Separation of radioactive cesium and iodine from contaminated soil by adsorption and decantation

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Approximately 19% of $^{131}$I was released in a solution mixing contaminated soil with distilled water and 10 wt% CaCl$_2$ solution, and the remaining $^{131}$I tightly bound to the soil. Approximately 12 to 15% of the radioactive cesium was found in the supernatant after mixing with saturated KCl aqueous solution, and the repeated treatment for 1 day indicated the additional release of 11 to 15% in the supernatant. Approximately 15 to 20% of radioactive cesium was found in the supernatant by mixing with saturated KCl solution and KCl powder, followed by heating. The repeated treatment with 40% KI solution led to a maximum release rate of 66% of radioactive cesium from the soil.

Key words: separation, radioactive cesium, radioactive iodine, soil, adsorption

1. Introduction

Radionuclides released in the aftermath of the Fukushima Daiichi nuclear plant accident spread over wide areas in many prefectures in the Tohoku and Kanto regions [1]. The released $^{134}$Cs and $^{137}$Cs fell to the soil and resulted in soil contamination [2]. The contaminated soil emitted $\gamma$-rays at 0.605 MeV for $^{134}$Cs and 0.660 MeV for $^{137}$Cs, and the air radiation dose increased to more than 1 $\mu$Sv/h in these areas [3, 4]. Therefore, decreasing the dose was desired and decontamination was required. Decontamination denotes the separation of $^{134}$Cs and $^{137}$Cs from the soil and their separate collection. Usually, the cesium ion has a positive charge and it is easy to be adsorbed into the clay interlayer [5]. Therefore, it is not easy to separate it from the soil.

In this study, the decantation of soil dispersed in an aqueous solution was carried out to separate the radioactive cesium contained in clay from the soil, and the radioactivity was determined. The removal of radioactive cesium from the soil was tested using potassium iodide and the condition of the removal is discussed.

2. Materials and Methods

2.1. Decantation

Approximately 200 g of radioactive-cesium-contaminated soil obtained on April 8, 2011 at about 25 km away from the Fukushima Daiichi nuclear plant, which was attached to spinach, was concentrated by decantation. First, the soil was washed with 100 mL each of distilled water, 10 wt% CaCl$_2$ aqueous solution, and hot water. Then, the soil was added to 100 mL of sodium bicarbonate solution (13 wt%) and mixed with shaking to make an alkali solution, and then it was allowed to stand for about 4 h to separate the soil mineral from the colloidal clay. These procedures were repeated five times and a colloidal solution was obtained. The colloidal solution was allowed to stand for one day to separate the supernatant from the precipitate. The $\gamma$-ray spectra were determined using a Ge semiconductor detector. Then, the precipitate was dried in a vessel with a silica gel for about one week to a solid gel. The solid gel was divided into three parts; one-third was divided into an upper part and a lower part. The vessel was V-1 type (5.5 cm diameter, 2 cm height).
2.2. Removal of radioactive cesium ions from contaminated soil

Trinuclear iron acetate, which expands the interlayer of the clay, potassium chloride, whose chemical properties are similar to those of cesium ion and is exchangeable with it, and CTAB, which is a surfactant and is easy to incorporate in the interlayer of the clay to expand it, were used to remove the radioactive cesium ions. After about 100 mL of the trinuclear iron acetate aqueous solution (4 wt%) was mixed with the five-times-washed precipitate and was allowed to stand for 30 min, it was centrifuged at 3500 rpm for 20 min and the $\gamma$-ray spectrum of the supernatant was determined. The precipitate was mixed with 100 mL of a saturated KCl aqueous solution and allowed to stand for 1 day. Then, it was centrifuged at 3500 rpm for 20 min and the $\gamma$-ray spectrum of the supernatant was determined. Again, the precipitate was mixed with the supernatant that included the saturated KCl solution and was allowed to stand for 1 day. After it was centrifuged at 3500 rpm for 20 min, the $\gamma$-ray spectrum of the obtained supernatant was determined. The precipitate was mixed with 100 mL of CTAB solution (4 wt%) and was allowed to stand for 1 day. After it was centrifuged at 3500 rpm for 20 min, the $\gamma$-ray spectrum of the obtained supernatant was determined. The precipitate was mixed with 100 mL of the saturated KCl solution and was allowed to stand for 3 days. After it was centrifuged at 3500 rpm for 20 min, the $\gamma$-ray spectrum of the obtained supernatant was measured. Then, the supernatant was again mixed with the supernatant, and 2.1 g of KCl was added. After they were heated on a hot plate, stirring with a magnetic stirrer was carried out for 1 h. After it was centrifuged at 3500 rpm for 20 min, the $\gamma$-ray spectra of the obtained supernatant and the stirrer piece attached to a magnetic iron in the soil were determined.

