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Short communication

Industrial countercurrent chromatography separations based on a cascade of centrifugal mixer-settler extractors

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ABSTRACT

In hydrometallurgy, traditional extraction technologies, in particular, for isolation and purification of rare-earth metals include a number of processing steps using up to hundreds of mixer-settler extractors. These technologies could be greatly simplified by using the methods of countercurrent chromatography (CCC) separation. However, the current CCC equipment cannot process large volumes of feed material formed during the industrial production of these metals. In this paper, the cascade of centrifugal mixer-settler extractors assembled as a multi-stage unit is suggested for industrial application of CCC and discussed.

1. Introduction

Separation processes largely determine the quality of the products in the production of organic and inorganic materials. An urgent problem is the production of pure and ultrapure substances. To address this issue, new, highly efficient processes of separation and purification of substances are being developed. Countercurrent chromatography (CCC) and centrifugal partition chromatography (CPC) separation processes, collectively called countercurrent chromatography (CCC) [1–13], combine the features of solvent extraction and partition chromatography [14,15]. Both separation methods are based on the different distribution of compounds of a mixture between two immiscible liquids, and the process of separation is controlled by the rates of interphase mass transfer and longitudinal mixing. These methods differ in the scale of the apparatus and in the mode of the separation process. Countercurrent extraction processes are usually carried out in the columns of large diameter or in a cascade of mixer settlers under steady state conditions. In CCC, to hold one of the phases stationary in a chromatography column, centrifugal devices of complex construction are used: in hydrostatic devices, a cascade of chambers is placed in a conventional centrifuge; in hydrodynamic devices, a tube in a spiral shape is wound in one or several layers onto the drum of a planetary centrifuge. A distinctive feature of chromatographic separations is non-steady state mass transfer where the sample is introduced into the column for a relatively short time. In CCC devices, the interphase mass transfer is enhanced due to the high degree of contact between the two phases in the alternating centrifugal force field of the coil planet centrifuge. In partition cells of CPC chromatographs with Z-type cells, a sufficient interfacial area is not provided to achieve rapid mass transfer. For example, the separation efficiency of the FCPC Kromaton Technologies apparatus (Annonay, France), containing 1320 partition cells, was equal to only 120 theoretical plates [16]. However, with newest twin-cell-design, up to 900 theoretical plates have been calculated for a 30 ml Kromaton FCPC rotor [17].

The complexity of centrifugal chromatographic devices imposes restrictions on their scale, and limits the productivity of the separation processes. Technical limitations of the larger instruments do not allow expanding the scope of application of CCC technologies, for example, in hydrometallurgy for separation of rare-earth metals. The purpose of this paper is to discuss the possibilities of using currently available countercurrent solvent extraction equipment, in particular, a cascade of centrifugal mixer-settler extractors, for conducting chromatographic separation processes including conventional CCC separation and more novel elution modes such as multiple dual mode [18–21] and closed-loop recycling [22–28] CCC and promote the application of CCC in the field of hydrometallurgy.

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2. Countercurrent chromatography separations in a cascade of centrifugal mixer-settler extractors

As mentioned above, a disadvantage of chromatographic apparatuses is relatively low throughput when addressing the needs of large volume industries, such as hydrometallurgy. They are incapable of throughput levels achievable by conventional solvent extraction equipment. The performance of mixer-settler extractors is several orders of magnitude higher than that of chromatographs and they are able to handle large volumes of dilute liquors. Multistage cascades of mixer-settler extractors are widely used in hydrometallurgy. A plant producing rare-earth elements may contain hundreds of stages of mixer-settlers [29]. Extractors can be assembled as a multi-stage unit, providing the required number of stages. This does not require inter-stage pumps.

A mixer-settler extractor basically consists of two – mixing and settling – chambers. In principle, CCC separations can be carried out in cascades of conventional mixer–settlers. However, in conventional extractors, the separation of the liquid phases occurs in the gravitational field, which causes a large volume of settling chambers. In the case of chromatography, this will enhance the broadening of chromatographic bands as they move along the cascade. Centrifugal extractors are free of this disadvantage; the volumes of mixing and settling chambers are approximately equal. In the mixing chamber – a stirred tank (TsENTREK, Russia [30]; Rousselet Robatel, France (www.rousselet.com – www.rousselet-robatel.com)) or the annular zone between the outside of a hollow rotor and the inside of the outer housing [31–33] – an intense mixing of two immiscible liquids occurs. In the settling chamber (inside the hollow rotor), the two phases are efficient and fast separated by centrifugal forces.

