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Xiuzhu Zhou[a]; Jiazhen Zhou[a]; Chengqun Zhang[a]; Wendong Yu[a]
[a] INSTITUTE OF NUCLEAR ENERGY TECHNOLOGY TSINGHUA UNIVERSITY, BEIJING, PEOPLE'S REPUBLIC OF CHINA

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Application of Annular Centrifugal Contactor on Separating Indium from Iron

XIUZHU ZHOU, JIAZHEN ZHOU, CHENGQUN ZHANG, and WENDONG YU
INSTITUTE OF NUCLEAR ENERGY TECHNOLOGY
TSINGHUA UNIVERSITY
BEIJING 100084, PEOPLE'S REPUBLIC OF CHINA

ABSTRACT

It is hard to separate indium from iron in sulfuric acid leachate without reducing Fe^{3+} to Fe^{2+} with di(2-ethylhexyl) phosphoric acid (HDEHP) by the equilibrium extraction process. A nonequilibrium extraction process with annular centrifugal contactors is studied through the extraction kinetics difference between In and Fe. The mass transfer velocities of In and Fe were determined. Laboratory- and industry-scale extraction tests with miniature and industry scale annular centrifugal contactors, respectively, were conducted. The results indicate the mass transfer velocity of In is much faster than that of Fe, the contact time between the two phases is very short in the contactor, and the In can be well separated from Fe in the nonequilibrium extraction process with annular centrifugal contactors.

Key Words. Annular centrifugal contactor: Separation: In; Fe; Nonequilibrium extraction

INTRODUCTION

Indium, as well as zinc, tin, iron, etc., always form paramagnetic minerals in the natural world. It is easy to separate indium from impurities, except iron, in sulfuric leachate by equilibrium solvent extraction with HDEHP as an extractant because most of iron exists as Fe^{3+} in sulfuric acid leachate, indium and Fe^{3+} both have a high distribution coefficient, and the content of iron in the feed is 10–100 times greater than that of indium. Therefore it is necessary to add a process step and equipment in order to reduce Fe^{3+} to Fe^{2+} so indium may be purified.

Provided the difference of the mass-transfer velocity between two components is large enough, they can be separated in nonequilibrium extraction even if they have the same distribution coefficient. Pushlenkov investigated the extraction kinetics and the separation of U, Pu, Ru, and Zr (1). However, no literature about the nonequilibrium extraction of In from Fe is available.

The objective of this investigation is to study the separation of indium from iron by nonequilibrium extraction with annular centrifugal contactors.

The operation of nonequilibrium extraction requires a suitable contactor to enable two phases to mix quickly and fully, and then to settle immediately. Thus the contact time between the two phases must be short enough that the component with a large mass-transfer velocity can be extracted into the organic solvent according to its distribution coefficient, while the component with a low mass-transfer velocity cannot be extracted due to deviation from thermodynamic equilibrium. Among the available contactors, only the centrifugal contactor satisfies the requirements (2–5).

Three types of annular centrifugal contactors (with rotor diameters of 20, 50, and 230 mm, respectively) used in the extraction tests were designed and manufactured by the Institute of Nuclear Energy Technology,

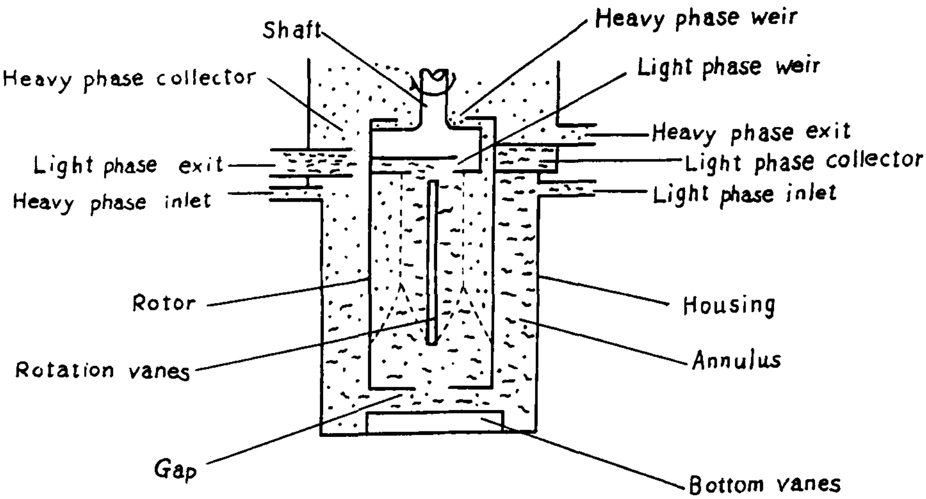

FIG. 1 Schematic diagram of the annular centrifugal contactor.

