

Cesium adsorption from aqueous solutions onto Japanese oak charcoal I: effects of the presence of group 1 and 2 metal ions

Shigeru Yamauchi · Takayuki Yamagishi ·
Kazuko Kirikoshi · Mitsuyoshi Yatagai

Received: 27 February 2014 / Accepted: 6 August 2014 / Published online: 12 September 2014
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Abstract To understand the mechanisms of adsorption of cesium ions onto charcoal from aqueous solution, adsorption isotherms were analyzed in terms of initial cesium concentration in aqueous solution. Aqueous cesium chloride solutions (CsCl concentration: 2.50×10^{-4} or 2.50×10^{-5} mol/L) were used as samples for the adsorption study. Japanese oak charcoal and activated charcoal passed through a 60-mesh sieve were adopted as adsorbents. Japanese oak charcoal displayed higher ability to adsorb cesium ions than activated charcoal. We examined the blocking effects of group 1 and 2 metal ions on the adsorption of cesium ions, because most charcoal or biochar probably contains considerable amounts of group 1 and 2 metals. The group 1 and 2 metals of lithium, sodium, potassium, magnesium, calcium, and strontium were added to 2.50×10^{-5} mol/L cesium chloride solution. A remarkable finding was that group 1 metal ions, which are monovalent cations, prevent cesium ions from attaching to Japanese oak charcoal much more strongly than group 2 metal ions, which are divalent cations.

Keywords Cesium · Adsorption · Japanese oak charcoal · Group 1 metal · Group 2 metal

Part of this report was presented at the 63rd and 64th Annual Meetings of the Japan Wood Research Society, Morioka, March 2013 and Matsuyama, March 2014.

S. Yamauchi (✉) · T. Yamagishi · K. Kirikoshi
Institute of Wood Technology, Akita Prefectural University,
11-1 Kaieisaka, Noshiro 016-0876, Japan
e-mail: sigeru@iwt.akita-pu.ac.jp

M. Yatagai
Supporting Organization for Research of Agricultural and Life
Sciences, 2-7-13 Uehara, Shibuya-ku, Tokyo 151-0064, Japan

Introduction

The hydrogen detonation at the Fukushima Daiichi nuclear power plants due to the Great East Japan Earthquake in March 2011 resulted in the large-scale scattering of radioisotopes. There is still a large radioactively contaminated area in Fukushima Prefecture, which is the most serious social and environmental problem in Japan. The key radioisotopes in the polluted area are ^{134}Cs and ^{137}Cs , and appropriate treatments for nuclear decontamination should be applied immediately and continuously. The first step of nuclear decontamination in forests and fields is to prevent radioactive materials from diffusing and moving. Therefore, it is of great importance to fix the radioisotopes using adsorbents.

Numerous reports of cesium adsorption using porous inorganic materials have already been published, while the number of studies on cesium adsorption through biochar has been gradually increasing [1–8]. We considered charcoal to be a key material that should be urgently examined in terms of its practical utility as an adsorbent of cesium [9].

To scatter a sufficient amount of adsorbent over a wide region, the adsorbent must not only have a high ability to adsorb cesium, but must also be producible on a massive scale using existing facilities in Japan. Charcoal has been attracting attention as adsorbent, and the amount of woody biomass for making charcoal is extremely abundant. Commercially available charcoal does not always have the same surface physical and chemical properties, and it may be expected that some charcoals produced under appropriate conditions exhibit remarkable ability for cesium adsorption. Thus, we focused our attention on Japanese oak charcoal, which is the most common charcoal in Japan.

In a previous work [10], we studied the cesium-adsorption behavior of commercially available Japanese oak and activated charcoal, whose manufacturing temperatures were estimated to

be 600–800 °C and more than 1000 °C, respectively, based on the Raman spectroscopic data [10, 11]. Investigations into cesium adsorption from aqueous solutions were carried out with respect to pH and surface structures of adsorbent, indicating that Japanese oak charcoal has a considerable ability to adsorb cesium ions from neutral or weakly basic aqueous solutions. From our adsorption experimental data, it was suggested that Japanese oak charcoal is superior to activated charcoal, which has a large specific surface area, in terms of adsorbing cesium, despite its smaller specific surface area and predicted that the primary interaction for cesium adsorption onto charcoal is electrostatic attraction induced by surface potential of charcoal, in other words, functional groups on charcoal surface. Moreover, it was confirmed that sodium ions strongly obstruct cesium adsorption onto Japanese oak charcoal.

