A simple method for quantifying iodate and iodide fractions in solution using Ag+-impregnated activated carbon

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Abstract
The speciation of iodine in the environment must be known in order to properly assess its geological fate and to study potential remediation materials for the decontamination of water containing radioactive iodine. In this study, the utilization of silver-impregnated granular activated carbon for separation between two iodine species, iodate and iodide, from each other was studied in different solution matrices with batch and column experiments. A high separation of the iodine species was achieved in the high concentrations of interfering ions, e.g. chloride, and in both μM and trace iodine concentrations. The method is suitable for both radioactive and non-radioactive iodine.

Keywords Iodide · Iodate · Iodine species separation · HPLC-ICP-MS · Silver-impregnated activated carbon

Introduction
Iodine is an abundant element found in Earth’s crust, atmosphere and oceans at notable concentrations. It is also a vital micronutrient to humans and other mammals as it is needed for the production of thyroid hormones and for the proper functioning of thyroid gland. For that reason, iodine is commonly added to table salt in the form of either iodide (I−) or iodate (IO3−) salt to supply populations the vital element [1, 2]. On the other hand, radioactive isotopes of iodine, 129I (t½ = 15.7 My) and 131I (t½ = 8 d), pose a radiological threat in the case of a nuclear fallout as the radioactive iodine tends to absorb effectively to the thyroid gland and causing an elevated risk of thyroid cancer. From the long-term perspective, iodine’s mobility in geochemical environments poses a major risk in nuclear waste disposal sites (i.e. [3]) and sets strict requirements for the long-term safety of the final disposal concepts for spent nuclear fuel [4]. The key attribute determining the geochemical fate of iodine is its speciation [3].

Iodine’s chemical and physical behaviour is strongly governed by its speciation. In general, IO3− is the dominant species in oxidizing environments, while I− becomes the main species in reducing conditions. In addition, molecular iodine (I2) exists in low pH and in natural environment, organic iodine compounds might be present [5]. It is crucial to notice that iodine can exist in several forms even at the same time. Further, the speciation of iodine affects essentially its geochemical behaviour and interactions with natural materials [5–7] or synthetic solid sorbents developed for iodine removal [8, 9]. To reliably assess the interaction of different remediation materials, it is essential to precisely control and survey the speciation of iodine.

In macro-concentrations, the speciation of iodine can be measured conventionally with a combination of chromatographic techniques (i.e. IC, LC or HPLC) and an appropriate detector, such as ICP-MS (inductively coupled plasma mass spectrometry) [10–15]. With radioactive samples, the availability of special instruments might be limited because of safety aspects. It is also rather common to perform experiments regarding iodine uptake with radioactive iodine tracers. Even though some experiments include alteration of iodine redox state, not all of the studies include or specify the verification of iodine speciation during the experiments or after the redox change. On the other hand, for
the environmental samples regarding iodine speciation, the instant separation of two major iodine species already at the time of sampling in the field would be beneficial to prevent any possible redox state changes between the sampling and analysis.

Silver-impregnated activated carbons (Ag-GAC) have been used in iodide removal for decades [16–18] and these materials are readily commercially available. The uptake of iodide is based on the precipitation reaction with silver cation [16]. AgI has extremely low solubility ($K_{sp} = 8.5 \times 10^{-17}$) compared to other silver halides, like AgCl ($K_{sp} = 1.8 \times 10^{-10}$) and AgBr ($K_{sp} = 5.4 \times 10^{-13}$). For the AgIO$_3$, the solubility product is also much higher ($K_{sp} = 3.17 \times 10^{-8}$) [19].

Based on the difference in the affinity of the two iodine species to silver, the separation of them can be obtained. Here, we demonstrate a simple method for separating iodide and iodate species from each other in liquid samples using a commercial Ag-GAC. While iodine separation from aqueous solution has been extensively studied in many studies (i.e. references [8] and [9]), usually studies are considering either total iodine removal or removal efficiency for different iodine species from contaminated water. Instead, Ag-GAC is selective only to one species of iodine (I$^-$) and not to another (IO$_3^-$). This high and reproducible separation performance can be utilized in the speciation analysis of iodine.

