, T = 30 min).

The results showed that the adsorption capacity for lead ions was the highest (83.5–94 mg/g), followed by cadmium (22.3–22.8 mg/g) and copper ions (15.1–15.8 mg/g). The order of adsorption of metals corresponds to the decreasing ionic radius: $Pb^{2+} > Cd^{2+} > Cu^{2+} > Fe^{2+} > Co^{2+}$.

In multicomponent systems, both synergistic and antagonistic interactions between ions were observed. For cadmium and cobalt ions, an ionic interaction coefficient in a sorption system (Rqe) ≥ 1 indicates synergism or the absence of interaction, while for iron and copper, Rqe < 1 indicates antagonism.

An increase in pH during the adsorption process demonstrated the buffering effect of carbon materials. Analysis of Langmuir and Freundlich isotherms confirmed the physical adsorption mechanism (R^2 = 0.94–0.99) and the heterogeneous nature of the surface.

The study confirms that natural carbon materials can be used as effective, environmentally friendly, and low-cost adsorbents for the removal of heavy metals from water containing many ions.

Keywords: military actions, waters, heavy metals, adsorption, carbon materials, isotherm.

1. Introduction

Military conflicts are an important factor in the social, political, and environmental transformations of humanity, which have a serious and long-term impact on the environment and ecosystems. Modern wars have significantly changed the nature and scale of environmental pollution, which is especially noticeable in terms of the dissemination of heavy metals. As a result of the use and destruction of military equipment, ammunition, explosives, infrastructure, and toxic metals such as lead, cadmium, etc., enter the environment. These metals accumulate in soil, water, and air, creating a long-term environmental threat and affecting human health [1, 2].

Purification of water contaminated with heavy metals is a significant ecological and technological task, which is associated with large economic costs, so the creation of inexpensive, environmentally friendly, and effective adsorbents based on waste is relevant [3, 4, 5, 6].

R. Agladze Institute of Inorganic Chemistry and Electrochemistry of Tbilisi State University have developed a technology that allows obtaining adsorbents from secondary agricultural waste, namely hazelnut and walnut shells, nectarine kernels, sawdust, and others [4, 9]. This technology is applicable to many types of raw materials; the process is single-stage and does not require preliminary processing of raw materials. Carbonaceous materials are obtained from secondary organic waste (in particular, cellulose-containing waste), which is characterized by a high specific surface and porous structure, which determines its good adsorption capacity. In addition, they are notable for their low cost.

It has been established that cellulose-based carbonaceous materials exhibit different adsorption capacity depending on the type of metal ion [7, 8, 9], which indicates their effectiveness in systems with complex polymetallic contaminants. This technology is a sustainable and promising approach to environmental protection, and the materials used are characterized by high adsorption activity with respect to heavy metal ions.

2. Methodology

Using the aforementioned original thermochemical method, finely dispersed carbonaceous powders with the required characteristics were obtained from hazelnut and walnut shells as experimental materials to test their sorption potential for heavy metals to neutralize them from aquatic ecosystems. A commercially available product, brand BAU A (GOST 6217-52) activated carbon, was used as a comparison material.

Table 1

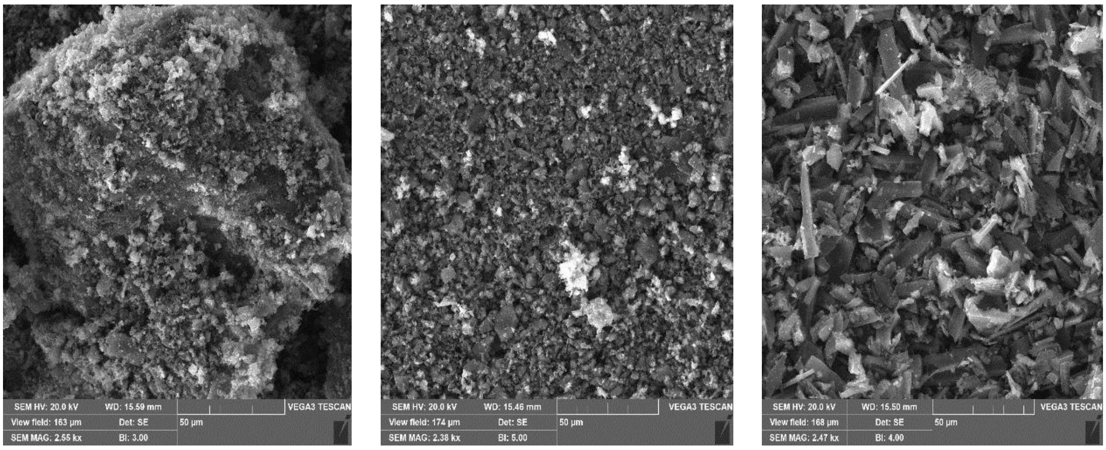
Characteristics of activated carbon and carbonaceous materials obtained from hazelnut and walnut

