CHAPTER 4

CONCLUSION

The main propose of this research is the study on the concentrations of Cr, Ni and Pb in human hair samples. For the determination of Cr, Ni and Pb in human hair samples, the hair samples were prepared by ultrasonic acid digestion. Then, the analyte was preconcentrated by cloud point extraction and followed by the analysis of Cr, Ni and Pb concentrations using FAAS. Various parameters that influence an ultrasonic acid digestion, such as acid mixture, presonication time, sonication time and temperature of the ultrasonic bath have been investigated. The suitable conditions for UAD were as follow: an acid mixture of 2 ml concentrated HNO_3 and 1 ml H_2O_2 , 10 min presonication time, 30 min sonication time and 60 ºC of temperature of the ultrasonic bath. After digestion, the sample solutions were preconcentrated by cloud point extraction. Chromium, nickel and lead were complexed with ammonium-pyrrolidine dithiocarbamate (APDC) and Triton X-114 was added as non-ionic surfactant. These complexes were extracted into the surfactant-rich phase, at above its cloud point temperature. The surfactant-rich phase was diluted with methanol prior to its analysis by FAAS. The chemical variables that affect the cloud point extraction, such as pH of solution, complexing agent concentration, Triton X-114 concentration and complexing time and temperature were optimized. Under optimal conditions, the cloud point was formed at 40°C with complexing time of 30 minutes, a 5 ml of sample solutions was used in the presence of 6×10^{-4} mol l^{-1} APDC and 0.10 %v/v Triton X-114 at pH 4. The selected conditions will be employed in real samples.

For the determination of Cr, Ni and Pb in real samples, the proposed method was applied to determine the concentrations of Cr, Ni and Pb in human hair samples. The human hair samples including untreated hair sample (A), hair sample from drug abuse person (B), hair sample from dyed color hair person (C-1 and C-2), hair sample from smoking person (D-1 and D-2) and hair sample from industry worker (E-1 to E-3) were studied. The concentration of Cr in human hair samples can be found in E-2. Sample A, C-1, D-1, D-2 and E-1 could not be detected the concentration of Cr by FAAS. The concentration of Ni in human hair samples can be ordered as following: E-3 > C-2. Sample A, B, C-1, D-1, D-2, E-1 and E-2 could not be detected the concentration of Ni by FAAS. The determination of Pb in human hair samples could not be detected the concentration of Pb but the content is below than LOD.

The accuracy expressed in term of percentage recoveries of this method was found in the range of 91.90 – 99.69%, 90.69 – 95.59 and 98.89 – 101.48% for Cr, Ni and Pb, respectively. The detection limits of Cr, Ni and Pb were 0.077 and 0.162 and 0.249 μg ml^{-1}, respectively. The precisions expressed in term of relative standard deviation (%RSD) were 1.27 % for chromium 1.59 % for nickel and 0.81 % for nickel. The linear range of calibration curves were in the range of 0.50-4.00, 0.50-4.00and 1.00-20.00 μg ml^{-1} for Ni, Cr and Pb, respectively. The results indicated that the proposed method was reasonable for trace analysis, resulting in good recoveries of Cr, Ni and Pb. The detection limits of the method are comparable level with the works in literature including cloud point extraction.

The proposed method was applied to analyze the human hair samples. For this purpose, appropriate amounts of Cr, Ni and Pb standard solutions were added into

hair sample solutions before digestion. The high concentration of Cr, Ni and Pb was found in hair sample from electronic and lens industry worker. The lower concentrations of Cr, Ni and Pb were found in hair sample from dyed color hair person and hair sample from smoking person. The minimum concentration of Cr and Ni was found in hair sample from drug abuse person. The concentration of Cr, Ni and Pb could not be detected in untreated hair sample.

In this study, a safe and rapid digestion method has been established for the reliable extraction of metals in human hair samples. The efficiency of ultrasonic acid digestion has demonstrated a good performance related to solid sample preparation. Several benefits offered by UAD are shortened sample preparation times, simplicity and enhanced safety compared to other more traditional methods. The low costs of the ultrasonic baths, the UAD can be considered as viable procedures for many laboratories. We have proposed the use of cloud point extraction as an alternative method for the preconcentration of metals as a prior step to their determination at micrograms per milliliter levels in human hair samples by FAAS which is available in most laboratories. CPE offers a simple, rapid, inexpensive and nonpolluting alternative to other preconcentration techniques. The presented method can be applied to the determination of metals in human hair samples by FAAS.