Chapter 9

Reuse of waste PET bottles through the production of activated carbon, an adsorbent to remove radionuclides from aqueous solutions

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Abstract

End-of-life polyethylene terephthalate (PET) bottles were used to prepare activated carbon by thermal carbonisation at 850 °C for 0.5 hours followed by chemical activation with a boiling aqueous solution of zinc chloride ($ZnCl_2$). The batch adsorption of mono-, di- and trivalent cationic radionuclides was then examined at ambient temperature.

The main physicochemical properties of the obtained material that may determine its adsorption properties have also been studied.

1. Introduction

A statistical Pole throws away almost one hundred polyethylene terephthalate (PET) bottles annually, which form tens of thousands of tons of these packaging per year [1]. The main problems with the waste bottles include that, when collected, they take up a lot of space and each piece decomposes in up to 500 years. In addition, in Poland, only 40% of PET bottles are fully recycled. The rest, if it does not go to landfills, is thrown directly to the environment [2]. For this reason, both public education and recycling of used PET packaging are so important in protecting the environment. In most other European Union countries, recycling rates of plastic bottles is higher, with Lithuania, Czech, and Netherlands leading the list [3]. In 2018, only six European countries processed less used PET packaging than Poland (see, Figure 1).

Figure 1: Recycling rate of plastic waste in Europe, 2018 [3]

Recycled PET bottles are not only used as a raw material in the production of packaging. Obtained granules from grinded plastic bottles can be used to produce fleece, which maintains good thermal properties. It has been estimated that about 35 bottles are needed to produce one blouse from this material. Other products in the clothing industry, such as ski clothes, tents, and backpacks, are also made of polyester fibers [1].

Recycling of PET bottles is a process conducted not only for ecological reasons. Apart from reducing the ever-increasing amount of waste, recycling also allows for obtaining materials that are potentially useful in other areas of life. In the process of pyrolysis of packages from PET, we can produce the activated carbons (ACs) that are specific materials widely used in industry [4-7]. ACs are materials that have a large chemical resistance, great specific surface area and high porosity, and are non-toxic [8,9]. ACs are used, among other things, to remove impurities (either in the gas phase or in liquids). The superiority of the ACs over other purifying materials lies not only in the high purity of the purified solvent but also in cheapness of the purifying material. In addition, designing and operating the purification processes is simple and does not require highly specialised staff. Thus, the use of solid extraction (sorption) of metals on the ACs in the wastewater management seems to be a potential method of treating the liquid radioactive waste (RLW) [10,11].

The aim of the presented work was to check whether an extremely simple recycling method of the PET bottles may be used for producing a material suitable for decontamination of water containing the radionuclides.

2. Experimental

2.1. Preparation of the carbon powder

Powdered carbon has been formulated by adapting a method proposed by Bratek *et al.* [12]. Used mineral water bottles made of PET were cut into small pieces (approx. 1 cm^2, each) and about 5g of sample were placed in quartz crucibles and high temperature carbonised (1098 K) in a nitrogen atmosphere (flux of about 10 $dm^3 \cdot h^{-1}$). A vacuum furnace (Nabertherm, VHT series) was used. The temperature of the process was raised from room temperature at a rate of 1 K/min, then kept constant for 30 minutes. The resulting carbon material was cooled at the same rate (full pattern of the temperature changes vs. time is shown in Figure 2). Yield of the process was assigned to about 20 %. The obtained material was grinded and analysed for the total organic carbon (TOC) (Analytic Jena Multi N/C 3100 with autosampler and high-temperature combustion chamber HT 1300). It was found that the material consisted of 99.32±1.03 % carbon.

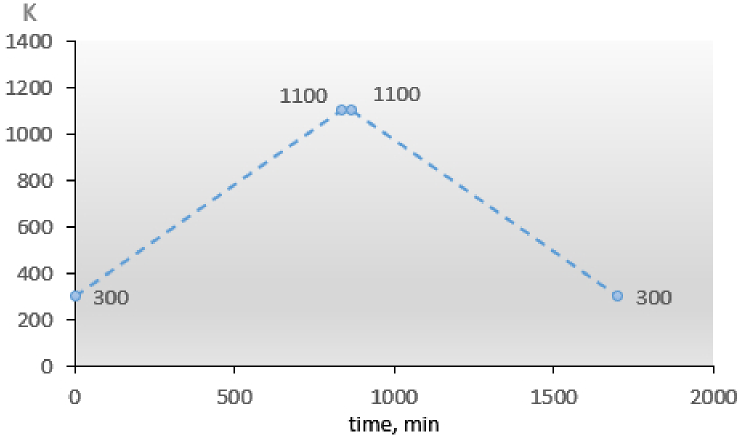

Figure 2: Time dependent temperature of the carbonisation process.

