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Application of Zinc-Silver Impregnated Activated Carbons in Removal of Lead(II) and Mercury(II) Compounds from Groundwater

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Abstract. Nowadays activated carbon is a material generating great interest, as it is characterized by a vast surface area due to a high number of pores in its structure. Therefore, the main purpose behind its use is the filtration of impurities from air and water that can be adsorbed with high efficiency. Activated carbon can be easily modified as well. The paper describes activated carbon modification with copper-, manganese-, silver- and zinc salts. The effects of the selected impregnates and their concentrations were examined. The products included 5 adsorbent samples: four universal adsorbents, impregnated with all the above-mentioned salts, and one specific adsorbent sample, designed to adsorb lead(II) and mercury(II) ions and impregnated with zinc- and silver salts only. The premise was to obtain pure drinking water. Properties, such as bulk density, methylene blue number or iodine number were determined for the modified activated carbons. To test the efficiency of an improved adsorbent, an experiment with water highly contaminated with Pb(II) and Hg(II) was carried out, and its results revealed that absorption efficiency for these heavy metals exceeded 99.9%. The adsorber samples were also observed under a digital microscope to compare their appearance.

Introduction

Activated Carbon (AC), thanks to its vast surface area, is a perfect catalyst carrier (support). Even though ACs impregnated with different compounds change their pore structure, such alteration also allows scientists to obtain a material which can serve as a chemisorbent for specific toxic substances [1]. Many compounds can be used for the impregnation process, in particular copper-, chromium-, silver-, potassium-, sodium-, zinc-, cobalt-, manganese-, vanadium-, molybdenum- and iron salts or some organic compounds e.g. pyridine and aromatic amines [2].

Impregnation process parameters are of key importance; even slight changes to the technological regime may cause a reduction in adsorption capacity by 20-30% and could result in completely different adsorbent properties [3]. Increased efficiency and selectivity in the removal of toxic substances is the effect of physical adsorption and chemical adsorption of substances at the AC’s surface or catalytic reactions with the impregnation compounds [4]. One of the most important carbon carriers include Metal-Impregnated Activated Carbons (MIAC). MIACs impregnated with copper, chromium and silver are called copper-chromium-silver (Cu/Cr/Ag) impregnated carbons. They deserve special attention due to their purification performance tested on air samples containing compounds, such as cyanogen chloride, hydrogen cyanide and arsines [5].

Current legal regulations are very restrictive in terms of heavy metal contamination, including mercury contamination of ground- and drinking water, as mercury and its compounds are highly-toxic substances and even in low concentration have a marked detrimental impact on the health and life of living organisms. The contaminants most commonly found in environmental samples

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include metallic mercury, methylmercury and phenyl mercury derivatives. It is believed that the paper industry, chemical industry, batteries production and agriculture are the main culprits and main sources of Hg contamination of the natural environment [6-8].

The efficiency of adsorptive removal of Hg(II) ions is largely dependent on the type of AC and activation process applied. Steam-activated ACs obtained from wood, coconut shells and coal show great capacity to adsorb mercury(II) ions from pH<5 solutions. It has been also discovered that the pH of the solution affects the amount of Hg(II) adsorbed by AC, where a general rule is the lower the pH, the greater the absorption rate observed [9]. The necessity of protecting waters from these contaminants was recognized a long time ago; the first World Health Organization's Standard for drinking water was published in 1958 [10]. It describes the exact maximum allowable concentration of lead in water, which was 0.1 mg/L, but it only briefly mentions mercury as a health threat. Back then, the issue was no sufficient detection methods. The original WHO Guidelines for Drinking Water Quality 3rd edition from 2008 [11] states mercury's acceptable concentration for 0.006 mg/L. Currently, according to WHO guidelines, the allowable concentration of mercury has been maintained, however, the allowable lead concentration has been reduced 10 times – to 0.001 mg/L [12]. Whilst considering the needs in the field of groundwater purification (the goal of which is to obtain drinking water of proper parameters in line with the current regulations), as well as efficiency in removing lead and mercury ions and innovative character of zinc-impregnated adsorbents, new production technology has been developed by the authors. The final products include two adsorbents: a universal (Cr-Cu-Ag-Zn) one and a silver-zinc-impregnated one.

