

Methods for determination of heavy metals biology essay



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There are various analysis methods applied by researchers to determine the heavy metal contents of a particular sample. Of the various analytical methods used, atomic absorption spectroscopic methods are the most commonly used methods to determine the amount of heavy metals present in a particular sample. Preconcentration and separation methods have been routinely used to eliminate matrix effects and cope with low metal levels. Different preconcentration technique for heavy metals such as cloud point extraction, solid phase extraction and acid preconcentration were performed by researchers before the samples being analyzed by flame atomic absorption spectroscopy.

2. 1. 1 Determination of heavy metals by using cloud point extraction before flame atomic absorption spectroscopy analysis

Chan and Teo (2001) used cloud point extraction (CPE) for the simultaneous pre-concentration of cadmium, copper, lead and zinc after the formation of a complex with 1-(2-thiazolylazo)-2-naphthol (TAN) before the sample undergoing analysis by flame atomic absorption spectrometry (FAAS) using octylphenoxypolyethoxyethanol (Triton X-114) as surfactant. Cloud point extraction is a separation and preconcentration procedure that has been extensively applied for trace metal determination in several different matrices. It is simple, rapid, high extraction efficiency and environmentally friendly. Its major advantages are simple experimental procedures, low cost, high preconcentration factors, and environmental safety. Cloud-point extraction is the preconcentration of target metal ion using surfactants such as non-ionic and anionic surfactants. Under the optimum conditions, the aqueous surfactant solution becomes clouding and separating into two

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phases which are aqueous and surfactant-rich phases. The interested analytes are extracted and concentrated into surfactant-rich phase. The surfactant rich phase with interested analytes is analysed by flame atomic absorption spectroscopy (F-AAS). The water samples tested in this experiment are tap water, river water, sea water and a standard reference material (SRM). The chemical variables affecting the separation phase and the viscosity affecting the detection process including 50mL of sample, temperature of 40°C, pH of 8.6, 2×10^{-5} mol l⁻¹ of TAN complexing agent, 0.05% non-ionic surfactant Triton X-114 were optimized.

Table 1: Limit of Detection and Sensitivity of F-AAS analysis for Cd, Cu, Pb and Zn in Water Samples (n= 10). (Chen and Teo, 2001)

Table 1 gives the calibration parameters, the relative standard deviation obtained for 10 analyte samples subjected to the complete procedure and the detection limits. In this case, linear relationships between the absorbance measured and the concentration of metal prepared for the calibration solution were obtained. The sensitivity of F-AAS for Cd, Cu, Pb and Zn analysis are 0.9982, 0.9987, 0.9995 and 0.9992 respectively. In addition, pre-concentration of 50 ml of sample in the presence of 0.05% Triton X-114 showed the detection of 0.099, 0.27, 1.1 and 0.095 ng mL⁻¹ for cadmium, copper, lead and zinc, respectively.

Table 2: Recovery analysis of Cd, Cu, Pb and Zn in water samples by F-AAS (n= 3) (Chen and Teo, 2001)

Table 2 shows the recovery percentage of Cd, Cu, Pb and Zn in tap water, river water and sea water by F-AAS. Recovery experiments for different <https://assignbuster.com/methods-for-determination-of-heavy-metals-biology-essay/>

amounts of Cd, Cu, Pb and Zn were carried out. The overall recovery percentages in this experiment for the four heavy metal elements were in the range of 98 % to 104 %.

2. 1. 2 Determination of trace elements in waters after preconcentration with 2-nitroso-1-naphthol

Gentscheva et al. (2012) determined heavy metals using F-AAS after pre-concentrating the water samples with 2-nitroso-1 naphthol. 2-nitroso-1-naphthol is a chelating agent that has been used for precentration of cobalt as well as for the precentration of cobalt as well as for the solid phase extraction of Th, Ti, Fe, Pb and Cr from waters. The purpose of this experiment was to study the possibilities for preconcentration of trace amounts of Cu, Ni, Co, Cd, and Pd using 2-nitroso-1-naphthol with analysis of F-AAS. The method developed was tested for reliability using a reference material which was SPS-WW2, Batch no. 108, Spectrapure Standards AS (Oslo, Norway) and this method was applied to the determination of traces of these elements in capture water and waste water. A comparison between ethanol and 1- ethanolic solution of 2-nitroso-1-naphthol was done by plotting calibration curves. It was found that 2-nitroso-1-naphthol suppressed the absorbance of the interested trace elements ideally.

