

## Cesium adsorption from aqueous solutions onto Japanese oak charcoal II: effects of metal ions eluted from the charcoal

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**Abstract** The major metal elements contained in Japanese oak (*Quercus serrata* Thunb. and/or *Quercus crispula* Blume) charcoal (OC) and activated charcoal (AC) were identified by X-ray fluorescence spectroscopy. The eluted amounts of sodium, potassium, magnesium, and calcium ions from OC and AC were measured using ion chromatography. We attempted to demineralize OC and AC by water-washing, and then determined the eluted amounts of the four metal ions from the washed OC and AC. The effects of the water-washing on the adsorption of cesium ions onto OC and AC were investigated with respect to  $2.50 \times 10^{-4}$  and  $2.50 \times 10^{-5}$  mol/L aqueous cesium chloride solutions. Remarkably, the washing improved the adsorption ability of OC for cesium ions in solutions whose initial cesium chloride concentration was  $2.50 \times 10^{-5}$  mol/L, although such effects were not so noticeable in solutions whose initial cesium chloride concentration was  $2.50 \times 10^{-4}$  mol/L.

**Keywords** Cesium · Adsorption · Japanese oak charcoal · Demineralization · Aqueous solution

### Introduction

The colossal tsunami in March 2011 cut off the electricity to the Fukushima Daiichi nuclear power plants.

The subsequent hydrogen detonation due to this power outage caused large-scale scattering of radioisotopes, preventing people from returning to their homes in the surrounding area. Moreover, the damaged power plants continue to generate large amounts of high-level radiation-contaminated water every day. These are very pressing issues, so appropriate measures for nuclear decontamination are urgently needed. Two radioactive nuclides,  $^{134}\text{Cs}$  and  $^{137}\text{Cs}$ , are the main targets of decontamination. To decontaminate radioactive cesium nuclides in soil or fresh water, it is of great importance to fix them. Therefore, we must identify an ideal adsorptive for such decontamination.

Many reports of cesium adsorption using porous inorganic materials such as zeolites have already been published, while in recent years the number of reports on cesium removal through biochar [1–3] has been gradually increasing. Generally, zeolite or clay exhibits much higher cesium-adsorption performance than biochar. An advantage of biochar is, however, the ability to reduce its volume markedly by combustion, which facilitates storage of the radioactive cesium isotopes. Thus, it is worthwhile examining whether charcoal made from woody materials has acceptable practical utility as an adsorptive for cesium [4].

An effective adsorptive must not only be highly efficient in terms of its adsorption ability, but it must also be producible at a massive scale at low cost because enormous amounts of it are needed for decontamination over a vast region. Woody biomass resources that are used as raw materials for making charcoal are ubiquitous and sustainable. The physical and chemical properties of commercially available charcoals are greatly dependent upon their production processes and vary widely. Therefore, it can be expected that some charcoals carbonized by appropriate

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methods show high performance for cesium adsorption. We first focused our attention on Japanese oak (*Quercus serrata* Thunb. and/or *Quercus crispula* Blume) charcoal (OC), which is the most popular charcoal manufactured in Japan. From investigations into its cesium-adsorption behavior from aqueous solutions, we found that OC has a certain ability to adsorb cesium ions and surface physical properties such as specific surface area are not controlling factors of cesium adsorption onto charcoal. Therefore, we predicted that the primary interaction for cesium adsorption onto charcoal is electrostatic attraction [5]. Furthermore, it was reported that group 1 and 2 metal ions, especially group 1 metal ions, showed blocking effects on adsorption for cesium ions from aqueous solutions [6]. Next, we investigated the effects of metal ions eluted from adsorbent charcoal on cesium adsorption because ordinary charcoal contains considerable amounts of group 1 and 2 metal elements [7, 8].

Our aim here is to understand the blocking effects of the metal ions eluted from OC, and to examine the changes in the adsorption ability of OC after demineralization. In this paper, the practical utility of OC as an adsorptive is evaluated in terms of its inherent adsorption ability.

## Experimental

### Materials

OC manufactured in Iwate Prefecture and activated charcoal (AC) 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 specific surface area (BET method), total volume of fine pore, and carbonization temperature of OC were 245 m<sup>2</sup>/g, 0.131 cm<sup>3</sup>/g, and 600–800 °C, respectively [5]. The OC used was not contaminated by artificial radioactive nuclides at all. The cesium chloride (CsCl) and other chemicals were commercially available and of special-grade. All cesium employed in this study was exclusively the stable <sup>133</sup>Cs isotope.

