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**A STUDY OF THE REMOVAL OF SOME TOXIC METALS
IN TAP WATER BY COFFEE GROUNDS**

Dissertation submitted in partial fulfillment for the Degree of
Bachelor of Science (Health) in Forensic Science

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2006

CERTIFICATE PAGE

CERTIFICATE

This is to certify that the dissertation entitled
“A Study of the Removal of Some Toxic Metals in
Tap Water by Coffee Grounds”
is the bonafide record of research work done by
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during the period of January 2006 to April 2006
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ACKNOWLEDGEMENT

It is difficult to imagine that this research project will come to completion without assistance and contributions from many individuals. I am flattered to have this opportunity to pen a few lines of gratitude and appreciation to those who has lent a hand to make my research become possible.

First of all, I would like to wish a million thanks to Dr. K. D. Henry for the unlimited ideas, guidance and time he spent for me through-out this project. From the very beginning until I finished my work, he directed and guided me at the same time observed me.

Secondly, a special thanks to En. Sahnusi for his time and attempt in helping me all along in this project since he is the one who in charge for the AAS instrument and without him it would have been impossible for me to complete the analysis. Also I like to extend my gratitude to Pn. Hafizah, Pn. Rosniah and En. Zulhairi for their assistance. I like to thank to UKM staff for letting me borrow the apparatus and equipment for my project.

Moreover, I would like to show my appreciations to my friend, Norazira Azizan as she has helped me a lot in my project, and her willingness to share and do the project together at the beginning. Last but not least, I thank my fellow friends, Khairulmazidah, Rina Haryani and Zulaiha, who accompanied me during the laboratory work, especially when I had to do my project after the office hours.

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ABSTRACT

This research is a study about the removal of toxic metals such as iron and chromium in tap water using coffee grounds as filters. Three samples of tap waters from different areas were collected, one sample from HUSM, one from Nurani Hostel and another one from D'Village (restaurant outside the campus). The purpose of collecting water samples from different areas is to determine whether the water from different tap water sources have different concentration of the metals concerned. Three different types of commercial coffee brands were selected to constitute different thickness of the coffee bed which were composed of fine, medium and coarse particles. This is to study the relevance of the coffee texture in the removal of the metals and the reason for selection of a variety thickness of coffee beds is to investigate the significance of the coffee bed thickness in the removal of metals. In the preparation of coffee, equal amounts of different types of coffee grounds were allowed to be in contact with different type of water, maintaining a constant time in which the coffee is in contact with water. In the first step, the concentration of the chromium and iron in the three samples of water was determined using the Atomic Absorption Spectrometry (AAS). The concentration of metals in the tap water samples were found to be very low, especially chromium. Therefore, the water samples had to be acid digested, complexed with a chelating agent and extracted by methyl isobutyl ketone (MIBK) to give a higher concentration of the metals. After the concentration of the metals in water samples were obtained, coffee was prepared using water from the same samples. As three different types of coffee were used and along with three different sources of water, a total of nine coffee extracts were prepared. The

coffee extracts were oxidized first to remove the organic matter. Concentration of total iron was determined by direct AAS while that of chromium was estimated after MIBK treatment.

INTRODUCTION

Toxic metals are unwanted additions to an average cup of tap water due to their toxicity. In areas with older distribution systems of water, the concentrations of some toxic metals can be high. Even though some metals are essential trace minerals that the body requires, long time exposure to metals can lead to toxicity and have a long-term toxic effect on humans.

Previous researches have established that metals like lead, copper and zinc in tap water can be removed by the coffee grounds [1]. It was found that the normal coffee grounds remove 78 to 90 percent of copper and lead from the water. It is argued that the mechanism of removal of these divalent metals is probably due to electrostatic attraction between the positive charges of the metals and uncharged or negatively charges of the coffee powder by forming a metal chelate. In addition, the deeper the coffee beds in the coffee maker, the more effective the removal of heavy metals. Besides lead, copper and zinc, the existence of other metals like iron and chromium in drinking water be potentially harmful to humans with a long time exposure of these metals in tap water. Therefore, the objective of this research project is to determine the effectiveness of coffee grounds in the removal of chromium and iron from the tap water.

