MICROEXTRACTION TECHNIQUES COMBINED WITH X-RAY FLUORESCENCE SPECTROMETRY FOR DETERMINATION OF DIFFERENT METAL IONS IN WATER

UNIWERSYTET ŚLĄSKI W KATOWICACH

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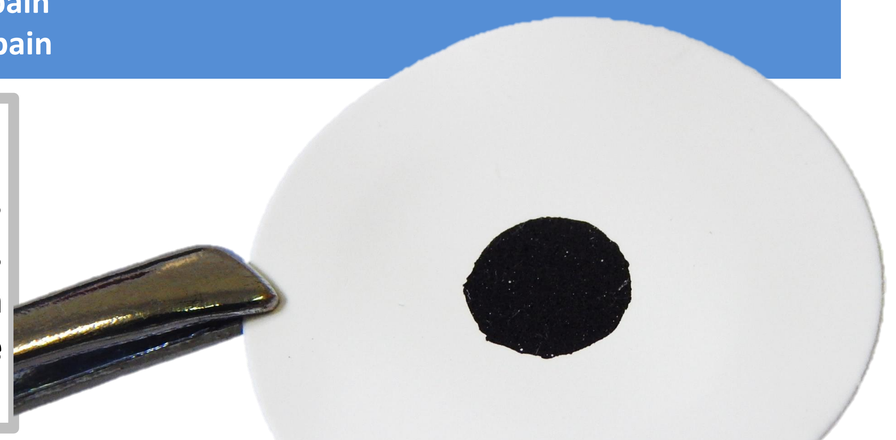
AIM

Direct XRF analysis of liquids does not allow the determination of trace and ultratrace elements. Therefore, an appropriate preconcentration procedure is necessary before the measurement. This work deals with the development of new analytical procedures enabling preconcentration and determination of a number of trace elements in water samples using combination of the microextraction and XRF techniques.

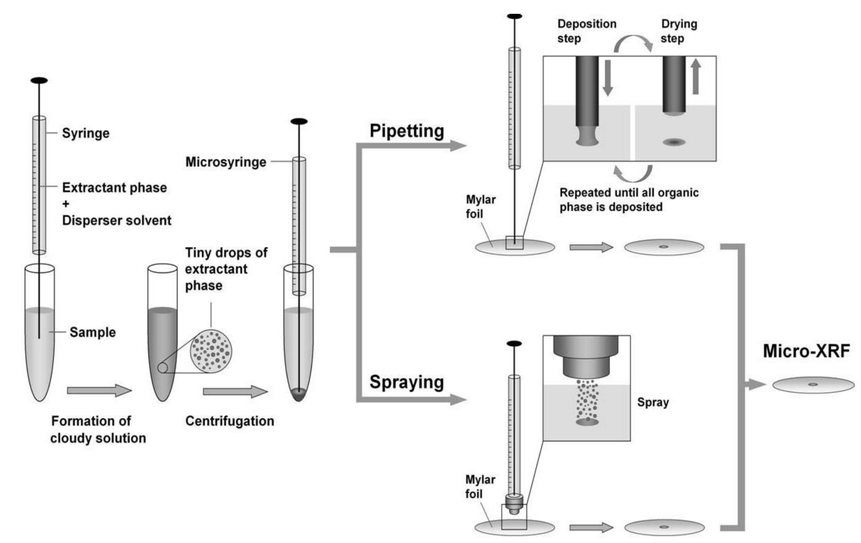


Preconcentration procedures

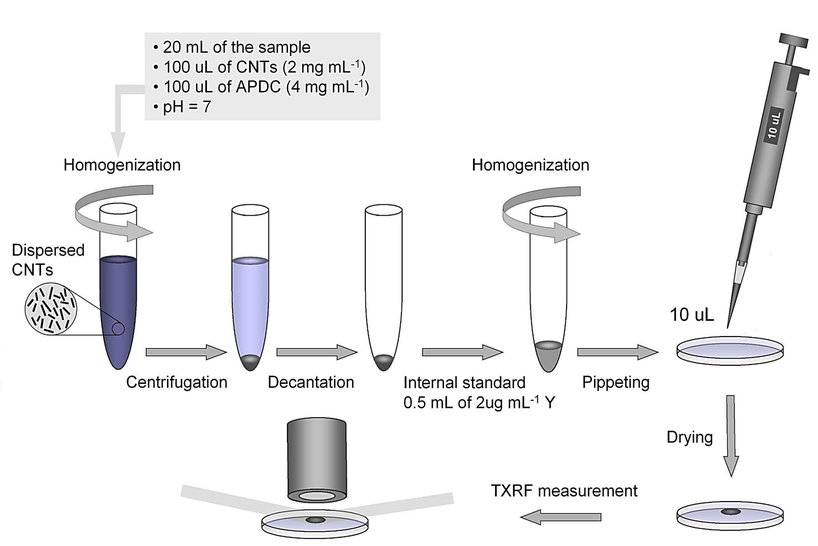
Proposed in this work procedures combine:

- **Dispersive liquid-liquid microextraction (DLLME) and energy-dispersive X-ray fluorescence spectrometry (EDXRF) for simultaneous determination of Fe, Co, Ni, Cu, Zn, Ga, Se and Pb ions in river water samples.**
- **Dispersive micro solid-phase extraction (DMSPE) and total-reflection X-ray fluorescence spectrometry (TXRF) for the determination of Cd and Pb ions in river, sea and tap water samples (sorbent: multi-walled carbon nanotubes)**
- **DMSPE and EDXRF for determination of Co, Cu, Ni and Pb ions and Se speciation in tap, lake and sea waters (sorbent: graphene).**