2.2. Activation of the carbon powder

During carbonisation, a certain porous structure is already formed, but its parameters are insufficient for practical application. Therefore, the product should be subjected to activation, aimed at 'developing' the pores. During the chemical charcoal activation in an industrial scale, the raw material is subjected to treatment with certain chemical activation agents. The most often used are phosphoric acid, natrium hydroxide, or zinc chloride. Then, the material is heated to 720−950 °C in an activation furnace. The resulting material is then washed with water until the activating agent is removed. The obtained activated carbon is dried and often sieved to get particles of the desired specific size range.

In this work, the crude carbon samples were chemically activated to obtain activated carbon (AC) in not so radical conditions. In these studies, the following activators were used: $ZnCl_2$, $CaCl_2$, H_3PO_4, KOH, HNO_3, HCl or H_2O_2, separately. A one-step process was carried out, according to Caturl *et al.* [13]. An aqueous solution containing one of the above-mentioned agents was mechanically mixed for eight hours. (Usually overnight) with a sample of carbon suspended in. A temperature of mixing was kept at 85 °C. Then, the samples were filtered, accurately washed with deionised water until an excess of the activating agent was removed and dried at 80 °C to a constant mass. After grinding, the material was exposed for one day to the ambient atmosphere and stored for further experiments.

In the case of using hydrogen peroxide for activation, about 30 g of the raw carbon was suspended in 200 ml of 10% H_2O_2 solution and stirred for 2 hours at room temperature [14]. The obtained AC solid was then rinsed with water, dried at 80 °C to a constant mass and after the 24 hours exposition to an air stored for the experiments.

The obtained material has been characterised by several methods, which are listed in Table 1. A more detailed description of the results obtained is presented in sections 3.1-3.5.

2.3. Chemicals and radionuclides

Chemicals used in this work were delivered from Merck-Sigma-Aldrich or Avantor Performance Materials (Polish branches) as the materials of pure p.a. grade. Throughout the work, deionised water was used.

The commercial charcoal adsorbent *Norit CNR116* was chosen to be a reference material in these studies. The commercially available product was treated with 10 % HCl for removal inorganic impurities.

Carrier-free radionuclides (i.e., free from any stable isotopes and radiochemically monoisotopic) of caesium-137 (t1/2=30.07 y; Eγ=661.7 keV), strontium-85 (t1/2=64.8 d; Eγ=514 keV), cobalt-60 (t1/2=5.3 y; Eγ= 1.173 and 1.332 MeV) and americium-241 (t1/2=432.2 y; Eγ=59.5 keV) were bought from POLATOM Świerk (Poland). Radionuclides Sr-85 and Tc-99m were used instead of Sr-90 and Tc-99 because of their easier radiometric detection.

NaTcO4(Tc-99m) was eluted from a Mo-99/Tc-99m commercial generator (GE Healthcare, supplied by Biker, Warszawa, Poland) as the 0.9% saline solution of ca 100 MBq·cm-3 specific radioactivity.

2.4. Studies on sorption of the radionuclides

Batch sorption experiments, also called static sorption, were performed by adding a known quantity of the AC sorbent to a solution containing impurities (in our case, radioactively contaminated) of known concentration. These suspensions were strongly shaken or vortexed throughout the desired time. Concentrations of the radionuclides in the initial solution, as well as in the final one, were detected radiometrically and their difference was treated as the amount of the radionuclide (q_e) be separated by a solid sorbent and used in Eq. 1.

$$q_e = (C_0 - C_e) \cdot \frac{V}{m} \tag{1}$$

where C0 and Ce are the initial and equilibrium concentrations of the radionuclides (any units of the radiation activity concentration), m and V stand as the mass of AC (g) and volume of the solution (mL) used in the experiments, respectively.