Experimental Procedure

Materials and sample preparation. *DT0* commercially-available activated carbon (produced by Gryfskand Sp. z o.o. company in Poland) was used in this experiment. It was first being oxidized in 20% nitric acid solution under periodical stirring for 24 hours [13]. Then AC was drained off and washed with deionized water until all the acid was removed. Afterwards, a washed activated carbon was kept a room temperature for 24h. At the end of the preparation process, the product was dried in a fluid bed dryer at 120°C for 20 minutes. The obtained adsorbents were divided into two groups:

- universal adsorbents (UA), containing zinc, copper, manganese, silver, and
- adsorbents containing zinc and silver (S-Hg/Pb) only.

The following metallic catalysts have been selected to produce UA:

- basic copper carbonate (Cu2CO3(OH)2) in aqua ammonia;
- potassium permanganate (KMnO4) in aqueous solution;
- silver nitrate (AgNO3) in aqueous solution;
- zinc nitrate (ZnNO3) in an aqueous solution.

After all the solutions were poured into one, the resultant mixture was poured into a beaker containing dried AC and stirred for a few minutes. The mixture was left over for 2 allowing the salts to precipitate. The next step was crucial for obtaining the desired properties of the MIACs: impregnated carbon was dried for 6 hours in total – 4 hours at the temperature of 120°C and 2 hours at 180°C. Copper was deposited on the carbon surface as a copper monoxide (CuO). CuO was obtained by heating a mixture of basic copper carbonate and ammonium carbonate diluted in an ammonia solution (25 wt%). First, a complex ion of hexaaminecopper(II) ion is formed [14]. Then, by its thermal decomposition, copper monoxide, carbon dioxide and gaseous ammonia are produced. CuO is adsorbed in ACs pores and the AC is thusly becoming a chemisorbent or a catalyst for the decomposition of toxic substances. A similar process can be described for potassium permanganate. However, it does not include any complex compounds, and permanganate ions are reduced to manganese dioxide particles [15]. This compound is a chemisorbent and catalyst for the decomposition of toxic substances as well. Silver and copper can also be deposited on activated carbon's surface. First, a diamine silver(I) carbonate complex is formed [16], and afterwards, as a result of thermal decomposition, metallic silver particles, silver(I) oxide, carbon dioxide and ammonia are obtained. The concentrations of each metal ion are shown in the table (Table 1).

The last MIAC (S-Hg/Pb) was specially designated to adsorb lead and mercury only. The key factor behind this process is the presence of zinc ions at the carbon surface, as they are known for their substantial heavy metal absorption properties [17]. Silver is added to obtain antibacterial traits [18] because this adsorbent is to be used to obtain drinking water from the surface and ground waters. Oxidized and dried activated carbon was impregnated with an aqueous solution of:
– silver nitrate ($AgNO_3$),
– zinc nitrate ($ZnNO_3$).

Those metals were deposited on the surface of the AC, as was previously mentioned in this chapter. The adsorbent obtained, S-Hg/Pb, was heated for 20 minutes in a fluid bed dryer, at the temperature of 120°C and then for another 2 hours at 350°C in a laboratory oven, with no air permitted. Finally, the sample was allowed to cool down. S-Hg/Pb contained 2.0% zinc (w/w) and 1.0% silver (w/w).