### Preparation of aqueous cesium chloride solutions

Two aqueous cesium chloride solutions (I and II) were prepared using cesium chloride dried at 100 °C. For solutions I and II, the initial cesium chloride concentrations were  $2.50 \times 10^{-4}$  mol/L (Cs<sup>+</sup> concentration: 33.2 mg/L) and  $2.50 \times 10^{-5}$  mol/L (Cs<sup>+</sup> concentration: 3.32 mg/L), respectively. Water used for preparing the aqueous solutions was deionized through a column filled with ion-exchange resins.

### Identification of elements in OC and AC by X-ray fluorescence spectroscopy

Elements in OC and AC whose atomic numbers exceeded eleven were detected by X-ray fluorescence measurements using a compact spectrometer (MESA-500, HORIBA, Ltd.). X-ray irradiation was carried out at ambient temperature. A sample chamber set in a cylindrical Teflon cell with a diameter of 20 mm containing about 0.5 g of powder OC or AC was evacuated. The measurement time was 2000s. X-ray tube voltages were 15 and 50 kV. Relative mass percentages of elements to total elements detected by X-ray measurement were calculated by a standardless fundamental parameter method [9, 10] from the characteristic X-ray intensities.

### Water-washing of OC and AC

OC or AC powder (ca. 10 g) was suspended in 500 mL of deionized water. The suspensions were filtered under reduced pressure after 24 h of stirring and the filtrate was discarded. This washing process was repeated four times. Acidic extractant such as hydrochloric acid was not used to avoid chemical changes of OC and AC surfaces as much as possible. OC and AC after the water-washing are referred to as W-OC and W-AC, respectively. W-OC and W-AC were used as adsorbents after drying at 105 °C for over a day.

### Determination of sodium, potassium, magnesium, and calcium ions eluted from charcoals

Each adsorbent powder (1.60 g) was suspended in 50 mL of deionized water. A sealed Erlenmeyer flask including the suspension was circularly shaken at  $23 \pm 2$  °C for 24 h. The powder from the suspensions was filtered out through a nylon-membrane microfilter (Millex-HN filters, SLHN033NS, Millipore Ireland, Ltd.). The filtrate (10 mL) was diluted ten times with deionized water. The concentrations of sodium, potassium, magnesium, and calcium ions in the diluted solution were determined using an ion chromatographic system (Dionex CS-3000, Nippon Dionex K. K.) according to the Japanese Industrial Standard for testing methods for industrial wastewater (JIS K 0102, 2008) [11]. An electrical conductivity meter was used as a detector.

### Determination of solid-phase cesium concentration

An aqueous cesium chloride solution (50 mL) containing powder adsorbent (0.10, 0.20, 0.40, 0.80, 1.60 g) in a sealed Erlenmeyer flask was circularly shaken and filtered out through the microfilter in the same way as mentioned

above. The pH of the filtrate was measured using a pH meter. The cesium ion concentration in the filtrate was determined using an ICP mass spectrometer (Agilent 7700x, Agilent Technologies, Ltd.) by an internal standard method (standard isotope:  $^{115}\text{In}$ ). The ICP mass measurements were performed three times for each 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 in the liquid phase at equilibrium. The solid-phase cesium concentration ( $W$ ) was calculated by dividing the decrease in mass ( $m$ ) of cesium ions in the liquid phase by the mass ( $M$ ) of powder adsorbent added, according to Eq. (1):

$$W = \frac{m}{M} \quad (1)$$

where the units of  $m$  and  $M$  are milligrams (mg) and grams (g), respectively.

## Results and discussion

The physical and chemical properties of OC and AC, such as the specific surface area, carbonization temperature [5] and content of major component elements [6], were reported previously.

### Metal elements contained in OC and AC

Relative mass percentages of the metal elements (>0.1 %) and silicon determined by X-ray fluorescence analysis are summarized in Table 1. We previously reported that the ash contents of OC and AC were 1.32 and 9.33 mass %, respectively [6]. Although several nonmetallic elements were detected by X-ray fluorescence spectroscopy, we will limit our discussion to the metal elements.

OC contained calcium and potassium in appreciable amounts compared with the other metals. The contents by relative mass percentage of calcium and potassium were about 64 and 30 %, respectively. Manganese was the tertiary metal element, but its content was only 2.21 %. Additionally, characteristic X-rays of iron and aluminum were clearly observed, but their contents were under 0.5 %.

