CHAPTER I

INTRODUCTION

The application of solid-phase extraction (SPE) technique for preconcentration of trace metals from different samples bring about several advantages such as the minimal waste generation, reduction of sample matrix effects as well as sorption of the target species on the solid surface in a more stable chemical form [1-3]. The core of solid phase extraction technique is the sorbent or supporting material that determines the degree of migration and separation of the components contained in the samples. Therefore, the development of SPE technique can be performed by developing the sorbent. The support using in the SPE is generally either polymer, e.g. polystyrene divinylbenzene resins or inorganic, e.g. silica, alumina and zirconia. Silica is the most popular support because of its resistance to mechanical deformation, hydrophilic properties, negligible swelling and chemical inertness [4-5]. However, the surface of silica is characterized by the presence of silanol groups, which are known as weak ion exchanger, causing low interaction, binding and extraction of the target analytes. For this reason, the silica available for inorganic trace element is not very selective. Therefore, it is possible to increase the selectivity of natural silica by functionalization processes [4-5].

To bring selectivity to the silica, impregnation or chemical derivatization with a chemical reagents are currently used [6]. The chemical derivatization is based on chemical bond formation between the silica surface groups and the organic modifier. This type of encapsulation generally requires several synthesis steps and has to be tailored for each organic reagent. With the impregnation technique, the reagent is physically adsorbed on the solid support. This technique is highly versatile and a large variety of reagents have already been loaded on silica. However, the binding force is quite low in this case and leaching is currently observed. Another route for immobilizing reagents is the direct encapsulation of a molecule by using sol-gel doping technology. In this case, the synthesis of silica is carried out in solution at low temperature, which allows the introduction of the organic molecule in a mixture of sol-gel precursors. The sol-gel doping technique is gaining popularity as a result of its generality and simplicity. The concept of the sol-gel method is intermediate between

impregnation and chemical derivatization technique and has provided a general and inexpensive route for the immobilization of reagent [7].

In addition, the pore size of silica is particularly crucial for extraction applications because this parameter controls the kinetics of the exchanges [6, 8]. Mesoporous silica (pore diameter 20-500 nm) is expected to provide superior extraction ability. This silica is prepared by micelle-templated sol-gel polymerization method which produces pore diameter adjustable between 2 and 300 nm, with narrow pore size distributions. The obtained material was synthesized by this method has also a great specific surface area [9-11].

In this study, 1-phenyl-3-methyl-4-stearoyl-5-pyrazolone (HPMSP) was encapsulated in mesoporous silica via a micellar route. The influence of concentration of NaOH as catalyst and amount of incorporated HPMSP on the synthesis of doped mesoporous silica were investigated. The adsorption-desorption behaviors of the obtained materials toward copper, cobalt and nickel were studied.

This thesis is divided into five chapters. Introduction of this work is described in the first chapter. Review of theory concerning solid-phase extraction, synthesis and properties of silica, method for functionalization of silica and method for characterization of solid materials are summarized in chapter II. In chapter III, the procedure for experiments is discussed. Next, details and discussion over obtained results are presented in chapter IV. Finally, the conclusion of this work is summarized in the last chapter.