Iron is one of the most common elements present in tap water. As iron makes up at least five percent of the earth's crust, it is one of the heavy metals that are present in the environment. Rainwater is filtered by the soil and the underlying geologic formations dissolves iron, which seep into aquifers and serve as the sources of underground water. The concentration of iron is influenced by geological structure of soils and rock formation, the

hydrological conditions of the area, the physical and chemicals that make up the surrounding rocks and soil, and the presence of the microorganisms [2]. Iron in tap water may be the result of corrosion of iron in pipes, tanks or plumbing systems.

Iron is mainly present in water in two forms, either the soluble ferrous iron (Fe^{2+}) or the insoluble ferric iron (Fe^{3+}). Water containing ferrous iron is clear and colorless because the iron it is completely dissolved. Due to air present in the atmosphere, certain kinds of bacteria utilize the iron and make the water turns cloudy and a reddish brown substance begins to form. This sediment is the oxidized or ferric form of iron that will not dissolve in water. Therefore, iron may be present as Fe (II) or Fe (III) in water. However iron may cause serious impairment of water quality. Environmental Protection Agency (EPA) recommends that the concentration of iron in drinking water should not exceed 0.30 mg/L [3]. Since the daily nutritional requirement is 1 to 2 mg, the standard is for aesthetic reasons rather than toxicity.

Although iron is essential for good health as it help to transport oxygen in the blood, if present at a high concentration in drinking water, may be toxic to the body. There are many problems that may result from iron toxicity, which include anorexia, oliguria, diarrhea, hypothermia, diphasic shock, metabolic acidosis, and death. In addition to these, the patient may experience vascular congestion of the gastrointestinal tract, liver, kidneys, heart, brain, spleen, adrenals, and thymus. As a result of iron storage disease, the liver becomes cirrhotic. Hepatoma, the primary cancer of the liver, has become the most common cause of death among patients with hemochromatosis. Also, when siderosis becomes severe in young people, myocardial disease is a common cause of death. Impotence may occur in young men, and

amenorrhea may occur in young women .Both these sexual related problems are due to iron loading in the anterior pituitary [4].

Like iron, chromium also occurs in nature. Even though it is widely distributed in soils and plants, it is rare in natural waters. The two largest sources of chromium emission in the atmosphere are from the chemical manufacturing industry and combustion of natural gas, oil and coal. It is also used in metal alloys such as stainless steel, protective coatings on metal, magnetic tapes and pigments for paints, cement, paper, rubber, composition floor covering and other materials. Other uses of chromium include chemical intermediates for wood preservatives, organic chemical synthesis, photochemical processing and industrial water treatment. In medicine, chromium compounds are used in astringents and antiseptics. They also are used in cooling waters, and in the leather tanning industry, in catalytic manufacture, and in fungicides, as an algacide against slime forming bacteria and in yeasts in brewery processing water and brewery warm water [5].

Chromium is unlikely to migrate to ground water. A field trial on the application of wastewater treatment sludge of soils found that movement of heavy metals, including chromium, from the soil surface is to a depth of 10 cm, but most of the metal remained in the upper 5 cm layer of soil [5]. Chromium compounds are persistent in water. Most of the chromium in surface waters may be present in particulate form as sediment. Some of the particulate chromium would remain as suspended matter and ultimately be deposited in sediments. Most of the soluble chromium in surface waters may be present as chromium (VI), and a small amount may be present as chromium (III) organic complexes. Hexavalent chromium or chromium (VI) is the major stable form of chromium in seawater, however,

chromium (VI) may be reduced to chromium (III) by organic matter present in water, and may eventually deposit in sediments.

There is no formal standard for chromium (VI) in tap water, but the state Office of Environmental Health Hazard Assessment proposed that the concentration of chromium in water should not exceed 0.2 parts per billion (ppb) [6]. However, according to Alan Hirsch who is the spokesman of state Office of Environmental Health Hazard Assessment, at that level, an estimated one million people could drink about two liters water a day for 70 years, one could contract cancer resulting from exposure to chromium (VI). Therefore, some scientist argued that it should not be present at all in water and water is safe for consumption because of insufficient scientist evidence linking chromium to illness. Chromium has the potential to cause the health effects such as damage to liver, kidney circulatory and nerve tissues, and dermatitis from the long-term exposures at levels that above 0.1 mg/L.