Our proposed method is especially suitable for the separation of iodate and iodide species of radioactive $^{125}$I measured with $\gamma$-detection, but it is not only limited to that but can be used for the separation of non-radioactive iodine species if combined with a suitable detector like for example ICP-MS. One advantage of the method is that it could be used already in the field for the separation of iodine species, while the analysis could be done in laboratory later on, avoiding possible changes in redox state of iodine. Another inorganic species of iodine is molecular iodine, I$_2$, which is a stable species in acidic pH. Iodine is routinely extracted in non-polar solvents like chloroform [20, 21] and the step could be easily added to our proposed methodology if needed. Organo-iodine compounds are beyond the scope of this particular study and if present require further separation not to interfere with the results of $\Gamma$/IO$_3^-$ separation.

Compared to a simple precipitation of AgI using silver salt like AgNO$_3$, the silver-impregnated activated carbon provides an alternative approach and helps to avoid unwanted and disturbing co-precipitation or oxidation–reduction reactions. As a reference, the performance of Ag-GAC is compared with strong basic anion exchanger, which often is used to separate different anions, e.g. iodide and iodate.

### Experimental

#### Chemicals

All reagents were of analytical grade (Alfa Aesar, Sigma-Aldrich, Riedel de Häen) and used without further purification. The radioactive $^{125}$I tracer was purchased from PerkinElmer. Silcarbon AG03 silver-impregnated carbon made from coconut shell (silver content ~0.3 wt%, specific surface area 1150 m$^2$ g$^{-1}$) was kindly supplied by Silcarbon Aktivkohle GmbH, Germany and used as received. Dowex$^\text{®}$ 1X4 Strong Anion Exchanger in chloride form (50-100 mesh) was purchased from Sigma-Aldrich and used without further pretreatment.

#### Instrumentation

In the experiments conducted with radioactive $^{125}$I tracer, the radioactivity was measured from 5 ml aqueous samples using Perkin Elmer WizardTM 3$^\text{rd}$ automated NaI-scintillation $\gamma$-detector with a 20 min counting time.

In the case of non-radioactive iodine, the concentrations of iodide and iodate were analysed using an anion-exchange chromatography column (Dionex AS11 4 x 250 mm analytical column and AG11 4 x 50 mm guard column) attached to an Agilent 1260 Infinity quaternary pump and autosampler HPLC-system connected to an Agilent 7800 ICP-MS via direct connection between the column and ICP nebulizer. The eluent was 50 mM sodium hydroxide (NaOH) and isocratic elution with flow rate of 0.8 ml/min was used. Argon gas was bubbled to the eluent container to minimize the concentration of dissolved carbon dioxide. The iodine species $\Gamma^-$ and IO$_3^-$ were separated based on their retention times obtained from the standard solutions prepared from KI and KIO$_3$. The quantification was done using external standards on the range 0–200 $\mu$g L$^{-1}$ of total iodine for both species and calculating the concentration from the peak areas. The ICP-MS was driven in the no-gas mode and the measured isotope was $^{127}$I. The quality control of measurements was done by measuring calibration blanks and standard samples (5 + 5 and 50 + 50 $\mu$g L$^{-1}$ of total iodine $\Gamma^-$ + IO$_3^-$) from separate dilution series after every calibration and between the sample measurements. The observed retention times remained stable and were 120 s for IO$_3^-$ and 360 s for $\Gamma^-$. A limit of detection (LOD) for HPLC-ICP-MS system was determined to be 0.5 $\mu$g L$^{-1}$ and 0.2 $\mu$g L$^{-1}$ for $\Gamma^-$ and IO$_3^-$, respectively, by using repeated injection method and measuring 8 replicates of 1 $\mu$g L$^{-1}$ calibration standard and calculating the LOD based on the literature [22].
Iodine speciation manipulation

KI and KIO₃ were used as analytes for I⁻ and IO₃⁻ in the case of non-radioactive experiments, but radioactive ¹²⁵I was only available in the form of Na¹²⁵I. For this reason, the oxidation and reduction procedures were first tested with non-radioactive starting materials and using HPLC-ICP-MS to determine the yield of the reactions with different concentrations of oxidant and reducing agent. NaOCl and NaHSO₃ were used to either oxidize iodine to IO₃⁻ or reduce to I⁻, respectively.

