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Determination of Low Level ^{137}Cs in Environmental Water Sample Using AMP Method and a Review Comparing with Other Adsorbents

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Abstract: A large amount of fission-yield ^{137}Cs product has been released into the environment due to nuclear power plant activity, nuclear weapon tests, and nuclear power plant accidents become a great concern to human health and ecological life. Radioactive cesium in the environment is potentially dangerous to aquatic organisms because it has a chemical similarity to potassium and could be quickly accumulated in internal organs. In this study, ammonium phosphomolybdate (AMP) was prepared and used as a coprecipitation adsorbent to separate radioactive ^{134}Cs and ^{137}Cs from environmental water samples. Optimized initial pH solution, CsCl as a carrier, and AMP amount were modified to achieve the adsorption's optimum conditions. Furthermore, the gamma rays emitted from ^{137}mBa as a daughter of ^{137}Cs were measured with a low background high purities Germanium detector. Using this method shows the right consistency with the result by γ-spectrometry and demonstrated a higher radiochemical recovery. AMP's chemical yield was ranged from 90% - 96%, and MDA for ^{137}Cs was 0.30 mBql^{-1}. Finally, this method was convenient for applying and suitable for analyzing low-level activity concentrations of ^{137}Cs in routine monitoring with a few improvements.

Keywords: ^{137}Cs, adsorption, ammonium phosphomolybdate, environmental water.

钼磷酸铵法测定环境水样中的低浓度 137 铯并与其他吸附剂比较

摘要：由于核电站活动、核武器试验和核电站事故，大量裂变产 ^{137} 铯产品被释放到环境中，成为人类健康和生态生活的重大问题。环境中的放射性铯对水生生物具有潜在危险，因为它与钾具有化学相似性，并且可以迅速积累在内部器官中。在本研究中，制备了磷钼酸铵并将其用作共沉淀吸附剂，以从环境水样中分离放射性 ^{134} 铯和 ^{137} 铯。优化的初始酸碱度溶液、氯化铯作为载体和磷钼酸铵 量被修改以达到吸附的最佳条件。此外，从 ^{137} 毫巴发射的伽马射线作为 ^{137} 铯 的子体是用低背景高纯度锗探测器测量的。使用这种方法显示出与 γ 光谱结果的正确一致性，并证明了更高的放射化学回收率。磷钼酸铵的化学产率范围为 90% - 96%，^{137} 铯 的最小可检测活动为 0.30 毫贝克勒每升$^{-1}$。最后，该方法应用方便，适用于常规监测中 ^{137} 铯的低活度浓度分析，稍有改进。

关键词：^{137}Cs，吸附，磷钼酸铵，环保水。

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1. Introduction

Significant sources of anthropogenic radionuclides have been produced and released into the environment by human nuclear activity, such as nuclear weapons tests, nuclear power plant activity, research reactors, and nuclear fuel reprocessing plants. Also, the accident of nuclear power plant facilities in Chernobyl (April 26, 1986) and Fukushima Dai-ichi Nuclear Power Plant on March 11, 2011, released a vast radioactive material into the atmosphere and terrestrial environment [1], [2], [3], [4], [5]. The high demand for energy consumption from nuclear power plants was increasing continually for the last 50 years. With a raised population growth and economic development, it has caused severe radionuclides contamination in our environment. In nuclear power plant reactors, radioactive cesium is one of the most abundant species and waste repositories due to its yield of about 6% from thermal fission of ^{235}U [6], [7]. There were 450 actives operational nuclear power plant units worldwide, and it could generate at least 396.4 GW(e) electricity for people's consumption [8]. Nuclear fuel reprocessing plants such as the Sellafield sites in the United Kingdom and La Hague site in France also contribute to the discharged radionuclides to the European sea for more than 40 years [9], [10]. The annual discharge of ^{137}Cs into the Irish Sea from 1974 to 1978 was 4000 TBq as part of the liquid effluent from the Sellafield reprocessing site and the primary source of ^{137}Cs to these waters [11], [12]. Since the Chernobyl nuclear power plant accident in 1986, concern about the behavior of ^{137}Cs and their impact on the environment has increased significantly. The total amount of Chernobyl-derived ^{137}Cs released was estimated to be 4.7 PBq [13], [14]. ^{137}Cs were radionuclides with long-lived fission products and had similar potassium and calcium and can be easily assimilated by terrestrial and aquatic organisms. Radioactive cesium could create an internal hazard in the body of organisms and will accumulate with a higher concentration in muscle tissues and lower in bones [15], [16], [17].

