



N,N'-dimethyl-*N,N'*-dicyclohexylsuccinamide: A novel molecule for the separation and recovery of Pd(II) by liquid-liquid extraction

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ABSTRACT

N,N'-dimethyl-*N,N'*-dicyclohexylsuccinamide (DMDCHSA) is investigated as a potential molecule for the liquid-liquid extraction of Pd(II) from chloride solutions for the first time. The effect of several parameters on Pd(II) extraction, such as the contact period between both phases, hydrochloric acid, extractant and hydrogen ion concentrations, is evaluated. Pd(II) extraction equilibrium is very fast (30 s) and the extraction percentage (%E) increases with the HCl concentration in the aqueous phases, being higher than 60% for [HCl] > 5 M. The loading capacity of DMDCHSA for Pd(II) is reasonable (molar ratio extractant/metal higher than 16). Several stripping agents (e.g. distilled water, 1 M HCl, seawater and 20 g/L chloride solution as NaCl) were successfully used to transfer Pd(II) to a new aqueous phase, and data obtained from five successive extraction-stripping cycles suggest a good DMDCHSA stability pattern. Attempts to replace 1,2-dichloroethane (1,2-DCE) by commercial and more environmentally friendly diluents showed much worse %E for Pd(II). Selectivity tests with binary, ternary and more complex metal ion solutions were carried out to evaluate the performance of DMDCHSA towards Pd(II) recovery from 6 M HCl, when in presence of Pt(IV), Fe(III), Zn(II), Al(III) and Ce(III), metal ions usually present in solutions that may result from the hydrometallurgical treatment of spent automobile catalytic converters. It was generally observed that the additional metal ions do not affect the recovery of Pd(II) by DMDCHSA, although Fe(III) and Pt(IV) were co-extracted in a great extent. A solvent extraction (SX) scheme is proposed, based on a previous separation of Fe(III) with tributylphosphate (TBP) and on the selective and sequential stripping of Pt(IV) and Pd(II) from the loaded DMDCHSA with 0.01 M thiourea in 0.5 M HCl and seawater, respectively. The dependence of the Pd(II) distribution ratios on DMDCHSA and acidity, complemented with UV-Visible spectroscopy data, points out to DMDCHSA: Pd(II) extracted species with a 2:1 molar ratio and suggests the occurrence of an outer-sphere ion pair reaction, in which both [PdCl₄]²⁻ and HCl are extracted.

1. Introduction

Platinum group metals (PGMs) have a high economic value and have been considered critical raw materials [1]. These facts are associated to the PGMs scarcity in the Earth's crust, to the difficult replacement of PGMs in many applications (fuel cells, electronics and catalysis) and also to the high supply risks associated to them [2].

Palladium belongs to the PGMs group, representing only $1.5 \times 10^{-6}\%$ of the Earth's crust; its demand and production led to an increase of its market price in the last few years [3–6]. Palladium has

been used for several purposes, including automobile catalysts, electronics, industrial catalysts, electronic circuitry, dental alloys, jewellery, and lately in the treatment of cancer [7–9]. Hence, the recovery and reuse of PGMs, namely palladium, from industrial wastes is crucial, for technical and economical reasons [10].

Industries have been using different methods to recover PGMs from aqueous solutions in the past few years: solvent extraction (SX) (also known as liquid-liquid extraction), ion exchange, evaporation, cementation, chemical precipitation, electrochemical and sorption methods [11–14]. PGMs recovery by hydrometallurgical processes

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