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Cloud Point Extraction and Separation of Scandium and Yttrium (III) with Triton X-114 in the Presence or Absence 8-Hydroquiloline as an Added Chelate

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Abstract: In this paper, the cloud point extraction and separation of scandium and yttrium (III) via use of Triton X-114 with and without 8-hydroquiloline (HQ) as an added chelate agent are investigated. The effects of various parameters, such as the aqueous phase pH, HQ concentration, Triton X-114 concentration, heating temperature, and incubation time, on the cloud point extraction of scandium and yttrium (III) are studied. The results demonstrate that, there are different extraction and separation behaviors for scandium and yttrium (III) with and without HQ as an added chelate. And in contrast to solvent extraction, cloud point extraction is an excellent method to extract and separate scandium and yttrium (III).

Keywords: Cloud point extraction, solvent extraction, 8-hydroquiloline, triton X-114, scandium (III), yttrium (III)

INTRODUCTION

Rare earths (scandium, yttrium, and lanthanides) are important elements from an industrial perspective. They are extensively used in astronavigation,

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luminescence, nuclear energy, and metallurgical industries (1–4). With the ever increasing demand of high purity rare earth and their compounds, the separation and purification of these elements have gained considerable importance in recent years.

Solvent extraction has been reported to be one of the most effective techniques to extract metal ion from various resources. However, the similar physical and chemical characters of rare earth ion (III) make their separation and purification still very difficult (5–7). At the same time, classical liquid–liquid extraction and separation methods are usually time consuming and require a relatively large volume of high purity solvents. Of additional concern is disposal of the solvent used, which creates a severe environmental problem as a result, restricting the further progress of solvent extraction (8).

Recently, cloud point extraction (CPE) has been an attractive subject as an alternative to solvent extraction (9, 10). Compared with conventional solvent extraction, CPE uses water and avoids the use of large amount of expensive toxic, and flammable organic solvents. In addition, CPE can contribute to higher recovery efficiency and a large preconcentration factor, since the presence of surfactant can minimize losses of analytes. There are some reports on the CPE method used for the preconcentration of rare earth (III) ions. The first example is the cloud point extraction of Er (III) with 2-(3,5-dichloro-2-pyridylazo)-5-dimethylaminophenol as chelating agent and PONPE-7.5 as non-ionic surfactant in 1997 by Silva et al. (11). Later a similar procedure was used for the preconcentration of Dy (III) as a step of the spectrophotometric determination of Dy (III) in urine (12). Recently, the separation of La (III) and Gd (III) through CPE technique with 8-hydroxyquinoline as chelating ligand and Triton X-114 as a non-ionic surfactant was studied by Micheline Draye et al. (13, 14). However, so far, no information is available for Sc and Y (III) extraction and separation through CPE procedure.

In this study, cloud point extraction and separation of scandium and yttrium (III) ions via use of Triton X-114 with and without 8-hydroquioline as an added chelate agent were investigated. Many excellent extraction characters of Triton X-114, such as lower cloud point temperature (23°C) (14), heavier surfactant-rich phase (14), and better solubility for 8-hydroquiloline (will be discussed in the section on RESULTS AND DISCUSSIONS), make it to be a suitable candidate for cloud point extraction of scandium and yttrium (III) ions. The aim of this work is to study in detail, the effect of experimental variables on the extraction efficiency and separation selectivity of scandium and yttrium (III), which is of great importance for industrial application. In addition, the solubility of 8-HQ in Triton X-114 surfactant was determined; the solvent extractions of yttrium (III) with Triton X-114 in the presence and absence of 8-hydroquiloline in chloroform solution were compared.

EXPERIMENTAL

Apparatus

A Shimadzu spectrophotometer model UV-365 with 10 mm-optical path cells was used to perform the absorption measurements. A pH meter model pHs-3C calibrated daily with 4.01 and 6.86 standard buffer solutions was employed for measuring pH value in aqueous phase. A thermostated water-bath was employed to maintain the experimental temperature. A centrifuge was used to speed up the complete phase separation of turbid solutions above the cloud point temperature.

Reagents

All reagents used were of analytical grade. Triton X-114 from Fluka Chmie Company) was used as received. A solution of Triton X-114 was prepared in a calibrated flask dissolving the appropriate amount in doubly distilled water, and mass concentration (w/v%) was used for describing the experimental condition in the whole experimental process. Solutions of scandium and yttrium (III) were prepared by dissolving their oxide in hydrochloric acid and standardized by EDTA titration using xylenol orange as an indicator. 8-Hydroquinoline without further purification was used by dissolving an appropriate amount into an alcohol solution in the cloud point extraction procedure, and into a chloroform solution in the liquid-liquid extraction procedure.