2.5. Details of methods used for characterisation of the AC materials and the devices used

See Table 1.

3. Results and discussion

3.1. Vibrational (FTIR) spectra: no chemical changes after activation of the raw carbon

Transmission spectra in the mid infrared region, both PET carbon and the activated PET carbon with the $ZnCl_2$ solution are presented in Figure 3.

A comparison of the spectrum registered for the crude PET carbon with this of the material treated with ZnCl2 shows that both spectra, even though they have rather poorly developed peaks, resemble each other. A reliable method for the numerical comparison of two spectra requires calculating the Pearson's correlation coefficient (P.c.c.), which must be between -1 and +1. A zero value describes spectra that do not correlate at all, that is, are completely different. On the contrary, values not far from ±1 suggest that both spectra are of great similarity, and therefore, that both compared substances are identical [15]. The computed value of the P.c.c., for the PET- and the ZnCl2 activated carbon is 0.998. Therefore, it may

Table 1: Methods of characterisation of the AC and the devices used

Characteristic	Methods	Devices
Confirmation of synthesis result	Recording of the FT-IR spectra of the dried materials	Nicolet iS10 (Thermo Fisher Scientific Instrument, USA)
Amorphous vs. crystalline structure	Powder X-ray diffraction	HZG-4 diffractometer (Seifert GmbH, Germany)
Specific surface area and pore volumes	N2 adsorption-desorption isotherms measured at 77.3 K	ASAP 2024 automatic sorption analyzer (Micromeritics, USA)
Charge of the surface	Determination of the zeta potential (PZ, ζn)	Zetasizer Nano ZS instrument (Malvern, UK)
Decomposition temperatures	Thermogravimetric (TGA) and derivative thermogravimetric (DTG) analyses	Linseis Stapt 1600 (Linseis Corporation, Selb, Germany)
Topography and elemental composition of the surface	Scanning Electron Microscopy	DSM 942 Scanning Electron Microscope (Zeiss-Leo, Germany
	Energy-dispersive X-ray spectroscopic (EDS) microanalysis	Quantax 400 (Bruker, Germany)
Adsorption properties	Batch adsorption with radiometric analyses	Heidolph Multi Reax test-tube shaker and MPW-251 centrifuge
		Perkin Elmer 2480 Wizard2© Automatic Gamma Counter
		Thermo Scientific Dionex ICS-5000 DC chromatographic set

be postulated that chemical activation does not result with the significant change in chemical structure of the material.

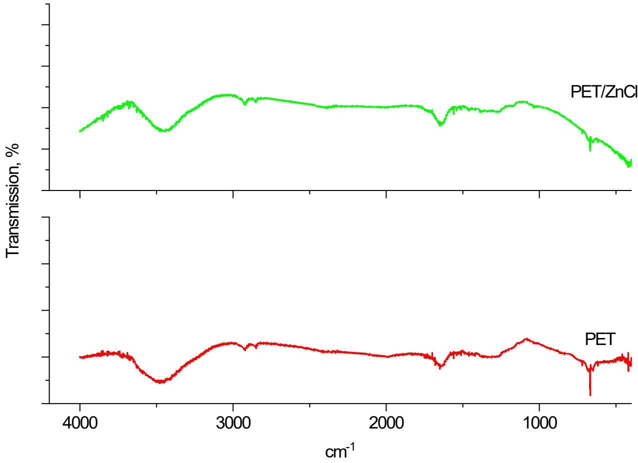

Figure 3: Mid infrared transmission spectra of the PET carbon and the ZnCl2 activated PET carbon sorbents.

In both spectra, the only wide bands can be observed in the region of about 3500–3600 cm^{-1}, and are accompanied by weak peaks in the regions of about 1650 cm^{-1} and about 670 cm^{-1}. As a rule, in the carbon spectra, the bands in the area of about 3500 cm^{-1} are characteristic for stretching vibrations of the -OH hydroxyl groups. Weak peaks at about 1650 cm^{-1} were attributed as stretching C=C of the aromatic rings [16]. Angular deformations of CH groups of aromatic rings outside the planes of the rings are usually related to low frequency bands (about 700 cm^{-1}) [17].