Chromium (III) is an essential nutrient as it works with insulin in the metabolism of sugar and stabilizes blood sugar levels, cleans the arteries by reducing cholesterol and triglyceride levels, and help in the transport of amino acids to places where the body needs them [7]. However, chromium is considered to pose risks to people in its hexavalent form chromium (VI), as it has been found to cause cancer.

REVIEW OF LITERATURE

In 2004, Tokimoto, T. et al. investigated the possibility of using coffee grounds to remove lead ions from drinking water. The study is about the lead ion adsorption characteristics to coffee beans and grounds by measuring the fat and protein content, adsorption isotherms for lead ions, and adsorption rates for lead ions. They found out that number of lead ions adsorbed by coffee grounds did not depend on the kind of coffee beans or the temperature at which adsorption tests were performed. The rate of lead ion adsorption by coffee grounds was directly proportional to the amount of coffee grounds added to water. When coffee grounds were degreased or boiled, the number of adsorbed lead ions decreased. When proteins containing coffee grounds were denatured, the lead ion adsorption was considerably reduced. The lead ion adsorption capacity of coffee grounds decreased with increased concentration of perchloric acid used for treating them and disappeared when 10% perchloric acid was used. These experiments demonstrated that proteins contained in coffee beans influence the adsorption of lead ion. This study gave an affirmative answer to the possibility of using coffee grounds, an abundant food waste, for removing lead ions from drinking water [8].

Boonamnuayvitaya, V. et al. (2002) investigated the preparation and utilization of coffee residues binding with clay as an adsorbent for removal of heavy metal ions in solution. Factors affecting the adsorption, such as pyrolysis temperature, weight ratio of coffee-residue to clay and particle size were investigated in this preliminary study on the adsorption of metal ions. The pyrolysis temperature of 500°C, weight ratio of coffee residue to clay of 80:20, and particle size diameter of 4 mm were found to be the suitable. Under these condition, the adsorption capacities of the coffee-clay adsorbent for Cd^{2+} , Cu^{2+} , Pb^{2+} , Zn^{2+} and Ni^{2+} were

determined by the Langmuir isotherm model. The adsorption increased in the order of $\text{Cd}^{2+} > \text{Cu}^{2+} > \text{Pb}^{2+} > \text{Zn}^{2+} > \text{Ni}^{2+}$. The effects of solution pH (8–10) and temperature (30–80°C) on the adsorption of Cd^{2+} has been investigated. The Cd^{2+} adsorption increased with increasing pH and temperature, and remained constant at high pH. The heat of adsorption ($\Delta H = -1.11$ kcal/g) implied that the adsorption was a physical exothermic adsorption. Desorption was easily achieved by leaching with distilled water which gave the recovery yield of 88–92%. The functional groups studied by Fourier Transform Infrared (FT-IR) indicated that hydroxyl, carboxyl and amine groups were the main functional groups present in the adsorbent. Electrical potential study showed that the coffee-clay adsorbent exhibited negative charge that was favorable to attract metal ion. The surface and pore study implied that the high fraction mesopores in the granular coffee-clay adsorbent contributed to the adsorption capacity [9].

Ohno, S. et al. (2002) has proposed for the successive determination of nanogram levels of copper and iron by a kinetic-catalytic spectrophotometric method, which is based on their catalytic effects on the oxidative coupling of p-anisidine with N,N-dimethylaniline (DMA) to form a colored compound (λ_{max} 740 nm) in the presence of hydrogen peroxide at pH 3.2. 2,9-Dimethyl-1,10-phenanthroline (neocuproine) acted as an activator for the copper catalysis, and 1,10-phenanthroline (phen) acted as an activator for the iron catalysis. The selectivity was improved in the presence of diphosphate as a masking agent. The determinable ranges were 0.16-10 ppb for copper and 1-100 ppb for iron, respectively. The relative standard deviations of copper and iron were 1.1 and 0.97% for five determinations of 10 ppb copper and 40 ppb iron. The method was successfully applied to the analyses of copper and iron in tap, well, river and pond waters [10].

