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Abstract: *N*-methyl-*N*-cyclohexyloctanamide (MCHOA) and *N*-methyl-*N*-phenyloctanamide (MPHOA) were synthesized, characterized, and tested to recover iron(III) from chloride media. Both compounds efficiently extract Fe(III) from HCl solutions above 4 M, although a decrease in extraction at 10 M HCl, involving MCHOA, is noticed. Stripping of Fe(III) by water may be easily and efficiently achieved. Results on the re-utilization of the organic phases are also presented. Preliminary selectivity experiments point out to a rather good performance of both compounds towards Fe(III) when in the presence of Cu(II), Ni(II), and Zn(II). Assuming that the extractants are under a monomeric form, average species such as $[3\ LH^+ \cdot FeCl_4^- \cdot 2\ Cl^-]$ (L, MCHOA or MPHOA) are proposed as being involved on Fe(III) extraction from 6 M HCl solutions.

Keywords: Amide derivatives, chloride media, Fe(III) extraction

INTRODUCTION

Chloride hydrometallurgy has been intensively developed in the last 20 years (1). There are some worldwide industrial and pilot plant facilities in operation involving chloride solutions, for instance, in the recovery of gold (2) and zinc (3).

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Iron is usually a contaminant in the pregnant leaching solutions, being traditionally separated by precipitation (4). However, this practice produces high amounts of waste whose disposal is environmentally undesirable (5). Additionally, those deposits usually contain important amounts of more valuable metals, this constraint causing operational and financial drawbacks that negatively affect the efficiency of the whole processes (5).

Some solvent extraction routes to recover/separate iron(III) from chloride leaching solutions have been appearing in literature and were briefly reviewed in 2006 (6). Most of the developed methods aim to efficiently extract other metal ions and leave Fe(III) in the feed aqueous phases. More recently, some pyrazolone derivatives to selectively extract V(V) over Fe(III) for hydrochloric acid solutions above 1 M were proposed (7), as well as the use of isoxazolones to extract Al(III) and Ti(IV) (8). Dibutylbutylphosphonate has also been successfully applied to the selective removal of Zn(II) in the presence of Fe(II) in chloride media (9), but the process was not viable to separate Zn(II) when Fe(III) was present. The mutual separation of Fe(III), Cu(II), and Zn(II) from a mixed 5 M Cl^-/0.5 M sulfate media has been reported when tributylphosphate (TBP), LIX 841, and Cyanex 923, respectively, were employed (10). Undiluted TBP has also been proposed as a good extracting agent to selectively recover Zn(II) over Fe(III) from pickling solutions resulting from hot dipping galvanizing baths (11).

Several *N,N′*-tetraalkylmalonamide derivatives have been systematically used to recover Fe(III) from concentrated HCl solutions (6,12–15), and the data achieved clearly reveal the enormous potential of these sorts of compounds to efficiently and selectively recover Fe(III) from concentrated chloride solutions.

In this work two amides were synthesized and characterized—*N*-methyl-*N*-cyclohexyloctanamide (MCHOA) and *N*-methyl-*N*-phenyloctanamide (MPHOA)—and subsequently tested to verify if they would present good Fe(III) extraction behavior by direct comparison with that of malonamides having identical structural moieties and a similar number of carbon atoms (*N,N'*-dimethyl-*N,N′*-diphenylmalonamide (DMDPHMA) (6,12) and *N,N′*-dimethyl-*N,N′*-dicyclohexylmalonamide (DMDCHMA) (6,14).

EXPERIMENTAL

Synthesis and Characterization

MCHOA and MPHOA were synthesized from the reaction of octanoyl chloride with *N*-methylcyclohexylamine and *N*-methylaniline,