Procedures

Solubility of HQ in Triton X-114 Solution

0.01 g of HQ was transferred to a 50 ml brown volumetric flask and dissolved to the mark by 0.5% Triton X-114 solution. Exactly 1.25 ml, 2.5 ml, 3.75 ml, 4.4 ml, and 5 ml portions of this HQ solution were transferred to five 25 ml brown volumetric flasks and filled up to the mark with 0.5% Triton X-114 solution. Using 0.5% Triton X-114 solution as a blank, the absorption spectra of HQ in 0.5% Triton X-114 solution was obtained. A calibration curve of HQ in 0.5% Triton X-114 solution was constructed by these standard solutions.

The solubility of HQ in Triton X-114 was obtained in the following way. The Triton X-114 solutions in various percent concentrations (0.2–1%) containing excess HQ were stirred for 24 h, stayed for 30 min, and centrifuged. The 0.5 ml supernatant solution was diluted to the 25 ml brown volumetric flask and filled to the mark by an appropriate concentration of Triton X-114. The absorbances of these solutions were measured, and from these data, the solubility of HQ in Triton X-114 solution was calculated.

Cloud Point Extraction Procedure

A Triton X-114 aqueous solution containing rare earth ion, acetate buffer, or a mixture of them with HQ was taken in a graduate centrifuge tube with a glass stopper. The mixture was diluted to 10 ml with doubly distilled water. After shaking for 2 min, the solution was heated up to 40° (without HQ) or 65°C (with HQ), then left to stand for 1.0 h at this temperature unless otherwise stated. Separation of the two phases was accomplished by centrifugation for 3 min at 3000 rpm. The upper aqueous phase was then removed, and the concentration of rare earth ion in this phase was determined by spectrophotometriclly at 672 nm for scandium (III), and 655 nm for yttrium (III) ion respectively, using Arsenazo (III) as an indicator at pH 2.8 in chloroactic acid-sodium hydroxide buffer solution. The final volume of the surfactant-rich phase was determined by measuring the phase volume after CPE. The pH values were maintained by acetate buffer ion and the desired pH values were obtained by addition of HCl or NaOH. The ionic strength was kept constant at 0.1 M with NaCl. The CPE of scandium or yttrium (III) from micelle solution was evaluated in terms of extraction efficiency *E*. The concentration of initial rare earth ion (III) was 1.0×10^{-4} M or 3.0×10^{-4} M, and the concentrations of Triton X-114 and HQ were changed in the range of 0.1–1.4% (w/v%) and 1.5×10^{-5} to 6.61×10^{-3} M, respectively.

Liquid-liquid Extraction

Liquid-liquid extraction was carried out by shaking equal volume (5.0 mL) of aqueous and organic phases in centrifuge tubes, using a mechanical shaker at 303 ± 1 K. The other experimental conditions and data treatment are the same as those of cloud point extraction.

RESULTS AND DISCUSSION

Solubility of HQ in the Triton X-114 Solution

The absorption spectra of HQ in various percent concentrations of Triton X-114 solution are shown in Fig. 1. The maximum absorption wavelength is found at 306 nm and HQ can be quantitatively dissolved in Triton X-114 micellar solution. The calibration curve of HQ is almost linear from 0 to 40 μg ml^{-1} ($y = 0.0187x - 0.041$, $R^2 = 0.9999$). From this calibration

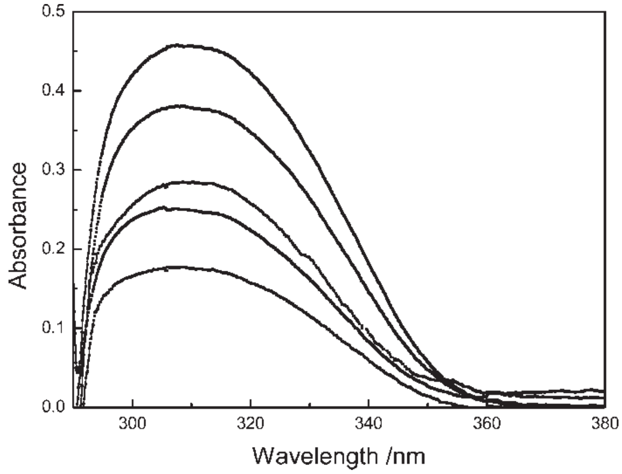

Figure 1. Absorption spectra of saturated HQ in various percent concentrations of Triton X-114 solution. The concentrations of Triton X-114 from top spectrum were 1.0, 0.8, 0.5, 0.4, and 0.2%, respectively. Theses absorption spectra were obtained after the saturated solutions were diluted as much as 50 times.

