



Review

Complex-forming organic ligands in cloud-point extraction of metal ions: A review

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ABSTRACT

Cloud-point extraction (CPE), an easy, safe, environmentally friendly, rapid and inexpensive methodology for preconcentration and separation of trace metals from aqueous solutions has recently become an attractive area of research and an alternative to liquid–liquid extraction. Moreover, it provides results comparable to those obtained with other separation techniques and has a greater potential to be explored in improving detection limits and other analytical characteristics over other methods. A few reviews have been published covering different aspects of the CPE procedure and its relevant applications, such as the phenomenon of clouding, the application in the extraction of trace inorganic and organic materials, as well as pesticides and protein substrates from different sources, or incorporation of CPE into an FIA system. This review focuses on general properties of the most frequently used organic ligands in cloud-point extraction and on literature data (from 2000 to 2012) concerning the use of modern techniques in determination of metal ions' content in various materials. The article is divided according to the class of organic ligands to be used in CPE.

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Abbreviations: 5-Br-PADAP, 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol; PAN, 1-(2-pyridylazo)-2-naphthol; PAR, 4-(2-pyridylazo)resorcinol; TAN, 1-(2-thiazolylazo)-2-naphthol; TAR, 4-(2-thiazolylazo)resorcinol; TAC, 2-(2'-thiazolylazo)-p-cresol; BDAP, 2-(2'-benzothiazolylazo)-5-(N,N-diethyl)aminophenol; MBT, 2-mercaptobenzothiazole; Br-TAO, 4-(5-bromo-2-thiazolylazo)resorcinol; Me-BTABr, 2-[2'-(6-methyl-benzothiazolylazo)]-4-bromophenol; Br-TACl, 2-(5-bromothiazolylazo)-4-chlorophenol; APDC, ammonium pyrrolidine dithiocarbamate; DDTc, diethylammonium-N,N'-diethyldithiocarbamate; 8-HQ, 8-hydroxyquinoline; DDTP, ammonium O,O'-diethyldithiophosphate; TMK, 4,4'-bis(dimethylamino) thiobenzophenone, e.g. Thio-Michler's Ketone; morin, 3,5,7,2'-4' pentahydroxy flavone; BPHA, N-benzoyl-N-phenylhydroxylamine; ACDA, 2-amino-cyclopentene-1-dithiocarboxylic acid; PMBP, 1-phenyl-3-methyl-4-benzoyl-5-pyrazolone; HEPTS, 4-ethyl-1-(pyridin-2-yl)thiosemicarbazide; HDEHP, di(2-ethylhexyl)phosphoric acid; NDDBH, 6-(4-nitrophenyl)-2,4-diphenyl-3,5-diaza-bicyclo[3.1.0]hex-2-ene; THF, tetrahydrofuran; DMF, dimethylfuran; BDTAC, benzyldimethyltetradecylammonium chloride; CTAB, cetyltrimethylammonium bromide; CPC, cetyl pyridinium chloride; SDS, sodium dodecyl sulfate; FAAS, flame atomic absorption spectrometry; ETAAS, electrothermal atomic absorption spectrometry; GFAAS, graphite furnace atomic absorption spectrometry; CVAAS, cold vapor atomic absorption spectrometry; HGAAS, hydride generation atomic absorption spectrometry; TS-FF-AAS, thermospray flame furnace atomic absorption spectrometer; ICP-OES, inductively coupled plasma optical emission spectrometry; ICP-MS, inductively coupled plasma mass spectrometry; ETV-ICP-OES, electrothermal vaporization inductively coupled plasma atomic emission spectrometry; USN-ICP-OES, inductively coupled plasma optical emission spectrometry with ultrasonic nebulization; CV-ICP-OES, cold vapour optical emission spectrometry; ETV-ICP-MS, electrothermal vaporization inductively coupled plasma mass spectrometry; ICP-DRC-MS, inductively coupled plasma mass spectrometry with dynamic reaction cell; SPF, spectrofluorimetry; CVAFS, cold vapor atomic fluorescence spectrometer; ETVAFS, electrothermal vaporization flame atomic fluorescence spectrometry; TLS, thermal lens spectrometry; FO-LADS, fiber optic-linear array detection spectrophotometry; CE, capillary electrophoresis; NAA, neutron activation analysis; HPLC, high-performance liquid chromatography

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1. Introduction

Determination of trace amounts of metals in a complex matrix has been usually complicated, despite modern analytical instrumentation now available, even with adequate sensitivity. In such matrixes, determination should be preceded by separation and preconcentration steps. This enables minimizing or even eliminating matrix effects and contaminants, lowering the detection limit of many metals with different techniques and enhancing the detectability for others. Thanks to green chemistry concept, modern analytical separation/preconcentration procedures should be simple, ecologically safe, sensitive, and selective for microcomponents' determination, combining concentration and further determination by physical or physico-chemical methods. Several strategies were adopted aiming at greener analytical procedures, involving replacement of toxic reagents, minimized reagent consumption, recycling and waste treatment. One of them is the use of surfactants, due to their low toxicity and relatively low cost. Moreover, interest in application of surfactants is related to their unique microheterogeneous structures capable of selective interaction with different solute molecules which can strongly modify solubility, chemical equilibrium, kinetics and the spectroscopic properties of analytes and reagents. Therefore, in the last few decades, development has been observed concerning surfactant-based methods in many chemical and pharmaceutical fields, organic synthesis and several industrial applications. Micellar systems are also widely utilized in analytical chemistry, especially in separation/preconcentration procedures and determination based on spectral methods.