Figure 1: Effect of sample volume on the recovery of Ni, Co, Cu, Cd and Pd with 2- nitroso-1-naphthol. (Gentscheva et al., 2012)

Figure 1 showed the effect of sample volume on the recovery of trace elements in the range from 20 to 250 ml. As can be seen, the maximum sample volume allowing a better quantitative recovery of the analytes which

was more than 95% was 160ml. At higher sample volumes the recovery gradually decreases. 11ml of optimum volume for 1% ethanolic 2-nitroso-1-naphthol was also determined.

Figure 2: Effect of time of complexation on the recovery of Ni, Co, Cu, Cd and Pb with 2-nitroso-1-naphthol (Gentscheva et al., 2012)

Figure 2 showed the effect of time of complexation on the preconcentration of trace elements in experiment. The effect of time of complexation was studied in the range of 15 to 60 minutes. The optimum time of complexation obtained was 40 minutes. Longer complexation time did not bring any effect on the recovery of the trace elements.

Table 3: Determination of Cu, Co, Ni, Cd and Pd in capture water from the region of an old copper mine (Bov, Bulgaria) and waste water from the copper plant "Assarel", Bulgaria

Table 3 shows the concentration of interested trace elements obtained from F-AAS analysis. From the table, copper was the trace element with the highest content not only in the waste water sample, but also in the capture water sample. The latter may be attributed to the high residual level of this element in the environment of the former copper mine. The Pd contents in water samples were below the detection limit of the method.

2. 1. 3 Solid-phase extraction of metal ions from environmental samples by flame atomic absorption spectrometry (FAAS)

Duran et al.(2007) used a column packed with Amberlite XAD-2010 resin as a solid-phase extractant for the preconcentration of Mn(II), Co(II), Ni(II), Cu(II), Cd(II), and Pb(II) ions based on their complex formation with the sodium diethyldithiocarbamate (Na-DDTC) subjected to flame atomic absorption spectrometric (FAAS) determinations. Metal complexes sorbed on the resin were eluted by 1 mol L⁻¹ HNO₃ in acetone. Effects of the analytical conditions for the preconcentration yields of the metal ions, such as pH, quantity of Na-DDTC, eluent type, sample volume and flow rate have been investigated.

Figure 3: Effect of pH on the retention of the metal ions (n= 4). (Duran et al., 2007)

Figure 3 showed that the effect of the pH that had been studied in experiment. pH of samples in the range 2 to 12 by adjusting the metal solutions with buffer solutions. Cu and Co were quantitatively recovered more than 95% in the pH range from 2 to 9. Quantitative recoveries were obtained in the pH range 3 to 9 for Ni, 4 to 9 for Cd, 5 to 9 for Pb, and 7 to 9 for Mn. According to the results, the optimum pH for multi-element preconcentration of the metals was 6, except for Mn for which the value is 8.

Figure 4: Effect of ligand quantity on the recoveries. (Duran et al., 2007)

Figure 4 showed the determination of amount of ligand needed for preconcentration of metal ions. From the figure above, it showed the

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preconcentration yields of the six metal ions were 11 to 21% without chelating agent. The preconcentration yields increased with the addition of Na-DDTC. The quantitative values were obtained after 2.0 mg of Na-DDTC. As the result, 5.0 mg of Na-DDTC was chosen as optimum value for the next works.

Figure 5: Effect of sample volume. (Duran et al., 2007)

Figure 5 showed the determination of sample volume in order to deal with real water samples which were containing very low concentrations of the metal ions. The recoveries were found to be stable until 500mL. Thus, 500mL was chosen as the optimum sample volume to work. In this work, the final solution volume to be measured by FAAS was 5.0 mL, hence the preconcentration factors were 100 for all the six metal ions.

Figure 6: Effect of flow rate on the recoveries. (Duran et al., 2007)

Figure 6 showed the study of sample flow rate in the experiment. The flow rate of sample solution through the Amberlite XAD-2000 column is a very important factor. The recovery percentage increases with the reduced flow rate, causing the preconcentration time increases. In order to obtain the quantitative recovery and to reduce the preconcentration time, the effect of sample flow rate was studied in the range 2 to 40 mLmin⁻¹. 10.0 mLmin⁻¹ was chosen as the sample flow rate. This flow rate is high enough to load the sample in a moderate short time and allowing metal/Na-DDTC chelates to interact with XAD-2010.

Table 4: Trace metals contents of real water samples with the proposed method (n= 3)

Table 4 showed the determination of concentration of trace metals in real water samples with the proposed method. Six sample collection sites were determined and under optimal conditions. There was no detection of Cd in all of the six water samples. There was also no detection of Co in Solakli stream, Kisarna mineral water, Of stream water and black tea while Ni was also cannot be detection by F-AAS in the water samples such as Kisarna mineral water and Of stream water. Overall relative standard deviations for Mn(II), Co(II), Ni(II), Cu(II), Cd(II), and Pb(II) ions were less than 5 %.