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respectively, according to well-established procedures (16). Octanoyl chloride was obtained by refluxing octanoic acid at about 76°C (39.60 mL, 0.22 mol) with thionyl chloride (19.50 mL, 0.27 mol) for 1 hour. The excess of thionyl chloride was removed in a rotary evaporator and the crude product (octanoyl chloride) was immediately used for the next syntheses. Hence, for each amide, half of the oily octanoyl chloride (17.88 g, 0.11 mol) was dissolved in n-hexane and *N*-methylcyclohexylamine (12.43 g, 0.11 mol) or *N*-methylaniline (11.77 g, 0.11 mol) were slowly added. Triethylamine was also added to each solution (15.30 mL, 0.11 mol) to guarantee an alkaline medium. Both reaction mixtures were left under stirring at room temperature for 3 hours. After washing with distilled water (2×100 mL), a 6 M HCl solution (1×100 mL) and again with distilled water (1×100 mL), both extracts were dried over anhydrous magnesium sulfate, filtered, and the solvent evaporated; both MCHOA and MPHOA were finally isolated. The yields were 87% for MCHOA and 83% for MPHOA. Several characterization methods were used—gas chromatography (GC, HP 4890), Fourier transform-infrared spectroscopy (Satellite FT-IR Mattson, with a resolution of 2 cm^{-1}) proton nuclear magnetic resonance (^1H, 400 MHz NMR, Bruker 400 Avance spectrometer), and gas chromatography coupled with mass spectroscopy (GC-MS, GC Agilent 6890A, capillary column TR-520232 TRB-5MS, MS Agilent 5973). The characterization results showed that both compounds had sufficient purity to be used for the solvent extraction experiments without further purification-see related data on Table 1.

Table 1. Physical, chromatographic and spectroscopic data obtained for MCHOA and MPHOA

Collected data	MCHOA	MPHOA
Physical appearance	Oily liquid orange-yellow	Oily liquid orange-yellow
FT-IR (ν_{max}, NaCl pellets/cm^{-1})	2928, 2855, 1644 (C=O), 1451, 1406, 1090	2927, 2856, 1660 (C=O), 1597, 1496, 1365, 702
^{1}*H NMR* (400 MHz; $CDCl_3$, Me_4Si, δ in ppm)	0.8 (3H, triplet, $-CH_3$ alkyl chain), 1.1–1.6 (10H, multiplet, $-(CH_2)_5-$ and 10H, multiplet, $[-(CH_2)_5+CH]$ cyclohexyl), 2.2 (2H, multiplet, $-CH_2CO$), 2.7 (3H, singlet, CH_3-N-)	0.8 (3H, triplet, $-CH_3$ alkyl chain), 1.2 (8H, multiplet, $-(CH_2)_4-$), 1.5 (2H, multiplet, $-\mathit{CH}_2CH_2CO-$), 2.0 (2H, multiplet, $-CH_2CO$), 3.2 (3H, singlet, CH_3-N-), 7.1–7.4 (5H, aromatic–CH)
GC-MS (retention time in min; % purity; m/z)	19.6; 99; 239 (molecular ion), 155 (base peak), 70	18.3; 99; 233 (molecular ion), 149, 107 (base peak)

Solvent Extraction Experiments

Hydrochloric acid solutions, with concentrations varying between 1 and 10 M and containing ~0.01 M of iron(III) (prepared by dissolving $FeCl_3 \cdot 6H_2O$ p.a.), were used as aqueous phases. 0.1 M solutions of each extractant in 1,2-dichloroethane (1,2-DCE) were generally used as organic solutions.

Extraction experiments were performed in stoppered Erlenmeyer flasks by stirring the aqueous and the organic phases for 15 minutes at a constant speed (1000 r.p.m.) and at room temperature, with an aqueous/organic volume ratio (A/O) of 1. Two replicate experiments were carried out for each condition. After stirring, both phases were placed in a separating funnel and subsequently separated.

The iron content in the aqueous phases was analyzed before and after extraction by flame atomic absorption spectroscopy (AAS) using Pye Unicam equipment, model SP9. For each sample, three aliquots were considered and the results were critically treated and only accepted if a reasonable standard deviation was achieved. The iron concentration in the organic phases was found by mass balance.

In the stripping experiments, the Fe(III) loaded organic phases were singly contacted with distilled water, a similar set of experimental conditions as the ones previously followed being again adopted.

The reutilization experiments consisted of four successive cycles of extraction/stripping. A solution containing 0.01 M Fe(III) in 8 M HCl was contacted with an organic solution of 0.1 M MCHOA or MPHOA in 1,2-DCE under the usual set of experimental conditions and, after each stripping stage with distilled water, the same organic extract was again equilibrated with a fresh portion of aqueous solution.