Further study is required to better understand the cesium-adsorption phenomena from aqueous solution. In the present work, the cesium-adsorption abilities of Japanese oak and activated charcoal were examined in more detail, using more dilute aqueous cesium chloride solutions than the solutions researched in our previous work [10]. We examined the blocking effects of group 1 and 2 metal ions on cesium adsorption onto Japanese oak charcoal in more detail because our previous work [10] is, to our knowledge, the only report on the blocking effects on biochar and ordinary charcoal contains several group 1 and 2 metals. Here, we investigate the adsorption mechanisms of cesium ions onto charcoal surfaces from aqueous solution and present a remarkable result that cannot be explained only by general knowledge on colloidal or interfacial science.

Experimental

Materials

Japanese oak charcoal produced in Iwate Prefecture and activated charcoal for column chromatography (Wako Pure Chemical Industries, Ltd., CAS[7440-44-0]) were used as adsorbents after passage through a 60-mesh sieve, and drying at 105 °C for over a day. The cesium chloride (CsCl) and other compounds used were commercially available special-grade chemicals. No radioactive cesium isotopes were employed in this study. Aqueous solutions were prepared using water purified through a column filled with ion-exchange resins.

Determination of nonmetallic elements and ash in Japanese oak and activated charcoal

The contents of nonmetallic elements and ash in Japanese oak and activated charcoal were determined according to

the Japanese Industrial Standard for coal and coke (JIS M 8813, 2004) [12]. Ashing of charcoal was carried out at 1350 °C.

Preparation of aqueous cesium chloride solutions

Aqueous cesium chloride solutions were prepared using cesium chloride dried at 100 °C. The initial cesium chloride concentration was 2.50×10^{-4} mol/L (Cs⁺: 33.2 mg/L, solution I) or 2.50×10^{-5} mol/L (Cs⁺: 3.32 mg/L, solution II). Cesium chloride solutions containing group 1 and 2 metal chlorides, lithium chloride (LiCl), sodium chloride (NaCl), potassium chloride (KCl), magnesium chloride (MgCl₂), calcium chloride (CaCl₂), or strontium chloride (SrCl₂), were prepared to examine the blocking effects of group 1 and 2 metal ions on cesium adsorption onto Japanese oak charcoal. The additional amounts of the group 1 and 2 metal chlorides were 5.1×10^{-3} , 5.1×10^{-2} , and 5.1×10^{-1} mol/L. The initial cesium chloride concentration of all the solutions supplemented with the group 1 and 2 metal chlorides was 2.50×10^{-5} mol/L (Cs⁺: 3.32 mg/L).

Determination of solid-phase cesium concentration

Sealed Erlenmeyer flasks including aqueous solution (50 mL) and powder adsorbent (0.10–1.60 g) were circularly shaken at 23 ± 2 °C for 24 h. The powder was then filtered from the suspensions through a nylon-membrane microfilter (Millex-HN filters SLHN033NS, Millipore Ireland Ltd.). Solutions I and II with no added adsorbent were filtered by the microfilter to estimate the amount of cesium ions attached to the membrane.

The pH of the filtrate was recorded using a pH meter. The cesium ion concentration was determined using an ICP mass spectrometer by an internal standard method (standard isotope: ¹¹⁵In). The ICP mass measurements were performed three times per sample solution. All the relative standard deviations were within 3.8 %. The cesium ion concentration in the filtrate was considered to be the cesium ion concentration at equilibrium. The solid-phase cesium concentration, namely, the cesium concentration on adsorbent, was calculated by dividing the decrease in mass (mg) of cesium ions in aqueous solution by the mass (g) of powder adsorbent added.

