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Electro analytical Procedure for determination of Heavy Metals in Brassica oleraceae ver. Botrytis

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Abstract: A fast and simple DC Polarographic method for the determination of heavy metals in a certain variety of cauliflower (*Brassica oleraceae ver.botrytis*) is described. This vegetable is commonly used in India. Using DC polarograms of dry digested cauliflower samples (flower), in acetate buffer (pH=4.7) and HCl (pH=2), quantities of Pb, Cd, Ni and Zn were determined. Half wave potentials were -1.10 V, -0.60 V, -0.42 V and -0.91 V for zinc, cadmium, lead and nickel, respectively. The heavy elements quantities in digested flower samples were as follows: Pb about 6.189 ppm, Cd 3.392 ppm, Ni 4.707 ppm and Zn 5.899 ppm.
Key Words: Cauliflower, Heavy metals (Pb, Cd, Ni, and Zn), DC polarography.

Introduction

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[21-23]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, Co, Ni, and Zn 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 Amanisha nala near Gurjar ki Thadi Jaipur city (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	5.893 6.001 6.674	6.189	0.0315	0.323111	0.423193
Cd	Acetate buffer	0.60	3.048 3.410 3.718	3.392	0.1396	0.229333	0.335362
Ni	Acetate buffer	0.91	4.461 4.700 4.961	4.707	0.1414	0.169111	0.250081
Zn	HCl	1.10	5.741 5.873 6.084	5.899	0.1499	0.123111	0.17301