For the selectivity experiments, a 6 M HCl solution containing 0.01 M Fe(III), Cu(II), Ni(II), and Zn(II)—from the corresponding chloride salts—was equilibrated with either 0.1 M MCHOA or MPHOA in 1,2-DCE. All the initial and final aqueous phases were analyzed by AAS and the metal ion contents in the organic extracts were found again by mass balance.

For the study of the dependence of Fe(III) extraction on extractant concentration, solutions of 0.01 M Fe(III) in 6 M HCl were prepared as mentioned before. They were subsequently put in contact with different organic solutions ($0.002\,\text{M} < [\text{MCHOA}] < 0.03\,\text{M}$ and $0.006\,\text{M} < [\text{MPHOA}] < 0.08\,\text{M}$), following the same experimental conditions. Two replicate experiments were carried out for each condition.

For the application of spectroscopic techniques (^1H NMR), equal volumes of a 8 M HCl solution and an organic phase containing 0.1 M MCHOA or MPHOA in dichloromethane were equilibrated, adopting

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the same scheme reported previously. After phase separation, the organic solutions were dried over anhydrous magnesium sulfate, filtered, and evaporated to dryness. The residues were dissolved in $CDCl_3$ with tetramethylsilane.

RESULTS AND DISCUSSION

Time Necessary to Reach Equilibrium

Preliminary experiments were carried out to evaluate how much time would be necessary for the extraction systems to attain equilibrium. Hence, the general set of experimental conditions was adopted for both amide derivatives, for which an aqueous solution 0.01 M Fe(III) in 5 M HCl was used. The equilibration time of the experiments varied between 30 and 1800 seconds. As can be seen in Fig. 1, equilibrium for both systems is soon reached; the percentage Fe(III) extraction values achieved after 60 s are practically similar to the ones obtained for longer periods of contact. It was then decided to adopt 900 s for the equilibration time involving MCHOA or MPHOA in the subsequent experiments. This period of time is similar to the one followed for the malonamide derivatives

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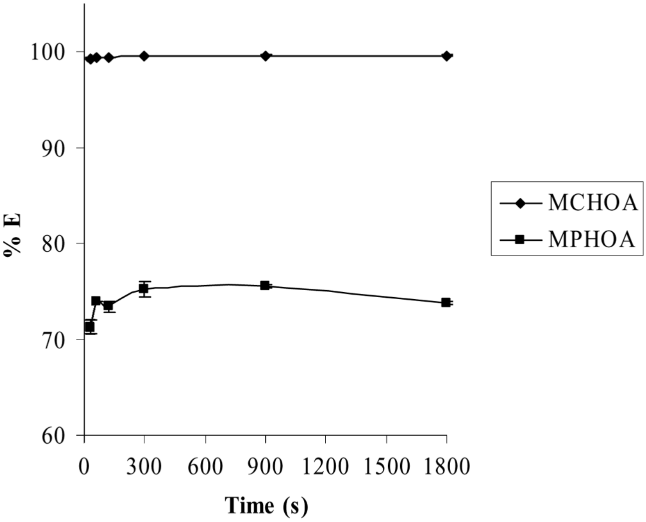

Figure 1. Evaluation of the time necessary to reach equilibrium (~0.01 M Fe(III) in 5 M HCl, 0.1 M MCHOA, or MPHOA in 1,2-DCE, A/O = 1, room temperature, 1000 r.p.m.).

(12–15). It is also observed that MCHOA is more efficient to extract Fe(III) than MPHOA.

Influence of Hydrochloric Acid Concentration

The results obtained for the study of the influence of HCl concentration on Fe(III) extraction show that the performances of MCHOA and MPHOA are different. The results displayed in Fig. 2 denote that MCHOA is more efficient than MPHOA for HCl concentrations below 6 M, at 3 M HCl Fe(III) is 90% extracted by MCHOA against 18% for MPHOA. Both amides give comparable Fe(III) extraction percentages at 8 M HCl (>99.5%) but, for 10 M HCl, a sharp decrease in Fe(III) extraction is verified for MCHOA; MPHOA maintains a high %E, 99.5%, whereas MCHOA only extracts 27% Fe(III). The reasons for this latter occurrence can possibly be related with a competition of HCl with Fe(III) for MCHOA, but can also be due to an irreversible and extensive degradation of MCHOA at such a high HCl concentration.

