

# Preconcentration of heavy metals by cloud point extraction

Mustafa Kocer<sup>1</sup>, Albert Maxima<sup>2</sup>, Anita Gonzales<sup>2</sup>

<sup>1</sup> xxxxxx University, Department of xxxxxxxx, Country  
E-mail address



## INTRODUCTION

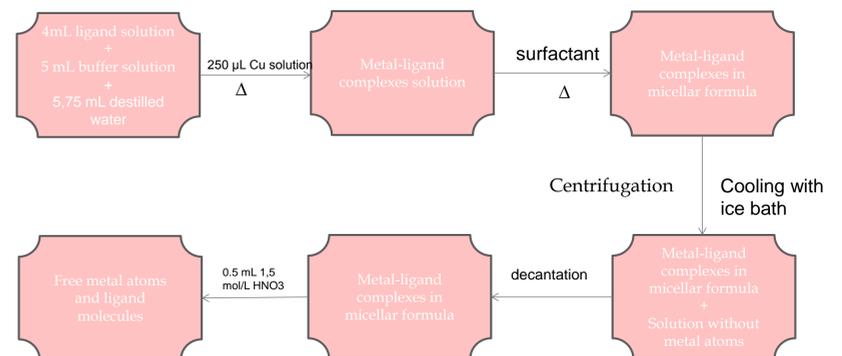
Determination of trace metal ions in natural water samples is interesting and important for analytical chemistry. To detect them, we can use several methods such as atomic absorption spectroscopy, inductively coupled plasma atomic emission spectrometry, and so on. However, it is difficult to determine trace metals in their samples directly, because of their low concentration and coexistence of matrix species. Therefore, pre-concentration and separation of analyte are needed before measuring. The most common procedures for the pre-concentration of analytes interested are the use of either liquid-liquid or solid sorbents extraction. Recently, a cloud point extraction of organic and inorganic compounds using non ionic surfactants have been concerned in analytical chemistry. [2,5-9] Cloud point extraction (CPE) is a separation and preconcentration method [4]. This method has been some advantages such as low cost, rapidly procedure and widely application field. Cloud point extraction has been applied to determination of trace metal ions from different matrices recently years. A cloud point extraction preconcentration technique is still being developed nowadays. If this technique was vested in the basic structure, which simply described as follows. Firstly, ligand solution is added into the solution of dissolved heavy metal atoms. Thus, metal-ligand complex is created then the surfactant is added to this solution. Thereafter, the resulting solution is heated. Thereby, the cloud point temperature is reached. Solution was heated at this temperature for a certain period of time. Micelle formation is provided. The micelle solution was centrifuged to collect together. After centrifugation, the solution and rich phase appropriately separated from each other. Finally, rich phase is dissolved back some of the solvents. Dissolved in a suitable concentration of metal atoms is analyzed using the spectrophotometric method. In these studies, optimized parameters; pH, metal concentration, the quantity the ligand, the cloud point temperature[1,3], clouding time and duration of centrifugation. [10-14]

## EXPERIMENTAL WORK

In this study, Copper (Cu) was selected as the metal atom. Model solutions, purchased at Merck 1000 mg / l of solution concentration were prepared. Quantity of metal, was chosen of 2.5 µg at all experimental works but in experiments of metal concentration, it was different. 5,7-diiodo-8-hydroxyquinoline as a ligand selected. NaH<sub>2</sub>PO<sub>4</sub>/K<sub>2</sub>HPO<sub>4</sub> solution was used as the buffer solution. 5 ml of buffer solution and 16 mg of ligand were used in the model solutions but in experiments of ligand concentration, quantity of ligand was used different. IGEPAL CO 520 purchased from Sigmaaldrich, was used as a surfactant. This surface active agent was chosen because it does not absorb the ligand without metal atoms. Surfactant was used 0.33% (v / v) for all model solutions. Cloud point temperature was set at 76 °C. Cloud point time was determined as 45 minutes. Centrifugation time was chosen 10 minutes. Spin speed of centrifugation was chosen 6000 rpm. 1.5 mol / L HNO<sub>3</sub> was used for recovery.

The model solutions are analyzed using inductively coupled plasma optical emission spectrometer (ICP-OES). Spectra - Optima 2100 DV model ICP-OES instrument used in all experiments. All preconcentration operations were performed in the volume of 15 ml polypropylene centrifuge tubes.

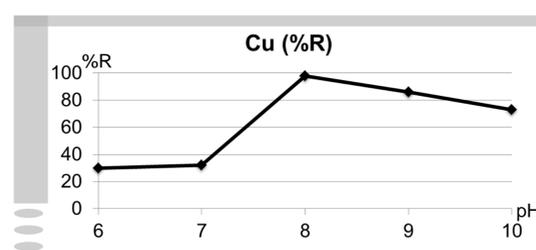
## METHODS



## RESULTS AND DISCUSSIONS

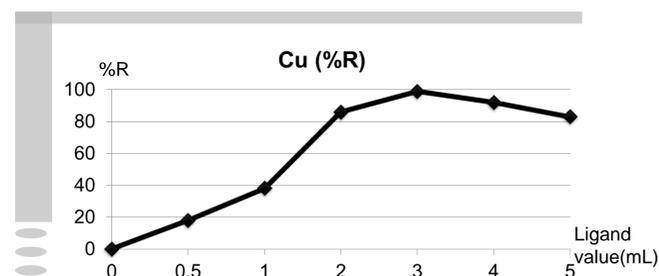
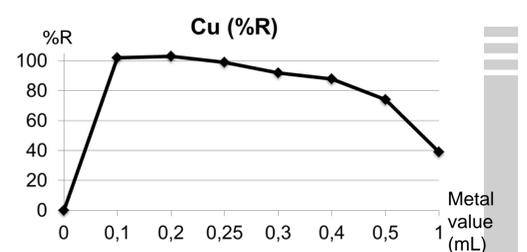
At the end of the experimental work in optimum pH, temperature, the quantity metal atoms and the quantity ligand molecules were analyzed. According to results, the copper atoms of the best conditions for recovery at pH 8, the temperature at 75 C, and 3 mL of 0.25 volume of the metal was analyzed in the volume of the ligand.

These important factors, such clouding time, centrifugation time, is planned to optimize factors such as speed centrifugation.

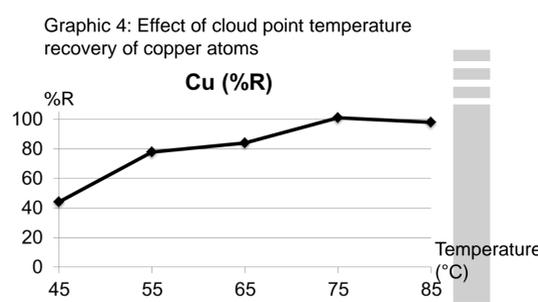


Graphic 1: Effect of pH recovery of copper atoms

Graphic 2: Effect of added metal value recovery of copper atoms



Graphic 3: Effect of added ligand value recovery of copper atoms



Graphic 4: Effect of cloud point temperature recovery of copper atoms

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