In order to obtain IO₃⁻ solutions, oxidation of I⁻ solutions (8 × 10⁻⁷ M) was achieved using 0.01–10 mM NaOCl in 10 mM NaOH for 24 h. Similarly, I⁻ was reduced from IO₃⁻ by using 0.01–10 mM NaHSO₃ in 20 mM HCl for 24 h. The concentration of I⁻ and IO₃⁻ were measured with HPLC-ICP-MS before and after the addition of oxidant or reductive agent.

Similar procedure was used for ¹²⁵I, where the starting material was Na¹²⁵I, but in much lower concentrations (~ 10⁻¹¹ M) compared to non-radioactive iodine solutions. The appropriate concentrations of oxidant or reductant was decided based on the experiments performed with non-radioactive materials.

Batch experiments

Iodide and iodate uptake properties of Ag-GAC and strongly basic anion exchanger were studied with batch experiments to test the effect of the matrix solution to the uptake of iodate and iodide. Sample series were made for both iodine species as a function of pH and with interfering anions (Cl⁻, NO₃⁻, SO₄²⁻) added as Na salt solutions in different concentrations (1–1000 mM for Cl⁻, 1–100 mM for the others).

In batch experiments, 20 ± 1 mg of adsorbent material (Ag-GAC or Dowex® 1X4 Strong Anion Exchanger) was weighed to a polyethylene vial and 10 ml of test solution were added. In the experiments where pH adjustment was needed, appropriate volumes of 0.1 M NaOH or HNO₃ solutions were added to samples before the addition of iodine. The initial iodine concentrations were 8 × 10⁻⁷ M in experiments with non-radioactive iodine and in experiments with radioactive iodine the radioactivity of carrier-free ¹²⁵I was between 100–250 Bq for a sample corresponding to concentrations on the scale of ~ 10⁻¹³ M. The samples were equilibrated for 24 ± 2 h and solid and liquid phases were separated by centrifuging (3000 G, 10 min) followed by filtering of 6 ml fraction with a 0.2 μm syringe filter (PVDF LC. Arcodisc, Gellman Sciences). The equilibrium pH was measured from the remaining supernatant using a Ross combined electrode. In the case of radioactive ¹²⁵I, 5 ml of filtered solution was pipetted to a scintillation vial and analysed with Wallac 1480 Wizard 3″ automated NaI-scintillation detector.

The sorption percentages were calculated from the concentrations obtained from the solution analysis using Eq. (1).

\[
S\% = \frac{c_i - c_f}{c_i} \times 100\%
\]

where \(c_i\) = initial concentration and where \(c_f\) = final concentration of iodide/iodate in the solution.

In the case of radioactivity measurements, the sorption percentages were counted from the net count rates using Eq. (2).

\[
S\% = \frac{R_i - R_f}{R_i} \times 100\%
\]

where \(R_i\) = initial net count rate and where \(R_f\) = final net count rate in the solution.

Column experiments

Column studies were performed with a simulant containing a high excess of SO₄²⁻, Cl⁻ and NO₃⁻ compared to iodine species (I⁻, IO₃⁻) concentrations. A self-prepared simulant with a simplified composition of Artificial Seawater ASTM D1141-98 [23] was used, described in Table 1.

In total, two identical column experiment sets were run with five different I⁻/IO₃⁻ concentration ratios in normal uncontrolled laboratory atmosphere. The columns were packed into open-cut 1 ml polyethylene disposable pipettes (i.d. ~4 mm) (Fig. 1) with a slurry containing 0.2 g of Ag-GAC and 5 ml of 10 mM NaNO₃. After the preparation, 5 ml of test solution was let to flow through the column to replace the solution remaining from the packing before collecting a 10 ml sample to a separate vial. The average flow velocity was 2.7 ± 1.0 ml/min. Samples of test solutions were analyzed with HPLC-ICP-MS before and after the contact

| Component | Concentration (mM) |
|-----------|-------------------|
| Na⁺       | 480               |
| K⁺        | 10                |
| Mg²⁺      | 55                |
| Ca²⁺      | 10                |
| Cl⁻       | 560               |
| Br⁻       | 1                 |
| SO₄²⁻     | 30                |
| pH        | 7.7               |

Table 1 The composition of the artificial sea water used in the experiments
with column and sorption percentages were calculated with Eq. (1).