Table 1. *Concentrations of metal ions in MIACs*

No.	Adsorbent	Copper [% w/w]	Manganese [% w/w]	Silver [% w/w]	Zinc [% w/w]
1.	SU-1	3.0	1.2	0.5	0.5
2.	SU-2	3.0	1.2	0.5	1.0
3.	SU-3	3.0	1.2	-	0.5
4.	SU-4	3.0	1.2	0.5	2.0

Sample characterization. The parameters of obtained adsorbents were characterized by spectrophotometric-, titration- and potentiometric methods. Both the universal adsorbents produced and S-Hg/Pb adsorbed metal ions on their surface, which was corroborated by the methylene blue (MBN) and iodine (IN) numbers study. The two are strictly related to the AC's micro and macropore volume – the greater the volume, the higher the MBN and IN. When these two number decreases, it means that the impregnation salts, or their derivatives, are bound on the surface of carbon grains. The authors experimented according to the Polish Defense Standard [19] which included titration being used to determine IN for all the MIACs samples, as well spectrophotometric measurement of MBN. To find out if there some visual changes on the surface of the impregnated carbons can be seen, the Keyence series VHX-7000 microscope with VH-ZST dual objective zoom lens was used. The authors presumed that the addition of zinc should increase the efficiency of mercury- and lead adsorption capacity, and to corroborate this hypothesis they conducted the required tests. Amount of 1 gram of each SU and S-Hg/Pb were added to two aqueous solutions, one containing lead and one containing mercury, and then they were stirred for 30 minutes. Final concentrations of the tested heavy metals were tested with inductively coupled plasma optical emission spectrometry (ICP OES) using the Thermo Scientific iCAP 7000 series spectrometer. Investigations were carried out with the following spectrometry parameters: lead wavelength: 283.306 nm; mercury wavelength: 253.652 nm or 184.950 nm using the cold vapor method with hydride generation system in case of the latter element.

Results and Discussion

In the beginning, the appearance of *DT0* and S-Hg/Pb was analyzed and the authors found that at the modified adsorbent grain there appeared clearly visible metallic silver aggregates (Fig. 1) that were too large to be deposited inside the pores. Selected parameters of every adsorbent were characterized and compiled in the table (Table 2.). Universal adsorbents' methylene blue number is 20-30% smaller than the MBN of unimpregnated *DT0* signifying that salts used for impregnation, or their derivatives, were deposited in mesopores and greater micropores of AC [20]. SU's iodine number is about 8-13% smaller than IN of plain activated carbon. As iodine is mainly kept in micropores, the lower number points out to the fact that zinc, manganese, copper and silver oxides were deposited in micropores as well [21].

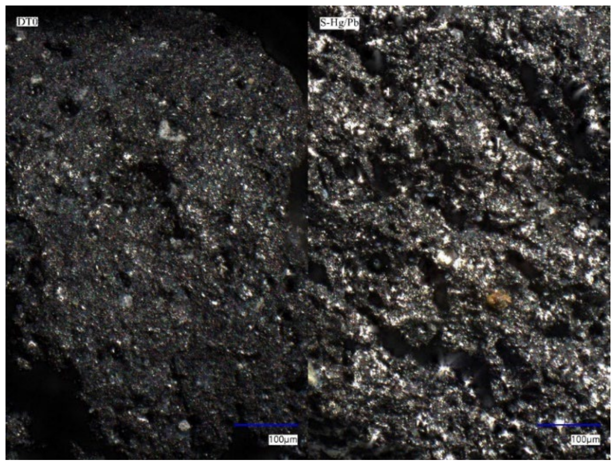

Fig. 1. The appearance of DT0 and S-Hg/Pb MIACs under a microscope

Table 2. *Adsorbent parameters*

No.	Parameter	Units	Adsorbent					
			DT0	SU-1	SU-2	SU-3	SU-4	S-Hg/Pb
1	Water content	%	5	4	4	4	4	3
2	Bulk density	g/cm^3	0.39	0.42	0.42	0.44	0.44	0.39
3	Methylene blue number (MBN)	cm^3	50	35	35	35	40	46
4	Iodine number (IN)	mg/g	1006	883	923	872	895	1045
5	Grain distribution: ≥0.750mm	%	0.40	1.52	1.99	1.16	1.68	2.36
	0.385-0,750mm		96.23	96.57	95.05	95.55	96.32	95.74
	≤0.385mm		3.37	1.92	2.96	2.32	2.00	1.89
6	Concentration in adsorbent: - manganese	%	-	1.2	1.2	1.2	1.2	-
	- copper		-	3.0	3.0	3.0	3.0	-
	- silver		-	0.5	0.5	-	0.5	1.0
	- zinc		-	0.5	1.0	0.5	2.0	2.0

