

Measurement of Trace Metals in Powder Activated Carbon (Pac) using Proton Induced X-Ray Emission (PIXE)

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We have used proton-induced x-ray emission (PIXE) to quantify ppm level of arsenic adsorbed in powder activated carbon (PAC), during the exposure of PAC to arsenic solutions. To evaluate different aspects of adsorption mechanisms in PAC, we have carried out equilibrium and kinetic adsorption experiments. After the exposure, arsenic concentration in adsorbent was quantified by means of PIXE (with 1.0 mm diameter ion beam). The As concentrations determined using PIXE compared favourably to those determined from inductively coupled plasma (ICP) measurements.

1. Introduction

Quantification of trace metals in any adsorbent directly with high accuracy and sensitivity is a challenging task. A rapid, high-sensitivity, non-destructive, quantitative, multi-elemental analytical method is best suited to investigate the issues associated with detecting trace elements. Proton induced x-ray emission (PIXE) is such an analytical tool and it is suitable for simultaneously quantifying trace elements with sensitivity of at least parts per million (ppm) [1]. In this study, trace arsenic concentrations in drinking water were investigated by PIXE. Since activated carbon (AC) can effectively remove hazardous trace elements from the drinking or wastewater, it is commonly being used to treat wastewater [2, 3]. As such, activated carbon is an excellent adsorbent for the investigation of the analysis of trace arsenic concentrations in water by characterisation techniques such as PIXE.

In this work, PAC has been used as an adsorbent to remove arsenic from drinking water. The amount of arsenic adsorbed was measured by PIXE. Various adsorption experiments were conducted to evaluate both the different aspects of adsorption mechanisms in PAC; and the detection limit and accuracy of PIXE measurements. The results are compared with those from inductively-coupled-plasma (ICP) measurements.

2. Experimental

We have carried out equilibrium and kinetic adsorption experiments to evaluate different aspects of adsorption mechanisms in PAC. In equilibrium experiments, different amount of PAC (between 0.1 and 5.0 g) was added to a solution with initial arsenic concentration of 1.0 mg and then mixed on a platform mixer for a period of 72 hours. In kinetic experiments, 5g of PAC was added to a solution with initial arsenic concentration of 1.0 mg and then agitated for between 5 minutes to 8 hours. After these treatments, the PAC was isolated from the solution and arsenic concentration in PAC was measured directly using PIXE.

PIXE measurements on these samples were carried out at Environmental Molecular Sciences Laboratory (EMSL) accelerator facility. The details of the accelerator facility and the end stations are described elsewhere [4]. The proton beam with energy of 2.5 MeV and 1.0 mm in diameter was used for our PIXE measurements. Theoretical simulations of PIXE experimental data were performed to determine elemental composition using the computer program GUPIX

[5]. These results were compared to the inductively-coupled-plasma (ICP) measurements. In addition, arsenic concentration in activated carbon was mapped out using micro beam PIXE with the beam size of 20-50 microns.

3. Results and Discussion

A typical PIXE spectrum from one PAC sample is shown in Fig. 1. All the characteristic peaks are identified. The area under each peak directly corresponds to the concentration of each element in the sample.

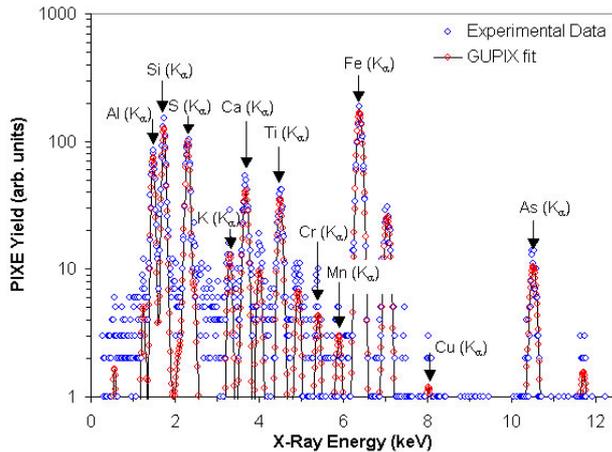


Fig. 1: A typical PIXE spectrum of PAC after an adsorption experiment. The 2.5 MeV proton beam was used for the PIXE measurements.

The results of equilibrium experiments are shown in Fig. 2. Arsenic concentration in PAC was also determined using ICP measurements after stripping all the arsenic from the PAC (ICP-direct). The concentration of remaining arsenic in treated solution was measured by ICP. The amount of arsenic adsorbed by PAC (o ICP) was calculated by subtracting arsenic concentrations determined in treated solution from the amount of arsenic present in the initial solution. The amount of arsenic adsorbed in PAC determined by PIXE agrees well with those measured by ICP-direct and ICP.

