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Analysis Optimization of I^- and NO_3^- ions using UV-Vis Spectroscopy

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1. Introduction

Nuclear energy, as a carbon-free energy, is a promising energy source. However, fission products, such as radioactive iodine isotopes (especially for iodide, I^-), generated from the NPPs operation can have harmful effects on human health and the environment. To solve this problem, sorption was considered as an effective method for the I^- treatment and Layered Double Hydroxide (LDH) could be utilized. This material can capture I^- within its structure through anion-exchange; for example, I^- can be exchanged with nitrate (NO_3^-) present in the interlayer of the LDH [1]. To confirm this mechanism, it is necessary to analyze the concentrations of target ions (e.g., I^- and NO_3^-) in the solution before and after sorption. Ultraviolet-Visible (UV-Vis) spectroscopy can be employed for this analysis, as it allows for qualitative assessment of target species in aqueous solutions based on the absorbance measurements. While techniques such as inductively coupled plasma mass spectrometry (ICP-MS) and ion chromatography (IC) are typically used for quantitative analysis of ionic species in solutions, these processes require high costs, long-term analysis, and specialized expertise, making accessibility limited. In contrast, UV-Vis spectroscopy is widely used and more accessible than ICP-MS and IC. Therefore, in this study, single solutions (I^- and NO_3^-) and mixed solution ($I^- + NO_3^-$) were measured using UV-Vis spectroscopy and calibration curves were plotted. Subsequently, calibration equations of the single solutions were derived, allowing the estimation of the concentration of unknown solutions. Based on this result, analysis of UV-Vis spectroscopy was optimized to demonstrate the anion-exchange mechanism between NO_3^- in the LDH interlayer and I^-.

2. Materials and methods

2.1. Synthesis and sorption experiments

Before optimizing the analysis, the sorbent was synthesized to prepare unknown samples. CoAl LDH (M^{2+}:M^{3+} molar ratio of 2.5:1) was synthesized by stirring, adjusting the pH to 8 with 2 M NaOH, and aging for 72 hours. The mixture was reacted at 110°C for 72 hours using Parr vessel, followed by cooling, washing with deionized water (DIW), and drying at ambient temperature for 3 days. Structural properties were confirmed using X-ray diffraction and Fourier transform-infrared spectroscopy.

For I^- sorption, 0.2 g of CoAl LDH was mixed with 10 mL of 40 mM I^- solution and stirred at 200 rpm for 3 days. After reaction, the mixture was centrifuged, and the supernatant was collected and filtered using 0.45-μm syringe filters to obtain the pre-wash sample. A sequential washing process was performed by adding 10 mL of DIW, centrifuging, and filtering the supernatant through 0.45-μm syringe filters. This process was repeated four times to collect washed samples.

2.2. Analysis optimization

UV-Vis spectroscopy (UV-2600i Plus, SHIMADZU) is an instrument that measures the amount of light absorbed or transmitted by a sample at different wavelengths of ultraviolet and visible light.

The absorbance of UV-Vis spectroscopy software was set to 1 with a wavelength range of 190-800 nm, and the background was measured using DIW. Then, 0.01, 0.025, 0.04, 0.055, and 0.07 mM solution was prepared and measured to obtain the each absorbance. Subsequently, a calibration curve was plotted based on the maximum absorbance values corresponding to each concentration. The absorbance of NO_3^- and $I^- + NO_3^-$ solutions was measured using same above method.

The concentrations of the unknown samples were measured using the calibration equation obtained through the above process.

3. Results and discussion

3.1. Calibration Curve Development

The concentration of the stock solution was set within the range of 0.01 to 0.07 mM to observe single species solution peaks below an absorbance of 1. Typically, I^- exhibited two peaks at around 226 nm (Spot 1) and 194 nm (Spot 2). A calibration curve was plotted based on the maximum absorbance at each concentration, as shown in Fig. 1.