3.2. Crystallographic studies

Carbonic materials can occur in various structural forms: some of them are the amorphous materials, others are crystalline (for example, graphite). Plots of the X-ray powder diffraction obtained for the ZnCl$_2$ activated PET carbon and Norit CNR116 materials are shown in Figure 4.

Diffractogram of the ZnCl$_2$ activated PET carbon reveals the existence of two distinct, but broad bands found at $2\Theta = 23.2$ and 43.7 °. It may be found in the literature that they are characteristic for a graphitic structure [18] and are commonly described as the (0 0 2) and (1 0 0) reflections of the graphitic carbon [19]. The corresponding diffractogram of Norit CNR116, in turn, is characteristic for the amorphous materials.

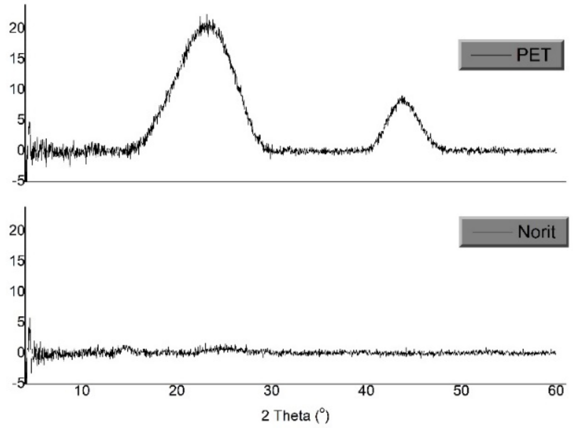

Figure 4: X-ray powder diffraction of the ZnCl2 activated PET carbon and Norit CNR116.

3.3. Scanning electron microscopy (SEM)

Scanning electron microscopy (SEM) equally to the transmission microscopy performed in the visible light and to the confocal laser scanning microscopy, gives us numerous interesting information on the surface topography of the studied material. This technique, however, allows for both a higher magnification and a deeper penetration of the surface. In turn, the SEM method with energy dispersive X-ray spectroscopy (EDX) provides information on the elemental composition of the sample surface. Both SEM and EDX are simple and non-destructive methods which permit for prompt measurements of different matrices.

Images shown in Figure 5 present the surface of the waste PET carbon particles, of the chemically activated with the $ZnCl_2$ solution, and of the Norit CNR116 reference material. Photographs made in the 100x magnification reveal that the surface of the PET carbon particles has crevices, gaps, and crystals of various sizes (Figures 5a and 5b). In addition, some crystals have large holes. The surface of the Norit material is more compact than that of the other materials. This observation corresponds well with the results of the XRD studies presented in part 3.2. Surfaces of the samples observed under the magnification of 5000x reveal, however, more distinct differences. That of the activated PET carbon is more uniform as compared with the Norit one and some micro agglomerates can be seen on the PET carbon surface. On the surface of the Norit material, one can observe the micropores leading to the interior of the particle.