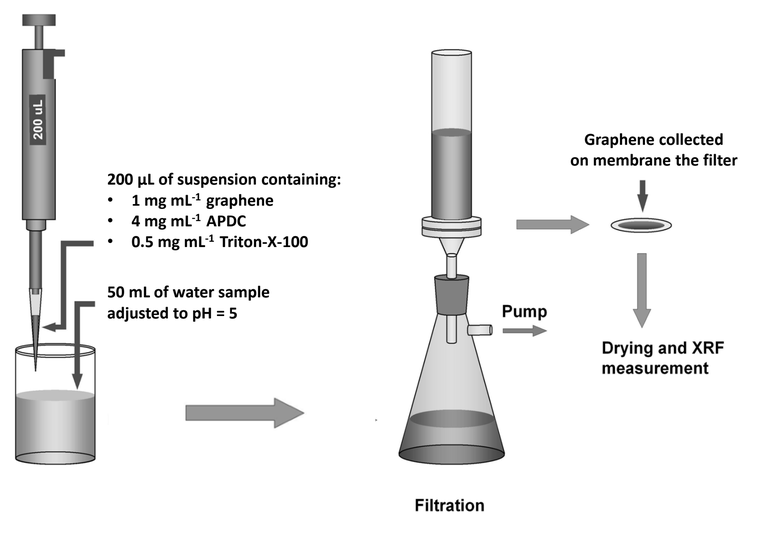
DLLME/EDXRF



DMSPE/TXRF



DMSPE/EDXRF



Obtained results

Analytical figures of merit of the proposed procedures

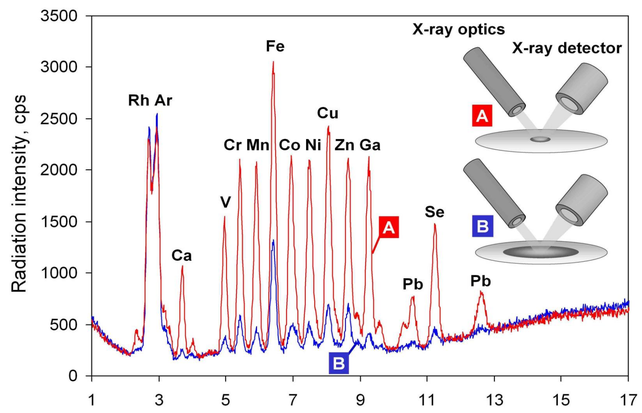
	DLLME/EDXRF	DMSPE/TXRF	DMSPE/EDXRF
Linearity (ng mL^{-1})	up to 400	up to 50	up to 100
DL (ng mL^{-1})	1.6 (Co) - 4.1 (Pb)	1.0 (Cd) - 2.1 (Pb)	0.23 (Co) - 1.1 (Pb)
RSD	7.5% (Co) - 14.4% (Se)	6.0% (Cd) - 10.5% (Pb)	2.6% (Ni) - 3.4% (Cu)
EF	250 for 5 mL sample	40 for 20 mL sample	418 (Pb) - 2553 (Cu)
extractant/adsorbent	**30 µL of CCl_4**	**200 µg of MWCNTs**	**200 µg of graphene**

Analysis of mineral water with DMSPE/EDXRF procedure

Sample	Element	Added, ng mL^{-1}	Found, ng mL^{-1}	Recovery (%)
Mineral water	Co	0	< DL	
		20	21.0 ± 0.39	104.9
		50	53.9 ± 0.60	107.8
	Ni	0	< DL	
		20	20.1 ± 0.47	100.3
		50	51.2 ± 0.85	102.4
	Cu	0	6.8 ± 0.31	
		20	26.4 ± 2.0	98.0
		50	59.3 ± 3.4	105.0
	Pb	0	< DL	
		20	20.2 ± 1.3	100.8
		50	48.6 ± 3.2	97.2

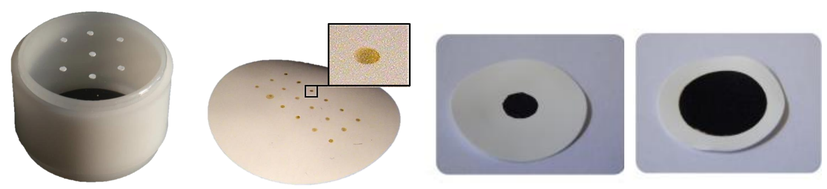
Benefits of the proposed preconcentration procedures

Improved sensitivity and DL



EDXRF spectra of two reference samples containing 0.5 µg of each element. Samples A and B of various diameters (ca. 1 and 5 mm, respectively) deposited onto the Millipore filter are excited with an X-ray beam of 900 µm focus spot size.

Nondestructive analysis



Unknown samples and more importantly calibration samples can be nondestructively measured with high precision many times.

Conclusions

The obtained results show that combination of microextraction techniques with XRF spectrometry allows low detection limits to be obtained in a very simple, non-time consuming and low-cost way. The advantage of this combination is the possibility of performing multielement analysis.

Acknowledgments

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