After the two supernatants and the precipitate after treatment with 2.3 wt% clay aqueous suspension were mixed again and centrifuged at 3500 rpm for 100 min, the $\gamma$-ray spectra of the obtained supernatant and precipitate were determined. The trinuclear-iron-acetate-treated precipitated soil was mixed with distilled water, and after it was centrifuged three times at 1000 rpm for 10 min, the supernatants were removed. The obtained precipitate was composed of different colored layers. The upper layers were light components and the lower layers were heavy components, and their $\gamma$-ray spectra were determined.

2.3. Removal of radioactive cesium ions from soil using potassium chloride method of Tokyo University

About 10 g of the contaminated soil was mixed with the following aqueous solutions and then allowed to stand for a given time. After the decantation was carried out and the supernatant was obtained, the $\gamma$-ray spectrum of the supernatant was determined.

(Method A)

1) Mixing with 20 mL of iodine aqueous solution (0.01 M) and standing for 18 h
2) Mixing with 5 mL of iodine aqueous solution (0.01 M) and 15 mL of KI aqueous solution (40%) and standing for 21 h
3) Mixing with 20 mL of KI aqueous solution (40%) and standing for 152 h
4) Mixing with 20 mL of distilled water and standing for 10 h
5) Mixing with 12 mL of KI aqueous solution (40%) and standing for 50 h
6) Determination of $\gamma$-ray spectrum of the remaining soil

(Method B)

1) Mixing with 20 mL of KI aqueous solution (40%) and standing for 42 h
2) Mixing with 20 mL of KI aqueous solution (40%) and standing for 152 h
3) Mixing with 20 mL of distilled water and standing for 10 h
4) Mixing with 10 mL of KI aqueous solution (40%) and standing for 50 h
5) Determination of $\gamma$-ray spectrum of the remaining soil

2.4. Health hazards associated with treatment using various solutions

Sodium bicarbonate is used as an agricultural chemical that is recognized to be safe to reduce the acidity of soil and as a detergent in the home. Potassium chloride (KCl) is a potassium fertilizer used for agriculture and in the kitchen and gardens. Trinuclear acetate is made from iron and acetate. Calcium dichloride dihydrate (CaCl$_2$・2H$_2$O) is a desiccant, it is used as an antifreeze to melt snow on roads, and its dissolution is exothermic. CTAB is a soaplike surfactant that kills germs. These reagents can be used in aqueous form without any adverse effects on health. However, when potassium iodide (KI) and iodine solution (I$_3^-$) are used, repeated contact with them can cause an allergic reaction to develop in susceptible
people [6]. These chemicals should be used in a well-ventilated environment. Finally, soil treated with these solutions should be washed with a large amount of water to remove these reagents.

3. Results and Discussion

3.1. Decantation

Fig. 1 shows a \( \gamma \)-ray spectrum of the contaminated soil measured on May 15, 2011. Four main peaks and two subpeaks were detected and were assigned as follows: \(^{129}\)Te at 0.460 MeV (7.7%); \(^{131}\)I at 0.365 MeV (81.7%); \(^{134}\)Cs at 0.569 MeV (15.4%); 0.605 MeV (97.6%), and 0.796 MeV (85.5%); \(^{137}\)Cs at 0.662 MeV (85.1% \(^{137}\)mBa) [7]. Percentages indicate emission rates of \( \gamma \)-rays (%).

