CHAPTER I

INTRODUCTION

Recently, solid-phase extraction (SPE) has been received much interest in the analytical field, especially for the extraction and separation of different organic compounds from environmental matrices [1-3]. This technique is superior to the liquid-liquid extraction in terms of simplicity, rapidity and the ability to obtain a high enrichment factor. Additionally, it reduces solvent usage and exposure, disposal costs and extraction time. However, the SPE method is still in need of development to obtain a more rapid, efficient and sensitive method for better extraction of the target species. One important factor of SPE is sorbent or solid support including silica, alumina, polymeric resin, activated carbon, etc. [4-7]. Among these materials, silica is the most widely used because of its good mechanical strength and thermal stability. However, commercial silica has very low selectivity especially for metal extraction. Thus, increasing of selectivity and efficiency of silica to support those requirements is an aspect of interest.

In general, three methods including chemical derivatization, impregnation and doping technique are used for silica modification to enhance its selectivity [8]. The doping technique is gaining popularity especially for its use in chemical or biochemical optical sensor, ion-selective electrode, solid-phase extraction and others due to its high versatility and simplicity [9-11]. For this method, the organic molecules are incorporated in silica through the sol-gel process. Consequently, the leaching of organic molecules could be reduced. In addition, the sol-gel route enables to control the physicochemical properties of the resulting materials.

For the selectivity improvement of silica, the use of chelating agent incorporated in silica is one of another route for this purpose. 1-Phenyl-3-methyl-4-stearoyl-5-pyrazolone (HPMSP) is an attractive ligand that could be immobilized on the surface of silica to raise the metal extraction selectivity of sorbent. This molecule has received much interest because of its potential to form different types of coordination compound due to several electron-rich donor centers and tautomeric enol and keto forms. Consequently, this molecule has frequently been used as a reagent for liquid-liquid extraction [12-14]. However, there are only a few reports on the use of HPMSP as a reagent for the much more efficient SPE technique. From our literature

survey, most of studies were concentrated on the synthesis of HPMSP modified microporous silica using impregnation method [15-16]. The HPMSP functionalization of silica using doping technique via sol-gel process was first introduced in 2000 [17-18]. The HPMSP doped mesoporous silica was found to be better than the HPMSP doped microporous gel in terms of facility and rapidity of the synthesis. Furthermore, these mesoporous materials had much more metal extractability than do the microporous sorbents due to their high surface area and better structural order. The influence of parameters on the synthesis and the metal extraction properties of HPMSP doped mesoporous sorbent was also investigated by the same research group [19]. However, the study was concentrated on only some metals and using only batch method for the extraction procedure.

Thus, the aim of this work is to investigate the metal sorption behavior of HPMSP doped mesoporous silica in both batch and column methods. The type of metal, the pH of metal solution, the presence of foreign ions and the concentration of $NaNO_3$ in metal solution are the factors to be evaluated in batch method. For the column method, the parameters to be optimized are the amount of metal, the concentration and the volume of eluent. The potential application of HPMSP doped mesoporous silica as a sorbent for metal extraction from food sample is also demonstrated in the last section.