It is worthwhile to point out that the corresponding malonamides, DMDPHMA (12) and DMDCHMA (14), present a similar tendency to extract Fe(III): DMDCHMA is a much better extractant for lower HCl

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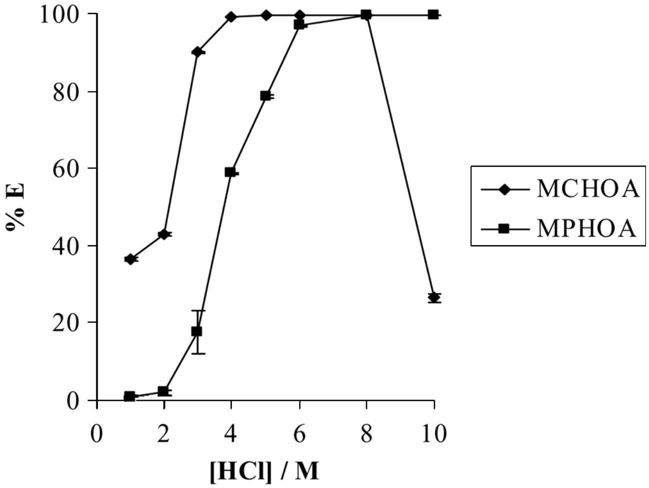

Figure 2. Dependence of Fe(III) extraction by MCHOA and MPHOA on hydrochloric acid concentration (~0.01 M Fe(III) in different HCl concentrations, 0.1 M MCHOA or MPHOA in 1,2-DCE, A/O = 1, 15 minutes equilibration, room temperature, 1000 r.p.m.).

concentrations than DMDPHMA, however, the abrupt decrease in Fe(III) extraction for the 10 M HCl solution has not been observed for DMDCHMA (14).

Stripping and Recycling

The Fe(III) loaded organic phases coming from the extraction experiments were subjected to an equilibration with an equal volume of distilled water, following the usual experimental set. The stripping results obtained are presented in Table 2. It can be generally observed that increasing HCl concentrations in the feed aqueous phases seem to enhance the efficiency of distilled water to strip Fe(III) to a new aqueous medium, a detail that has previously been detected for DMDPHMA (12) and DMDCHMA (14). In fact, more HCl concentrated loaded organic phases somehow help the stripping of Fe(III) itself. This effect can particularly be observed when MPHOA is involved. For MCHOA that tendency is not so evident, as there is an unexpected decrease in Fe(III) stripping for the initial 5 M HCl aqueous feed. Fe(III) is efficiently stripped by water when the loaded MCHOA and MPHOA were previously equilibrated with 8 M HCl aqueous media.

In order to check if both compounds would maintain their high %E Fe(III) if reused, four sequential extraction-stripping experiments were carried out. The initial aqueous phase contained ~0.01 M Fe(III) in 8 M HCl. The Fe(III) extraction results obtained for all the cycles are displayed in Fig. 3. It can be seen that both MCHOA and MPHOA keep their ability to extract Fe(III) along the four experiments; however, it was noticed that, after the first cycle, water does not strip Fe(III) completely—for MCHOA, the Fe(III) stripping values varied between 95% and 80% for the four cycles, whereas for MPHOA the range obtained was from 90%

Table 2. Fe(III) stripping by distilled water from the loaded organic solutions originating from extraction tests (variable [Fe(III)] in 0.1 M MCHOA or MPHOA in 1,2-DCE, distilled water, A/O = 1, 15 minutes equilibration, room temperature, 1000 r.p.m.)

