EVALUATION OF DIFFERENT METHODOLOGIES FOR THE ANALYSIS OF TOXIC ELEMENTS IN POLYMER SAMPLES BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY

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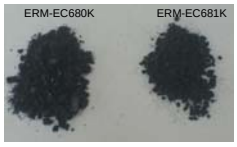
INTRODUCTION

Current European standard regulate the content and migration levels of heavy metals in different polymer products. Previous interlaboratory studies have shown that these methodologies lack of accuracy and reproducibility. In one hand, sample preparation (i.e. digestion/extraction) is not reproducible. In the other hand, the spectroscopy techniques recommend (i.e. Flame or Hydride Generation Atomic Absorption Spectrometry) are time consuming since just one element can be determined by experiment. In addition, information about matrix effects and calibration strategies are unknown and the analyte concentration in polymeric materials is too close, or even lower that the detection limits of the analytical techniques recommended. Recently, Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) has been evaluated as detection technique for heavy metals in polymeric samples. The results obtained shows that ICP-AES fulfils the requirements of this type of analysis but a careful selection of the plasma view mode (i.e. axial or radial) and the sample introduction system is required. However, current drawbacks of sample preparation treatments remain unsolved. The aim of this work is to evaluate and improve current methodologies for the digestion of polymeric samples. To this end, two different microwave ovens (MSP-1000 and Mars 5) were employed for the digestion of heavy metal certified polyethylene samples. Parameters studied were sample amount, temperature, pressure, time and acid concentration. Digested samples were analyzed by means ICP-AES.

POLYMER DIGESTION

Metal content certified LDPE samples



Home-made PVC samples

- 1.00 g sample
- 5 mL H_20
- 5 mL (1/3 65% HNO_3+2/3 36% HCl)

CEM-MDS-1000

- 0.25 g sample
- 8 mL 65% HNO_3

CEM-MARS 5

Table 1. LDPE and PVC digestion program for MSDS-10000 MW

Step	Power (W)	Temperature (°C)	Time (min)	Max pressure (psi)
1	430	–	4	0
2	-	–	1	0
3	360	–	4	0
4	500	–	10	40
5	500	–	10	80
6	500	–	10	120
7	500	–	10	160

Table 2. LDPE and PVC digestion program for MARS 5 MW oven.

Step	Power (W)	Temperature (°C)	Time (min)	Max pressure (psi)
1 (Ramp)		0-205	20	260
2		205	15	260

The higher temperatures and pressures generated by Mars 5 when compared to MDS-1000 improves polymer digestion efficiency and speed

INCOMPLETE SAMPLE DIGESTION!!

COMPLETE SAMPLE DIGESTION!!

ICP-AES ANALYSIS

Digested samples

Conventional sample introduction system (CS)

Hydride generation (HG)

ICP-AES

Analyte recovery depends on the sample introduction system!

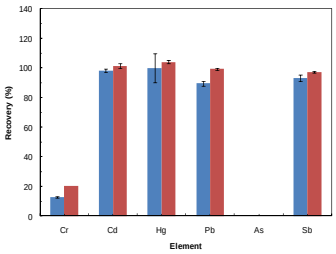


Figure 1. Analyte recovery in metal certified LDPE samples with a CS. (■) ERM-EC680K; (■) ERM-EC681K

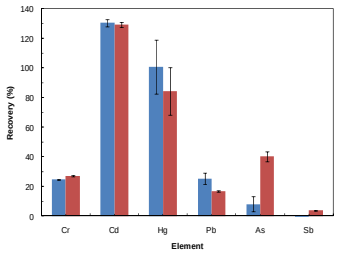


Figure 2. Analyte recovery in metal certified LDPE samples with a HG system. (■) ERM-EC680K; (■) ERM-EC681K

Analyte recovery depends on the additive employed!

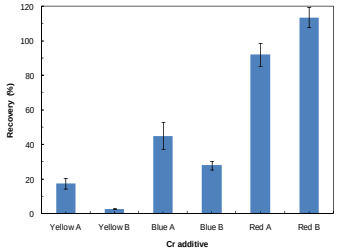


Figure 3. Influence of the chromium additive on the analyte recovery in home-made PCV samples with a CS

CONCLUSIONS

- **POLYMERIC MATERIALS** ARE **EFFICIENTLY DECOMPOSED** BY MEANS OF A **MW DIGESTION** TREATMENT. SAMPLE DIGESTION STRONGLY **DEPENDS ON** THE MW **OVEN CHARACTERISTICS** AND ON THE **EXPERIMENTAL CONDITIONS** EMPLOYED
- **METALS IN PLASTIC** MATERIALS ARE EASILY **DETERMINED BY ICP-AES**. **ANALYTICAL RESULTS** STRONGLY **DEPENDS ON** THE **SAMPLE INTRODUCTION SYSTEM** AND ON THE **ADDITIVE USED**

ACKNOWLEDGEMENTS

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