After the soil was stirred with water and 10 wt% CaCl\(_2\) aqueous solution, about 19% of \(^{131}\)I was detected in the solutions, 13% in H\(_2\)O, and 6% in the CaCl\(_2\) aqueous solution. After a subsequent stirring in hot water, there were no released components. Furthermore, after stirring in 10 wt% CaCl\(_2\) aqueous solution, only 3% of \(^{131}\)I was detected. \(^{134}\)Cs and \(^{137}\)Cs were not detected in their solutions, indicating that they were tightly bound to the soil. It was found that about 22% of \(^{131}\)I adsorbed on the soil was dissolved in water. The chemical form of the dissolved \(^{131}\)I is iodide and the remaining one may be iodine or iodine-adsorbed aerosol. Thus, several chemical forms of \(^{131}\)I were found in the contaminated soil. However, \(^{134}\)Cs and \(^{137}\)Cs were not released from the contaminated soil by the above procedures. Therefore, the separation of the radioactive species adsorbed in clay from the soil was attempted. Generally, the soil contains clay, which is composed of SiO\(_2\) and Al\(_2\)O\(_3\), and the clay has an interlayer structure [8]. The interlayer is charged negatively and a positive ion binds there to neutralize the charges. In an alkali solution, the clay surface is charged negatively and is easily dispersed in the solution without coagulation. Therefore, a 13 wt% sodium bicarbonate aqueous solution, which is easily obtainable at home, was added to the soil to disperse it in an alkali solution.

Table 1 shows peak areas of \(^{131}\)I, \(^{134}\)Cs, and \(^{137}\)Cs in the supernatant after repeated washing. The peak areas of the radioactive materials were decreased by the repeated washing from 1st to 3rd, indicating that the radioactive materials adsorbed on the clay with a limited size.

Table 2 shows peak areas of \(^{131}\)I, \(^{134}\)Cs, and \(^{137}\)Cs in the supernatants that were allowed to stand for 0 and 4 h. As shown in Table 2, the peak areas of the radioactive materials in the supernatant that was allowed to stand for 0 h (immediately after washing with sodium carbonate solution) became larger than those in the case of 4 h, indicating that the radioactive materials adsorbed and precipitated with the clay.

### Table 1  Peak areas of \(^{131}\)I, \(^{134}\)Cs, and \(^{137}\)Cs in the supernatant after repeated washing.

| Supernatant | \(^{131}\)I | \(^{134}\)Cs | \(^{137}\)Cs |
|-------------|----------|----------|----------|
| 1st         | 229      | 282      | 199      |
| 2nd         | 149      | 217      | 193      |
| 3rd         | 78       | ND       | ND       |

### Table 2  Peak areas of \(^{131}\)I, \(^{134}\)Cs, and \(^{137}\)Cs in the 3rd supernatant that was allowed to stand for 0 and 4 h.

| 3rd Supernatant | \(^{131}\)I | \(^{134}\)Cs | \(^{137}\)Cs |
|-----------------|----------|----------|----------|
| 0h              | 108      | 115      | 98       |
| 4h              | 78       | ND       | ND       |
Separation of radioactive cesium and iodine from contaminated soil

Thus, under the alkali condition, colloidal clay was able to separate from the soil owing to the electrostatic repulsion of each negative charge.

Table 3 shows the peak areas of $^{131}$I, $^{134}$Cs, and $^{137}$Cs in the collected colloidal precipitate and the remaining soil after washing three and five times. By increasing the number of washings from three to five, the amounts of radioactive species decreased. This indicates that the clay fraction that contained radioactive species was removed as colloidal clay.