2. 2 Sample Analysed For Determination of Heavy Metals

Heavy metals can be found in various materials, not only in water samples.

By performin different analytical methods, most researchers had determined the presence of heavy metals, commonly, Cd, Pb, Cr and Cu to name a few, in environmental samples which include water sample, food samples, biological samples and plastic materials as well.

2. 2. 1 Environmental samples

Among the environmental samples that had been tested to determine trace toxic metals level are the water samples such as capture water and waste water (Gentscheva et al., 2012); river water, tap water and sea water (Chan and Teo, 2010) and sea water; soil samples heavy metals determination which carried out by Capitelli et al. (2002) in order to investigate the contents of chromium, copper, iron, manganese, nickel, lead and zinc by laser induced breakdown spectroscopy.

2. 2. 2 Food samples

Many experiments for determining heavy metal contents have been performed on food samples. Ghaedia et al. (2008) determined the presence of chromium, iron, copper, iron, nickel, cobalt and zinc in cow meat, spinach, liver and fish samples. Muñoz and Palmero (2004) determine the presence of cadmium, lead and copper in milk by using potentiometric stripping analysis while Tripathi et al. (1999) determined zinc, lead, copper and cadmium in human milk, milk powder and baby food. Vegetable and fruits that we usually consumed as part of our daily diet intake were also found to have trace amount of heavy metals. Radwan and Salama (2006) determined the heavy metals such as zinc, copper, cadmium and lead contents in strawberry, apples, banana, eggplant, lettuce and many more.

Determination of cadmium, cobalt, chromium, copper, iron, nickel, lead and zinc in beverage such as tea, coffee, dairy products, fruit juices, carbonated soft drinks and wines was carried out by Onianwa et al. (1999), using an air-acetylene flame atomic absorption spectrophotometer.

2. 2. 3 Biological Samples

Other than environmental samples and food samples, several researches had also been carried out to determine heavy metal contents in biological samples such as human urine and blood which are the most common tested samples for determination of heavy metals. Suleiman et al. (2008) determined the presence of cadmium, cobalt, nickel and lead in human urine, blood and serum samples by black stones online coupled with ICP-OES. In addition, Aguiar et al.(2012) determined the presence of calcium,

copper, iron, magnesium, selenium, and zinc which are essential for reproduction by using ICP-MS.

2. 2. 4 Plastic samples

The plastic samples that commonly tested to determine the heavy metal contents are the plastic toys (Omolaoye, Uzairu and Gimba, 2010); waste electrical and electronic equipment such as mobile phones (Nnoroma and Osibanjob, 2010), computer mouse, printer, keyboards, refrigerators, washing machines, personal computer and television (Onwughara et al., 2010); and small waste electrical and electronic equipment which includes plastic materials from household appliances such as vacuum cleaners, pocket calculators, video games, body care appliances, clocks and watches as well.(Dimitrakakis et al., 2009)

2. 3 Permissible Limits of Heavy Metals in Waste Water Samples

For every product that available in the market, there must be a limit of the substances added that will gives the details of the contents of that particular product to ensure the safety of the product to consumer. According to Environmental Quality Act (Sewage and Industrial Effluents) Regulation 1979, the maximum contaminant level of copper, cadmium, chromium, lead and nickel in waste water are 0. 20, 0. 01, 0. 05, 0. 20 and 0. 20 ppm respectively. This means that if waste water sample was found to contain either one of the heavy metals mentioned having values exceeding the maximum contaminant level, that particular waste water sample could pose health hazards and environmental pollution to human being.

2. 4 Improving the Sensitivity of Heavy Metals in F-AAS Analysis

High sensitivity of the analysis of the presence of heavy metals present in a waste water sample are needed to give a reliably accurate and precise result in some cases where there is only a slightly difference in the amount of heavy metals. The sensitivity of various heavy metals in F-AAS analysis can be enhanced by several methods.

2. 4. 1 Copper, Cadmium and Lead

The sensitivity of F-AAS analysis for the detection of Cu, Cd and Pb can be enhanced by performing online sensitivity enhancement method involving the implementation of various designs of quartz tubes of slotted tube atom traps (STAT) system as published by Yaman (2005). In this technique, quartz tubes which had a 5 and 6 mm diameter and 2. 3cm for an upper slot length, produced the best sensitivity as high as with the factor of 6- and 10-fold enhancement for Pb and Cd respectively.

2. 4. 2 Other Heavy Metals

The sensitivity of F-AAS analysis for the detection of heavy metals can be increased by using pre-concentration methods such as chromatographic method, extraction method, drying and ashing method, electrochemical method and miscellaneous method published by Rottschäfer, Boczkowski and Mark (1971).