In their study, Kolayly, S. et al. (2003) found that the complex formation capacity of caffeine, which is present highly-consumed tea and coffee component, was determined for Ca, Mg, Fe, Zn, Pb, Mn, Co and Cr metal ions. The binding constants of metal ion–caffeine complexes for the metals chosen were determined spectrophotometrically. The results were compared with the known stability constants of metal ion–EDTA complexes, EDTA being known for its high metal binding capacity. Furthermore, iron chelating activity of caffeine, using the ferrozine reference method, was studied and compared with that of EDTA. The results showed very little complex formation capacity of caffeine with binding constants of 29.6, 22.4, 59, 396, 55, 9.3, 83 and 592 M⁻¹ for Ca, Mg, Fe, Zn, Pb, Mn, Co and Cr metal ions, respectively, in contrast to that of EDTA. The iron chelating activity of caffeine was also found to be 6%, which was considered to be quite low compared with EDTA [11].

Manios T., et al. (2002) studied about the *Typha latifolia* plants, commonly known as cattails, grown in a mixture of mature sewage sludge compost, commercial compost and perlite (2:1:1 by volume). Four Groups (A, B, C and D) were irrigated (once in every two weeks) with a solution containing different concentrations of Cu, Ni, and Zn, while in the fifth (group M) tap water was used. At the end of the 10 weeks experimental period substrate and plants were dried, weighed and analysed for heavy metals. The amounts of all three metals removed from the irrigation solution, were substantial. In the roots and leaves/stems of *T. latifolia* the mean concentration of Zn reached values of 391.7 and 60.8 mg/kg of dry weight (d.w.), respectively. In the substrate of Group D all three metals recorded their highest mean concentrations of 1156.7 mg/kg d.w. for Cu, 296.7 mg/kg d.w. for Ni and 1231.7 mg/kg d.w. for Zn. Linear correlation analyses suggested that there was a linear relationship between the concentration of metals in the solutions and the concentration of metals in the substrates at the

end of the experiment. The percentage removal of the metals in the substrate was large, reaching 100% for Cu and Zn in some groups and almost 96% for Ni in group D. The total amount of metals removed by the plants was considerably smaller than that of the substrate, due to mainly, the small biomass development. A single factor ANOVA test (5% level) indicated that the build up in the concentration of metals in the roots and the leaves/stems was due to the use of metaliferous water solution and not due to the metals pre-existing in the substrate. The contribution of the plants (both roots and leaves/stems) in the removing ability of the system was less than 1% [12].

Lunvongsa, S. (2005) has developed a flow injection spectrophotometric method for the determination of dissolved and total amounts of iron in tap and natural water samples. The method for the determination of iron employs a sample acidification step in order to decompose iron hydroxide and iron-complexes into free iron, Fe(III) and Fe(II). The amounts of free iron were detected using a catalytic action of Fe(III) and Fe(II) on the oxidation of *N,N*-dimethyl-*p*-phenylenediamine in the presence of hydrogen peroxide. Increase in absorbance of oxidized product was detected spectrophotometrically at 514 nm. The proposed method allows 0.02 and 0.06 $\mu\text{g L}^{-1}$ of limit of detection (LOD) and limit of quantification (LOQ), respectively, with relative standard deviation below 2%. The accuracy and the precision of the method were evaluated by the analysis of the standard reference material, river water. The developed method was successfully applied to other water samples [13].

Monteiro, M.I.C. et al. (2002) studied three different analytical methods comprising a colorimetric method with 1,5-diphenyl-carbazide, electrothermal atomic absorption spectrometry and flame atomic absorption spectrometry were utilized in a study to determine traces of chromium in synthetic tannery effluent from laboratory scale treatment process

variations. All the results obtained using the three different methods showed good agreement and met the requirement of Brazilian regulation for total chromium for effluent discharges ($<0.5 \text{ mg L}^{-1}$). However, electrothermal AAS has been the proposed method because it was faster, less laborious, required smaller volume of sample and presented a lower limit of quantification ($\text{LOQ} = 2.2 \mu\text{g L}^{-1}$) [14].