The use of the cascade of centrifugal mixer-settlers (CCMS) as a chromatography plant offers a variety of options for performing industrial CCC separation processes. Fig. 1 shows the cascade of centrifugal mixer-settler extractors of the TsENTREK type operating in CCC separation modes. The stirrer 1 and the hollow rotor 2 (centrifugal separator) are mounted on a common shaft. Due to the large interfacial area created by the intense mixing, and the efficient centrifugal phase separation, each stage in the cascade can be considered as an equilibrium (theoretical) stage. In the case when the

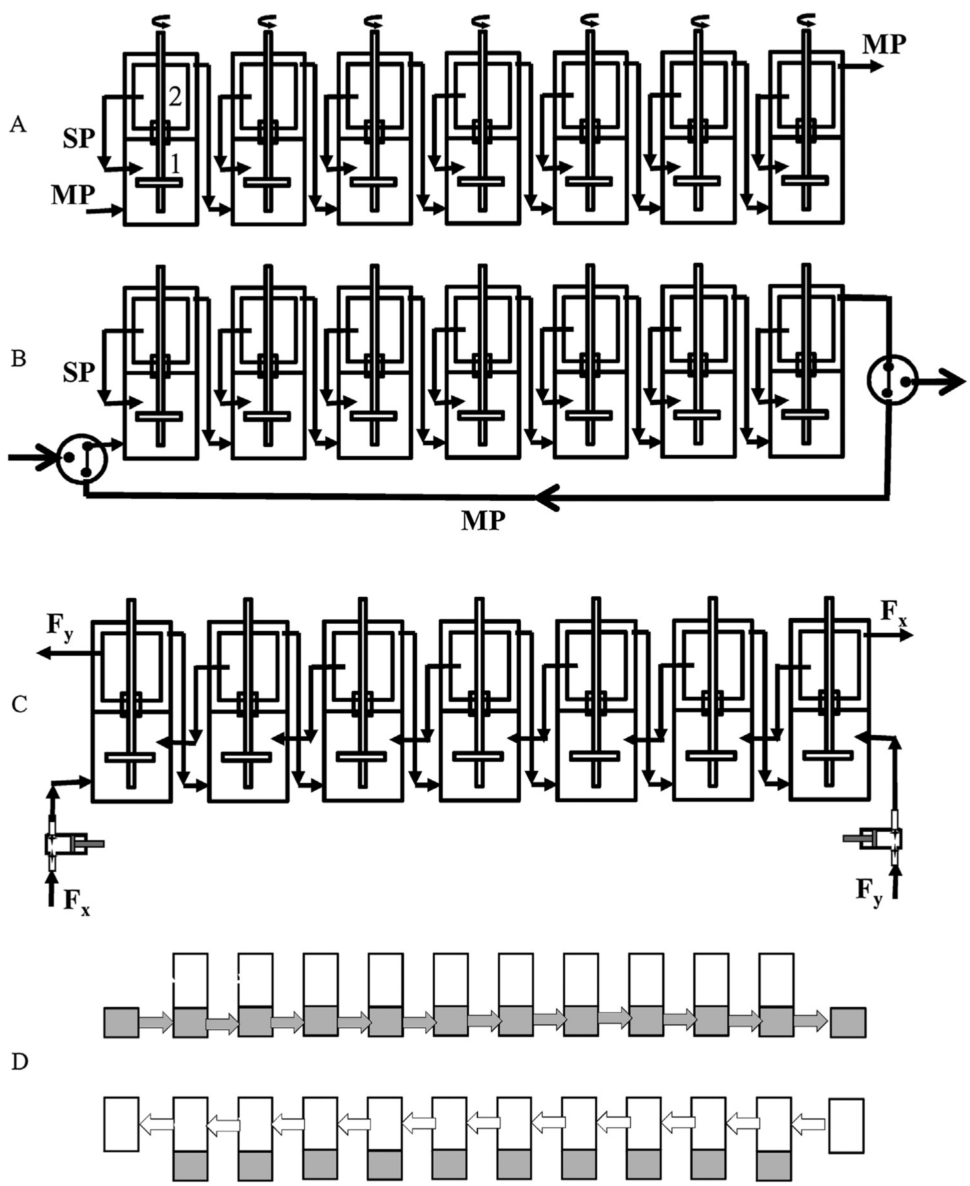

Fig. 1. Cascade of centrifugal mixer-settler extractors operating in CCC separation modes: A – conventional isocratic elution mode; B – CCMS CCC separation in the closed-loop with a long recycling pipe; C, D – Craig's counter-current distribution method.