For AC, calcium was also the primary metal element, with a content of ca. 37 %. Iron, whose content exceeded 25 %, was the secondary metal element, but the content of potassium was ca. 1.6 %, which is an extremely low value compared to that of OC. Other metals having contents greater than 1 % were magnesium (4.58 %), aluminum (1.81 %), and manganese (1.25 %).

Accordingly, the concentrations of these metals in the liquid phase of the sample suspensions should increase significantly by elution from OC or AC. However, iron(III) elution would be negligible because iron(III) hardly dissolves in aqueous solution under nonacidic conditions due to the very small solubility product of  $\text{Fe}(\text{OH})_3$  [12, 13], and all the sample suspensions including OC or AC were neutral or weakly basic. Moreover, it can be assumed that iron(III) scarcely exists as a species having a positive charge in nonacidic aqueous solution even if iron(III) elution occurs [14], unlike for group 1 and 2 metals. From the viewpoint of chemical behavior in aqueous solution, aluminum is similar to iron(III). In contrast, iron(II) is more soluble in weakly basic aqueous solutions than iron(III) because the solubility product of  $\text{Fe}(\text{OH})_2$  is much larger than that of  $\text{Fe}(\text{OH})_3$  [12, 13]. Thus, the eluted amount of iron(II) from AC may be large enough to affect its adsorption ability for cesium. Nevertheless, with respect to OC, it is likely that the effects of iron(II) elution on cesium adsorption are not prominent due to the small content of iron.

It is difficult to evaluate manganese species in the suspension samples in detail because manganese can change its oxidation state from +2 to +7 and form various oxo or hydroxo complexes in aqueous solution [15]. If manganese exists as these complexes, no blocking effects would be observed because they are anionic or electrically neutral species. However, from the viewpoint of the solubility product of manganese hydroxide [ $\text{Mn}(\text{OH})_2$ ] [12, 13], it is suggested that the hydroxide is not an insoluble compound when the aqueous solution is weakly basic. Thus, the concentration of manganese ion ( $\text{Mn}^{2+}$ ) in the liquid phase may significantly increase by the addition of OC or AC, although the contents of manganese are not so large. Nonetheless, it is predicted that manganese ion shows no significant blocking effect, because it is comparable to magnesium ion ( $\text{Mg}^{2+}$ ) in terms of ionic valence and radius [6, 16].

**Table 1** Relative mass percentages of metal elements and silicon to total elements detected by X-ray fluorescence analysis

Charcoal	Element (Mass %)									
	Mg	Al	Si	K	Ca	Ti	Mn	Fe	Sr	
OC	ND	0.40	0.88	30.0	64.2	ND	2.21	0.45	0.19	
AC	4.58	1.81	19.6	1.62	36.6	1.10	1.25	25.4	0.24	

ND means not detected, OC Japanese oak charcoal, AC activated charcoal

Determination of sodium, potassium, magnesium, and calcium ions eluted from charcoals

As we reported previously [6], group 1 and 2 metal cations obstruct the adsorption of cesium ions onto charcoal from aqueous solution. Thus, the concentrations of potassium, magnesium, and calcium, which are detected as major elements among the metals contained in OC or AC, in the liquid phase of the sample suspensions were determined. Although sodium was undetectable by X-ray fluorescence analysis, its concentrations were also measured because ordinary biochar would contain a certain amount of sodium [7, 8].

As can be seen in Table 2, the four metal ions in deionized water used in this study were undetectable by ion chromatography, but this does not necessarily mean that the deionized water is completely free from the four metal ions. The filtrate obtained from the sample suspension was diluted ten times with deionized water as described above. Thus, the concentration of a metal eluted from charcoal should be in excellent agreement with the 10-fold value of the concentration of the metal measured by ion chromatography if the original concentration of the metal contained in deionized water is small enough compared to the concentration of the metal eluted from charcoal. However, if not, the concentration of the metal eluted from charcoal should be significantly smaller than the 10-fold value of the measured concentration of the metal. Consequently, for the four metals, two possible concentrations, the maximum and minimum, were calculated from the measured concentration, assuming that (a) deionized water is free from the four metals or (b) the concentrations of the four metals in deionized water are equivalent to their detection limits. Table 2 contains a summary of the maximum and minimum concentrations of the four metal ions eluted from OC, AC, W-OC, and W-AC. The pH in the filtrate solutions is shown as well.