Fig. 2 shows a $\gamma$-ray spectrum of 30 g of the dried solid gel, which was prepared by allowing the collected colloidal precipitate to stand for one week in a closed vessel that contained silica gel. $^{131}$I was not detected on June 10, 2011 because of its short half-life of 8 days. The weight of the dried solid gel increased by about 4 g by absorbing water from the air. The dried solid gel was divided into part (A), and this part was divided into an upper part (B) and a lower part (C), as shown in Fig. 3. Table 4 shows peak areas of $^{134}$Cs and $^{137}$Cs in the dried solid gel, a part of the dried solid gel (A), and the divided upper part (B) and lower part (C), and the weight of each. Comparison of the peak areas of $^{134}$Cs and $^{137}$Cs in the dried solid gel and in the (A) part of the dried solid gel shows that their peak area ratio (35-36%) almost agreed with their weight ratio (30%). However, the peak area ratio of the lower part was larger than

![Fig. 2 $\gamma$-ray spectrum of the dried solid gel determined on June 10, 2011.](image)

![Fig. 3 Photographs of dry soil, part of soil (A), lower part (B), and upper part (C).](image)

| Table 3 | Peak areas of $^{131}$I, $^{134}$Cs, and $^{137}$Cs in the collected colloidal precipitate and the remaining soil after washing three and five times. |
|---------|----------------------------------------------------------------------------------|
|         | Peak area                           | $^{131}$I | $^{134}$Cs | $^{137}$Cs |
| Collected colloidal precipitate | 362 | 591 | 475 |
| 3rd remaining soil             | 561 | 910 | 745 |
| 5th remaining soil             | 211 | 392 | 350 |

| Table 4 | Peak areas of $^{134}$Cs and $^{137}$Cs in the dried solid gel, part of the dried solid gel (A), and the divided upper part (B) and lower part (C), and weight of each. |
|---------|----------------------------------------------------------------------------------|
|         | Peak area | Weight/g |
| Dry Solid | $^{134}$Cs | $^{137}$Cs | 30 |
| Part of Dry Solid | 368 | 306 | 9 |
| Lower Part | 346 | 243 | 4 |
| Upper Part | 123 | 98 | 5 |
that of the upper part, indicating that the radioactive cesium was present in a large size of clay and a large size distribution of the clay containing cesium. Otherwise, the weight of the dry solid gel increased by about 44% in air, indicating that the solid gel is hygroscopic. Furthermore, the lower part was dispersed in a montmorillonite clay suspension and was fractionated by decantation. Table 5 shows peak areas of $^{134}$Cs and $^{137}$Cs in a supernatant, a supernatant near precipitate, and precipitate: approximately 41 to 44% for the precipitate, 24 to 29% for the clay supernatant, and 30-31% for the supernatant near the precipitate. After the centrifugation at 3500 rpm for 100 min, approximately 92 to 94% of the radioactive cesium was detected in the precipitate. These results indicate that the component containing radioactive cesium is closely size-distributed. Finally, it was found that the separation of most radioactive cesium could be performed by decantation of its contained clay suspension.

3.2. Removal of radioactive cesium from contaminated soil using various materials

It is known that radioactive cesium is particularly adsorbed on the clay interlayer of the soil [5]; therefore, various materials were used to remove the radioactive cesium as follows.

Trinuclear iron acetate, which has a high volume [9], will widen the interlayer; thus, the radioactive cesium is easy to replace with it and is expected to be released. The release rates of $^{134}$Cs and $^{137}$Cs using trinuclear iron acetate were found to be only 2 to 4% in the supernatant. The precipitate was stirred and it was centrifuged three times at 1000 rpm for 10 min. The precipitate indicated double layers made of the upper part and the lower part, and each layer contained almost 50% of $^{134}$Cs and $^{137}$Cs. This indicates that the centrifugation was useful to selectively obtain the radioactive cesium.

Potassium chloride was used for an ion exchange with the radioactive cesium ions. Table 6 shows the release rates of $^{134}$Cs and $^{137}$Cs and the adsorption rate of $^{40}$K in the KCl. Approximately 12 to 15% of $^{134}$Cs and $^{137}$Cs were released in the supernatant and about 14% of $^{40}$K adsorbed to the soil. The weight of the adsorbed $^{40}$K was calculated from the concentration of the saturated KCl (100 mL) and was about 0.2 mg. This value is sufficient to replace the radioactive cesium. This indicates that the radioactive cesium ions were replaced with $^{40}$K.