curve, the amount of HQ saturated in 0.5% Triton X-114 solution is calculated to be 0.005 M at 20°C and pH 6.5. From Fig. 1, it is shown that the solubility of HQ also increased in proportion to the percent concentration of Triton X-114 ($y = 0.3444x + 0.1106$, $R^2 = 0.9992$).

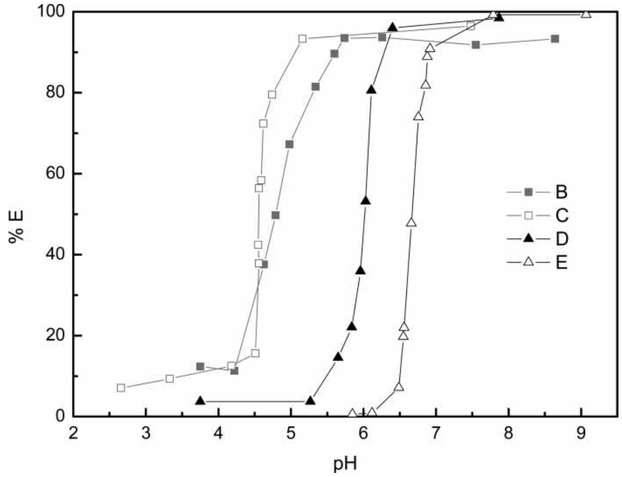

Figure 2. The effect of pH on the CPE of Sc and Y (III) ion with and without 8-HQ as an added chelate. B. $ScCl_3$ 0.3 mM, Triton X-114 0.5%, HQ 1 mM; C. $ScCl_3$ 0.1 mM, Triton X-114 0.5% D. YCl_3 0.3 mM, Triton X-114 0.5%, HQ 1 mM; E. YCl_3 0.1 mM, Triton X-114 0.5%.

The Effect of pH on CPE of Scandium and Yttrium (III)

Figure 2 shows the effect of pH on the CPE of scandium and yttrium (III) in the presence and absence of HQ. It can be found that with increasing pH, the extraction efficiency increases. For the system of Triton X-114-Sc (III), high quantitative extraction can be achieved at $pH \geq 5.0$, while for the Triton X-114-Y (III) system, that can be achieved at $pH \geq 7.0$. When HQ as a chelate agent is added to the cloud point extraction system, it is obvious that the extraction efficiency increases for Y (III) and decreases for Sc (III), and high quantitative extraction turns to $pH \geq 5.5$ for Sc (III) and $pH \geq 6.0$ for Y (III) respectively. Therefore, we should select the Triton X-114-HQ-Y and Triton X-114-Sc systems for further study instead of Triton X-114-Y and Triton X-114-HQ-Sc systems. Since the increase of pH can result in the hydrolysis of rare earth ions (III) and their complexation, it is reasonable to consider that $(RE^{3+})(Cl^{-})_3$ species, $RE(H_2O)(OH)_m^{3\text{-}m}$ species or $RE_n(H_2O)_m(Cl^{-})x^{(3n\text{-}x)+}$ polymerization can be extracted into the surfactant-rich phase (RE denotes the rare earth ion (III)). According to Asiya Mustafina (13), the sharp increase in extraction efficiency of Sc (III) without HQ as an added chelate at pH about 4.5 should indicate that, hydroxyl complexes $Sc(H_2O)(OH)_m^{3\text{-}m}$ are extracted into micellar pseudo-phase much more efficient than ionic associates $(Sc^{3+})(Cl^{-})_3$. Similarly, the sharp increase in the extraction the efficiency of Y (III) at pH equal to 6.0 with HQ and pH equal to 6.5 without HQ should suggest that, hydroxyl complexes $Y(H_2O)(OH)_m^{3\text{-}m}$ are extracted into the micellar pseudo-phase which is much more efficient than the ionic associates $(Y^{3+})(Cl^{-})_3$. Furthermore, a white (without HQ) or yellow (with HQ) precipitate, and gel-like solid, perhaps a polymer of $RE_n(H_2O)_m(Cl^{-})x^{(3n\text{-}x)+}$-Triton X-114 or HQ-$RE_n(H_2O)_m(Cl^{-})x^{(3n\text{-}x)+}$-Triton X-114 is observed in the surfactant-rich phase at higher loading, and similar phenomena is observed in the liquid-liquid extraction of yttrium and lanthanides (15, 16).