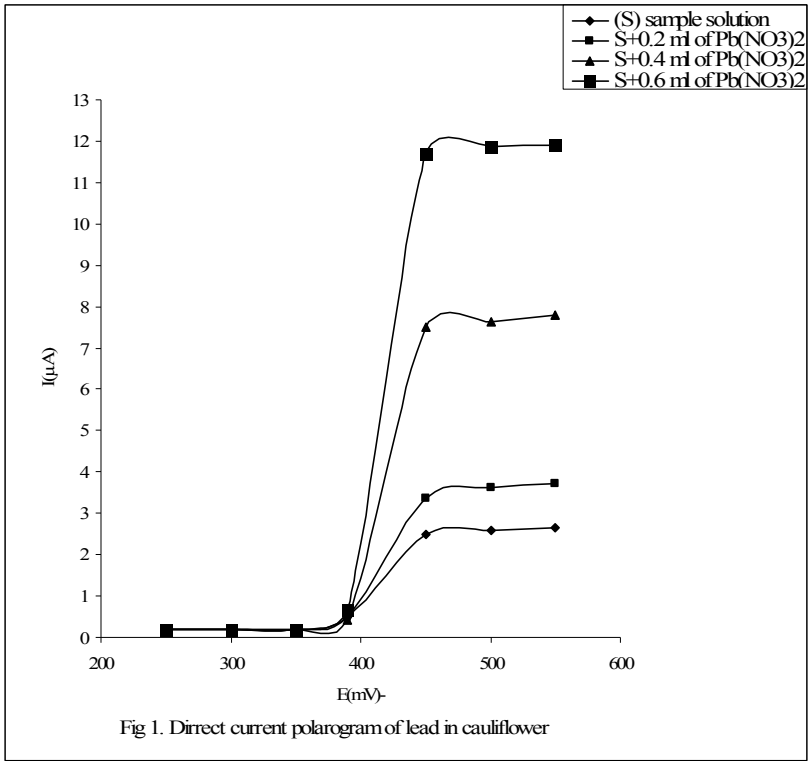

Fig 1. Dirrect current polarogram of lead in cauliflower

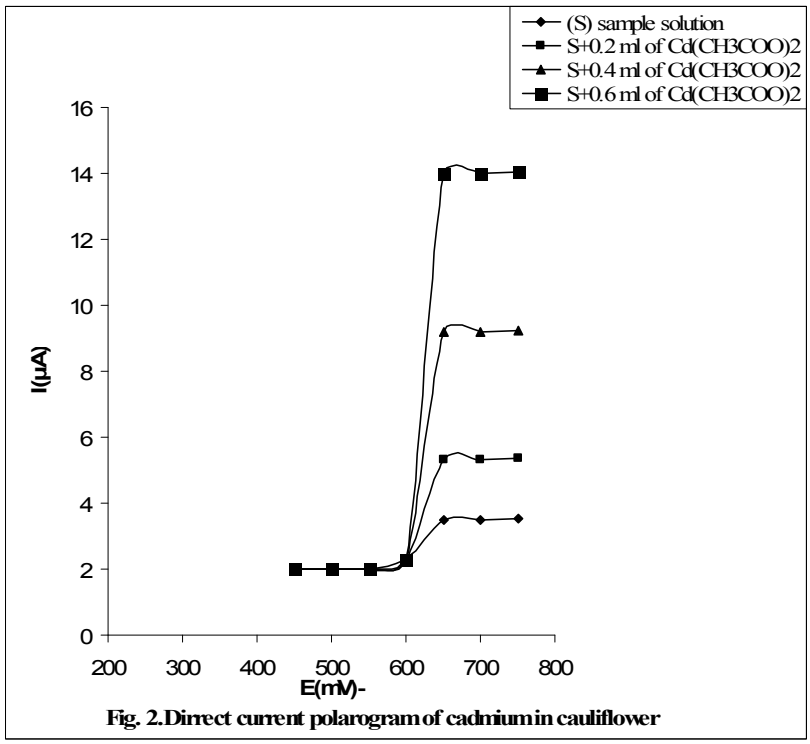

Fig. 2.Dirrect current polarogram of cadmium in cauliflower

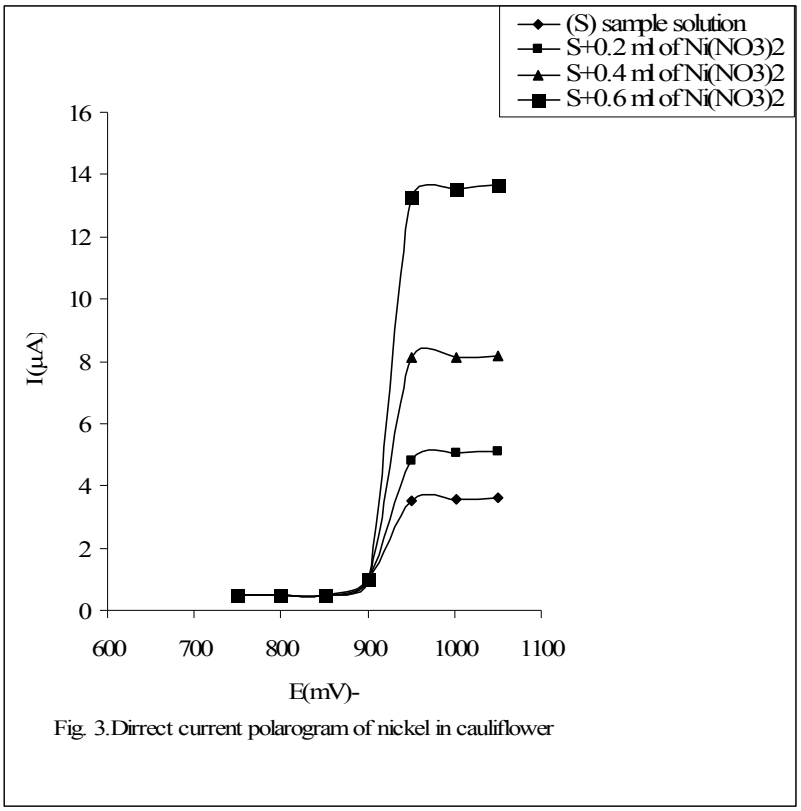

Fig. 3.Dirrect current polarogram of nickel in cauliflower

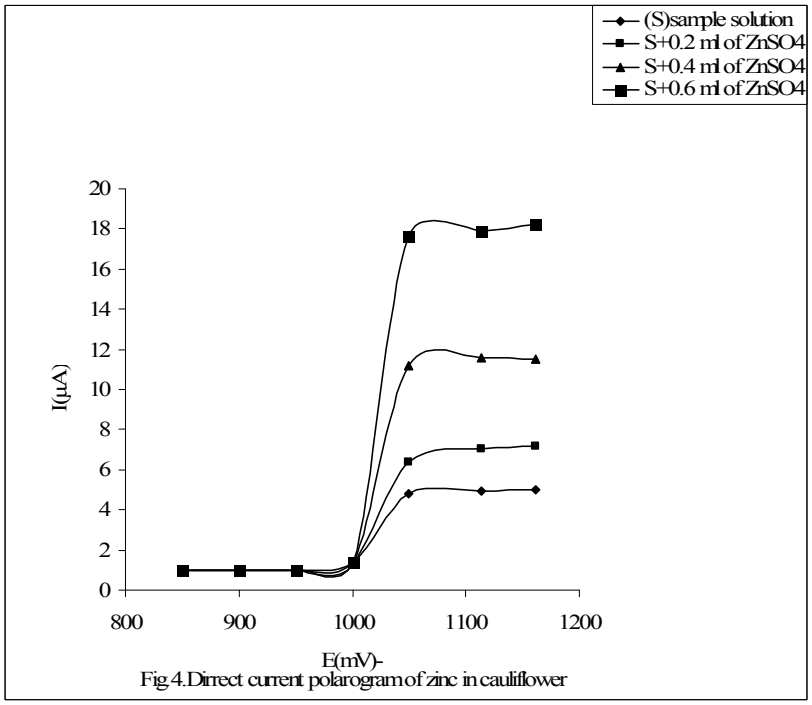

Fig.4.Dirrect current polarogram of zinc in cauliflower

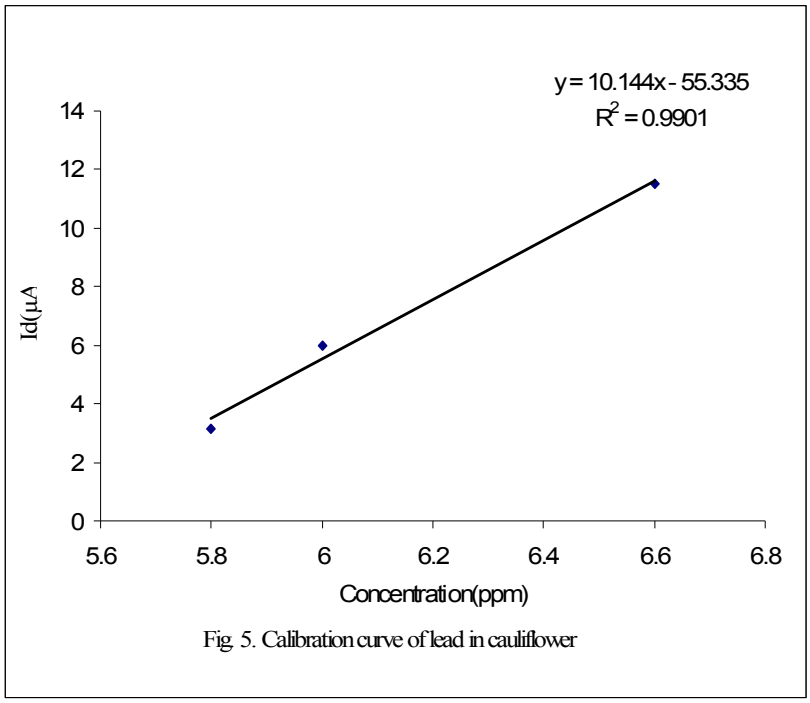

Fig. 5. Calibration curve of lead in cauliflower

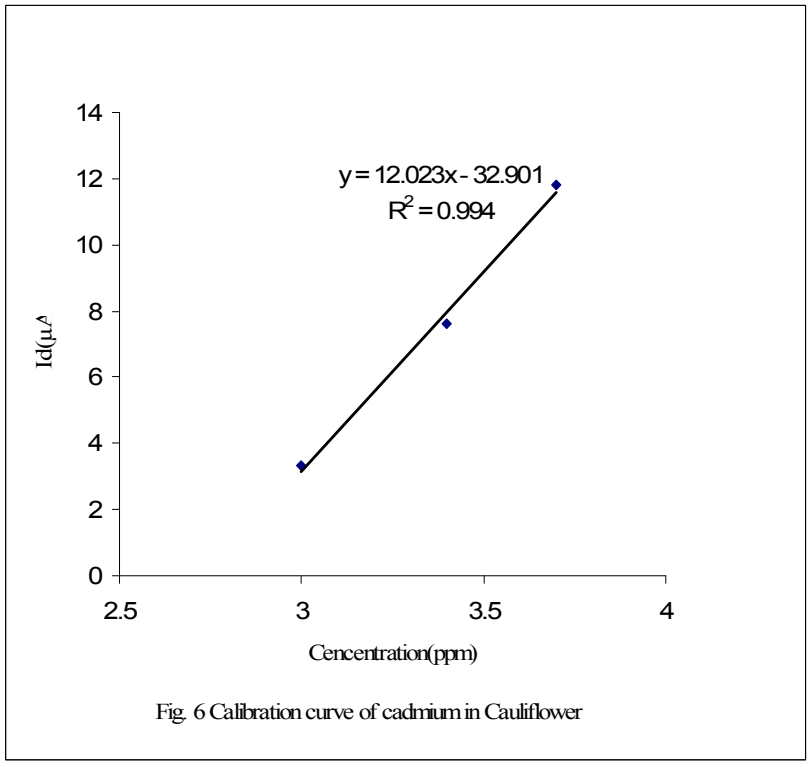

Fig. 6 Calibration curve of cadmium in Cauliflower

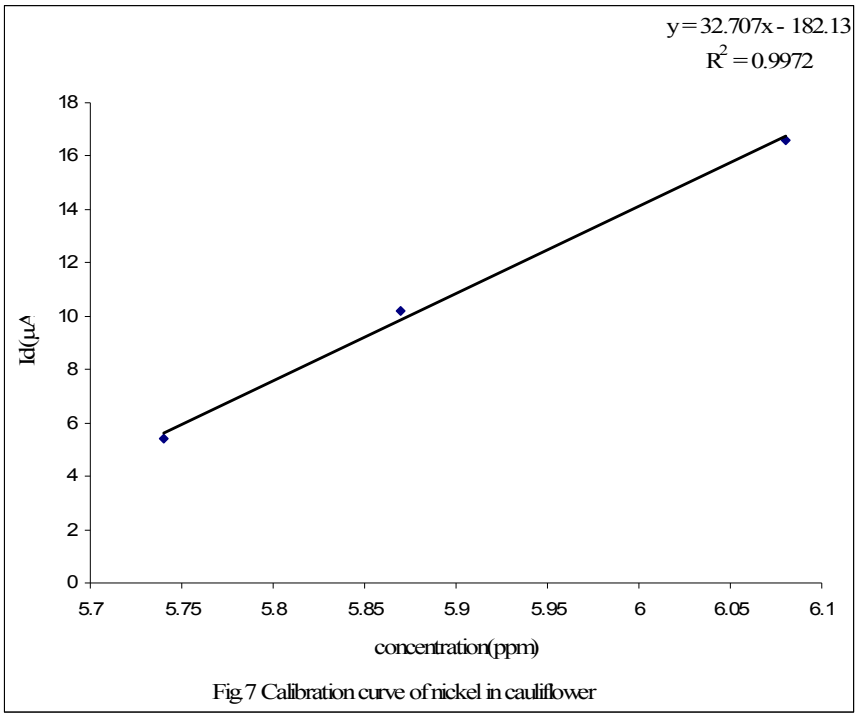

Fig.7 Calibration curve of nickel in cauliflower

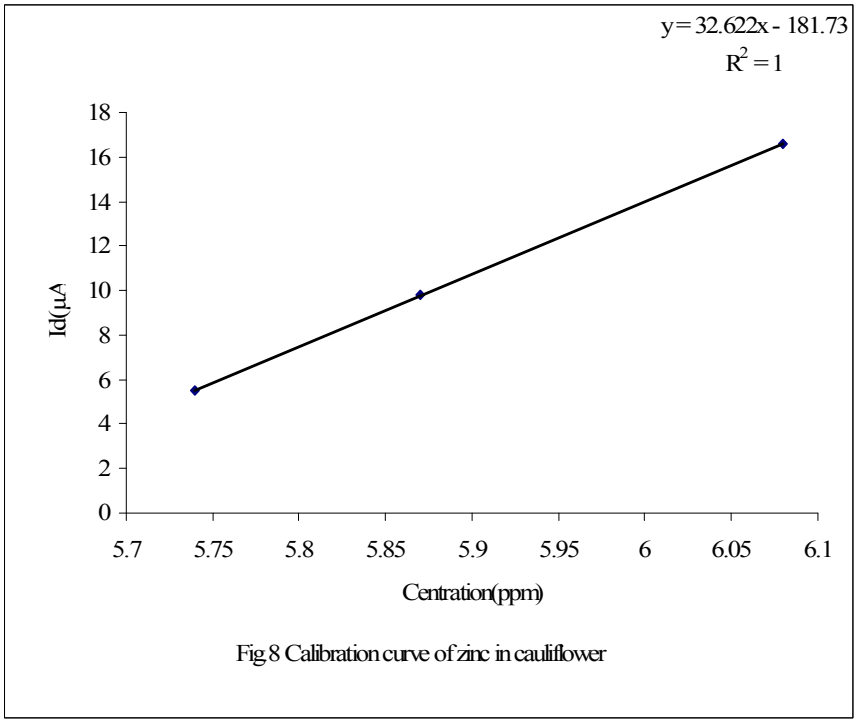

Fig.8 Calibration curve of zinc in cauliflower

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, 4.707, 5.899 and percentage error is small as 0.0315, 0.1396, 0.1414, 0.1499 respectively for Pb, Cd, Ni and Zn in acetate buffer and HCl buffer. Mean deviation is found to be 0.323111, 0.229333, 0.169111, 0.123111 and standard deviation is found to be 0.423193, 0.335362, 0.250081, and 0.17301 respectively for Pb, Cd, Ni and Zn. 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[24-25]. The allowed values of metals such as Pb 0.030ppm, Cd 0.001 ppm, Ni 0.50 ppm, and Zn 5.0 ppm are not harmful, but whenever the concentration of these metals exceeds from this value, it may pose harmful effects on our body as follows-

1. The most ubiquitous of toxic metals in vegetables is lead. The toxic effects of lead can lead to nerve and brain damage. Children are especially sensitive Exposure may also lead to kidney damage, and blood disorders
2. High levels of cadmium cause kidney problem, anemia and bone marrow disorders.
3. Small amount of nickel are needed by the human body to produce red blood cells, in excessive amount can cause decreased body weight, heart and liver damage.
4. High levels of zinc cause nauses, gastric ulcer, pancreatitis, anemia and excessive salivation.

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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