Results and discussion

Characterization of Japanese oak and activated charcoal

The contents of nonmetallic elements and ash in Japanese oak and activated charcoal are summarized in Table 1.

Table 1 Contents of nonmetallic elements in Japanese oak and activated charcoal

Charcoal	Percentage by mass (dry basis, %)					
	Carbon	Hydrogen	Oxygen	Nitrogen	Sulfur	Ash
Japanese oak charcoal	83.86	2.36	11.86	0.45	0.04	1.32
Activated charcoal	69.30	1.12	18.07	0.46	0.24	9.33

These contents were calculated on the basis of the dry mass of charcoal. The carbonization temperatures estimated from Raman spectroscopic measurements and surface physical properties such as specific surface area have been reported previously [10].

For charcoal, it is generally agreed that carbon content increases and hydrogen content decreases as carbonization temperature rises. The carbon and hydrogen contents in Japanese oak charcoal are not inconsistent with those of common charcoal made from wood in terms of the carbonization temperature (600–800 °C) [13, 14], while the carbon content in activated charcoal is considerably small despite more than the 1000 °C carbonization temperature and the ash content is unusually large as for wood charcoal. Thus, it is suggested that the raw material of activated charcoal is not ordinary wood.

Adsorption isotherms of cesium ions onto Japanese oak and activated charcoal

Our previous work reported the adsorption behavior of cesium ions onto Japanese oak and activated charcoal from concentrated aqueous cesium chloride or nitrate solutions (Cs^+ : ~330 mg/L) [10]. In this study, more dilute aqueous cesium chloride solutions were adopted to understand more clearly the adsorption behavior of cesium ions. Figure 1 shows the plots of equilibrium cesium ion concentrations in aqueous solution (C : mg/L) against cesium concentration on Japanese oak and activated charcoal (W : mg/g), namely, isotherms of cesium adsorption onto Japanese oak and activated charcoal from aqueous solutions with initial cesium ion concentrations of 33.2 mg/L (solution I). Figure 2 depicts isotherms of cesium adsorption onto Japanese oak and activated charcoal from aqueous solutions with initial cesium ion concentrations of 3.32 mg/L (solution II). All the suspensions were weakly basic after 24 h of shaking. Additionally, reduction rates in cesium ion concentration in solutions I and II by filtration were less than 2.5 %. Thus, it is concluded that the number of cesium ions attached to the nylon membrane of the microfilter was negligible.

From Figs. 1 and 2, it is apparent that the cesium concentrations on activated charcoal are lower than those on Japanese oak charcoal. In other words, the cesium-adsorption capacity of activated charcoal is inferior to that of Japanese oak charcoal. This agrees with our previous

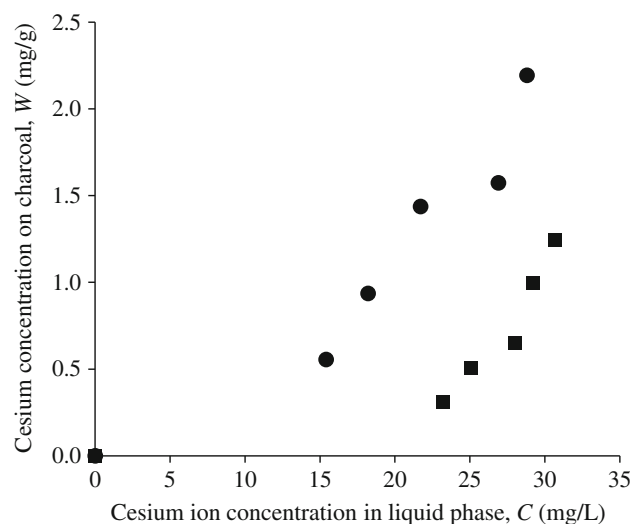


Fig. 1 Isotherms of cesium adsorption onto Japanese oak and activated charcoal from 2.50×10^{-4} mol/L $CsCl_2$ solution (Cs^+ : 33.2 mg/L). The pH values of the suspension samples ranged from 8.8 to 10.1. Adsorbent: filled circles, Japanese oak charcoal: filled square, activated charcoal