On March 11, 2011, the Tohoku earthquake created an enormous tsunami that, magnitude 9.0, and caused the Fukushima Dai-ichi nuclear power plant accident. This accident resulted in considerable contamination of radioactive materials on terrestrial, rivers, and sea around Fukushima Prefecture [2], [3], [4], [18]. The total amount of radioactive cesium released into the atmosphere was estimated to be 20 PBq [19], [20] and about 20 % deposited on land, and about 80% was transported to the Pacific Ocean [21], [22]. Radioactive cesium is considered the most abundant and dangerous element because it emitted gamma radiation and has a long half-life of 2.06 years for ^{134}Cs and 30.17 years for ^{137}Cs [23].^{137}Cs, emitting β- with energy Emax = 1176 keV, and about 5.4% of the cases, ^{137}Cs can decay directly to stable ^{137}Ba. In 89.9 % of cases, beta emission decays to a metastable state of ^{137}Ba and attains a stable isotope of ^{137}Ba by emitting a γ-photon with energy 661.6 keV. Furthermore, ^{134}Cs with a shorter half-life have a much more complicated decay scheme. Towards the ground state stable isotope, ^{134}Cs was emitted β-with energy of gamma rays of 604.72 keV (97.63 %), 569.33 keV (15.37 %), 563.25 keV (8.34 %), 475.37 keV (1.48 %) [24]. The determination of ^{137}Cs in environmental waters is essential to protect the population during nuclear incidents and protect the environment from possible contamination consequences. In emergency cases, rapid analytical methods are needed to provide adequate information for a fast response. Many developed methods for rapid determining of ^{134}Cs and ^{137}Cs in environmental waters have been applied. It was difficult to measure the activity in some cases because its concentration may too low and require many samples. Also, the chemical characteristic of cesium has a high solubility in water [25], [26].

The separation of radioactive cesium was conducted using preconcentration from a large volume of water and is typically carried out using specific selective ion exchangers. In the future, the ion exchange or adsorption method is the most promising analysis due to its relatively straightforward operation, good chemical stability in the presence of nuclear radiation, and high efficiency. Composite materials and inorganic cation exchange are the most common adsorbents due to high selectivity towards certain radioisotopes, such as cesium [25]. Hexacyanoferrates and molybdophosphate such as AMP have been successfully developed and used in cesium removal as inorganic adsorbents. Still, an increasing scale of its application for a large water sample is not proving yet because the adsorbents have a low stability mechanic form in particular conditions [27], [28]. Sebesta and Stefula [29] reported that the composite ion-exchanger AMP-polyacrylonitrile (PAN) with an improving AMP's mechanical dust properties was suitable for selective separation and removal ^{137}Cs from a seawater sample.

In this paper, we propose an improved AMP procedure. We used the advantage of the high selectivity and efficiency of ammonium phosphomolybdate (AMP) to analyze ^{134}Cs and ^{137}Cs activity concentration seawater and river water contaminated from Fukushima Dai-ichi nuclear power plant accident. The process's main parameters are studied, and AMP adsorption methods in the low-level activity of ^{137}Cs were also reported. Modified engagement of high selectivity and efficiency of AMP with optimized initial pH solution, CsCl as a carrier, and AMP amount could be very effective for measurement radiocesium nearly minimum detectable activity.