The Effect of Triton X-114 Concentration on CPE of Scandium and Yttrium (III)

Figure 3 shows the effect of the Triton X-114 concentration on E% of Sc and Y (III). It can be found that if there is no HQ as an added chelate, the extraction efficiency would increase with the increasing Triton X-114 concentrations, and then level off (for Triton X-114-Sc (III) system), which means that there is a strong complexation between Sc (III) and Triton X-114. On the contrary, if there is HQ as an added chelate, the extraction efficiency would decrease rapidly (for Triton X-114-HQ-Y system) when the concentration of Triton X-114 beyond a certain value. A reasonable explanation is that, when the concentration of Triton X-114 is low, with increasing concentrations of Triton X-114, the solubility of HQ into Triton X-114 micellar aqueous solution increases, which leads to increasing extraction efficiency, and when

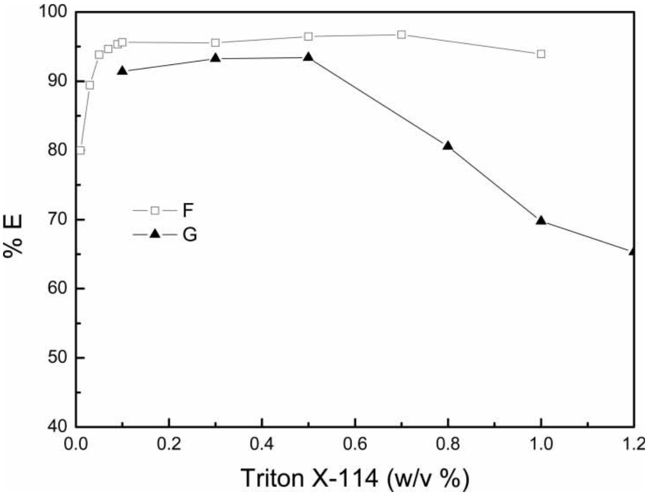

Figure 3. The effect of Triton X-114 concentration on the E% F. $ScCl_3$ 0.1 mM, pH = 5.20; G. YCl_3 0.1 mM, pH = 6.20, HQ = 1 mM.

the concentration of Triton X-114 is beyond a certain value, strong interaction between excess Triton X-114 and HQ should repulse the extraction of Y (III). Because the volume of the surfactant-rich phase is small, the HQ should not be solubilized well in the Triton X-114 solution and extraction process becomes more difficult. In this work, 0.5% Triton X-115 solution is chose for cloud point extraction of Sc and Y (III).

The Effect of HQ Concentration on the CPE of Scandium and Yttrium (III)

The effect of HQ concentration on the cloud point extraction of Sc and Y (III) is illustrated in Fig. 4. It can be found that with increasing concentration ratios of HQ to RE (III), the extraction percentages increase, and the high quantitative extraction can be realized when the concentration ratio of HQ to RE (III) is more than 10. It is evident that, the influence of concentration ratio of HQ to RE (III) on the E% for Y (III) is more prominent than that for Sc (III), which may be due to competitive extraction of Triton X-114 to Sc (III) and no contribution for Y (III) at pH 6.0.

The Effect of Heating Temperature and Incubation Time on CPE of Scandium and Yttrium (III)

The effects of incubation time and heating temperature on the cloud point extraction of scandium without HQ and yttrium (III) with HQ using a