Sample of Carbonaceous Material	S BET m^2/g	S micro phores, m^2/g	V micro phores, sm^3/g	Ash, %	Moistness %
Hazelnut shell	637.3	427.65	0.20	2.9	1.4
Walnut shell	499.0	380.26	0.15	3.5	4.3
Activated carbon	708.70	473.78	0.21	4.2	1.3

Table 2

Elemental composition of samples of activated carbon and carbonaceous materials obtained from hazelnut and walnut

Sample	Elemental composition of samples % (average)												
	C	O	Ca	K	Si	S	Fe	Ni	Cu	Zn	Al	F	Cr
Hazelnut shell	85	11	2	0.5	0.1	0.1	0.4	0.3	0.2	0.2	0.0	0.0	0.0
Walnut shell	89.5	7.4	0.8	1.4	0.0	0.4	0.15	0.04	0.2	0.1	0.0	0.0	0.0
Activated carbon	89.5	8.6	0.7	0.4	0.01	-	0.09		0.4	0.01	0.0	0.2	-



(a) **(b)** **(c)**

Fig. 1. Morphology of carbonaceous sorbents (SEM): a) Hazelnut, b) Walnut, c) Industrial carbon.

Experiments were conducted on combinations of various metal ions:

1. Pb^{2+}, Cd^{2+}, Co^{2+};
2. Fe^{2+}, Cu^{2+}, Cd^{2+};
3. Fe^{2+}, Cd^{2+};
4. Pb^{2+}, Cd^{2+}, Co^{2+}, Cu^{2+}

Carbonaceous materials with a particle size fraction of 40–20 μm derived from hazelnut and walnut shells by the above-mentioned thermochemical method were used for the experiments. The mass of the sorbent was 1 gram **(**m_sorb = 1 g), the volume of the solution was 100 mL **(**V_sol = 100 mL**),** and the experiments were conducted at 25 °C (t = 25 °C). As a reference sample, commercial activated carbon BAU A (GOST 6217-52) was used.

The studies were carried out using model solutions of various metals at a concentration of 0.01 M, specifically $CoCl_2$ (for Co^{2+} ions), $CdCl_2$ (for Cd^{2+} ions), $Pb(NO_3)_2$ (for Pb^{2+} ions), $CuSO_4 \cdot 5H_2O$ (for Cu^{2+} ions), and $FeSO_4 \cdot 7H_2O$ (for Fe^{2+} ions).

These tests were aimed at evaluating the adsorption capacities of the carbonaceous materials toward each of these metal ions.

3. Results and discussion

3.1. Adsorption capacity

As can be seen from the results below (Table 3 and Table 4), in different systems the effect of ionic interaction of cadmium on walnut shell carbonaceous material is equal to or greater than 1, which indicates synergism or lack of interaction; the cadmium ion is not an inhibitor in the adsorption of other cations. The cobalt ion is also not an inhibitor for the considered multi-cationic systems. In the case of iron, copper, and lead, the coexistence of the other ions significantly hindered the adsorptive removal of individual ions from different multi-cationic systems.

Table 3

Adsorption of heavy metals for single- and multi-component systems and the effect of ionic interaction for walnut shell carbonaceous material

Multicationic system	Adsorption, mg/g in a single-component system on walnut shell carbonaceous material				Adsorption, mg/g in a multicomponent system of walnut shell carbonaceous material				Rqe, The effect of ionic interaction			
Pb,Co,Cd,Cu	**Pb**	**Co**	**Cd**	**Cu**	**Pb**	**Co**	**Cd**	**Cu**	**Pb**	**Co**	**Cd**	**Cu**
	83	6	21	32	35	11.5	21.7	15	0.42	1.9	1	0.47
Pb,Co,Cd	**Pb**	**Co**	**Cd**		**Pb**	**Co**	**Cd**		**Pb**	**Co**	**Cd**	
	83	6	21		88	12.6	22.8		1.06	2.1	1	
Fe,Cu,Co	**Fe**	**Cu**	**Cd**		**Fe**	**Cu**	**Cd**		**Fe**	**Cu**	**Cd**	
	21	32	6		9.8	15.8	21.8		0.46	0.5	3.6	
Fe,Cd	**Fe**	**Cd**			**Fe**	**Cd**			**Fe**	**Cd**		
	21	21			17.5	30			0.83	1.43		