It is interesting to note that there was little correlation between the eluted amounts of metal ions and the contents of metals in the charcoals. The most abundant metal in the filtrate was not calcium but potassium, although calcium was the primary element in both OC and AC. In particular, for AC, the eluted amount of potassium was higher than those of magnesium and calcium despite its smaller content in the solid phase. Additionally, it was confirmed that considerable amounts of sodium eluted from OC and AC. The eluted amount of potassium from OC was more than twice that from AC, while the total eluted amounts of the other three metals from AC were much greater than those from OC.

As is evident from Table 2, the numbers of sodium, potassium and magnesium ions eluted from OC or AC were drastically decreased by the water-washing as expected. However, the results shown in Table 2 reveal that the eluted

**Table 2** Ranges of concentrations of sodium, potassium, magnesium, and calcium ions eluted from OC, AC, W-OC, and W-AC

Adsorbent	Molar concentration (mol/L)				pH
	Na <sup>+</sup>	K <sup>+</sup>	Mg <sup>2+</sup>	Ca <sup>2+</sup>	
No adsorbent (blank)	<1.3 × 10 <sup>-6</sup> <sup>a</sup>	<5.1 × 10 <sup>-7</sup> <sup>b</sup>	<8.2 × 10 <sup>-7</sup> <sup>c</sup>	<1.0 × 10 <sup>-6</sup> <sup>d</sup>	
	Maximum	Maximum	Maximum	Maximum	Minimum
OC	4.8 × 10 <sup>-5</sup>	2.8 × 10 <sup>-3</sup>	3.3 × 10 <sup>-5</sup>	3.0 × 10 <sup>-5</sup>	2.0 × 10 <sup>-5</sup>
AC	7.4 × 10 <sup>-4</sup>	1.2 × 10 <sup>-3</sup>	6.2 × 10 <sup>-4</sup>	1.8 × 10 <sup>-4</sup>	1.7 × 10 <sup>-4</sup>
W-OC	1.3 × 10 <sup>-5</sup>	1.0 × 10 <sup>-4</sup>	8.2 × 10 <sup>-6</sup>	1.7 × 10 <sup>-4</sup>	1.6 × 10 <sup>-4</sup>
W-AC	7.0 × 10 <sup>-5</sup>	2.8 × 10 <sup>-5</sup>	2.8 × 10 <sup>-4</sup>	3.5 × 10 <sup>-4</sup>	3.4 × 10 <sup>-4</sup>

OC Japanese oak charcoal, AC activated charcoal, W-OC washed Japanese oak charcoal, W-AC washed activated charcoal

<sup>a</sup> Detection limit of sodium ion concentration

<sup>b</sup> Detection limit of potassium ion concentration

<sup>c</sup> Detection limit of magnesium ion concentration

<sup>d</sup> Detection limit of calcium ion concentration

amount of calcium from OC and AC increased distinctly after the water-washing, contrary to our expectations. The total eluted amount of the four metal ions from W-OC or W-AC became much smaller than that before the washing. This is in accordance with the fact that the pH in the filtrate solutions was decreased by the washing.

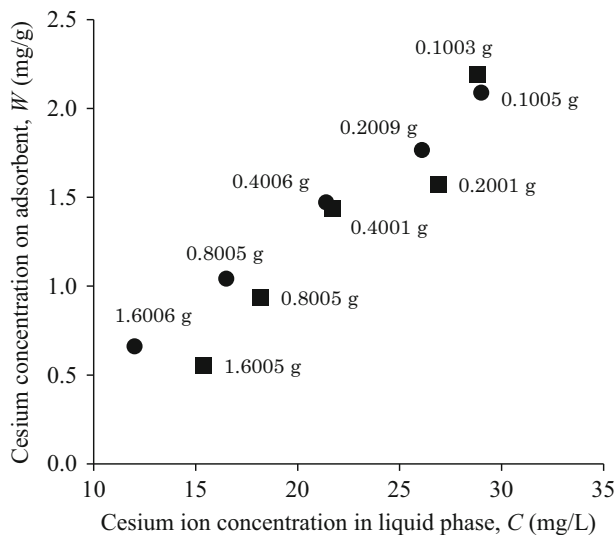
Adsorption isotherms of cesium ions onto W-OC and W-AC

From the data shown in both Table 2 and previous work [6], it is assumed that the group 1 and 2 metal ions eluted from OC or AC, especially potassium ions, significantly prevented cesium ions from being adsorbed onto charcoals. Positive improvements in adsorption ability are expected to be achieved by the washing even if the metals cannot be completely removed from OC to AC. This is because the blocking effects of magnesium and calcium were relatively small and the eluted amounts of sodium and potassium showing the strong blocking effects were decreased drastically by the washing. To confirm this, the adsorption behavior of cesium ions was investigated using W-OC and W-AC as adsorbents.