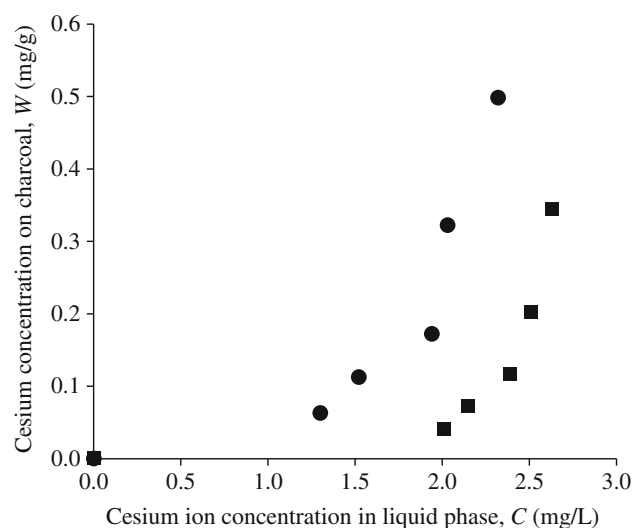


Fig. 2 Isotherms of cesium adsorption onto Japanese oak and activated charcoal from 2.50×10^{-5} mol/L $CsCl_2$ solution (Cs^+ : 3.32 mg/L). The pH values of the suspension samples ranged from 9.1 to 10.2. Adsorbent: filled circles, Japanese oak charcoal: filled square, activated charcoal

Table 2 Gradient (α), y-axis intercept, (β) and determination coefficient (R^2) of Freundlich equation

Solution	Gradient	Y-axis intercept	Determination coefficient	Adsorbent
Solution I	1.953	-2.524	0.9333	Japanese oak charcoal
Solution II	3.415	-1.600	0.9443	Japanese oak charcoal
Solution I	4.715	-6.936	0.963	Activated charcoal
Solution II	7.419	-3.644	0.9697	Activated charcoal

Solution I: 2.50×10^{-4} mol/L CsCl₂ solution; solution II: 2.50×10^{-5} mol/L CsCl₂ solution

experimental observation for more concentrated cesium solutions [10].

It is difficult to make a decision on the type of isotherms [15] shown in Figs. 1 and 2 from observation alone, because these isotherms could contain break points. Thus, the four isotherms were analyzed based on Freundlich equation. The gradients (α) and y-axis intercepts (β), and the coefficients of determination (R^2) calculated by the least-squares method on the assumption that the equation, $\log W = \alpha \log C + \beta$, holds are summarized in Table 2. This equation is generally called Freundlich equation. In the case of Japanese oak charcoal, it is evident that $\log W$ is linearly correlated with $\log C$, because the two coefficients of determination are 0.9333 and 0.9443. The values (1.953 and 3.415) of the gradient for Japanese oak charcoal are much greater than 1, suggesting that the adsorption isotherms are on a concave curve [15]. The gradient value for solution II is larger than that for solution I.

The adsorption isotherms of activated charcoal are likely to be more concave than those of Japanese oak charcoal, although they might have a break point. This is supported by the result that the gradient values were 4.715 (solution I) and 7.419 (solution II) for activated charcoal. In the same way as for Japanese oak charcoal, the gradient value for solution II is also far greater than that for solution I. For activated charcoal, both the coefficients of determination values (0.9630 and 0.9697) are sufficiently close to 1. Accordingly, the gradient value of Freundlich equation demonstrates a tendency to increase with decreasing cesium ion concentration in solution. Nevertheless, we cannot discuss here the numerical values of gradient and y-axis intercept in terms of physics or chemistry, because Freundlich equation is merely empirical [15].