$[HCl]_{initial}$/M	% Fe(III) stripping from MCHOA	% Fe(III) stripping from MPHOA
4	66	74
5	36	76
6	80	79
8	95	94

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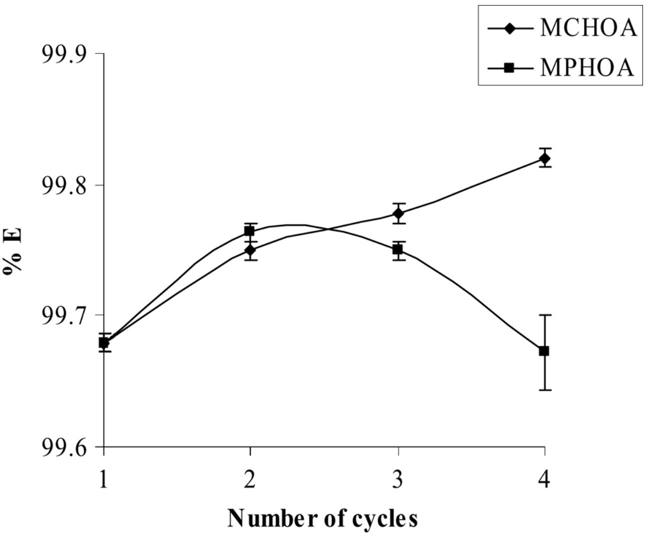

Figure 3. Percentages of Fe(III) extraction by MCHOA and MPHOA in four successive cycles of extraction and stripping [(Extraction: ~0.01 M Fe(III) in 8 M HCl, 0.1 M MCHOA, or MPHOA in 1,2-DCE; Stripping: loaded Fe(III) in organic phases, distilled water), A/O = 1, room temperature, 1000 r.p.m.].

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to 56%. These experiments also denote that MCHOA and MPHOA do show a very good loading capacity to accommodate Fe(III), as they keep the high %E towards the metal ion even when the latter is only partially stripped from the corresponding organic phases.

Selectivity

The selectivity of both amide derivatives towards Fe(III) when in the presence of other metal ions has been tested for a 6 M HCl aqueous phase containing 0.01 M Fe(III), Zn(II), Ni(II) and Cu(II). The extraction profiles obtained are displayed in Fig. 4. It can be observed that both compounds maintain their high %E for Fe(III) and all the other metal ions are not significantly extracted, as the major %E obtained are 18% Zn(II) for MCHOA and 8% Cu(II) for MPHOA. In general MPHOA exhibits a better selectivity profile than MCHOA, and that situation has also been verified for the correspondent malonamides (6). Lower HCl concentrated aqueous feed solutions will probably affect the equilibria involving the different metal ion chlorocomplexes and would somehow modify the selectivity profile presented, but significant changes are not likely to be expected.

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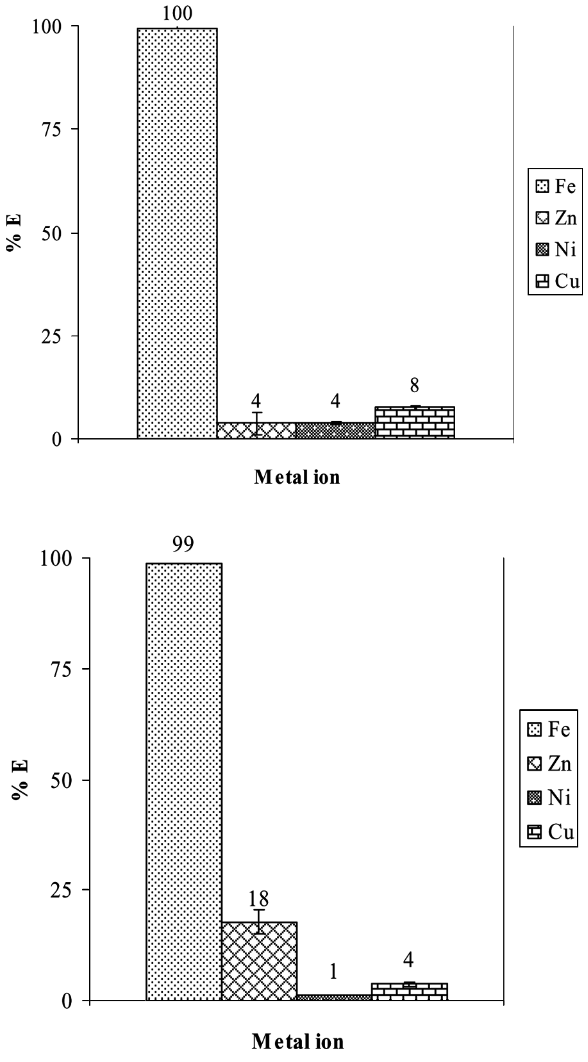

Figure 4. Extraction results for Fe(III), Zn(II), Ni(II) and Cu(II) by MPHOA and MCHOA, respectively (~0.01 M Fe(III), Zn(II), Ni(II) and Cu(II) in 6 M HCl, 0.1 M MCHOA, or MPHOA in 1,2-DCE, A/O = 1, 15 minutes equilibration, room temperature, 1000 r.p.m.).