7	Concentration in water (with 10m/h flow): - manganese	mg/dm^3	-	0.02	0.02	0.03	0.02	-
	- copper		-	0.02	0.02	0.02	0.02	-
	- ammonia		-	0.9	1.1	1.0	0.9	-
	pH	-	8.5	7.86	9.2	9.4	9.4	7.8

The main goal of this study was to obtain improved properties of activated carbon in terms of lead and mercury adsorption capacity. The test results are shown in Table 3. Even though universal adsorbents are effective adsorbents of heavy metals but not enough to qualify filtered water as drinking water. The most efficient AC proved to be SU-4; it removed almost 80% of lead(II) ions and nearly 90% of mercury(II) ions from water, while other adsorbents adsorbed 58-82% of these elements. Among all the SUs, SU-4 was impregnated with the highest amount of zinc salt, which had been proven to be to be a good heavy metal binder. Thus, a specially designed MIAC – S-Hg/Pb, containing zinc and silver only, removes lead with more than 99.9% efficiency, while in the case of mercury, it is 99.999% efficient.

Table 3. *Obtained test results.*

Adsorbent	Concentration [mg/l]					
	Lead			Mercury		
	Before adsorption	After adsorption	Percentage removed	Before adsorption	After adsorption	Percentage removed
SU-1	1	0.32	68%	1	0.21	79%
SU-2		0.42	58%		0.18	82%
SU-3		0.38	62%		0.22	78%
SU-4		0.22	78%		0.11	89%
S-Hg/Pb		<0.1	99.9%		0.001	99.999%

Conclusions

In the present study activated carbon was modified with copper-, manganese-, silver- and zinc salts. The effects of selected impregnates and their concentrations were examined. Methylene blue number and iodine number were used to determine if the impregnation process was efficacious or not. The results showed a decrease of MBN and IN by 20-30% and 8-13%, respectively, which means that the process was successfully accomplished and the salts or their derivatives were deposited in AC's pores. There are two more proofs that the deposition occurred; the first of which included the microscopic image o S-Hg/Pb with visible silver aggregates, while the second is the change of bulk density that was caused by the embedding of elements heavier than carbon. The testing of the efficiency of lead(II) and mercury(II) adsorption was done by the comparison of the measurements of SUs and S-Hg/Pb filtrate samples concentrations with ICP OES. While universal adsorbent showed good absorption performance, when heavy metals were concerned, the results for the proprietary adsorbent were excellent. The final concentration of mercury was lower than the reference for drinking water, however, the lead concentration may be higher [11]. Because ICP OES is not accurate enough, another method, e.g. GC-MS, should be used [12]. It can be said that the product designed by the authors performs well, however, further development and testing could be beneficial. Issues related to the removal of heavy metal pollutants from water are strongly linked to production quality [22-24] and management level in businesses [25]. Such pollutants are a common problem in industries that utilize metals [26-28], including special alloys [29], as well as technologies associated with surface layer modification [30] and the application of special coatings [31-33]. The creation of DLC [34, 35] and ESD [36, 37] coatings involve technologies that generate both liquid and gas pollutants, similar to welding processes [38, 39]. Similar pollutants are also formed during the operation of machinery [40], railway rolling stock [41], chemical installations [42], and the use of chemicals in civil engineering [43-45]. The purification processes need to be highly efficient, requiring optimization and careful planning [46]. These processes are multifactorial, and it is beneficial to perform dimensionality reduction [47] prior to optimizing them using statistical methods in industrial settings [48-50] to avoid detrimental correlations. It is also advantageous to utilize non-parametric methods [51-53] that employ a *data-driven* approach, as they are not limited to predefined model assumptions.

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