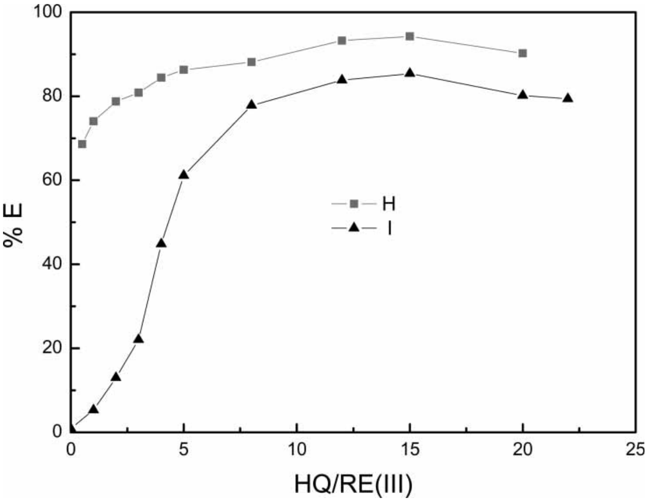

Figure 4. The effect of HQ concentration on the CPE of scandium and yttrium (III) H. $ScCl_3$ 0.3 mM, pH = 5.2; I. YCl_3 0.3 mM, pH = 6.0.

0.5% Triton X-114 solution at pH 6.0 were investigated in the range of 10–60 min and 40–70°C, respectively. The extraction percentage (E%) of RE (III) reached up to 95% after 30 min, and 60 min was enough to achieve the entire extraction. At the same time, it can be found that with increasing temperatures, the extraction percentages for Y (III) almost linearly increase from 40°C to 65°C, and are independent beyond 65°C, as presented in Fig. 5, which suggests that the complexation of HQ with Y (III) in Triton X-114 solution is endothermic reaction. On the contrary, the higher extraction percentage obtained at lower temperature clearly indicates that the complexation of Triton X-114 with Sc (III) is exothermic reaction. The conclusion is in accordance with that of ref (17), where the usual exothermic complexation between nonionic POE surfactant and metal ion is described.

Separation of Scandium and Yttrium (III)

If the values of $pH_{1/2}$ are used to compare extraction ability, $\Delta pH_{1/2}$ for separation ability and S for separation coefficient ($S = D_{Sc}/D_{Y}$), Table. 1 clearly project the results that the extractability increases with increasing aqueous phase pH, and the order of extractabilities for Sc and Y (III) in different extraction systems is Triton X-114-Sc (III) > Triton X-114-HQ-Sc (III) > Triton X-114-HQ-Y (III) > Triton X-114-Y (III). Though the extractability of Sc (III) with HQ in Triton X-114 micellar solution is lower than that with Triton X-114 without added chelate, the larger separation coefficient with

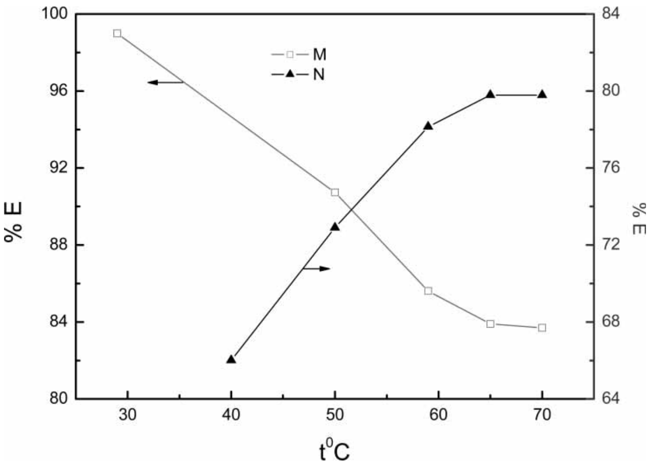

Figure 5. The effect of heating temperature on the CPE of scandium and yttrium (III). M. $ScCl_3$ 0.3 mM, pH = 5.2; N. YCl_3 0.1 mM, pH = 6.0, HQ = 1 mM.

Y (III), especially at lower pH (Table 2), shows a potential practical application in rare earth separation.

Liquid-liquid Extraction of Yttrium (III) with Triton X-114, 8-HQ and their Mixtures in Chloroform Medium

In order to compare the extraction efficiency with solvent extraction, the effect of pH on the liquid-liquid extraction of Y (III) has been performed, as illustrated in Fig. 6. It can be seen that the extraction efficiency is very low with Triton X-114, HQ and their mixtures, and even with 0.01 M HQ, the extraction percentage is still less than 40%. Therefore, we can say that cloud point extraction is superior to the liquid-liquid extraction. In addition, a phenomenon that the lower extraction efficiency of the mixtures of Triton X-114 and HQ than that of any extractant alone can be seen, this may be due to the strong interaction of Triton X-114 with HQ repulsing the extraction of Y (III).