Aqueous solutions of many non-ionic and zwitterionic surfactant micellar systems become turbid over a narrow temperature range, when the experimental conditions have been changed. This temperature is named "cloud point temperature" (CPT) or "lower consolute temperature" (LCT). The value of CPT depends on the structure and concentration of the surfactant, and the presence of additives such as salts, alcohols, other surfactants, polymers, and some organic or inorganic compounds, which can cause an increase or decrease of CPT. Therefore, in micellar systems two types of phase separation can be observed: upper consolute type (occurring above CPT) and lower consolute type (occurring below CPT). The lower consolute type of phase separation is rarely obtained in micellar systems. Nevertheless, in some cases the cloud point was attained below CPT and even at room temperature, by changing pressure or using various additives, including electrolytes [1–4]. In micellar media, the upper consolute type of phase separation dominates. In this case, clouding phenomenon is observed especially with polyoxyethylene surfactants and can be ascribed to the efficient dehydration of hydrophilic portion of micelles at higher temperature conditions, and due to the interaction of nonionic surfactant micelles via an attractive potential, whose well-depth increases with temperature. These micelles attract each other and form clusters with the approach of the cloud point. However, the mechanism behind the lower consolute behavior of nonionic surfactant systems still remains obscure. Earlier, the phase separation was attributed to the micellar growth, the micellar coacervation, or the changes in poly (oxyethylene) chain conformations with temperature. However, recent experimental and theoretical investigations put forth the view that the formation of the connected micellar network or the

strongly orientation-dependent interactions (H bonds) between water and the surfactant molecules could be responsible for the lower consolute behavior. Moreover, the roles of oscillation in the critical concentration, and of micellar growth as mechanisms for the clouding phenomenon are still controversial [5–7]. More details about the clouding behavior of ionic and nonionic surfactants and influence of different kinds of additives on CPT can be found in reviews by Mukherjee et al. and Bezerra et al. [7,8].

Clouding behavior of micellar solutions is widely exploited as CPE for the extraction and preconcentration of various metal ions, organic and inorganic industrial pollutants, pesticides and proteins. The typical cloud point methodology used for extraction of metal ions is given in Fig. 1.

Cloud point extraction (CPE), as an effective separation/preconcentration technique, first studied by Watanabe and co-workers in the early 1980s [9–12], has been demonstrated to have the distinct merits of low cost, simplicity, speed, lower toxicity to the environment than extractions that use organic solvents and a high capacity to concentrate a wide variety of analytes of widely varying nature with high recoveries and high concentration factors. On the other hand, simple combination with spectral, atomic absorption, chromatographic, and electrochemical analyses allows using cloud-point extraction for elaborating high-sensitive and convenient analytical methods. The frequency of use of analytical techniques combined with CPE that were proposed in the past 12 years is given in Fig. 2. As can be seen, flame atomic absorption spectrometry (FAAS) is by far the most widely employed technique for analyte determination, followed by CPE preconcentration. FAAS has been a very attractive technique for routine metal determinations due to well-known interferences, its ease of operation, its low acquisition and operating costs compared with electrothermal atomic absorption spectrometry (ETAAS), graphite furnace atomic absorption spectrometry (GFAAS) or inductively coupled plasma mass spectrometry (ICP-MS), and its high sample throughput.

In the CPE technique, surfactant is one of the key reagents. The surfactants which are used in cloud point extraction are mostly of nonionic type, such as Triton X-114 (polyoxyethylene-7.5-octylphenoxy ether), Triton X-100 (polyoxyethylene-9.5-octylphenoxy ether) or PONPE (polynonylphenyl ether). In some cases, mixtures of nonionic and cationic/anionic (CTAB, CPC, BDTAC, and SDS)

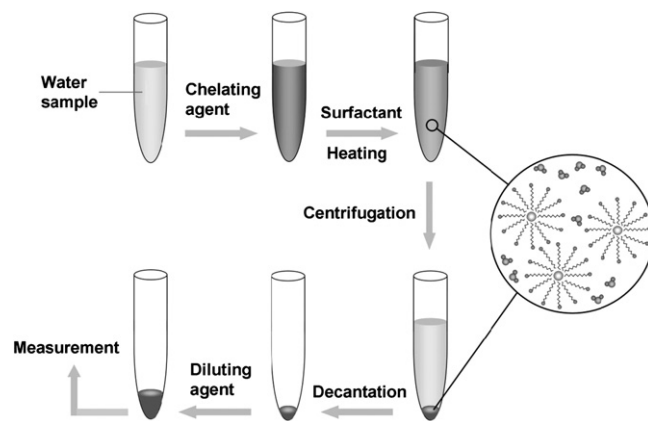


Fig. 1. Schematic representation of a conventional CPE to metal preconcentration.

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