In addition, although Hayashi et al. [16] calculated the adsorption constants of xyloglucan to cellulose in aqueous solution based on Langmuir plots, Langmuir's equation

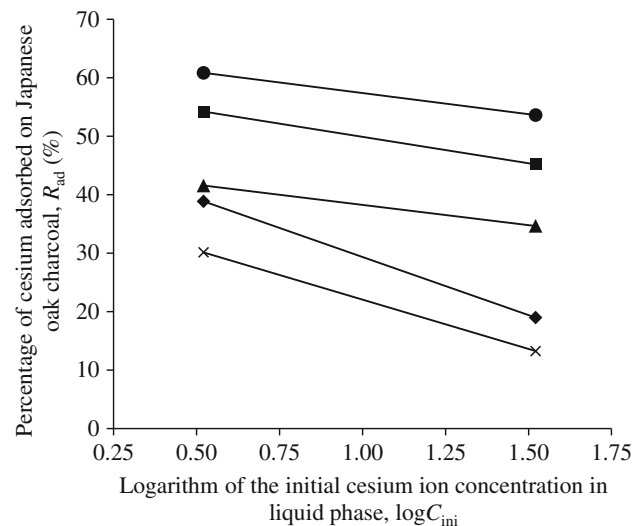


Fig. 3 Plot of percentage of cesium adsorbed on Japanese oak charcoal against logarithm of initial cesium ion concentration in liquid phase. Mass of Japanese oak charcoal powder added to 50 mL sample solution: filled circles, 1.60 g; filled squares, 0.80 g; filled triangle, 0.40 g; filled diagonal, 0.20 g; times, 0.10 g

does not hold for cesium adsorption onto Japanese oak and activated charcoal.

Rate of cesium adsorption onto Japanese oak and activated charcoal

Plots of the percentage of cesium adsorbed on the solid phase (R_{ad}) against logarithm of the initial cesium ion concentration (C_{ini} : mg/L) in the liquid phase are shown in Figs. 3 (Japanese oak charcoal) and 4 (activated charcoal). The percentage of cesium adsorbed on the solid phase in the sample suspension is written as follows:

$$R_{ad} = 100(1 - C/C_{ini}).$$

It can be seen that the percentage of cesium adsorbed on the solid phase increases with a decrease in logarithm of the initial cesium ion concentration in the liquid phase. From Figs. 3 and 4, it is evident that Japanese oak charcoal has considerable cesium-adsorption capacity in weakly basic aqueous solution compared with activated charcoal. This is in agreement with the results in our previous work [10] and suggests that specific surface area or pore volume of charcoal is not the main factor controlling cesium adsorption. For example, the specific surface areas measured by the BET method and total pore volumes of Japanese oak and activated charcoal were 245 m²/g, 0.131 cm³/g and 674 m²/g, 0.477 cm³/g, respectively [10]. By the addition of 1.60 g Japanese oak charcoal into a 50 mL solution II (2.50×10^{-5} mol/L), more than 60 % of cesium ions transfer from the liquid phase to the solid

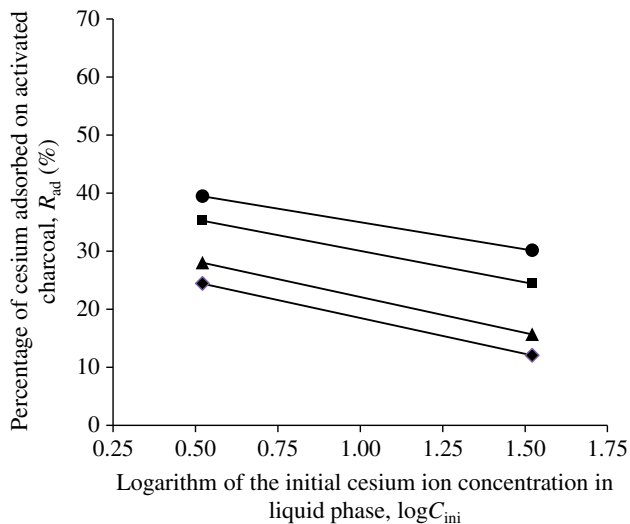


Fig. 4 Plot of percentage of cesium adsorbed on activated charcoal against logarithm of initial cesium ion concentration in liquid phase. Mass of activated charcoal powder added to 50 mL sample solution: filled circles, 1.60 g; filled squares, 0.80 g; filled triangle, 0.40 g; filled diamond, 0.20 g

phase. However, it is most likely that the increase in the percentage of cesium adsorbed on the solid phase is not proportional to the additional amount of adsorbent. It seems that the greater the amount of adsorbent, the lesser is the rate of increase in solid-phase cesium concentration.