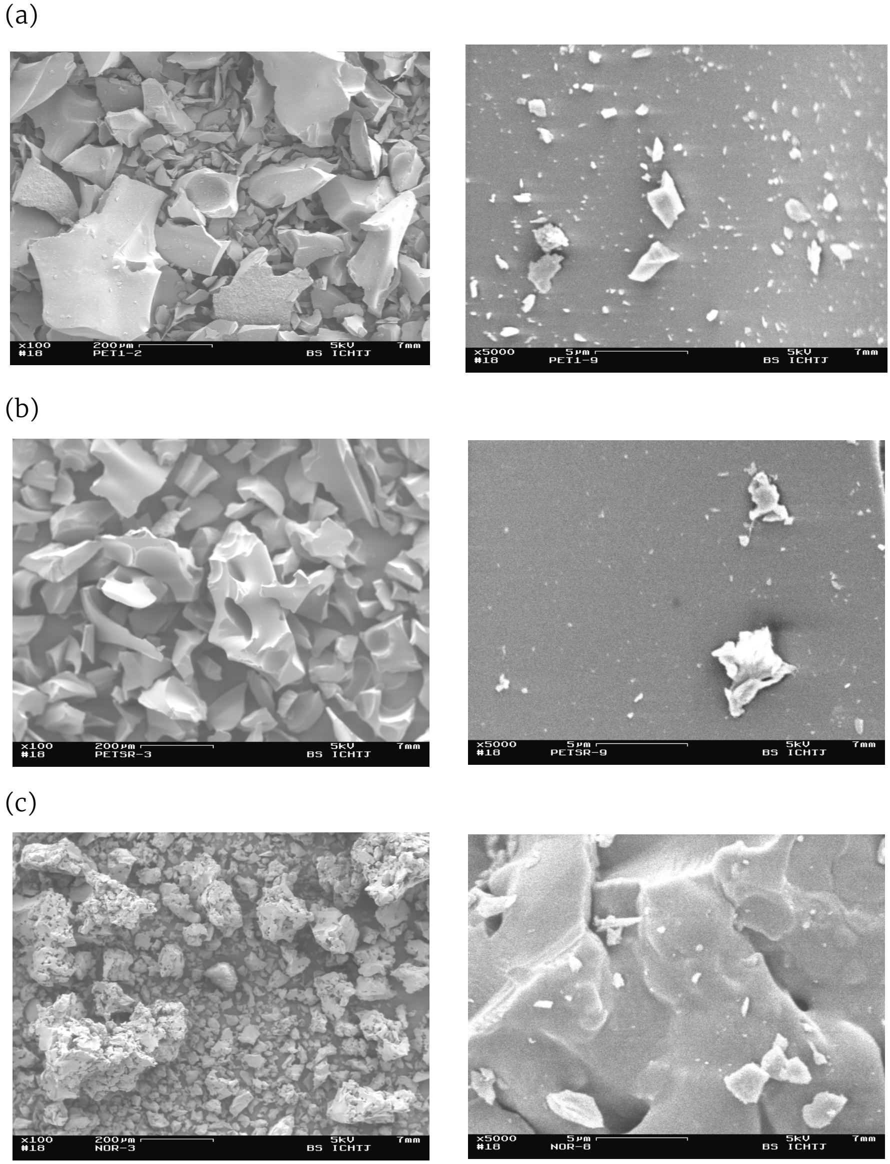

Figure 5: SEM images (100x magnification - left, and 5000x – right pictures) of the PET carbon: raw material (a), sorbent saturated with $SrCl_2$ (b); Norit CNR116 reference material (c)

3.4. Point of zero charge (PZC) of the sorbent surface

Among the main properties of the sorbent that contribute to adsorption of the ionic species is its point of zero charge (PZC). The PZC parameter describes the pH, for which the surface of the adsorbent is not charged (meaning, is neutral). It means that the surface contains the equal number of the positively and negatively charged binding groups. Below the pH described as the PZC, the surface is positively charged and over this pH, it is negatively charged. So, comparing the PZC with the pH of the solution to be purified informs if a metal cation ion can be sorbed by a sorbent: cations interact preferably with a negatively charged surface.

Values of the PZC for the crude PET carbon and the $ZnCl_2$ activated were determined to be: 1.1±0.1, 0.7±0.1, and 3.3±0.3 for the raw material, $ZnCl_2$ activated, and Norit, respectively. When comparing the above values with the pH dependent speciation of the radionuclides studied (Figure 6) it can be seen, that for pH of the solutions greater than one (the PZC), either the AC or the raw carbonic sorbent or Norit are mainly positively charged. Therefore, for pH values greater than 1, they are capable of sorbing the radionuclides.

