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THE DEVELOPMENT OF PLANTS EXTRACTION METHOD FOR GAS CHROMATOGRAPHIC DETERMINATION OF PCBs

Diana Puiu, Mariana Popescu, Marcela Niculescu, Madalina Mihalache, Luoana Florentina Pascu, Vasile Iancu

National Research and Development Institute for Industrial Ecology-ECOIND, 71-73
Drumul Podu Dambovitei, district 6, 060652, Bucharest, diana_puiu@ymail.com,
Romania

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Introduction

Polychlorinated biphenyls (PCBs) are classified by International Agency for Research on Cancer (IARC) as persistent organic pollutants, carcinogenic to human organism. Due to their high liposolubility PCBs can easily enter in food chain and bioaccumulate in adipose tissue. Atmospheric deposition of PCBs generated by waste combustions represents a risk of contamination over large areas.

The half-lives of PCBs in soil between 1-20 could beslowly diminished to 50 days – 11 years by plant uptake or by rhizoremediation with specific enzymes.

Organic pollutants determination from complex plant matrix requires the development of a method which is characterized by a linear range, a proper quantification limit, good precision and accuracy. The factors that affect the loss of PCBs, like soil biodegradation, volatilization of less chlorinated compounds, the activity of roots enzymes and the uptake to plant must be carefully analysed. The storage of these toxic compounds varies with different plant species. Besides crops, the contamination risk of medicinal plants is less studied.

This work is devoted to the development of an extraction method of various tetra-heptachlorinated biphenyls (PCB28, PCB52, PCB138, PCB153, PCB 180) which are retained in medicinal plants. The method is desired to be less time consuming, simplest and reliable. In order to ensure the method suitability for PCBs extraction, the results were evaluated in terms of quantification limit, relative standard deviation and recovery.

Materials and methods

The PCBs determinations from soils and plants were performed by gas chromatographic analysis of residues extracted through solid-liquid technique. Before extraction, the samples were milled and properly homogenized. Due to high sensitivity of capture electron detector to halogenated compounds, the purifying method of extracts was investigated in order to remove the interfering compounds: by separating 5 mL of extract solubilized in hexane on a column filled with 3 g of basic alumina and 1 g of anhydrous sulfate or by oxidation with 8 mL H₂SO₄ 96%.

Results and conclusions

The soil and plant samples extracted with hexane: acetone 50:50 volume ratio by 30 min sonication were colorful and have suspensions matter, which makes the extracts unsuitable for GC analysis. The presence of various hydro and liposoluble chemical

compounds (ex. carotenoids, polyphenolcarboxylic acids, flavonoids, volatile oils, etc) decrease the PCBs peaks resolution and intensity in chromatogram.

The coloured extract shows that purification is ineffective on smaller amounts of alumina or acid. The differences between the methods are observed into the chromatograms, where as is seen in Figure 1 the extracts purified with alumina was more clear of unknown peaks, while interferences degradation with highly oxidative H₂SO₄ 96% generate compounds with smaller molecular mass which can be detected on ECD. Instead, the response signal is 2 times higher on sulphuric method, but the recoveries for selected PCBs was the same (Figure 2). It was found that while the method sensitivity increase with PCBs chlorine atom numbers, the recoveries decreased to 50%. Combining the alumina method with H₂SO₄ had a decreasing effect on PCBs recoveries for at least 10%.

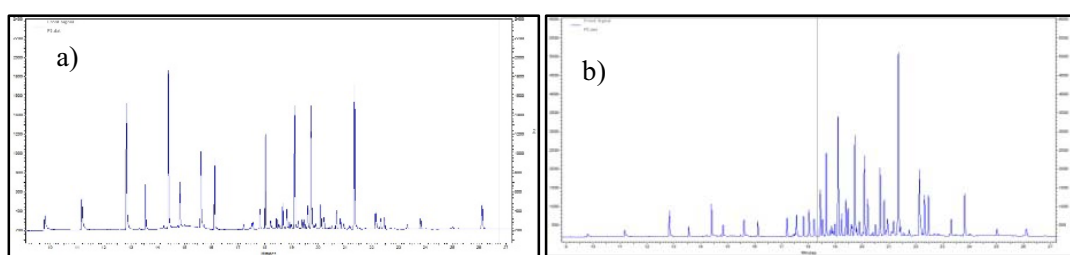


Figure 1. Chromatogram of PCBs extract purified with a) basic alumina and b) H₂SO₄ 96%

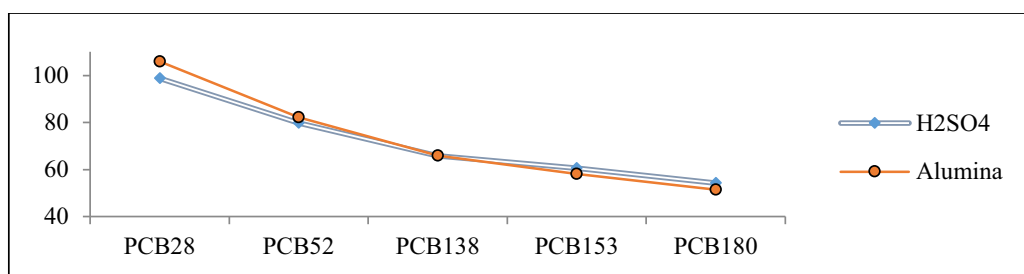


Figure 2. Recoveries of selected PCBs for the 2 methods

It was established that based on a signal-to-noise ratio value higher than 10, the limit of quantification was higher than 0.004 mg / kg for 0.5-4 g root, stem and leaves samples. The peaks are characterized by a gaussian form and good resolution. From the experimental data was observed that duplicate plants samples have relative standard deviations up to 80%. This fact is attributed to PCBs distribution in soil which differentiate the quantitative uptake in plants, rhizomerization processes, roots surface area, plants uptake in time and extraction method, processes which are shaped by physico-chemical proprieties of PCBs. Also, it was evidenced that these compounds are not evenly distributed into the plant tissue as are systemic pesticides. It was assessed the variation between the PCBs number of chlorine atoms and compounds uptake in plant and compared the calculated bioconcentration factor with other plants.

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