In general, zeolite, clay, or Prussian blue is superior to biochar in cesium adsorption. To cite an example, some zeolites and mordenites display nearly 100 % adsorption rates for concentrated solution (Cs^+ : ca. 500 mg/L) when 0.3 g of the adsorbents is added to 30 mL of the cesium solution [17]. By contrast, for many kinds of charcoal made from woody material, the cesium-adsorption rates range from 35 to 85 % under the same addition amount of charcoal even when a dilute solution (Cs^+ : 1.0 mg/L) is used and ordinary Japanese oak charcoal shows medium ability to adsorb cesium [7]. However, biochar has a great advantage of reducing its volume markedly by combustion. Moreover, it has been reported that the cesium-adsorption ability of biochar increases as the production temperature falls [7]. It can, therefore, be expected that Japanese oak charcoal is improved as a practical adsorbent for cesium.

Effects of the presence of group 1 and 2 metal ions on cesium adsorption

Figure 5 depicts Freundlich plots for solution II including lithium chloride (a), sodium chloride (b) [10], and potassium chloride (c). The adsorbent was Japanese oak charcoal and the added amounts of group 1 metal chlorides were 5.1×10^{-3} (■), 5.1×10^{-2} (▲), and 5.1×10^{-1}

mol/L (●). The data (◆) of the reference suspension, suspension to which no group 1 metal chlorides were added, are also shown in Fig. 5. All the sample suspensions were in the pH range of 7.8–10.2.

As is evident from Fig. 5, the amounts of adsorption of cesium decrease sharply with an increase in the added amount of group 1 metal chlorides, indicating that group 1 metal ions seriously obstruct cesium adsorption onto Japanese oak charcoal. It is conceivable that the electrostatic shield grows with ionic strength in aqueous solution and the growth of the shield reduces the absolute value of the surface potential of the fine particles [18–21]. The shield by cations other than cesium ions can adequately account for the experimental data illustrated in Fig. 5. Furthermore, it is likely that the obstruction effect increases in the order of lithium < sodium < potassium. This may suggest that the ionic radius has some influence on the obstruction.

Freundlich isotherms for solution II to which group 2 metal chlorides [magnesium chloride (a), calcium chloride (b), strontium chloride (c)] were added are shown in Fig. 6. As with the group 1 metal chloride solutions, the adsorbent was Japanese oak charcoal powder and additional amounts of group 2 metal chlorides were 5.1×10^{-3} (■), 5.1×10^{-2} (▲), and 5.1×10^{-1} mol/L (●). The suspensions to which magnesium chloride, calcium chloride, and strontium chloride were added were in the pH ranges of 8.1–9.6, 7.1–9.1, and 6.1–9.2, respectively. For solutions to which strontium chloride (5.1×10^{-1} mol/L) was added, two sample suspensions showed pH values lower than 6.70, which is the point of zero charge by the pH indication of Japanese oak charcoal in aqueous solution [10].

Although the three group 2 metal chlorides probably also produce some blocking effects on cesium ion adsorption, we obtained a surprising result in that the decrease of adsorption by the addition of group 2 metal chlorides was much smaller than that by the addition of group 1 metal chlorides. In general, it is believed that the electrostatic shield increases with ionic strength as described above. Thus, group 2 metal ions should show a greater shield than group 1 metal ions when the two concentrations are equivalent, since group 1 and 2 metal ions are monovalent and divalent cations, respectively, and it is commonly recognized that group 1 and 2 metal elements exist mostly as free ions unless aqueous solutions dissolving them are strongly basic [22]. However, our experimental data are completely opposite to the prediction derived from the published data [23–26]. The fact that considerable cesium adsorption onto Japanese oak charcoal from solutions at lower pH than the point of zero charge of Japanese oak charcoal took place is interesting, suggesting that cesium adsorption was caused by not only surface potential, namely, electrostatic attraction, but also other factors, for instance, chemical interactions of residual functional