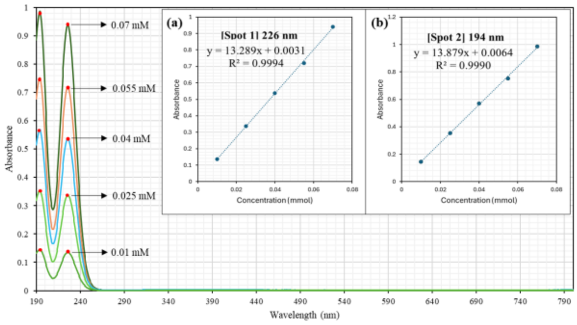

Fig. 1. Calibration curve of iodide (0.01 ~ 0.07 mM); inset information – (a) Spot 1 (226 nm) and (b) Spot 2 (194 nm)

Based on the calibration curve equation, y represents the absorbance value obtained from the unknown sample, and x denotes the I^- concentration. By measuring the absorbance of an unknown sample and substituting it into the calibration equation, the I^- concentration can be estimated. Meanwhile, the peak of NO_3^- was observed at around 203 nm (Spot 3). A calibration curve was plotted based on the maximum absorbance at each concentration, as shown in Fig. 2.

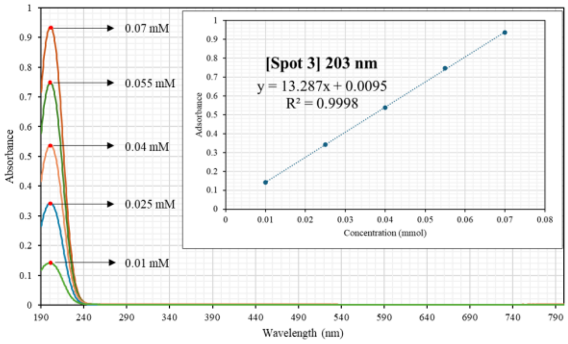

Fig. 2. Calibration curve of nitrate (0.01 ~ 0.07 mM); inset information – Spot 3 (203 nm)

3.2. Peak deconvolution

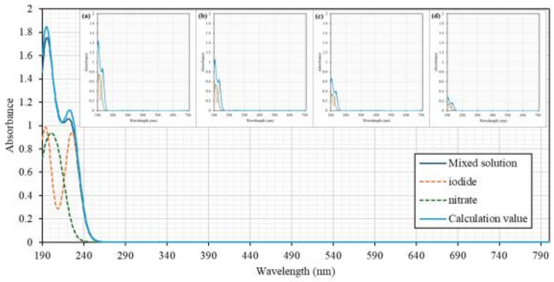

Fig. 3. Peak deconvolution of $I^- + NO_3^-$ solution (0.07 mM; inset information – (a) 0.055 mM, (b) 0.04 mM, (c) 0.025 mM, and (d) 0.01 mM

In the mixed solution, the peaks at around 223 nm (Spot 4) and 195.5 nm (Spot 5) were typically detected as shown in Fig. 3. However, the absorbance values of the mixed solution were 5-8% (avg: 6.78%) lower than that of the calculation values indicating the sum of single species cases.

Considering this variation, the analysis method was established to semi-quantitatively determine the concentrations of I^- and NO_3^- in unknown mixed solutions using the following procedure:

(1) Measure the absorbance of the unknown $I^- + NO_3^-$ solution using UV-Vis spectroscopy and obtain the absorbance values at Spot 4 and Spot 5.

(2) Multiply the absorbance values of the two peaks by a factor of (1 + 0.0678) to correct for the variation between the mixed solution and the calculated solution.

(3) Determine the I^- concentration in the mixed solution using the calibration equation of I^- for Spot 1.

(4) Calculate the expected absorbance for I^- at Spot 2 based on the obtained I^- concentration by (3).

(5) Since the measured absorbance at Spot 2 includes contributions from both I^- and NO_3^-, subtract the calculated I^- absorbance to separate the absorbance of NO_3^-.

(6) Determine the NO_3^- concentration using the Spot 3 derived from the single NO_3^- solution.

This study established an effective method for the separation and semi-quantitative analysis of I^- and NO_3^- concentrations in mixed solutions.

4. Conclusion

This study optimized I^- and NO_3^- peak deconvolution in UV-Vis spectroscopy. The UV-Vis spectroscopy techniques can provide semi-quantitative assessment methods compared to ICP-MS and IC and offer more cost-effective and accessible methods. As a result, this study can be expected to provide a strategy for relatively facile method for estimating concentration in aqueous solution.

REFERENCES

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