Table 4

Adsorption of heavy metals for single- and multi-component systems and the effect of ionic interaction for hazelnut shell carbonaceous material

Multicationic system	Adsorption, mg/g in a single-component system on huzelnut shell carbonaceous material			Adsorption, mg/g in a multicomponent system of huzelnut shell carbonaceous material			Rqe, The effect of ionic interaction		
Pb,Co,Cd	**Pb**	**Co**	**Cd**	**Pb**	**Co**	**Cd**	**Pb**	**Co**	**Cd**
	73	10	35	83.5	13.2	22.5	1.14	1.32	0.64
Fe,Cu,Co	**Fe**	**Cu**	**Cd**	**Fe**	**Cu**	**Cd**	**Fe**	**Cu**	**Cd**
	24	20	35	10	15.8	22.5	0.42	0.79	0.64
Fe,Cd	**Fe**	**Cd**		**Fe**	**Cd**		**Fe**	**Cd**	
	24	35		18	30		0.75	0.86	

For the hazelnut shell carbonaceous material, the effect of ionic interaction for iron, copper, and cadmium ions in the considered multicationic systems is less than 1, which means that antagonism occurs, while in the case of lead and cobalt ions, their values are greater than 1, which indicates that their adsorption in this multicationic system is not affected by the coexistence of other ions.

Table 5

Adsorption of heavy metals for single- and multi-component systems and the effect of ionic interactions on activated carbon

Multicationic system	Adsorption, mg/g in a single-component system on activated carbon			Adsorption, mg/g in a multi-component system on activated carbon			Rqe, The effect of ionic interaction		
Pb,Co,Cd	**Pb**	**Co**	**Cd**	**Pb**	**Co**	**Cd**	**Pb**	**Co**	**Cd**
	84	10	26	94	12.25	22.5	1.11	1.22	0.86
Fe,Cu,Co	**Fe**	**Cu**	**Cd**	**Fe**	**Cu**	**Cd**	**Fe**	**Cu**	**Cd**
	26	26	26	9.25	15.5	31.25	0.35	0.6	1.2
Fe,Cd	**Fe**	**Cd**		**Fe**	**Cd**		**Fe**	**Cd**	
	26	26		18.5	31.3		0.71	1.2	

For activated carbon, in the considered multicationic systems, for the ions of iron and copper, we observe the similar practice as for the carbonaceous material from hazelnut and walnut shells. In the case of lead and cobalt ions, their values are greater than 1, which indicates that in this multicationic system their adsorption is not affected by the coexistence of other ions. In the multicationic system of Pb, Co, and Cd, the adsorption of cadmium ions was significantly hindered by the coexistence of other metal ions, while in the other considered systems, the adsorption of its ions is not slowed down.

During the adsorption process, the removal of one metal ion from a solution in a multi-component system can be affected by the coexistence of other metal ions. Also, the effect of the interaction between metals depends not only on the coexistence of other metal ions but also on the nature of the material.

Lead ions showed the highest adsorption capacity (83.5–94 mg/g), which is due to the large radius of the ion (1.21 Å) and electronegativity (2.33). The adsorption of cadmium, copper, and iron ions was relatively low.

The order of magnitude of adsorption corresponds to the order of decreasing ionic radius of the metals: $A(Pb^{2+}) > A(Cd^{2+}) > A(Cu^{2+}) > A(Fe^{2+}) > A(Co^{2+})$.

3.2. Ionic interaction

In multi-component systems, the following trends were noted:

- For cadmium and cobalt ions — synergistic effect (Rqe ≥ 1);
- For iron and copper ions — antagonism (Rqe < 1);
- For lead ion — the results varied depending on the composition of the system.

This indicates that the interaction between ions depends on both their physicochemical characteristics and the structure of the adsorbent.

3.3. The pH change

During the adsorption process, the pH of the solution increased, which is associated with the buffer properties of the adsorbent and the hydrolysis of ions. On the hazelnut and walnut shell carbonaceous materials, the pH increased from 5.0 to 7.9.

3.4. Isothermal analysis

To evaluate the adsorption mechanism, Langmuir and Freundlich isotherm models were used. The Langmuir isotherm model showed a high correlation with the experimental data (correlation coefficient - R^2 = 0.92–0.99) among the studied heavy metals. Lead ions (Pb^{2+}) exhibited the highest adsorption capacity (q_m = 95.24 mg/g) on hazelnut shell carbon material and q_m = 90.09 mg/g on walnut shell carbon, indicating the strong affinity of these materials for lead ions.