Table 1. The values of $pH_{1/2}$ and $\Delta pH_{1/2}$

System	$pH_{1/2(Sc)}$	$pH_{1/2(Y)}$	$\Delta pH_{1/2}$
Triton X-114	4.5	6.5	2.0
Triton X-114-HQ	4.8	6.0	1.2

$RECl_3$ 0.3 mM, HQ 1 mM, Triton X-114 0.5% (w/v).

Table 2. Effect of 8-hydroquinloline/lanthanides ratio on distribution coefficient and Sc^{3+}/Y^{3+} selectivity

8-HQ/Ln(III) mole ratio	pH = 5.2			pH = 6.0		
	$D_{Sc^{3+}}$	$D_{Y^{3+}}$	$S_{Sc^{3+}/Y^{3+}}$	$D_{Sc^{3+}}$	$D_{Y^{3+}}$	$S_{Sc^{3+}/Y^{3+}}$
0	67.4	4.25	15.8	84.7	0.54	154.6
1	8.64	3.51	2.64	43.5	4.04	10.7
2	15.8	3.73	4.77	46.3	9.99	4.63
3	27.2	5.65	5.42	47.5	16.9	2.80
8	34.6	5.12	6.76	176.2	155.5	1.13
10	35.9	5.20	7.67	184.2	165.9	1.11
12	39.8	5.23	7.61	186.5	167.5	1.11
14	41.2	5.38	7.67	188.5	170.8	1.10
15	41.8	5.41	7.73	180.4	160.3	1.12

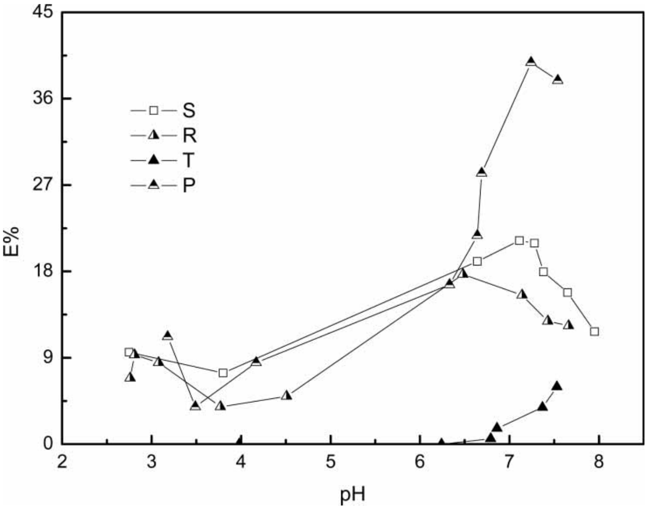

Figure 6. The effect of pH on the yttrium (III) extraction percentage YCl_3 0.3 mM: NaCl 0.1 M, t = 30°C S. 0.5% (w/v) Triton X-114; T. 1 mM HQ; R. 0.5% (w/v) Triton X-114 + 1 mM HQ; P. 10 mM HQ.

CONCLUSIONS

In this paper, the following conclusions can be drawn:

1. Triton X-114 is an excellent candidate for cloud point extraction and separation of scandium and yttrium (III). The high quantitative extraction can

be realized at pH value more than 5.0 for Sc (III) and 7.0 for Y (III) with Triton X-114 concentration of 0.5%, heating temperature of 40°C and incubation time of 60 min.

2. 8-Hydroquioline is a very stable and strong complexation reagent, and its solubility in 0.5% Triton X-114 is 0.005 M. Though its adulteration into the Triton X-114 solution can slightly decrease the extraction efficiency of scandium (III), it is good candidate for yttrium (III) extraction. The high quantitative extraction can be realized at pH value more than 5.5 for Sc (III) and 6.5 for Y (III), when the concentration of 8-hydroquiloline is 1 mM in the 0.5% Triton X-114 solution, with a heating temperature of 40°C and incubation time of 60 min.
3. Though the extractability of Sc (III) with HQ in Triton X-114 micellar solution is lower than that with Triton X-114 without added chelate, it is still a good choice for the separation Sc (III) and Y (III) by CPE method.

In conclusion, in contrast with liquid-liquid extraction, cloud point extraction shows higher extraction efficiency and larger separation selectivity for scandium and yttrium (III), and so many characters, such as green, rapid, and easy and so on, that it is hoped that solvent extraction in further for purification and separation of rare earth will take place.

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