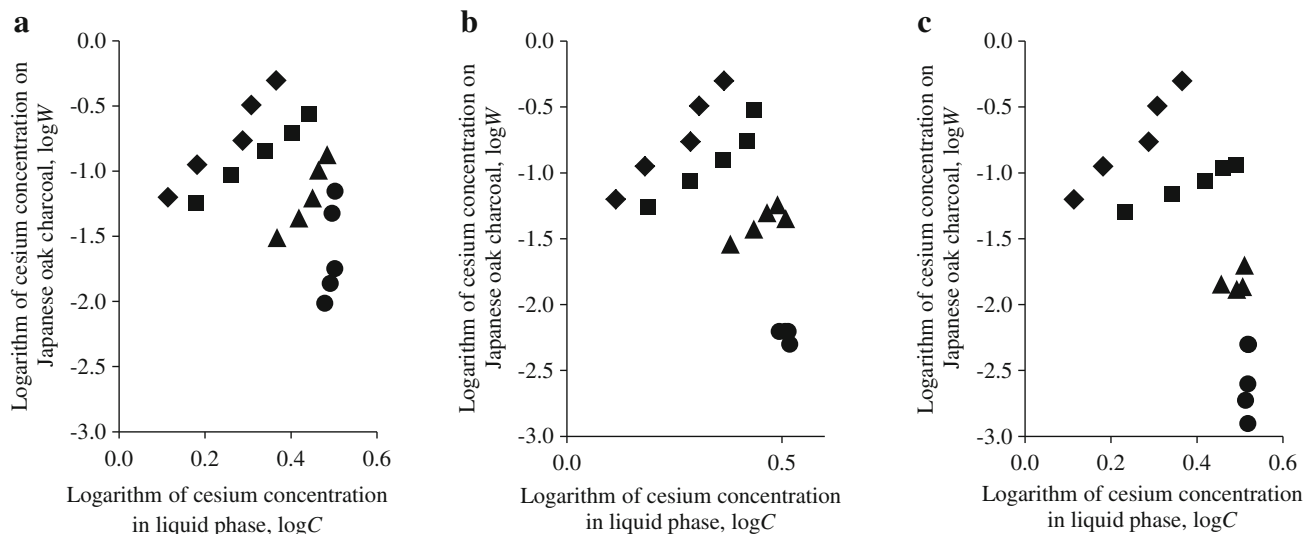


Fig. 5 Freundlich plots for 2.50×10^{-5} mol/L CsCl_2 solution including LiCl (a), NaCl (b), and KCl (c). The pH ranges of the suspension samples shown in (a), (b) and (c) were 8.9–10.1, 7.8–10.2 and 8.7–10.1, respectively. Molar concentrations of metal chloride

added to the cesium chloride solution: *filled diagonal*, no addition; *filled square*, 5.1×10^{-3} mol/L; *filled triangle*, 5.1×10^{-2} mol/L; *filled circles*, 5.1×10^{-1} mol/L