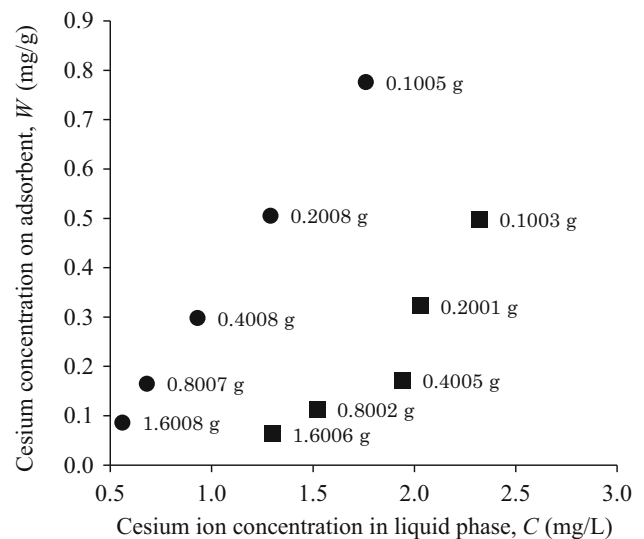
Figure 1 depicts plots of cesium ion concentration ( $C$ : mg/L) in aqueous solution against cesium concentration ( $W$ : mg/g) on W-OC (solid circle) and OC (solid square), namely, adsorption isotherms of cesium adsorption onto W-OC and OC from solution I. The pH in the liquid phase of W-OC suspensions ranged from 7.6 to 9.0. The plots of OC have been published previously [6], but are shown in Fig. 1 for comparison to W-OC. There are no remarkable differences

in curve profiles between the plots of W-OC and OC. It seems, however, that the liquid-phase cesium concentration of the W-OC suspension was clearly lower than that of the OC suspension upon addition of 1.60 g of adsorbent. This suggests that the blocking effects of the metal ions eluted from adsorbent charcoals appeared distinctly when the amount of added adsorbent exceeded a certain value. Presumably, potassium ions produced the strongest blocking effects in the metal ions eluted from adsorbent.

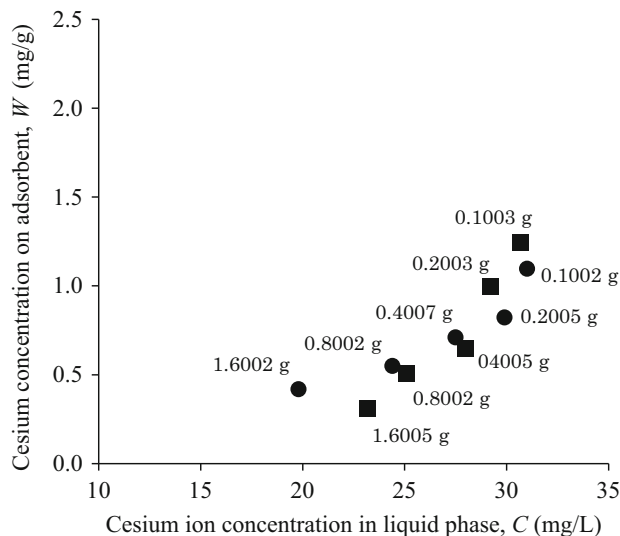
The adsorption isotherms for solution II are given in Fig. 2. The adsorbents are also W-OC (solid circle) and OC (solid square), and the pH range in the filtrate of W-OC suspensions was 7.7–9.0. Unlike in Fig. 1, the results shown in Fig. 2 make it clear that the liquid-phase cesium concentration of the W-OC suspension was much lower than that of the OC suspension over the whole range. In the case of solution II, the rate of cesium in the solid phase was about 60 % when the amount of added OC was 1.60 g, while the rate increased up to 83 % by addition of the same amount of W-OC. The adsorption isotherms of cesium adsorption onto W-AC (solid circle) and AC (solid square) from solutions I and II are displayed in Figs. 3 and 4, respectively. The data of AC have already been reported [6] and are shown as a reference. The pH ranges in the liquid phase of W-AC suspensions for solutions I and II were 7.8–8.4 and 7.7–8.5, respectively. From Fig. 3, it can be seen that the effect of the water-washing does not appear clearly on the adsorption isotherms, except after addition of 1.60 g of adsorbent. Comparing Fig. 3 with Fig. 1, W-AC was far inferior to W-OC in terms of its ability to adsorb cesium ions. As seen in Fig. 4, for solution II, the water-