Table 5  Peak areas of $^{134}$Cs and $^{137}$Cs in a supernatant, a supernatant near precipitate, and precipitate.

| Sample                  | $^{134}$Cs | $^{137}$Cs |
|-------------------------|------------|------------|
| Supernatant             | 76         | 67         |
| Supernatant near ppt    | 60         | 65         |
| Precipitate             | 109        | 92         |

Table 6  Release rates of $^{134}$Cs and $^{137}$Cs and adsorption rate of $^{40}$K.

| Material  | Release rate/% | Adsorption rate/% |
|-----------|----------------|------------------|
|           | $^{134}$Cs     | $^{137}$Cs       | $^{40}$K          |
| 1st KCl   | 12             | 15               | 14               |
| 2nd KCl   | 11             | 15               | 8                |
Since the CTA molecule has a hydrophobic part with a positive charge and is easily adsorbed into the clay interlayer, the remaining concentration of the radioactive cesium may only replace the radioactive cesium, and the adsorption site of the radioactive cesium was different from that of CTAB. Further addition of KCl and heating led to an increase to 14 of 16% in the release rates of $^{134}$Cs and $^{137}$Cs in the supernatant. Nonetheless, $^{40}$K did not adsorb on the clay. That is, heating is a cause of the increase in the release rate. Fig. 4 shows the γ-ray spectrum of the supernatant, and the typical peaks of $^{134}$Cs, $^{137}$Cs, and $^{40}$K were clearly observed. This indicates that heating is effective in the presence of much KCl. Furthermore, a black magnetic iron component, which was contained in the solution, was adsorbed on the magnetic stirrer and had about 3 to 4% of the radioactive cesium. This indicates that magnetic iron can adsorb the radioactive cesium and is easy to remove using a magnet.

Finally, CTAB was the most useful for removing the radioactive cesium from the soil. The hydrophobic part may be useful for removing the adsorbed radioactive cesium from the soil.

On the other hand, Nogawa et al. reported that potassium iodide (KI) was effective in removing the radioactive cesium from the soil [10]. Therefore, the consecutive use of KI was tested, and an iodine solution (I$_3$) containing hydrophobic molecules of I$_{-}$, was also investigated.

Table 8 shows the release rates of $^{134}$Cs and $^{137}$Cs after some treatments using 0.01 M I$_{-}$ aqueous solution, 40% KI aqueous solution, and distilled water. In the case of the I$_{-}$ solution, although the concentration is much lower than that of KCl (3.3 M), as shown in Table 6, the release rate for I$_{-}$ became higher than that for KCl. This indicates that I$_{-}$ in I$_{-}$ played an important role in the removal of the radioactive cesium. 40% KI is about 2.4 M and the release rate depends on the standing time. In the case of only 40% KI, after the water treatment, approximately 29 to 33% of the radioactive cesium was released in the supernatant. Although this value was larger than that of 7 to 8% even in the same 40% KI, this may be the reason why the remaining concentration of the radioactive cesium was twofold higher than in the case of only 40% KI.

**Table 7**  Release rates of $^{134}$Cs, $^{137}$Cs, and $^{40}$K in the supernatant added with CTAB, KCl aqueous solution, and KCl powder, and then heated.

| Treatment | $^{134}$Cs Release rate/\% | $^{137}$Cs Release rate/\% | $^{40}$K Release rate/\% |
|-----------|-----------------------------|-----------------------------|-------------------------|
| CTAB      | 8                           | 5                           | 47                      |
| 1st KCl   | 16                          | 15                          | 100                     |
| 2nd KCl   | 14                          | 16                          | 87                      |

