

Feasibility of high purity flexible graphite digestion using pyrohydrolysis for further chlorine and sulfur determination by IC

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Introduction

The flexible graphite (FG) has attractive properties as resilience, impermeability to fluids, chemical inertness and high-temperature stability. FG is used in different applications as gasket, packing and sealing in different industries. In nuclear industry, this material has been used because shows high resistance to nuclear radiation. During FG production many non-metallic impurities can be introduced during production of this material. The most important non-metallic impurities are chlorine (Cl) and sulfur (S) that are present in FG material due to residual amount of decomposed intercalate. Additionally, Cl and S cause corrosion of metallic tubes by permeating stainless steel chromium oxide film.¹ In this sense, Cl and S should be determinate in FG material used for nuclear applications. However, specific method for Cl and S determination in FG material is not available in literature. The objective of this work was to develop a pyrohydrolysis method for further determination of Cl and S by IC.

Results and Discussion

Some pyrohydrolysis parameters such as influences of air and water flow-rate through the reactor, reaction time, absorbing solution, use of V₂O₅ and sample mass were evaluated. For initial studies FG masses of 250 mg and 10 min of reaction time were used. In agreement with previous work published in literature pyrohydrolysis reaction was carried out using 1100 °C.² Maximum air and water flow-rate pumped through the reactor of 0.1 L min⁻¹ and 0.5 mL min⁻¹, respectively should be used to minimize that sample was carried together with air and water flow-rate. Pyrohydrolysis reaction time of 30 min was adopted for total volatilization of analytes (Figure 1). Quantitative recoveries of Cl and S were only achieved for 250 mmol L⁻¹ of H₂O₂. Use of H₂O₂ was necessary for complete conversion of S compounds to sulphate. The use of V₂O₅ as accelerator for pyrohydrolysis reaction was not necessary. For comparison, Cl and S were also determined in digests obtained using microwave-induced combustion (MIC). Statistical differences between pyrohydrolysis and MIC results were not

observed. Limits of detection (LOD) of 1.0 µg g⁻¹ were found for both Cl and S using IC analysis.

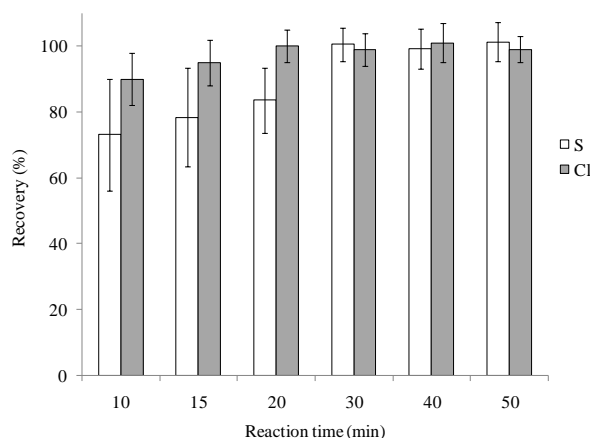


Figure 1. Evaluation of reaction time for volatilization of S and Cl from FG by pyrohydrolysis. (n = 5, mean ± standard deviation).

Accuracy was also evaluated using certified reference material (CRM) of coal (SARM 19) and agreement better than 95% was obtained.

Table 1. Results obtained for determination of Cl and S using pyrohydrolysis and MIC (n=3)

	Pyrohydrolysis (µg g ⁻¹)		MIC (µg g ⁻¹)	
	Cl	S	Cl	S
FG I	35 ± 3	880 ± 40	38 ± 6	890 ± 45
FG II	< 1.0	375 ± 25	< 5.0	400 ± 31
FG III	< 1.0	398 ± 20	< 5.0	350 ± 30
FG IV	< 1.0	320 ± 24	< 5.0	310 ± 28
SARM 19	35 ± 3	5045 ± 825	34 ± 7	5045 ± 987

SARM 19: 4900 ± 700 µg g⁻¹ for S and 32 µg g⁻¹ for Cl (certified values).

Conclusions

Pyrohydrolysis was a suitable sample preparation method for quality control of Cl and S in high purity FG samples. Use of H₂O₂ was necessary for complete recovery of sulfur for IC analysis.

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