2. Materials and Methods

2.1. Reagents and Materials

All chemicals were of analytical reagent grade. Pure ammonium phosphomolybdate (AMP) was used for the adsorption of radiocesium in the dissolved phase. Analytical grade of CsCl solution was used as a carrier. Nitric acid (HNO_3) 65% and Milli-Q deionized water were used to prepare all solutions to precipitated radiocesium in river and seawater samples. Millipore filters of cellulose acetate (0.45 μm Whatman type) were used in the AMP/Cs separation process by suction filtration. Polyethylene bags were used to packed the AMP samples to measure ^{137}Cs and ^{134}Cs. In this experiment, seawater samples were collected from OnahamaBay, in Fukushima Prefecture and river water samples from the Tone River, in Gunma Prefecture from 2018 to 2019.

2.2. Instrumentation

Continuous flow centrifugation (Kokusan, H-2000B) separated suspended solid from the river water. Two High-Purity Germanium (HPGe) planar detectors were used to count radioactivity at the Low-Level Radioactivity Laboratory (LLRL) and Ogoya Underground Laboratory (OUL), Kanazawa University. The planar type gamma-spectrometer efficiency was 18-34% with 1.8 - 1.98 keV at FWHM for the 1332 keV gamma-ray line of ^{60}Co. The HPGe detector's efficiency was calibrated using a multiple gamma-ray emitting standard source to achieve high-quality gamma-ray spectra, including nine radionuclides (Japan Radioisotope Association, Japan).

2.3. Analysis Samples

In the laboratory, the suspended solids from river water samples were separated using continuous flow centrifugation at a speed of 14000 rpm, 20° C, with a water flow rate of 0.3 l min^{-1}. Then dissolved fractions were put in a 20 L container for the next procedures. Dissolved radioactive cesium was measured as concentrations per liter of water ($mBql^{-1}$). The river water and seawater samples (20L) were adjusted by adding concentrated HNO_3 to pH 1.0 - 1.6 before analysis. ^{134}Cs and ^{137}Cs in a dissolved phase were quantitatively separated by coprecipitation with ammonium phosphomolybdate (AMP)/Cs in a low pH solution. The samples were stirred for 30 minutes after adding 0.26 g CsCl and then adding a 4.0 g AMP as a Cs^+ adsorbent, stirred for 60 minutes, then left at room temperature overnight. The same process was also conducted in seawater samples without the separation of suspended solid due to a low concentration of particulates. The precipitated AMP/Cs compounds were separated by vacuum filtration in 0.45 μmmillipore, and the dried weight yield was calculated after drying in the oven at 60°C. The dried AMP/Cs were packed in polyethylene bags into two types of geometry for 4 g AMP (3.5 cm x 7.0 cm) and 8 g AMP (5.5 cm x 5.5 cm).

2.4. Measurements and Calibration for ^{137}Cs Efficiency

^{134}Cs and ^{137}Cs were measured to quantify activity concentration (Bq kg^{-1} dry weight of the samples) using low-background gamma-ray spectrometry for 86,400 s – 259,200 s the best results statistically. The efficiency of radioactive cesium concentrations in the sand samples was compared with reference materials (JSAC0472) in similar geometry, based on the G-ray peaks at 604.7 keV for ^{134}Cs and 661.7 keV for ^{137}Cs. The brick shape of passive lead was used to housing each of the detectors to a minimum of the radiation background, with a thick wall that was 20-25 cm and close with an iron block (25 cm thick). A multichannel analyzer (MCA) was performed to determine the detectors' resolution and accumulated energy spectrum from each radionuclides' definitive source with high resolution (4096-channels). A representative sample was prepared with approximately the same chemical composition and geometry as AMP samples to calibrate the energy of ^{134}Cs and ^{137}Cs activities. Radioactivity of ^{134}Cs and ^{137}Cs was evaluated using a detection efficiency curve obtained from the representative samples. The analytical precision for measuring ^{134}Cs was 1-30% based on the counting statistics' standard deviation. All of the ^{134}Cs and ^{137}Cs activity data were decay-corrected to each sampling date.