**Fig. 1** Isotherms of cesium adsorption onto OC (solid square) and W-OC (solid circle) from solution I. The numerical values next to the plotted symbols (solid square and solid circle) are the amounts of added adsorbent



**Fig. 2** Isotherms of cesium adsorption onto OC (solid square) and W-OC (solid circle) from solution II. The numerical values next to the plotted symbols (solid square and solid circle) are the amounts of added adsorbent



**Fig. 3** Isotherms of cesium adsorption onto AC (*solid square*) and W-AC (*solid circle*) from solution I. The numerical values next to the plotted symbols (*solid square* and *solid circle*) are the amounts of added adsorbent

washing effect clearly appeared in AC as well. It is, however, apparent that the adsorption ability of W-AC was much less than that of W-OC, by comparison of Figs. 2 and 4.

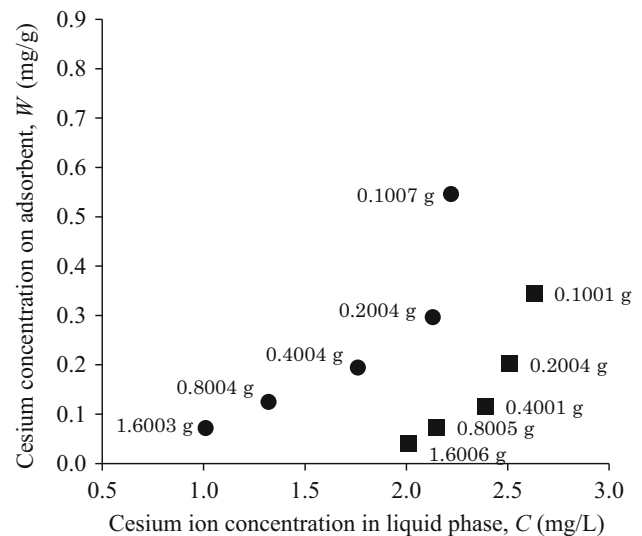
Accordingly, it was elucidated that demineralization is an essential process for precise evaluation of the adsorption ability of charcoal for cesium ions. Moreover, it is expected that the lower the initial concentration of cesium in aqueous solution, the greater the effects of the water-washing, namely, the greater the rate of cesium in the solid phase.

From a practical viewpoint, it is considered that OC cannot sufficiently exhibit its adsorption ability for cesium ions in soil due to the metal ions it contains, especially potassium ions. Therefore, the data in this study lead us to the conclusion that demineralization of OC as a pretreatment is indispensable for making an effective adsorbent for the decontamination of cesium, although decontamination of cesium in soil would be much more difficult than that of cesium in fresh water because cesium ions would adhere strongly to soil constituents [17, 18]. To make the blocking effects of metal ions, especially sodium and potassium ions, as small as possible is a crucial issue for decontamination of radioactive cesium.

Further study is required to confirm whether the demineralization treatment by water-washing in this study is sufficient to estimate the intrinsic cesium-adsorption ability of OC.

## Conclusion

This study shows that sodium and potassium ions that exhibit strong blocking effects on the adsorption of cesium



**Fig. 4** Isotherms of cesium adsorption onto AC (*solid square*) and W-AC (*solid circle*) from solution II. The numerical values next to the plotted symbols (*solid square* and *solid circle*) are the amounts of added adsorbent

ions are washed out from OC and AC more easily than magnesium and calcium ions. A noteworthy outcome of the present work is that demineralization by water-washing enhances the cesium-adsorption ability of OC and AC for solution II ( $\text{CsCl}$ :  $2.50 \times 10^{-5}$  mol/L), although such enhancements are not observed for solution I ( $\text{CsCl}$ :  $2.50 \times 10^{-4}$  mol/L). This allows us to postulate that W-OC has considerably high adsorption ability when the cesium concentration is extremely low, as in actual contaminated soil, if the blocking effects of other metal ions are negligibly small.

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