**Results and discussion**

**Iodine speciation manipulation**

Iodine speciation is a crucial factor affecting the element’s chemical behaviour and thus its behaviour in purification processes and fate in environment. Two procedures for iodine speciation manipulation were used to change the oxidation state of iodine. The procedures were tested with $^{127}$IO$_3^-$ and $^{127}$I$^-$ to ensure the suitability for the redox manipulation of radioactive $^{125}$I. For $^{127}$I, solid starting materials KIO$_3$ and KI are readily available but for radioactive tracers the iodine is quite often only available in the form of K$^{125}$I. Table 2 shows the concentrations of both I$^-$ and IO$_3^-$ species for all the samples with different concentration of redox agents NaOCl and NaHSO$_3$ used for oxidation and reduction, respectively, of iodine. It seems that oxidation in basic conditions is reproducible as > 95% of I$^-$ was oxidized to IO$_3^-$ in all of the samples.

What should be noted, is that also raising concentrations of I$^-$ were detected with rising concentration of NaOCl which seems a bit controversial. It might also be just measurement artefact. On the other hand, reduction of IO$_3^-$ to I$^-$ worked fine although decreasing efficiency was observed with rising concentration of NaHSO$_3$. One reason could be stabilization of molecular iodine with the decreasing pH [24]. No extra peaks were detected in chromatograms, although the chromatogram for a sample after the reduction of IO$_3^-$ to I$^-$ showed tailing (see supplementary information).

**Batch experiments**

The effect of solution matrix elements on iodine uptake of Dowex 1X4 Strong Basic Anion exchanger and SilCarbon Ag-GAC were studied with a series of batch experiments. For Ag-GAC, also the effect of pH was studied. The experiments with Dowex were done only with macroscopic iodine concentrations ($8 \times 10^{-7}$ M), while the experiments with Ag-GAC were done with both macroscopic and trace iodine concentrations using radioactive $^{125}$I$^-$ and $^{125}$IO$_3^-$ ($\sim 10^{-13}$ M).

Dowex 1X4 Strong Basic Anion exchanger showed considerably higher affinity towards I$^-$ than IO$_3^-$ (Fig. 2) as expected, but in low matrix concentrations also notable fraction of IO$_3^-$ was removed from the solution. With raising matrix concentrations, the uptake of both iodine species declined, the effect being more drastic for IO$_3^-$ . The results show that Dowex Strong Basic Anion Exchanger does not show enough of selectivity to either of iodine species (I$^-$, IO$_3^-$) to be used as analytical tool for $^{125}$I in the conditions of the conducted batch experiments.
The identical experiments in macroscopic and trace iodine concentrations were done with Ag-GAC (Fig. 3). The I$^-$ removal was almost 100%, while the uptake of IO$_3^-$ was negligible in the range of 1-100 mM for all competing ions. Even high concentration of another halide, Cl$^-$, did not reduce the I$^-$ uptake significantly, as it stayed as high as 97.3 ± 0.1% even in 1000 mM Cl$^-$ matrix. In MilliQ water, the uptake was lower, although the samples showed a high variation. We suspect this is due to the degradation of the Ag-GAC material itself in such a low ionic strength solution but was not furthermore studied because such a pure analysis matrix is not relevant from the method application point-of-view.

The experiments with trace concentration of iodide showed somewhat different results (Fig. 4a) compared to macroscopic concentrations. In MilliQ water the iodide removal was 97.3 ± 1.7% but it decreased to 71.8 ± 2.0% in 1000 mM Cl$^-$. With other ions and in lower Cl$^-$ concentrations the removal percentages were rather same regardless of iodine concentrations.

In theory, the competition between Cl$^-$ and I$^-$ can be described by the Eq. (3).

![Fig. 2](image1.png) The sorption of I$^-$ (a) and IO$_3^-$ (b) sorption to Dowex Strong Basic Anion exchanger in different matrices. The iodine measurements were done with HPLC-ICP-MS and equilibrium pH's were 4.8–5.9. The error bars show the uncertainty of individual sample measurements caused by the uncertainty of the analysis method (see supplementary information for the detailed description). (n = 1)

![Fig. 3](image2.png) The effect of concentration of different competing ions to $^{127}$I$^-$ (a) and $^{127}$IO$_3^-$ (b) sorption (total iodine concentration $8 \times 10^{-7}$ M) to Ag-GAC. The iodine measurements were done with HPLC-ICP-MS and equilibrium pH's were 7.2–8.5. Results represent the mean values of parallel samples and error bars show the standard deviation. (n = 3)
AgCl(s) + I⁻(aq) ⇌ AgI(s) + Cl⁻(aq)  