3. Results and Discussion

The separation of selective low-level radioactive cesium from the water remains a challenge, not only in neutral pH solution but also in the presence of high concentration competing ions such as Na, Mg, Ca, and K [30]. The AMP compound as a cation exchange shows remarkably selectivity for concentrating ^{137}Cs from an aqueous sample with distribution coefficient value Kd = ~190. Still, an AMP application using columns has low flow rates due to its fine crystalline form [25], [31]. However, these difficulties can be solved by packing the AMP compound in a conventional filtration kit with a mesoporous filter paper and vacuum filtration using low-pressure suction. The AMP procedures have become a convenient procedure for radioactive cesium determination by gamma(γ)-spectrometry. This study uses the same adsorption methods from Aoyama et al. [32] for determining low-level ^{137}Cs in seawater and river water samples.

3.1. ^{137}Cs Efficiency Recovery Using AMP Methods

To validated the radiochemical recovery of ^{137}Cs in the AMP compound, we used the same result data and same procedure from previous research with the recovery value of 96 - 100 % activity of ^{137}Cs [32],

[33]. In most cases, approximately 60 - 120 l samples should be used to determine ^{137}Cs in seawater to get a radioactivity level higher than MDA (minimum detectable activity) with γ-spectrometry. However, in this study, 20 - 40 l amounts of seawater and river water samples are enough to analyze^{137}Cs activity because its concentration was higher than MDA. Table 1 shows that the average concentration value of ^{137}Cs in river water was 1.64 mBq l^{-1} with a range of 0.62 – 3.57 mBq l^{-1} and 3.82 mBq l^{-1} average seawater 1.40 – 5.91 mBq l^{-1}. All results data shows the activity concentration was higher than the MDA value 0.30 mBq l^{-1}. By assuming the radiochemical yield recovery of 90-96 %, these results indicate that this study's method is suitable for analyzing low-level ^{137}Cs concentration in a small sample of seawater and river water. The sample analysis's preparation time takes about 4 hours for radiochemical separation, excluding overnight precipitation and drying time. While the measurement time takes about 1-3 days, counting depends on the sample's activity concentration. This result was suitable for routine monitoring or emergencies to determine ^{137}Cs with a high-efficiency measurement rapidly.

Table 1 Result of seawater and river water samples by AMP adsorption method

AMP used	4 g
CsCl carrier	0.260 g
Sample volume used	20 L
The activity of ^{137}Cs in river water, mean (range)	1.64 mBq l^{-1} (0.62 – 3.57 mBq l^{-1})
Activity of ^{137}Cs in seawater, mean (range)	3.82 mBq l^{-1} (1.40 – 5.91 mBq l^{-1})
MDA, approximate	0.30 mBq l^{-1}
Chemical yield	90-96

3.2. Activity Ratios of ^{134}Cs and ^{137}Cs

The gamma-spectrometer efficiency was calibrated using a multiple gamma-ray emitting standard source, including nine radionuclides from JRA (Japan Radioisotope Association). The samples' concentration of ^{137}Cs in the sample was quantifying and validated using gamma-spectra comparison with a reference material of JSAC 0472 containing ^{134}Cs and ^{137}Cs standard. An error measurement from MCA spectra should be below 30 % to get a confidential statistic. All the AMP/Cs samples used were prepared in the same weight and geometry as a standard before measurement and sealed to prevent contamination of detectors from the samples and avoid a gaseous leak of ^{222}Rn. ^{137}Cs activity was determined by measuring the gamma-ray with 661.7 keV from decaying ^{137m}Ba to a stable ^{137}Ba using conditions of measurement established from previous research [32]. The samples were counted at a minimum of 72 h depending on their activity level and correction error less than 0.3. Appropriate software was used to analyze ^{134}Cs and ^{137}Cs peak spectra with a 95% confidence level, and the activity ratios were calculated and corrected to accident date March 11, 2011. The ratios of ^{134}Cs/^{137}Cs in seawater and river water samples range from 0.9 to 1.1. This result indicated that the contamination of radioactive cesium was from the FDNPP accident.