Tsinghua University (6). A schematic diagram of the annular centrifugal contactor is shown in Fig. 1. When an aqueous phase and an organic phase are fed through their inlets into the annular gap of the centrifugal contactor, the two phases are violently stirred by revolution of the rotor and mass transfer between the two phases occurs. Then the mixed phases are sucked into the rotor through the orifice at its bottom. The heavy aqueous phase is thrown toward the inner wall of the rotor and the light organic phase is extruded and moves to the center. The separated two phases flow into their collecting chambers through their weirs and enter the next stage contactor or containers.

MATERIALS

The organic phase used in the tests is 30% HDEHP–kerosene, and the aqueous phase feed used has three different contents:

Aqueous phase feed I, In 0.126 g/L, Fe 20.5 g/L, H_2SO_4 20.0 g/L
Aqueous phase feed II, In 0.156 g/L, Fe 21.6 g/L, H_2SO_4 20.0 g/L
Aqueous phase feed III, In 0.1 g/L, Fe 20.0 g/L, Zn 100–120 g/L H_2SO_4 20.0 g/L, and small amounts of Cu, Co, As, Sb, etc.

The aqueous phase feed III used in the industrial scale tests was H_2SO_4 leachate supplied by a factory, and the other two were simulated feeds made in our laboratory.

PROCEDURES

Separatory Funnel Tests

The mass-transfer velocity of indium and iron was determined by separatory funnel tests. A 50-mL separatory funnel was filled with the two phases (aqueous phase feed I and organic phase) in a given phase ratio (A/O = 10/1) and was strenuous shaken for varying lengths of time and then the two phases were settled. The content of indium and iron in the organic phase was determined by the neutron activation method and spectrophotometry, respectively.

Extraction Tests in ϕ 20 mm and ϕ 50 mm Annular Centrifugal Contactors

The mass-transfer velocity of indium and iron and the influence of the rotor speed on the extraction yield were examined in these tests with aqueous phase feeds I and II.

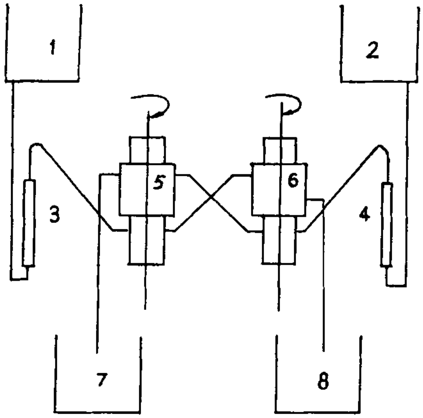

FIG. 2 Two-stage operation system of annular centrifugal contactors: (1, 2) organic, aqueous feed tank; (3, 4) rotameter; (5, 6) centrifugal contactor; (7, 8) organic, aqueous phase receiving tank.

Industry-Scale Extraction Tests

Single-stage tests and then two-stage cascade tests were carried out with aqueous phase feed III in ϕ 230 mm annular centrifugal contactors. The aqueous feed was filtrated before it entered the contactors in order to remove solid impurities. The stage efficiency of indium was determined in the tests. The two-stage operation system of annular centrifugal contactors is shown in Fig. 2.

RESULTS AND DISCUSSION

Mass-Transfer Velocity of In and Fe

The effects of the contact time between the two phases on indium and iron extraction yield in the separatory funnel and in the miniature annular centrifugal contactors (rotor diameter; ϕ 20 mm) is shown in Figs. 3 and 4, respectively.