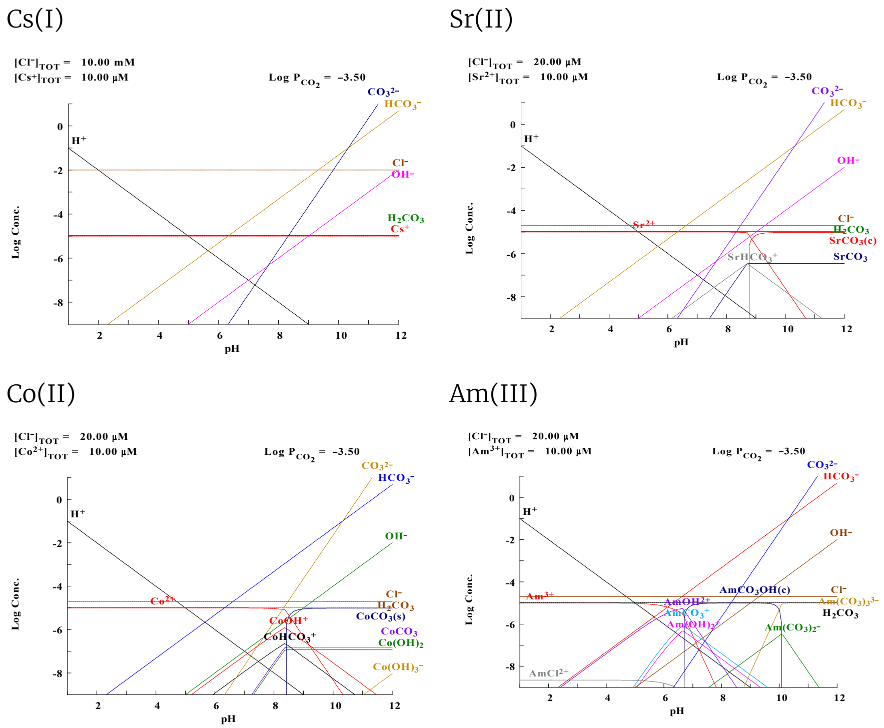

Figure 6: Speciation of Cs(I), Sr(II), Co(II) and Am(III) complexes in aqueous solutions of different pH. Simulation computed using the Medusa program [20].

3.5. Surface potential and adsorption of metals

One of the most important properties of the material that determines its sorption properties is the charge of the surface. To describe the magnitude of this charge, we often use–a quantity called the zeta potential (ZP), which tells us about the potential difference on both sides of the boundary between the solids and liquids. In this work, we have determined the ZP for a broad range of the solution pH, namely for 3.5, 4.2, 6.9 and 10.4. For the raw carbonic materials, we have found that the values are 18.1 mV, -29.3 mV, -49.6 mV, and -47.2 mV, respectively. The corresponding values for the $ZnCl_2$ activated carbon, being correspondingly -10.6, -31.3, -50.1, and -44.4 mV, are presented in the upper part of Figure 7. It can be seen that the data do not differ each other and the most negative values are in the region of the neutral solutions (pH around 7). It has also been found that even in the strongly acidic solutions reach in protons, both examined materials preserve their negatively charged groups. These negative charges come most of all from the presence of the carboxyl groups, which are reported to occur on the surface of the activated carbon [21]. It can be seen that with decreasing acidity of the solutions, i.e. with decreasing number of protons, in accordance with the law of mass action, dissociation of these carboxyl groups is favoured. So, a charge of the surface (ZP) becomes more negative.

Detailed data on the dependence of sorption of the radioactive metals determined in this work will be presented in section 3.7. However, already now, in order to compare them with the changes of ZP along the pH series in the lower part of Figure 7, selected data for strontium (II) are presented. It can be seen that both patterns are reversed. When the ZPs of the activated carbon surface become more negative, the electrostatic attraction with the positively charged cations increases and vice versa.

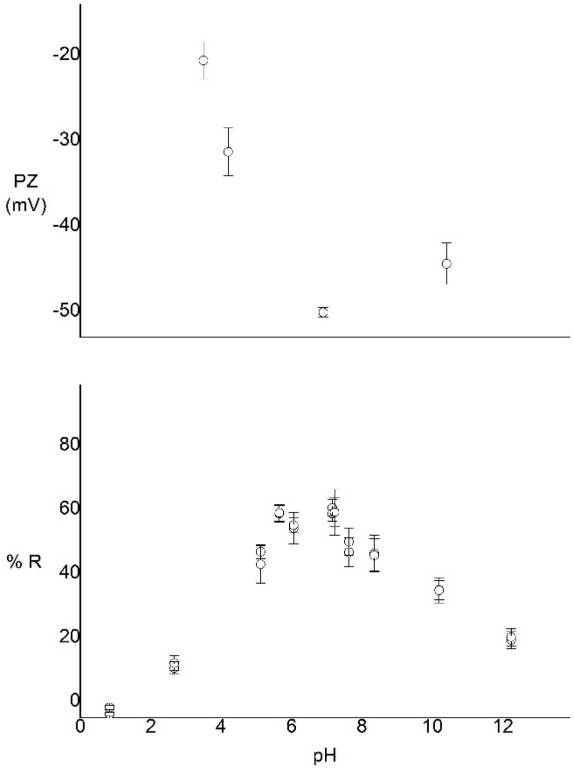

Figure 7: Changes of zeta potential (ZP) of the $ZnCl_2$ activated carbon with pH (upper part) and the adsorption properties of strontium (II) by this material (bottom).

