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Electro Analysis of Lead and Cadmium in Vegetables

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Abstract: Heavy metal contamination in vegetables is a growing concern due to its adverse effects on human health. In this study, an electro analytical approach using Differential Pulse Polarography was employed for the simultaneous determination of lead (Pb) and cadmium (Cd) in vegetable samples. The samples were pretreated through acid digestion to break down organic matter and release metal ions into solution. The polarographic analysis was conducted using a dropping mercury electrode, where current responses were recorded as a function of applied potential. Well-defined peaks corresponding to Pb^{2+} and Cd^{2+} were obtained, allowing their identification and quantification. Calibration and standard addition methods were used to ensure accuracy and minimize matrix effects. The method exhibited excellent sensitivity, reproducibility, and low detection limits. The study confirms that differential pulse polarography is an effective and reliable technique for monitoring toxic metals in food samples and contributes to food safety assessment. Differential Pulse Polarography is applied to simultaneous determination of lead and cadmium in dry ash vegetables in nitric acid at 450±50°C. Determination of the elements was made in acetate buffer (pH 4.7) and HCl (pH=2) with a scan rate of 60 mVs -1 and a pulse height of 50 mV by Mercury Dropping Electrode. The solution was stirred during pre-electrolysis at -1150 mV (vs. Ag/AgCl) for 90 s and the potential was scanned form -1150 mV to +100 mV(vs. Ag/AgCl). Precision of the method, expressed by relative standard deviation for lead and cadmium, was calculated as 4.5% and 3.5%, respectively. Under these conditions, the limits of detection were 0.005 and 0.005 mg/kg for lead and cadmium, respectively. In our study the concentrations of lead was found significantly higher than those of standard and labeled values (P<0.001), respectively.

Keywords: Trace element; Lead; Cadmium; Differential pulse polarography

Introduction

Heavy metal contamination in food has become a significant environmental and public health concern in recent years. Among various contaminants, lead (Pb) and cadmium (Cd) are particularly hazardous due to their high toxicity, non-biodegradability, and ability to accumulate in living organisms. Vegetables, being an essential part of the human diet, can absorb these metals from contaminated soil, irrigation water, and atmospheric deposition. Continuous consumption of such contaminated vegetables may lead to serious health disorders, including Lead poisoning and Cadmium toxicity. Therefore, the detection and quantification of trace levels of Pb and Cd in vegetables are crucial for ensuring food safety and protecting public health. Various analytical techniques are available for heavy metal determination, but

electroanalytical methods have gained prominence due to their sensitivity, simplicity, and cost-effectiveness. One such technique is Differential Pulse Polarography, which is widely used for trace metal analysis. Differential Pulse Polarography is based on measuring current changes resulting from the reduction of metal ions at a dropping mercury electrode under controlled potential conditions. The technique offers high resolution and allows the simultaneous determination of multiple metal ions, as each metal produces a characteristic peak at a specific potential. This makes it particularly suitable for analyzing complex samples such as vegetable extracts. In this study, Differential Pulse Polarography is employed to determine the concentration of lead and cadmium in vegetable samples. The method provides accurate and reliable results, contributing to the monitoring and control of heavy metal contamination in food sources.

Trace elements play a very important role in human nutrition. They are essential to life, trace metals are useful at very low concentrations, but these are toxic at high concentrations. Cadmium and lead are heavy metals which are widely distributed, naturally occurring, and potentially toxic elements. With increasing industrial use, environmental pollution and associated toxic exposure, concern has increased about their long term exposure and potentially toxic effects on human health specially infants and young children which are at the pick of growth [1-3]. Since they are taken mostly from the diet, their determination in food is very important .The chemical state and concentrations of trace elements in biological material are such that different techniques and methods of analysis are usually required for their determination. Using electro thermal atomic absorption spectrometry (ETAAS)[4], Se in wheat, fruits, fish and meat has been analyzed[5]. Neutron activation techniques have high sensitivity, but they are not frequently used because of the specialized techniques, time and cost involved[6]. Other techniques such as atomic emission spectrometry with inductively coupled plasma excitation (AES-ICP) [7]and X-ray fluorescence (XRF) [8] are very expensive and do not offer sufficient sensitivity for accurate determination of trace elements and heavy metals detection in vegetable by Differential pulse polarography (DPP) [9]. Differential Pulse Stripping Voltammetry (DPSV) is relatively inexpensive and is one of the most sensitive and selective techniques in the determination of trace amounts of metals at natural levels[10-17]. Electrochemical methods have the advantage that they require relatively inexpensive instrumentation, have demonstrated ability for multi element determination[18-20]and are capable of determining elements accurately at trace and ultra tracelevels[21]. Cauliflower is one of the several vegetables in the species *Brassica oleracea*, in the family *Brassicaceae*. It is an annual plant that reproduces by seed. Typically, only the head (the white curd) is eaten while the stalk and surrounding thick, green leaves are discarded. Cauliflower is very nutritious, and may be eaten cooked, raw or pickled. Cauliflower samples have been analyzed for their trace element contents using DC. Polarography. The aim of this work was to determine the toxic elements in a certain type of cauliflower, which is a very important vegetable because of its high consumption in the India.