Tunceli A. et al. (2002) have developed a simple and a sensitive method for the speciation, separation and preconcentration of Cr(VI) and Cr(III) in tap water. Cr(VI) has been separated from Cr(III) and preconcentrated as its 1,5-diphenylcarbazone complex by using a column containing Amberlite XAD-16 resin and determined by Flame AAS (FAAS). Total chromium has also been determined by FAAS after conversion of Cr(III) to Cr(VI) by oxidation with KMnO_4 . Then, concentration of Cr(III) has been calculated by subtracting Cr(VI) from the total. The effect of acidity, amount of adsorbent, eluent type and flow rate of the sample solution on the preconcentration procedure has been investigated. The retained Cr(VI) complex was eluted with 10 mL of $0.05 \text{ mol L}^{-1} \text{ H}_2\text{SO}_4$ solution in methanol. The recovery of Cr(VI) was 99.7 ± 0.7 at 95% confidence level. The highest preconcentration factor was 25 for a 250 mL sample volume. The detection limit of Cr(VI) was found as $45 \mu\text{g L}^{-1}$. The adsorption capacity of the resin was found as 0.4 mg g^{-1} for Cr (VI). The effect of interfering ions has also been studied. The proposed method was applied to tap water samples and chromium species have been determined with the relative error $<3\%$ [15].

OBJECTIVES

Tap water is the main water supply to most part of the country. Most importantly we use tap water for drinking, cooking and other activities. Recent studies had showed that tap water contain some toxic metals like copper, zinc, lead and lately iron and chromium. For health reasons, we are to drink at least two liters of water per day. Thus we can imagine the amount of toxic metals that have accumulated in our body.

The objectives of this project are;

- (i) to determine the concentration of the toxic metals like iron and chromium in tap water.
- (ii) to investigate how effective are the coffee grounds in the removal iron and chromium from tap water.
- (iii) to gain information about the toxic effects of iron and chromium in human body.
- (iv) to be aware of safe concentration limits of the toxic metals in our drinking water.
- (v) to achieve better knowledge and experience in handling AAS instrument.

MATERIALS AND METHODS

A. Determination of metals concentration in samples.

In the determination of the iron and chromium concentration in the pure tap water, three methods that were employed include;

- I. direct analysis of the elements in samples of water
- II. acid digestion
- III. determination of metals by complexing

Oxidation of metals in tap water by Acid Digestion Method

100 mL of tap water sample was transferred to a 250 mL heating vessel. 2 mL of 1 M HNO_3 and 10 mL of 1 M HCl were added to the sample. The vessel was covered and heated on the boiling bath with the temperature was adjusted to 92 to 95°C for two hours or until the volume sample was reduced to between 25 to 50 mL. The sample was cooled and filtered to remove insoluble material. Finally, the volume of samples was adjusted to 100 mL with distilled water.

Determination of metals in tap waters using Extraction Procedure

This method describes the determination of iron, chromium and several other metals which are present in low concentrations in tap waters. The metals were simultaneously extracted by chelation with diethyldithiocarbamic acid followed by extraction into methyl isobuthyl ketone (MIBK).

i. Preparation of Diethyldithiocarbamate (DDC) reagent

20 g of diethyldithiocarbamic acid-sodium salt was dissolved in 380 mL of distilled water and filtered through filter paper. The filtrate was extract twice with MIBK, and aqueous samples were used.

ii. Preparation of Phthalate buffer

102 g of potassium biphthalate was dissolved in 500 mL of distilled water, 14 mL of 1 M HCl was added and the solution was diluted to 1 liter with distilled water.

iii. Preparation of sodium hydroxide solution (NaOH), 1M

4 g of NaOH was dissolved in 100 mL of distilled water.

iv. Preparation of samples of water

A 100 mL of water sample is filter through filter paper and then acidify with 16 drops of concentrated (conc.) HCl. The water was transferred into 250 mL Erlenmeyer flask. 2 mL of phthalate buffer was added and the pH of sample was adjusted to 3.6 ± 0.1 . 7 mL of DDC solution was added. The sample was transferred to 500 mL separatory funnel and 15 mL of MIBK was added. The sample was shaken vigorously for 30 seconds, and allowed to separate. MIBK layer which contained the metal complexes was drawn off into a glass-stoppered test tube and the samples were ready for analysis.