It can be seen that the mass-transfer velocity and the extraction yield of indium are much higher than those of iron. The results indicate that when the contact time in the separatory funnel reaches 6 seconds, the mass-transfer process of indium has already reached the thermodynamic equilibrium (stage efficiency approaches 100%), and the extraction yield of indium is more than 90% while that of iron is less than 3%. The weight

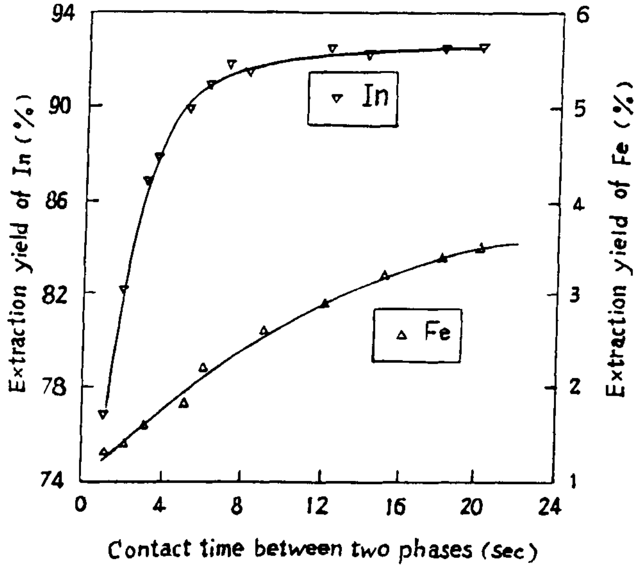

FIG. 3 Influence of the contact time between the two phases on the extraction yield of indium and iron with the separatory funnel aqueous phase (g/L): In 0.126, Fe 20.5, H_2SO_4 20.0. Organic phase: 30% HDEHP–kerosene. Phase volume ratio: A/O = 10/1.

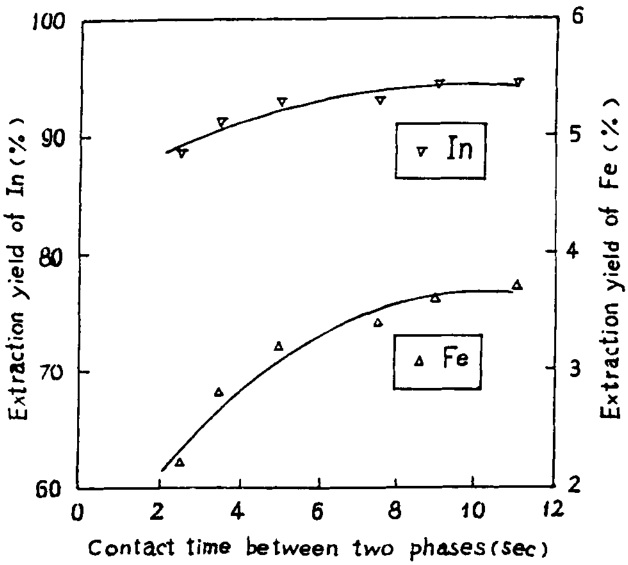

FIG. 4 Influence of the contact time between the phases on the extraction yield of indium and iron with the annular centrifugal contactor aqueous phase (g/L): In 0.126, Fe 20.5, H_2SO_4 20.0. Organic phase: 30% HDEHP–kerosene. Flow ratio: A/O = 10/1.

ratio of iron to indium is about 5.2 after one stage extraction and the same ratio reaches 160 before separation. The results with ϕ 20 mm annular centrifugal contactors are similar. The extraction yield of iron is only 3.8% even if the contact time is 11 seconds, but the indium yield is more than 93%. As a result, the iron content in indium is reduced by a factor of 20 after a single stage of extraction. It is obvious that indium and iron can be separated in the nonequilibrium extraction, and the iron content in indium is reduced to an allowable value.

Influence of the Rotor Speed on the Extraction Yield

Figure 5 shows the influence of rotor speed on the extraction yield of indium and iron.

The extraction yield of indium increases slightly with the rotor speed, while that of iron remains almost unaltered. Thus, an increase in rotor speed slightly enhances the separation of indium from iron. When the flow ratio and the contact time remain constant, the increase in rotor speed results in more mixing of the two phases. Therefore the extraction yield of indium increases slightly due to its high mass transfer velocity but that of iron remains constant due to its very low mass transfer velocity.

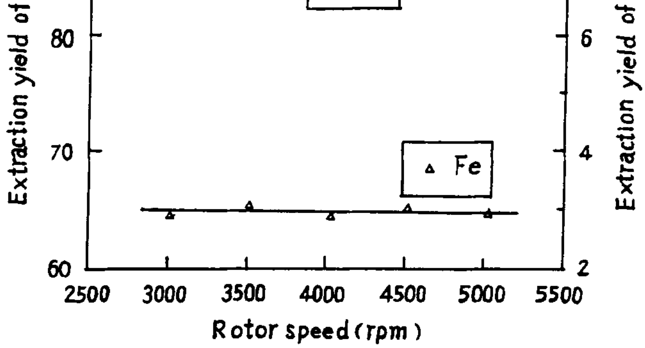

FIG. 5 Influence of the rotor speed on the extraction yield of indium and iron aqueous phase (g/L): In 0.156, Fe 21.6, H_2SO_4 20.0. Organic phase: 30% HDEHP–kerosene. Flow ratio: A/O = 10/1. Contact time: 3.5 seconds.