Although lead and cadmium ions showed high q_m values, their low adsorption equilibrium constant - KL values suggest a physical adsorption mechanism, likely related to weak van der Waals forces and electrostatic interactions. The Langmuir isotherm did not fit the experimental data for copper ions well. Since the Langmuir model describes a monolayer adsorption system, this may indicate that the adsorbent surface is not fully utilized, and only certain parts are active in adsorption.

The Langmuir isotherm results emphasize selective adsorption behavior towards different metal ions and the heterogeneous nature of the carbonaceous adsorbent surface. Iron ions (Fe^{2+}) showed relatively lower adsorption capacity but a significantly higher KL (0.229 L/mg), indicating strong binding, possibly due to strong electrostatic attraction. These results highlight both selective adsorption behavior and the heterogeneous nature of the adsorbent surface.

The findings suggest that walnut and hazelnut shell carbonaceous materials exhibit high selectivity and efficiency towards heavy metal ions, with efficiency varying depending on the metal type.

Regression analysis of the Freundlich isotherms showed weaker or more heterogeneous adsorption for lead and cadmium ions, as confirmed by the adsorption equilibrium constant - Kf and n values for these materials. The carbon materials are characterized by surface heterogeneity, meaning there are strong and weak active sites with different energies. Strong active sites promote further adsorption, a characteristic of heterogeneous surfaces.

Low Kf values in the Freundlich model indicate weak adsorption, suggesting that physical aggregation or mechanical trapping (i.e., pollutants physically accumulating on the surface) rather than strong adsorption might be occurring on the carbon materials. The degree of freedom in the Freundlich equation is – the n value less than 1 indicates competitive adsorption behavior, where adsorption becomes easier as more molecules accumulate on the surface.

The Freundlich isotherm model showed variable adsorption behavior for different heavy metal ions on walnut shell carbon. For Co^{2+} ions, the Kf values were relatively high (0.525 for walnut shell and 0.926 for hazelnut shell carbon), indicating strong interaction with the heterogeneous carbon surface. Pb^{2+} showed the weakest adsorption (Kf = 0.0052 for walnut shell and 0.0020 for hazelnut shell), despite moderate model fitting, indicating minimal surface interaction.

Copper ion adsorption could not be effectively explained by the Freundlich model due to a low R^2 value (0.26), despite its moderate Kf value.

An n < 1 value indicates the dominance of a physical adsorption mechanism. The results show that despite the heterogeneous surface of the carbonaceous materials, their adsorption efficiency depends on the specific metal ion involved.

The adsorption process of heavy metals (Cd^{2+}, Co^{2+}, Pb^{2+}) fits well with the heterogeneous surface multilayer model ($R^2 \geq 0.90$) on walnut shell carbon material and (Cd^{2+} Cu^{2+}) on hazelnut shell carbon material. However, for all elements, n < 1 suggests the presence of weak adsorption interactions. Higher Kf values for Fe and Co reflect greater adsorption capacity, while lead (Pb), which showed high adsorption in the Langmuir model, exhibited low Kf values in the Freundlich model. These findings confirm that the carbonaceous materials have heterogeneous surfaces with selective behavior toward different metals.

4. Conclusion

It was found out that obtained carbonaceous materials effectively absorb lead, cadmium, copper, cobalt, iron, etc. ions in solution both separately and simultaneously. The absorption efficiency depends on the concentration and pH of the solution, the adsorbent retention time, and the amount of carbonaceous material. The adsorption of heavy metals in polymetallic systems with the simultaneous presence of several metal ions (Pb^{2+}, Cd^{2+}, Co^{2+}, Cu^{2+}, Fe^{2+}) was studied. The purpose of the analysis was to evaluate the effect of ionic interactions between metals on the selectivity of the adsorbent in a complex medium.

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ВОЗМОЖНОСТИ СОРБЦИОННОГО ИЗВЛЕЧЕНИЯ ИОНОВ ТЯЖЁЛЫХ МЕТАЛЛОВ ИЗ ВОД, ЗАГРЯЗНЁННЫХ В РЕЗУЛЬТАТЕ ВОЕННЫХ ДЕЙСТВИЙ, С ИСПОЛЬЗОВАНИЕМ УГЛЕРОДСОДЕРЖАЩИХ МАТЕРИАЛОВ, ПОЛУЧЕННЫХ ИЗ ВТОРИЧНЫХ ОТХОДОВ

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Резюме: Целью исследования было изучение адсорбционного поведения ионов тяжелых металлов (Pb^{2+}, Cd^{2+}, Co^{2+}, Cu^{2+}, Fe^{2+}) в многокомпонентных водных растворах с использованием природных углеродсодержащих материалов. В качестве адсорбентов были выбраны углеродсодержащие материалы на основе скорлупы фундука и грецкого ореха, а также активированный уголь. Исследование проводилось как в однокомпонентных, так и в многокомпонентных системах при стандартных условиях (ms = 1 г, V_x = 100 мл, t = 25 °C, T = 30 мин).