Experimental

Apparatus

A digital DC Recording polarograph CL-357 was used for the measurement of current-voltages data. This apparatus has three electrode assembly, dropping mercury electrode as working electrode, calomel as reference electrode and platinum as counter electrode. DC polarograms were

recorded by the Strip chart recorder LR-101P, under the conditions of 150 mV per minute span rate and 100 nA per division sensitivity. Elico digital pH meter was employed to measure the pH of solution.

Reagents

All reagents used were of analytical reagent grade purity (AR). The mercury used in the dropping mercury electrode was obtained from Merck. Standard stock solutions (0.025 M) of Pb and Co were prepared with triply distilled water from their nitrate and sulfate salts. The C-V data for test solution were recorded after passing pure nitrogen gas in the test solution and 0.001%triton-X-100 was used as maxima suppressor.

Glassware

All glassware were soaked in 2 M nitric acid for at least 7 days, washed three times with distilled deionizer water, soaked in 0.1 M hydrochloric acid until ready for use. In distilled deionizer water and finally soaked

Procedure

Sampling and digestion

The Brassica type Cole, which is commonly used in the Rajasthan (India) as the main vegetables, was collected in samples from the area of Karauli (Rajasthan India), at first separated as leaves and flower, cut into small pieces after washing and then dried in an oven at about 105 °C until constant weight. Then it's digested by dry ash method,

Electro analytical determination

A total of 10 mL electrolyte was de-aerated by a stream of nitrogen gas (99.999 %) for about 15 min. Polarograms were taken by scanning the potential in the negative direction from 0.0 to –1.5 V, depending on pH, at a scan rate of 5 mV/s. to the sample solution taken in Pyrex polarographic cell including 2.0 ml. of suitable buffer solution we add 0.1 ml. of 0.001% triton-X-100 and remaining required volume of distilled water. After that the polarographic cell was de-aerated by a stream of nitrogen gas for about 15 minutes. To ascertain the presence of the metal ions in the sample, a known quantity of stock standard solution of each metal ion was added to the analyte and polarograms were recorded. An increase in the wave height of the ion signal was observed without any change in its $E^{1/2}$ values confirming the presence of Pb, Cd, Ni and Zn in cauliflower sample solution.

Table 1. Trace analysis of Pb, Cd, Ni and Zn in Cauliflower Vegetable

Heavy metal	Supporting Electrolyte	Half wave Potential as E1/2 in volts	Conc. in ppm	Mean	% Error	Mean deviation	Std. deviation
Pb	Acetate buffer	0.42	5.993 6.001 6.674	6.189	0.0315	0.323111	0.423193
Cd	Acetate buffer	0.60	3.148 3.410 3.718	3.392	0.1396	0.229333	0.335362

Results and discussion

The results obtained from the study of toxic metals in *Brassica oleraceae ver.botrytis* (Cauliflower) in part per million ranges are presented in table 1. Which shows mean concentration in ppm as 6.189, 3.392, and percentage error is small as 0.0315, 0.1396, respectively for Pb and Cd, in acetate buffer and HCl buffer. Mean deviation is found to be 0.323111, 0.229333, and standard deviation is found to be 0.423193, 0.335362, respectively for Pb, and Cd, Linearity of calibration curves was obtained in all cases with the value of correlation factor (r) near to one. Linear relationship between concentration and diffusion current (Id) has been proved statistically by applying straight line equation to all calibration curves.

Heavy metals are important environmental pollutants they are a threat to the environment and to human health, because they are not biodegradable as they are retained indefinitely in the ecological systems and in the food chain . The allowed values of metals such as Pb 0.030ppm, Cd 0.001 ppm, are not harmful, but whenever the concentration of these metals exceeds from this value, it may pose harmful effects on our body .

Conclusion:

The described DCP method for the determination of Pb, Cd, Ni and Zn in vegetables is specific, sensitive and rapid with a simple approach comprising low cost instrumentation compared to the mass spectrometry and atomic absorption spectrophotometry. The results obtained by DCP are quantitative and in good agreement in terms of precise measurement.

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