(3)

The reaction occurs because of much lower solubility of AgI compared to AgCl but only proceeds as long as Cl⁻/I⁻ molar ratio is less than 2.1 × 10⁶ [25]. In the case of the batch experiments of our current study, the Cl⁻/I⁻ ratio was 1 × 10¹³ in 1000 mM Cl⁻ solution. This is several orders of magnitude over the equilibrium constant of Eq. (3) and no AgI should precipitate. However, iodide adsorption can occur even in these unfavourable Cl⁻/I⁻ ratios because of formation of mixed crystal silver halides [16]. AgI and AgCl form mixed crystals to the extent of 1–5 mol % [26, 27], which is much higher compared to the Cl⁻/I⁻ ratios in our study and can explain the iodide sorption in the conditions of the batch experiments.

Regardless of the matrix solution, the results looked similar for ¹²⁵I₂Ο₅⁻ (Fig. 4b) as the results of experiments with macroscopic IO₃⁻ concentrations.

The effect of pH on IO₃⁻ removal was studied in a solution containing 10 mM of all NaCl, NaNO₃ and Na₂SO₄ (Fig. 5). At very low pH, IO₃⁻ concentrations decreased (pH 3.0: 45%, pH 3.5: 14%) and this is most probably explained by the formation of volatile I₂. In samples with a pH value higher than 5.0, where the probability of IO₃⁻/I₂ transition is not favoured, negligible (or very low) iodate removal was observed. In the case of I⁻, no iodide was detected on pH range between 2.9 and 9.4 (18 samples, not shown in graph).

**Column experiments**

The batch experiments showed high selectivity of Ag-GAC to I⁻ species over IO₃⁻. In these experiments the contact time was rather long, 24 h, and system was expected to be in equilibrium. To evaluate the performance in dynamic conditions more relevant to speciation determining applications, a series of column experiments were carried (Fig. 6). With a
as 1000 mM Cl\(^-\) did not interfere the I\(^-\)/IO\(_3\)\(^-\) separation. In \(\mu\)M iodide concentration level even as high reaction that is needed for analytical applications. Compared with other studies regarding iodine removal from aqueous solution (i.e. refs. [8] and [9]), this approach only offers a simple way to perform I\(^-\)/IO\(_3\)\(^-\) separation between the two iodine species.

Conclusions

We have shown that Ag-GAC can be readily used in quantitative separation of I\(^-\) and IO\(_3\)\(^-\) species. Iodide is adsorbed efficiently on the material while iodate remains in solution. Iodide and iodate species fractions can be easily calculated if the total iodine concentration is known before and after the contact. However, in simultaneous high Cl\(^-\) concentrations (> 100 mM) and trace level iodide concentrations, also the competition between Cl\(^-\) and I\(^-\) should be taken into account. In \(\mu\)M iodide concentration level even as high as 1000 mM Cl\(^-\) did not interfere the I\(^-\)/IO\(_3\)\(^-\) separation. Compared with other studies regarding iodine removal from aqueous solution (i.e. refs. [8] and [9]), this approach only removes iodide and not iodate, offering a tool for the separation between the two iodine species.

Chemically stable and commercially available Ag-GAC offers a simple way to perform I\(^-\)/IO\(_3\)\(^-\) species separation in variable matrices, even in the case of high salt content. No special equipment for separation is needed as long as total iodine concentration can be measured by some analysis method. Especially suitable this method is in the case of using radioactive iodine tracers, i.e. \(^{125}\)I, which can easily be detected. Moreover, the proposed method can be used in the field to achieve the separation of iodine species right after the sampling without the risk of sample alteration because of uncontrolled reduction and oxidation reactions between the sampling and the analysis. This could be beneficial even with non-radioactive iodine analysis.

The method relies on the highly favoured interaction between silver in activated carbon and iodide, while iodate does not have notable affinity towards the material. The separation was shown to be quantitative both in batch and column type separations. This study does not concern organic iodine compounds or molecular iodine. Another or modified approach is needed if those are expected to be present in samples. On the other hand, the proposed methodology can be readily combined with other separation techniques, e.g. liquid–liquid extraction, for comprehensive iodine speciation analysis even when other iodine species like organic iodine compounds and elemental iodine are present.

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