Fig. 1 shows the activity ratios of ^{134}Cs/^{137}Cs corrected to the accident date in seawater and river water samples versus the sample's date. A clear relationship between the percentage error of ^{134}Cs/^{137}Cs ratios was increased when the sampling date increased in river water. That phenomenon can be explained by the influence of ^{134}Cs with a short half-life of 2.04 years. The sampling process from 2018 to 2019 makes the ratio activity concentration of ^{134}Cs in seawater and river water decreased over time. This result was supported by previous studies that reported the dissolved ^{134}Cs, and ^{137}Cs concentration in Fukushima river water decreases with time [34], [35]. The low concentration of ^{134}Cs in the samples makes it difficult to measure and increases the error of the ratio ^{134}Cs/^{137}Cs. The activity of ^{134}Cs from 2019 was below the detection limit in both samples due to its almost four times half live with 1/16 of ^{134}Cs concentration remaining after the accident date. The activity of ^{137}Cs in seawater rapidly decreased after the FDNPP accident in association with current ocean movement [36], and during the period of our study, the ^{137}Cs activity was range 1 – 10 mBq l^{-1} [37]. Thus, the ^{134}Cs activity in seawater is very low.

3.3. Comparison with the Previous Method

Interfering other radionuclides in the sample matrix may influence the efficiency of gamma-ray spectrometric and radiochemical recovery measurements such as insoluble salts of transition metals with hexacyanoferrate(II) can incorporate metal ions into their crystal lattices.

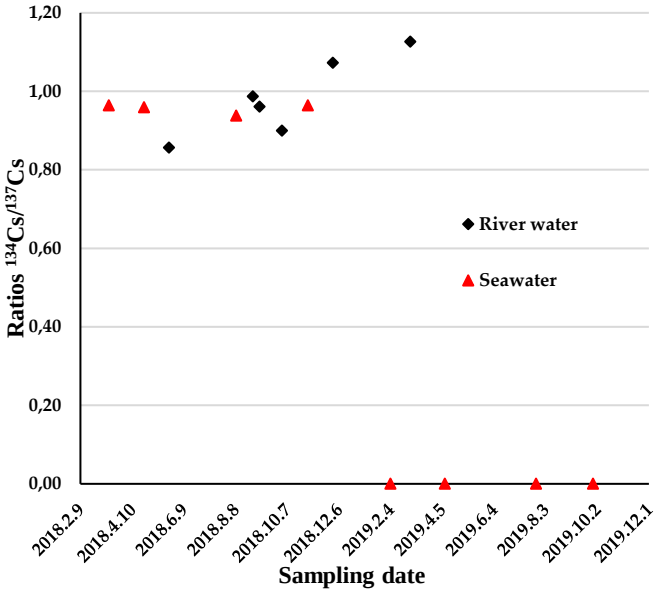


Fig. 1 Activity ratios of ^{134}Cs/^{137}Cs in seawater and river water samples

Several adsorption methods for removing radioactive cesium were developed for selective separation in the presence of other radionuclides. Promising inorganic cation-exchange materials are probably the essential ion exchanger due to good chemical stability [34].

Table 2 shows several research developments in methodology for separating radioactive cesium in the water samples matrix. Nilchi et al. [38] studied about modification of copper hexacyanoferrate-polyacrylonitrile composite (CHCF-PAN) as an ion exchanger. Some parameters should be adjusted to obtain the optimum adsorption using this method, such as the temperature solution, contact time, interference from other cations, and the influence of pH at an initial aqueous solution. The efficiency from this modified adsorbent column was obtained around 63.78 % of the recovery. Prihatiningsih and Suseno [39] developed methods to determine ^{137}Cs efficiency using ammonium phosphomolybdate, hexacyanoferrate, and hexacyanoferrate impregnated cartridge filter 100 l seawater samples with the presence of ^{137}Cs. The measurement activity concentration of ^{137}Cs was counted using a gamma spectrometer. The results show efficiency value of removal ^{137}Cs was 93.6 % for ammonium phosphomolybdate, 90.3 % for hexacyanoferrate, and 92 % for hexacyanoferrate impregnated cartridge.