3.6. Sorption properties of the carbonaceous material: chemical activation.

Carbon materials are used as popular sorbents effective in the removal of metal ions from aqueous solutions because of their valuable properties, for example, their relatively great porosity and big surface area, high adsorption capacity, low price and, most importantly, high chemical inertness [22-24].

There are cases where carbon materials do not adsorb fully metals that exist in aqueous solutions in trace or ultra-trace concentrations. So, many times, authors have proposed some preliminary methods for their surface modification (often, activation) in order to enhance their adsorption properties [25-29].

Besides some physical methods (e.g. high-temperature heating), this goal may be reached by chemical methods that apply certain chemical agents. Typically, the latter provides better results than the former [30]. Nevertheless, the chemical activation method also has some limitations. Among the most important, one should mention the need of removal an excess of the activation agents by washing the carbon with a large volume of water, which may result in the adverse environmental problems.

The result of the action of different activating chemicals was checked by comparing the adsorption property of each AC sample with this of raw material. The cumulative graph of such comparison is presented in Figure 8, where the raw material is named PET. One may see that using the $ZnCl_2$ containing aqueous solution results in the best results. The adsorption properties are comparable to those of the commercial Norit CNR116 AC adsorbent.

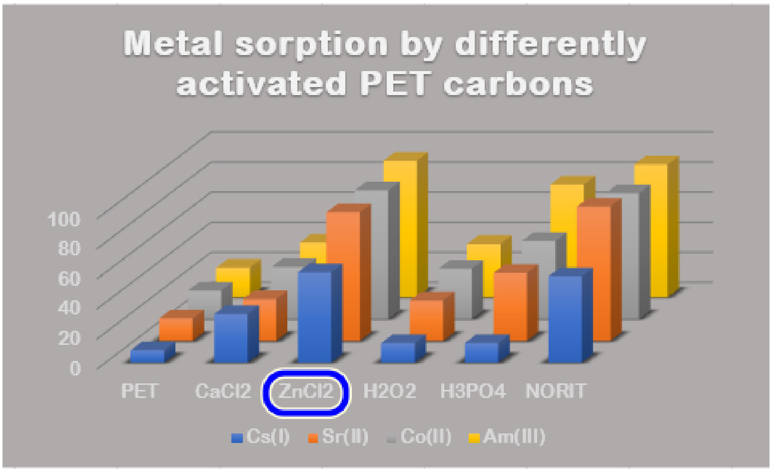

Figure 8: Sorption of the radioactive metal ions by carbon samples activated with different chemicals. Sorption by Norit CNR116 reference material is also shown.

3.7. Sorption properties of the carbonaceous material: the pH and sorbent dosage dependence.

When designing a sorption process on an industrial scale, it is important to know the dose of the sorbent that ensures the efficient removal of metals. In the present work, eight different doses of the chemically activated carbon sorbent were examined. They ranged from 1 to 20 $g{\cdot}L^{-1}$.

The obtained results are shown in Figure 9. It has been found that up to the adsorbent dose of about 5 g·L−1, the removal efficiency improves for each metal. Then, the removal effectiveness reaches a plateau value. Such an increasing trend may be explained in terms of the growing number of sorption centres, for the homogenous surface sorbents being related to the mass of these materials. Above the dose of 5 g˙L-1, the number of the sorption centres exceeds this of the sorbate metal ions, especially if they are in the nca[1] concentration.

In conclusion, a dose of 5 $g{\cdot}L^{-1}$ of the sorbent seems to be the suitable amount for the proposed procedure.