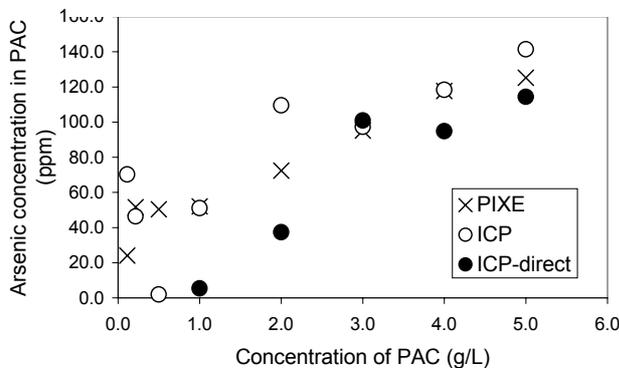


Fig. 2: PIXE and ICP measurements of arsenic concentration in PAC after the equilibrium experiments.

The results of kinetic experiments are presented in Fig. 3. As in the case of equilibrium experiments, after the treatment, the PAC was isolated from the solution and arsenic concentration in PAC was determined using PIXE and ICP measurements. As observed in the equilibrium experiments, there is a good agreement between the results from PIXE and ICP measurements.

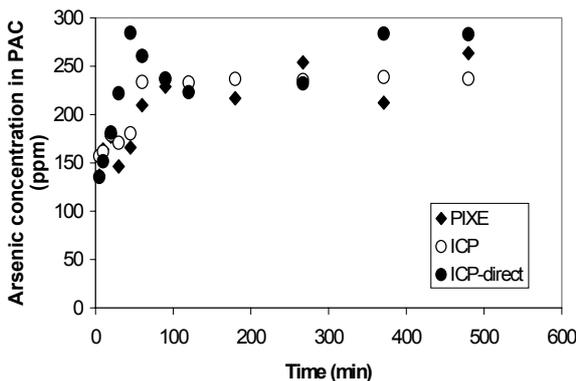


Fig. 3: PIXE and ICP measurements of arsenic concentration in PAC after the kinetic experiment.

Although there is a general agreement between the arsenic concentrations determined using PIXE and ICP measurements, some differences were observed in both equilibrium and kinetic experiments. This may be due to the fact

that the area on the PAC that the PIXE measurements are being taken may not be representative of the arsenic concentration in the whole sample. To understand the inhomogeneous adsorption of arsenic in activated carbon, micro beam PIXE measurements were carried on some of the activated carbon granules. The results from these measurements clearly showed that different regions within the each granule adsorb different amounts of arsenic (results are not provided). Therefore, the differences are mainly due to inhomogeneous structure of PAC. This includes the variable surface properties such as surface area and pore sizes in each particle. Different sampling techniques have to be employed to get better representation of arsenic concentration in these types of inhomogeneous samples.

4. Conclusion

PIXE technique can be used to determine the concentration of trace elements in adsorbent such as PAC. Inhomogeneous structure of PAC makes it difficult to make consistent PIXE measurements. Arsenic adsorption characteristics of PAC can be studied by fitting these experimental data with various isotherms including Freundlich [6] and Langmuir [7] isotherms and detailed analyses are in progress.

Acknowledgments

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References

- [1] G. W. Grime, *X-ray spectrometry* 27 221-231 (1998).
- [2] J. Pattanayak, K. Mondal, S. Mathew, S.B. Lalvani, *Carbon* 38 (2000) 589-596.
- [3] U.S. Army Corps of Engineers (2001), Adsorption design guide DG 1110-1-2. www.usace.army.mil/inet/usace-docs/design-guides/dg1110-1-2/toc.pdf (1 March 2001).
- [4] S. Thevuthasan, C.H.F. Peden, M.H. Engelhard, D.R. Baer, G.S. Herman, W. Jiang, Y. Liang, and W.J. Weber, *Nucl. Instr. Meth. A* 420 81-89 (1999).
- [5] J. A. Maxwell, J. L. Campbell, W. J. Teesdale, *Nucl. Instr. & Meth. B* 43 218-230 (1989)
- [6] H. Freundlich, Uber die Adsorption in Losungen, *Z. Phys. Chem.* 57 385 (1907)
- [7] Langmuir, The adsorption of gases on planes of glass, mica and platinum. *J. Am. Chem. Soc.* 40 1361 (1916).