Another method was recently reported by Cho et al. [40] using sodium alginate hydrogel beads-based adsorbent containing chemically bound Prussian blue (PB). The technique was to make a crosslinked sodium alginate with Fe(III) ions as a dual function to form hydrogel beads. The reaction with hexacyanoferrate could form Prussian Blue formation using Fe(III) ions to serve as a crosslinking agent. This study result shows that the amount of embedded PB in the composite beads is more than two times larger than in conventional sodium alginate-PB composite beads. This method's advantage was that the composite beads have ions adsorption capacity 2-5 times higher to remove the radioactive cesium in water. The effective removal of radioactive ^{137}Cs was observed values as high as 99 %.

Wu et al. [30] developed an adsorbent of Cs using a novel porous silica loaded with ammonium molybdophosphate (AMP/SiO_2) to decontamination system for the Fukushima Daiichi Nuclear Power Plant. The technique was to make novel porous silica as the adsorbent with the AMP as an active site composite throughout impregnation, precipitation, and freeze-drying methods. The surface area of the composite was 72.2 m^2 g^{-1} with a pore diameter of 254.2 A. The efficiency of removal ^{137}Cs from this study shows promising results with a radiochemical recovery of 97.1 % in low-level seawater concentration. The results indicated that AMP/SiO_2 has effectively treated cesium contamination even in the presence of highly concentrated competing ions in seawater.

Table 2 A comparison of developed material adsorbent for the Cs removal from waters

Adsorbent	Matrix	% removal of Cs	References
Copper hexacyanoferrate-PAN composite	Aqueous solution	63.78	[38]
Ammonium phosphomolybdate	Water	93.64	[39]
Hexacyanoferrate Cartridge		90.30	
Hexacyanoferrate		91.97	
AMP/SiO_2	Seawater	97.1	[30]
Prussian blue [$K_4[Fe(CN)_6]$ in sodium Alginate hydrogel	Water	99.0	[40]
AMP with CsCl carrier	River water and seawater	96-100	This study, [32]

In this study, a simple ^{137}Cs separation technique using ammonium molybdophosphate (AMP) was used to analyze radioactive cesium in environmental water samples. AMP coprecipitation was applied as a more comfortable and more rapid preconcentration technique for a large water volume. The procedure that we used in this study is achievable within 3 hours of laboratory work in 1 sample from precipitation to preparation for gamma counting, excluding settling and drying time that no longer than 24-48 h. This result indicates that the AMP coprecipitation method was more rapid and easy to use when an emergency occurs than the previous procedure. Recovery of chemical yield AMP during the whole procedure was relatively high, ranging from 90% to 96% with radiochemical yield >90%. The analytical results using AMP are in good agreement with previous research data using modified AMP or another adsorbent (Table 2). The method of AMP precipitation to selectivity removal ^{137}Cs from environmental samples is acceptable for long-term monitoring due to its ease of applied field sampling. In the previous procedure reported by Aoyama et al. [32], higher sensitivity measurements are necessary to determine the presence of low-level concentration ^{137}Cs in smaller volumes of water. Therefore, high sensitivity gamma spectrometry instruments were used with very low background radiation. Furthermore, the proposed method is suitable for ^{137}Cs monitoring seawater or other aqueous samples derived from global fallout and the Fukushima Daiichi Nuclear Power Plant accident in Indonesian marine waters. It can also combine with the previous method or improve the sensitivity of gamma spectroscopy for the measurement of low-level ^{137}Cs from the deeper seawater monitoring.

4. Conclusions

Environmental seawater and river water samples obtained from Onahama Bay and the Tone River were analyzed using the AMP coprecipitation technique and measured by gamma-spectrometry. This study showed that the AMP precipitation from optimized pH 1-1.6 was selectively enabled to separate radioactive cesium from a relatively small water sample and showed high efficiency with the result by γ-spectrometry. The chemical yield of AMPdemonstrated a higher radiochemical recovery was approximately 90%, and MDA for ^{137}Cs was 0.30 mBql^{-1}.The proposed method is suitable for long-term monitoring seawater or other aqueous samples in an emergency and routine condition. In the future, we will use the improved procedure to determining the low-level concentration of ^{137}Cs in marine water for the deeper layers to find the temporal change of ^{137}Cs derived from global fallout and FDNPP accident. Therefore, a higher sensitivity of gamma spectrometry and larger samples are necessary to obtain high-resolution water profiles.

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