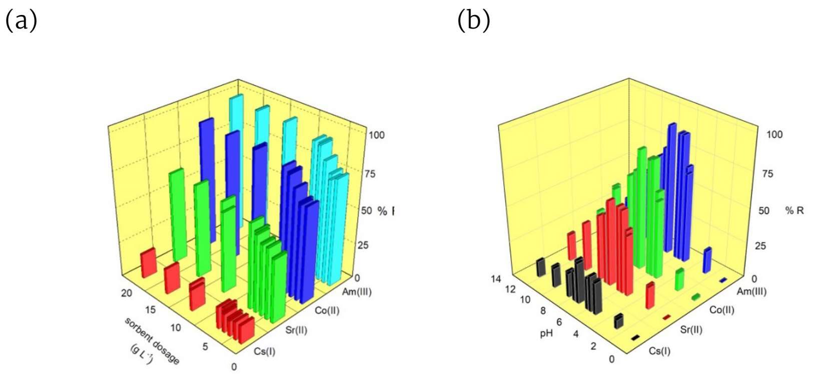

Figure 9: (a) Dependence of the adsorption efficiency, %R, of the radioactive metals on the adsorbent dose; (b) Relation between the acidity of the solution and the efficacy of mono-, di-, and trivalent radioactive metals removal by the PET carbon.

Another factor of the greatest importance in determining the metal sorption is the acidity of the solution. It affects both the charge of the adsorbent surface and the speciation of metals. In this work, the effect of the pH was studied within the pH range of about 11 units. Results presented in Figure 9 show that the %R values for each radioactive metal arise its maximum value placed in the range from pH 5 to pH 8. These features have been found already for sorption by different activated carbon materials, e.g. in [28, 29].

3.8. Sorption properties of the carbonaceous material: metal sorption capacity of the PET carbon

A specific amount of metal that may be adsorbed (called as adsorption capacity, CEC), was determined in the present work using non-radioactive

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[1] No Carrier Added (nca) radionuclide solution means that no carrier atoms have been added and that during its preparation special actions have been undertaken to minimise contamination with stable isotopes of the element in question., However, it does not mean the monoisotopic radioactive substance [Glossary: Definitions and Terminology; https://link.springer.com/content/pdf/bbm%3A978-81-322-2607-9%2F1.pdf].

caesium(I), strontium (II) or cobalt (II) chlorides, respectively. Aqueous solutions containing any metal salt in the concentration of 10 $g \cdot L^{-1}$ were agitated for six hours. with a weighed amount of sorbent. Then, the phases were separated, and the initial and equilibrium solutions were subjected to the metal content analysis using a method of ion chromatography.

It has been found that the adsorption capacity of strontium (II) sorbed at pH 6 was 2.1 $mg \cdot g^{-1}$ for the raw carbon and 2.7 $mg \cdot g^{-1}$ for the $ZnCl_2$ activated carbon. The reference value for the Norit CNR116 is two times larger (5.2 $mg \cdot g^{-1}$). However, one should remember that the procedure for the carbonisation of the PET material and further activation may be not optimum. Data found in the relevant literature on the different activated carbon materials produced in the optimum conditions show that the CEC varies from only 2 $mg \cdot g^{-1}$ to about 180 $mg \cdot g^{-1}$ [31].

In turn, adsorption capacity values for caesium(I) and cobalt (II) have been determined to be 4.4 $mg \cdot g^{-1}$ and 4.1 $mg \cdot g^{-1}$, respectively. The lack of correlation between the adsorption capacity, metal ionic radii, and the ionic charges suggests that the total sorption capacity depends on the adsorption of different hydration forms of these metals present in the mother solution.

4. Conclusion

- About 80 % carbon content of the produced sample indicates that better pyrolysis conditions should be found so that the result in more carbon-containing (less impurities) materials.
- The structure of the material does not change upon activation and differs from the commercial charcoals.
- The obtained material meets the requirements necessary for use in the purification of radioactively contaminated water.

5. Additional information

- This study was supported by the Institute of Nuclear Chemistry and Technology through the financial support of the Polish Ministry of Climate and Environment for the statutory activity of the INCT.
- Author contributions: L. F. conceptualisation, physicochemical and sorption studies, writing - original draft preparation; I. H.-K.: physicochemical and sorption studies, writing - review and editing; M. R.: synthesis of the carbonic material.
- All authors have read and agreed to the published version of the manuscript.
- The authors declare no competing interests.

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