Результаты показали, что наибольшая адсорбционная емкость наблюдалась по ионам свинца (83.5–94 мг/г), далее следовали ионы кадмия (22.3–22.8 мг/г) и меди (15.1–15.8 мг/г). Порядок адсорбции металлов соответствует убыванию ионного радиуса: $Pb^{2+} > Cd^{2+} > Cu^{2+} > Fe^{2+} > Co^{2+}$.

В многокомпонентных системах наблюдались как синергетические, так и антагонистические взаимодействия между ионами. Для ионов кадмия и кобальта коэффициент ионного взаимодействия в сорбционной системе (Rqe) ≥ 1 свидетельствует о синергизме или отсутствии взаимодействия, тогда как для железа и меди Rqe < 1 – об антагонизме.

Повышение pH в процессе адсорбции продемонстрировало буферное действие углеродных материалов. Анализ изотерм Ленгмюра и Фрейндлиха подтвердил физический механизм адсорбции (R^2 = 0.94–0.99) и гетерогенность поверхности.

Исследование подтверждает, что природные углеродные материалы могут быть использованы в качестве эффективных, экологически чистых и недорогих адсорбентов для удаления тяжелых металлов из воды, содержащей большое количество ионов.

Ключевые слова: военные действия, воды, тяжёлые металлы, адсорбция, углеродные материалы, изотерма.

İKİNCİ DƏRƏCƏLİ TULLANTILARDAN ƏLDƏ OLUNAN KARBON MATERİALLARDAN İSTİFADƏ EDƏRƏK, HƏRBİ ƏMƏLİYYATLAR NƏTİCƏSİNDƏ ÇİRKLƏNMİŞ SULARDAN AĞIR METAL İONLARININ ADSORBSİYA ÜSULU İLƏ ÇIXARILMASI POTENSİALI

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Xülasə: Tədqiqatın məqsədi təbii karbon materiallarından istifadə edərək çoxkomponentli sulu məhlullarda ağır metal ionlarının (Pb^{2+}, Cd^{2+}, Co^{2+}, Cu^{2+}, Fe^{2+}) adsorbsiya davranışını öyrənmək idi. Adsorbent kimi fındıq və qoz qabığı karbon materialları və aktivləşdirilmiş karbon seçilmişdir. Tədqiqat həm təkkomponentli, həm də çoxkomponentli sistemlərdə standart şəraitdə aparılmışdır (ms = 1 q, V_x = 100 ml, t = 25°C, T = 30 dəq).

Nəticələr göstərdi ki, qurğuşun ionlarının adsorbsiya qabiliyyəti ən yüksəkdir (83.5–94 mg/g), ardınca kadmium (22.3–22.8 mg/g) və mis ionları (15.1–15.8 mg/g) gəlir. Metal ionlarının adsorbsiya sırası azalan ion radiusuna uyğundur: $Pb^{2+} > Cd^{2+} > Cu^{2+} > Fe^{2+} > Co^{2+}$.

Çoxkomponentli sistemlərdə ionlar arasında həm sinergik, həm də antaqonist qarşılıqlı təsirlər müşahidə edilmişdir. Kadmium və kobalt ionları üçün adsorbsiya sistemində ion qarşılıqlı təsir əmsalı (Rqe) ≥ 1 sinergizmi və ya qarşılıqlı təsirin olmamasını, dəmir və mis üçün isə Rqe < 1 antaqonizmi göstərir.

Adsorbsiya prosesi zamanı pH-ın artması karbon materiallarının tamponlama təsirini nümayiş etdirdi. Langmuir və Freundlich izotermlərinin təhlili fiziki adsorbsiya mexanizmini (R^2 = 0.94–0.99) və səthin heterogen təbiətini təsdiqlədi.

Tədqiqat göstərir ki, təbii karbon materialları çoxionlu sudan ağır metalların çıxarılması üçün effektiv, ekoloji cəhətdən təhlükəsiz və ucuz adsorbentlər kimi istifadə oluna bilər.

Açar sözlər: hərbi əməliyyatlar, sular, ağır metallar, adsorbsiya, karbon materialları, izoterm.