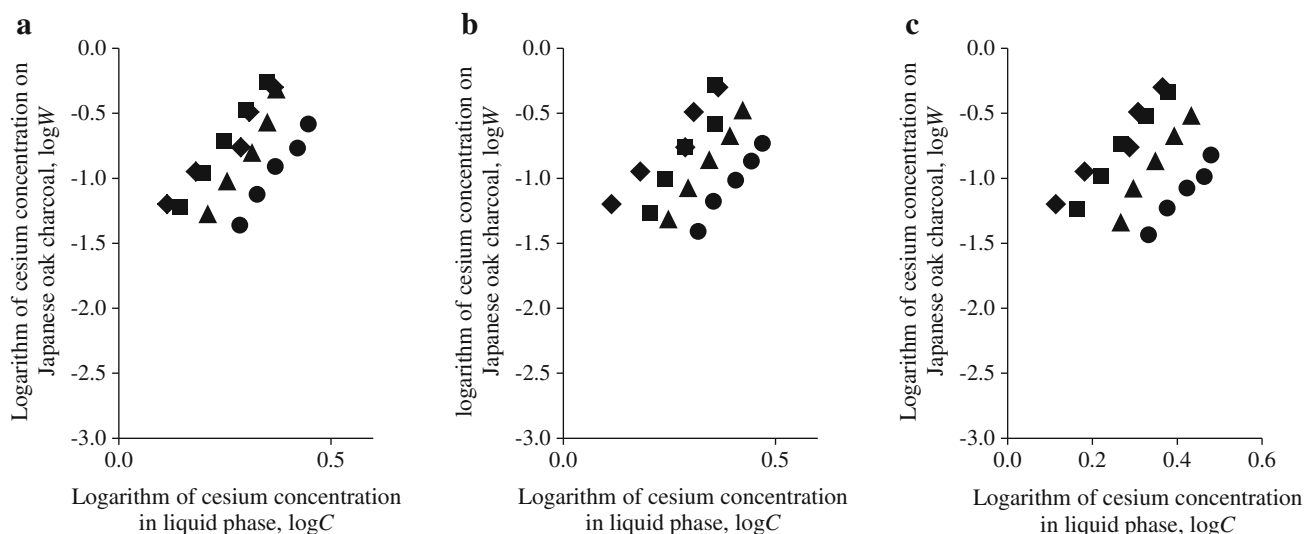


Fig. 6 Freundlich plots for 2.50×10^{-5} mol/L CsCl_2 solution including MgCl_2 (a), CaCl_2 (b), and SrCl_2 (c). The pH ranges of the suspension samples shown in (a), (b) and (c) were 8.1–9.8, 7.1–9.1 and 6.1–9.1, respectively. Molar concentrations of metal

chloride added to the cesium chloride solution: *filled diagonal*, no addition; *filled square*, 5.1×10^{-3} mol/L; *filled triangle*, 5.1×10^{-2} mol/L; *filled circle*, 5.1×10^{-1} mol/L

groups on the Japanese oak charcoal surface with the metal ions.

Furthermore, our results reveal that the determination of metal ions eluted from charcoal is required to evaluate precisely the cesium-adsorption capacity of charcoal from aqueous solution, because ordinary biochar would contain certain amounts of group 1 and 2 metals. If the isotherms shown in Figs. 1 and 2 have break points, the discontinuities may be due to the metal ions eluted from charcoal.

The next issue is to investigate the effects of eluted metal ions on cesium adsorption.

Conclusion

The present work confirmed several important and remarkable findings about cesium adsorption onto charcoal from aqueous solution.

The adsorption isotherms of cesium onto Japanese oak and activated charcoal are concave curves. In other words, the gradients of Freundlich equation are greater than 1. The gradient for Japanese oak charcoal is lower than that for activated charcoal when the initial concentrations of cesium ions in solution are equal. Moreover, the smaller the initial concentration of cesium ions in solution, the larger is the gradient of Freundlich equation.

The rate of cesium adsorbed onto charcoal increased with a decrease in the initial concentrations of cesium ions in solution and an increase in the amount of charcoal added. The maximum value of the percentage of cesium adsorbed in the solid phase of Japanese oak charcoal in this study was 60.8 % when 1.60 g of the adsorbent was added to 50 mL solution II, while the maximum value of activated charcoal under the same conditions was less than 40 %. Thus, it was concluded that the cesium-adsorption ability of Japanese oak charcoal was greater than that of activated charcoal.

Group 1 and 2 metal ions evidently obstruct cesium adsorption onto charcoal from aqueous solution, and it is interesting that group 1 metal ions have much stronger blocking effects on the adsorption of cesium than group 2 metal ions. A possible explanation for this are differences in the adsorption sites on the charcoal surface between group 1 and 2 metal ions. However, further experimental evidence is required to elucidate the mechanism of the blocking effects by group 1 and 2 metal ions. Furthermore, to provide accurate assessment of adsorption ability, it is necessary to determine the metals eluted from charcoal. The next task will be adsorption tests of cesium using demineralized charcoal as an adsorbent.

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