Extraction Reactions

In order to identify the reactions responsible for Fe(III) extraction, the dependence of the Fe(III) distribution coefficients from 6 M HCl on the

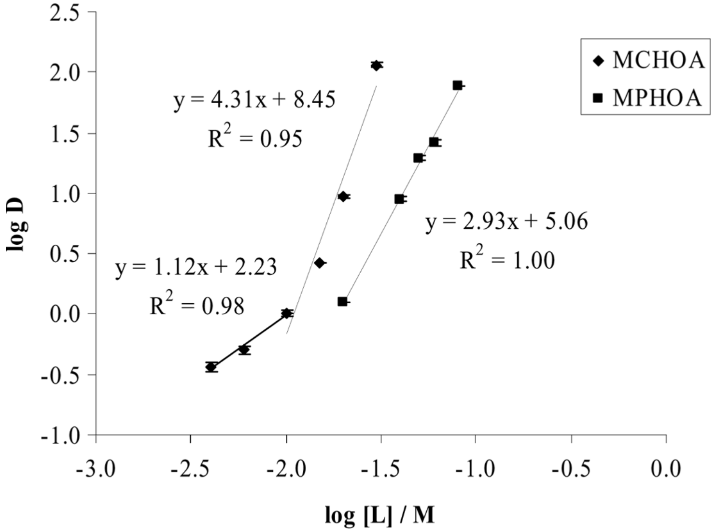

Figure 5. Dependence of the Fe(III) distribution coefficient on the initial extractant concentration (~0.01 M Fe(III) in 6 M HCl, MCHOA, or MPHOA in 1,2-DCE, A/O = 1, 15 minutes equilibration, room temperature, 1000 r.p.m.).

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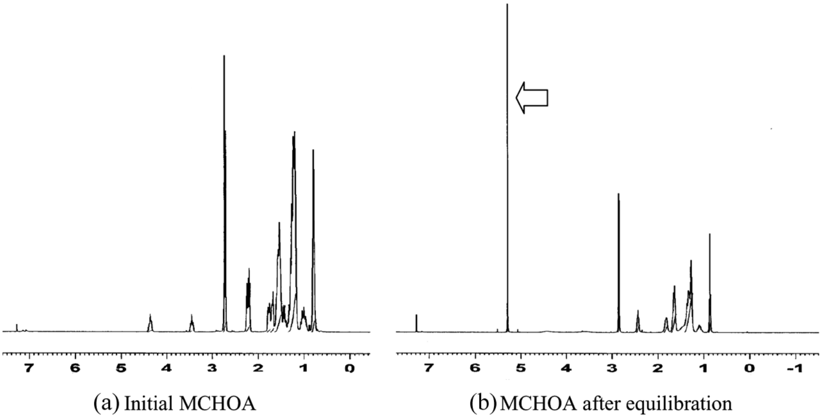
initial concentrations of both amide derivatives was investigated. The data obtained from the correspondent log-log plots are presented in Fig. 5.

The experimental points obtained for MPHOA adjust well to a straight line within the concentration range tested, but the same does not occur for MCHOA; hence two regions of linearity have been considered for this latter amide. The log-log profile shown by MCHOA suggests that the stoichiometry of the extractant:Fe(III) species may be significantly changing within the extractant concentration range considered—the slope value for the straight line corresponding to MCHOA lower concentrations is about one, but that value increases to about four for higher concentrated MCHOA solutions. This particular behavior suggests that possibly MCHOA may present some tendency to form aggregate species in solution; however, this equilibrium data does not allow any conclusive statement about this feature.

Based on the slope values obtained, it can generally be estimated that an average of three MPHOA or MCHOA molecules for each Fe(III) should be involved in the metal ion extraction.