Consecutive treatment with KCl was carried out as follows: the saturated KCl aqueous solution was mixed with the above pretreated soil and allowed to stand for 1 day. Approximately 11 to 15% of $^{134}$Cs and $^{137}$Cs were released in the supernatant and about 8% of $^{40}$K from the saturated KCl adsorbed to the soil, as shown in Table 6. This indicates that repeated treatment with the KCl aqueous solution increased the release rates of $^{134}$Cs and $^{137}$Cs, reaching a constant of about 11 to 15%. However, it was still tightly adsorbed on a different site in a different manner. To increase the release rate, the CTAB molecule, which is a long molecule and a surfactant having a hydrophobic carbon chain and a hydrophilic ammonium ion, was used. The hydrophilic part has a positive charge and is easily adsorbed into the clay interlayer [5]. Therefore, when CTAB was in the clay interlayer, the hydrophobic long parts were also incorporated and expected to physically push out the radioactive cesium.

Approximately 5 to 8% of $^{134}$Cs and $^{137}$Cs were released by adding CTAB to the KCl-treated soil, as shown in Table 7. However, approximately 47% of $^{40}$K was also released in the supernatant. This indicates that most of CTAB was replaced with $^{40}$K in the clay interlayer. Furthermore, the addition of KCl and allowing to stand for 3 days led to an increase of 15 to 16% in the release rates for $^{134}$Cs and $^{137}$Cs in the supernatant. Since the CTA molecule in the CTAB tightly bound to the soil, $^{40}$K in KCl did not adsorb on the soil, and the preadsorbed $^{40}$K was released. Therefore, the release rate may increase over time, or K’ may only replace the radioactive cesium, and the adsorption site of the radioactive cesium was different from that of CTAB.

**Table 8**  Release rates of $^{134}$Cs and $^{137}$Cs after some treatments using 0.01 M I$_{-}$ aqueous solution, 40% KI aqueous solution, and distilled water.

| Treatment | $^{134}$Cs Release rate in the supernatant/\% | $^{137}$Cs Release rate in the supernatant/\% | Treatment | $^{134}$Cs Release rate in the supernatant/\% | $^{137}$Cs Release rate in the supernatant/\% |
|-----------|--------------------------------------------|--------------------------------------------|-----------|--------------------------------------------|--------------------------------------------|
| 0.01M I$_{-}$ for 18h | 22                          | 23                          | 40% KI for 42h | 19                          | 20                          |
| I$_{-}$ and 40% KI for 21h | 23                          | 26                          | 40% KI for 152h | 17                          | 14                          |
| 40% KI for 152h | 19                          | 23                          | Water for 10h | 5                           | 6                           |
| Water for 10h | 7                           | 7                           | 40% KI for 50h | 29                          | 33                          |
| 40% KI for 50h | 7                           | 8                           |               |                             |                             |
Finally, the repeated treatment using KI led to a maximum release rate of 66% of the radioactive cesium from the remaining soil. There was an error of about 4 to 20% for the radioactivity of the radioactive cesium between the supernatant and the remaining soil.

4. Conclusions

Approximately 19% of $^{131}$I was released in a solution mixing the contaminated soil with distilled water and 10 wt% CaCl$_2$ solution, and the remaining $^{131}$I tightly bound to the soil. $^{134}$Cs and $^{137}$Cs were not released using sodium bicarbonate, CaCl$_2$, distilled water, and hot water. The soil was separated easily in the colloidal clay and precipitated by dispersing in 13 wt% sodium bicarbonate aqueous solution. $^{131}$I, $^{134}$Cs, and $^{137}$Cs were found both in the colloidal clay and precipitated soil. The clay containing $^{134}$Cs and $^{137}$Cs was dried as a solid gel, and the radioactive materials were distributed more in the upper part than in the lower part. The solid gel was hygroscopic. Approximately 2 to 4% of the radioactive cesium was found in the supernatant after mixing with trinuclear iron acetate. Approximately 12 to 15% of the radioactive cesium was found in the supernatant after mixing with saturated KCl aqueous solution, and the repeated treatment for 1 day indicated the additional release of 11 to 15% in the supernatant. Approximately 5 to 8% of the radioactive cesium was found in the supernatant after mixing with saturated KCl aqueous solution, and the repeated treatment using KI led to a maximum release rate of 66% of radioactive cesium from the soil.

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