B. Preparation of Standard

Prior to the analysis of the samples, the standard solutions have to be prepared for each element concerned.

For chromium, three standard solutions were prepared, in which the concentrations are 1.0, 2.0 and 3.0 ppm. For 1.0 ppm, the 0.1 mL of stock standard solution of chromium is added into 100 mL volumetric flask and the volume was adjusted to 100 mL with E-pure water (double distilled water). For 2.0 ppm standard, 0.2 mL of stock standard solution of chromium used and for 3.0 ppm, 0.3 mL of standard solution is used and E-pure water is added to adjust the volume up to 100 mL. The standards were shaken vigorously before analysed with the AAS.

For iron standard, three standard solutions were prepared by serial dilution method. The concentrations of standards prepared were 1.0, 2.0 and 4.0 ppm. Starting with concentration 4.0 ppm first, 0.4 mL of stock standard solution of iron is added into 100 mL volumetric flask and the volume is adjusted to 100 mL with E-pure water. The solution was shaken vigorously. For preparation of 2.0 ppm standard, 50 mL of the 4.0 ppm solution was transferred into another 100 mL volumetric flask by using 50 mL pipette. The volume in this solution was also adjusted to 100 mL by adding the E-pure water. Then, again 50 mL of second solution is transferred to third volumetric flask for preparing 1.0 ppm standard. E-pure water was added to make up the solution up to 100 mL and the solution was shaken vigorously before analysed with AAS.

C. Description of AAS Instrument

Type Perkin-Elmer Precisely

Model AAnalyst 800

Flames The two oxidant or fuel combinations used exclusively in atomic absorption are air-acetylene and nitrous oxide-acetylene.

Air-Acetylene The temperature of the air-acetylene flame is about 2300°C and the acetylene flow is about 4 liters/minute, or 8.5 cubic feet/hour, using a heat combustion value of 1450 BTU per cubic foot. The heat given off would be approximately 12,300 BTU per hour.

Nitrous Oxide-Acetylene The nitrous oxide-acetylene flame has a minimum temperature of about 2900°C and the acetylene flow is about 14 liters per minute or 30 cubic feet per hour. Using a heat combustion value of 1450 BTU per cubic foot, the heat given off would be approximately 43,000 BTU per hour.

Light Sources The main source used for atomic absorption is the hollow cathode lamp.

Standard Atomic Absorption Conditions for Chromium

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Sensitivity check (mg/L)	Linear Range (mg/L)
357.9	0.7	1	0.078	4	5

Standard Flame Emission Conditions for Chromium

Wavelength (nm)	Slit (nm)	Flame
425.4	0.2	Nitrous oxide-acetylene

Stock Standard Chromium, 1000 mg/L. Dissolve 3.735 g of potassium chromate, K_2CrO_4 in deionized water and diluted to 1 liter with deionized water.

Flames The determination of chromium requires a fuel-rich (yellow) air-acetylene flame. The absorption is sensitive to the fuel-to-air ratio.

Standard Atomic Absorption Conditions for Iron

Wavelength (nm)	Slit (nm)	Relative Noise	Characteristic Concentration (mg/L)	Sensitivity check (mg/L)	Linear Range (mg/L)
248.3	0.2	1	0.100	5	5

Standard Flame Emission Conditions for Iron

Wavelength (nm)	Slit (nm)	Flame
372.0	0.2	Nitrous oxide-acetylene

Stock Standard Iron, 1000 mg/L. Dissolve 1 g of iron wire 50 mL of (1+1) HNO₃.

Solution used Diluted to 1 liter with deionized water.

Light sources With multielement lamps containing cobalt, an interference may occur when using the 248.3 nm iron line.