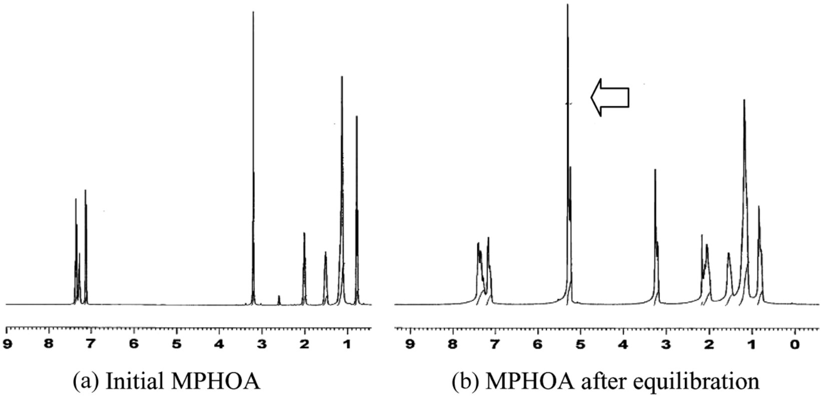
An equilibration experiment of both MCHOA and MPHOA with an 8 M HCl aqueous solution was carried out to check by ^{1}H NMR if the amide derivatives would show any tendency to protonate (12). Hence, the NMR spectra of the extractants were plotted and directly compared



(a) Initial MCHOA (b) MCHOA after equilibration

Figure 6. ^{1}H NMR spectra of MCHOA (a) before and (b) after equilibration with 8 M HCl (the appearance of a new peak is identified with an arrow).

with the ones obtained for the initial organic compounds. Figures 6 and 7 present the results achieved. For the MCHOA and MPHOA spectra obtained after equilibration, new and intense peaks at 5.3 ppm appeared. It can then be assumed that there is an extensive protonation of both compounds when put in contact with concentrated HCl solutions, a situation that also occurred with DMDPHMA, a new peak identified at 5.3 ppm (12), but not with DMDCHMA, as the spectrum of this latter compound did not suffer any modification (14). This extensive protonation can also be considered as an indication that aggregate species may be



(a) Initial MPHOA (b) MPHOA after equilibration

Figure 7. ^{1}H NMR spectra of MPHOA (a) before and (b) after equilibration with 8 M HCl (the appearance of a new peak is identified with an arrow).

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present in both extractant solutions, but again only specific techniques to check this sort of phenomena can elucidate such an assumption (17).

According to the data collected and considering that $FeCl_3$ is always the predominant Fe(III) species in the hydrochloric acid concentrations tested (18), the reaction proposed as being responsible for Fe(III) extraction by either MCHOA or MPHOA is displayed in Eq. (1). This equation is only valid when the extractants are considered to exist under a monomeric form in the organic solutions before and after Fe(III) extraction.

$$3\mathrm{L}\ (\mathrm{org}) + \mathrm{FeCl_3}\ (\mathrm{aq}) + 3\mathrm{HCl}\ (\mathrm{aq}) \longrightarrow 3\mathrm{LH^+} \cdot \mathrm{FeCl_4^-} \cdot 2\mathrm{Cl^-}(\mathrm{org}) \quad (1)$$

In Eq. (1), L stands for MCHOA or MPHOA.

Finally it is reasonable to assume that the Fe(III) extraction reactions by the amide derivatives described in this study seem rather complex and, accordingly, further research to better understand all the phenomena involved should be considered.

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CONCLUSIONS

The results presented in this work demonstrate that the two amide derivatives investigated, MCHOA and MPHOA, compare well with the corresponding malonamides DMDCHMA and DMDPHMA when used for the recovery of Fe(III) from concentrated chloride solutions; in fact, it seems that the existence of two carbonyl groups in the extractant skeleton of malonamides is not a crucial factor in determining their extracting properties towards Fe(III). The data achieved for Fe(III) extraction generally denote that MCHOA and MPHOA allow a fast and good recovery, show a quite good loading capacity and do exhibit promising selective properties towards the metal ion. The results collected to estimate the Fe(III) extraction reactions by MCHOA and MPHOA indicate that both amides suffer extensive protonation. Further research to elucidate the eventual aggregation of the amide extractants in the tested organic media would be worthwhile.

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