Influence of the Total Flow-Rate and the Flow Ratio on Stage Efficiency

The industry-scale extraction tests were carried out with ϕ 230 mm annular centrifugal contactors. In the two-stage cascade tests the flow ratio (A/O) is from 15/1 to 30/1, the flow rate of the feed is 2 to 5 m^3/h, and the contact time is about 2 seconds. The indium extraction yield for the two contactors is 91.8 and 94.4%, respectively, and the total yield is about 95.6% in the two contactor cascade tests while that of iron always remains lower than 0.5%.

The stage efficiency of indium is plotted in Fig. 6 as a function of the total flow rate, and in Fig. 7 as a function of the flow ratio. The stage efficiencies of indium only slightly increase with the total flow rate. When the total flow rate increases, the mixing of the two phases is more complete while, at the same time, the contact time becomes a little shorter. The former makes the stage efficiency increase but the latter makes it decrease slightly.

It is more difficult for the two phases to contact fully with an increasing flow ratio. Therefore the stage efficiency of indium decreases slightly with an increasing flow ratio (A/O). The stage efficiency of indium in single-stage tests is higher than 90%.

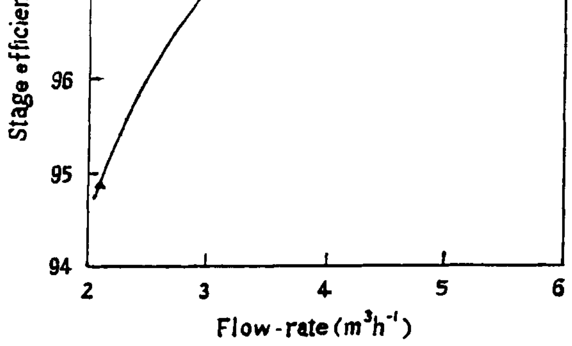

FIG. 6 Effect of aqueous phase flow-rate on stage efficiency.

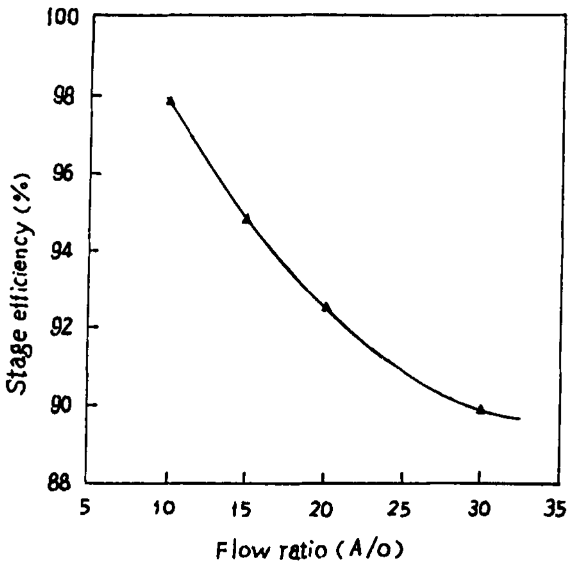

FIG. 7 Effect of flow ratio on stage efficiency.

CONCLUSIONS

1. There is a large difference in the mass transfer velocities of indium and iron in the extraction process. Indium, which has a high mass-transfer velocity, quickly reaches thermodynamic equilibrium after some second contact, and iron, which has a low mass-transfer velocity, still deviates from thermodynamic equilibrium even after 10 seconds of contact.

2. Indium can be well separated from iron in nonequilibrium extraction due to the large difference in the mass transfer velocities of indium and iron. The total extraction yield of indium is about 95.6% in the two-stage cascade tests while that of iron always remains less than 0.5%. The stage efficiency of indium in single-stage tests is higher than 90%. The iron content in indium can be reduced to an allowable value by selecting a suitable stage number.

3. Among available liquid–liquid contactors, only the centrifugal contactor satisfies the requirements for nonequilibrium extraction. It has the advantages of a short contact time and a wide operational flow ratio, and it can be continuously operated and easily maintained.

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