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Development and Performance of a Glass Fiber Reinforced Plastic Centrifugal Contactor

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Abstract: Centrifugal contactors are efficient extraction equipments and have been successfully used in some industrial fields. But many of the extraction systems contain H_2SO_4 or HCl, therefore, centrifugal contactors that their main material is the stainless steel or titanium can be corroded by these acids not to be used in these systems. In order to solve the problem, a centrifugal contactor with 70 mm of the rotor diameter that the main material is the glass fiber reinforced plastic (GFRP) has been developed at Institute of Nuclear and New Energy Technology, Tsinghua University, China (INET). The mechanical performance of GFRP satisfies the operating requirements of the centrifugal contactor. The maximum total flow of the single stage contactor can reach 200 L/h at suitable operating conditions for H_2O-kerosene system. The extraction stage efficiency is greater than 95% at suitable operating conditions for both extracting Co^{2+} with 35% N_{235} (tertiary amine)-20% iso-octyl alcohol-45% kerosene from $CoCl_2$ solution and stripping Co^{2+} with 0.1 mol/L HCl solution from the organic phase (35% N_{235}-20% iso-octyl alcohol-45% kerosene) containing Co^{2+}.

Keywords: Centrifugal contactor, glass fiber reinforced plastic, mechanical performance, hydraulic performance, mass transfer performance

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INTRODUCTION

Centrifugal contactors are efficient extraction equipments in extraction processes. Compared with conventional contactors such as mixer-settlers and pulsed columns, centrifugal contactors offer the following advantages (1–3):

- low liquid inventories;
- excellent phase separation;
- high mass transfer efficiency;
- compact and short therefore low capital costs;
- do not lose the steady-state when shut-down;
- rapid start-up, shut-down, and wash out of the process liquors;
- direct interconnection with any desired number of contactors for multi-stage processes.

The primary centrifugal contactor was the paddle type, and had been successfully developed and operated for many years at Savannah River Plant (SRL). In the late 1960s, the paddle type centrifugal contactor was modified to the annular type at the Argonne National Laboratory (ANL). The ANL centrifugal contactor was reliable, easy to operate, and maintain (4–6). In the late 1970s, a series of annular centrifugal contactors have been successfully developed with the rotor diameter from 10 mm to 230 mm and used in some industrial fields at INET (7–9).

At present, the main material of centrifugal contactors is stainless steel or titanium that can be corroded by H_2SO_4 or HCl, so they cannot be applied in extraction systems containing these acids. The glass fiber reinforced plastic (GFRP) has many desirable properties over conventional metallic materials including high corrosion resistance to acids, high strength and stiffness to weight ratio, low cost, and can easily be optimized to suit any applied load (10–12). In order to make the centrifugal contactor be applied in systems containing H_2SO_4 or/and HCl, a centrifugal contactor with 70 mm of the rotor diameter that the main material is GFRP has been developed to use in the industrial scale test for separating rare earths to obtain the high purity yttrium at INET. The design goal of the GFRP centrifugal contactor is as follows:

- the mechanical performance is good;
- the expected total flow (F) of two phases can reach 100 L/h;
- the entrainment is less than 0.5%;
- the range of flow ratio (aqueous/organic) is from 10/1 to 1/10;
- the rotor speed can be varied from 2000 r/min to 2800 r/min by frequency modulation;
- the mass transfer efficiency can reach 95%.

In this paper, both design and operation of the GFPR centrifugal contactor are described. Experimental results on the mechanical, hydraulic, and mass-transfer performance of a single-stage contactor are presented.

DEVELOPMENT OF THE GFRP CENTRIFUGAL CONTACTOR

Radius of Light and Heavy Phase Weirs

In designing a centrifugal contactor, determination of the weirs radii is one of the most important problems, since the weirs determine the interface and the interface must be kept in reasonable region under the expected change of the operation conditions. The light and heavy phase weirs of annular centrifugal contactors must be satisfied with the following equation (4, 13):

$$r_i = \sqrt{\frac{F_A R_A^2 - (\rho_o/\rho_A) F_o R_o^2}{1 - \rho_o/\rho_A}} \tag{1}$$

where r_i is the radius of interface (cm), $F = (r/R)^2$, R is the radius of phase weir (cm), r is the radius of liquid surface when flowing over the weir (cm), footnotes "A" and "O" represent heavy and light phase respectively.

Two facts must be considered when designing a contactor. First, the radius of the rotor inlet is somewhat smaller than that of the light phase weir for the dispersed liquids to be pumped through the rotor. Second, the radius of the light phase weir is smaller than that of the heavy phase weir. In addition, once the rotor of the centrifugal contactor has been processed, the radius of the light phase weir is determined, and the radius of the heavy phase weir is the only parameter that can be changed.

The Maximum Throughput and the Dispersion Number

The dispersion number concept proposed by Leonard et al. can be used to obtain the maximum throughput of the centrifugal contactor. The dispersion number N_{Di} that depends on the system, mixing intensity and the type of dispersion, is defined as (14):

$$N_{Di} = \frac{Q}{V}\sqrt{\frac{\Delta Z}{\bar{r}\omega^2}} \tag{2}$$

where Q is the maximum throughput of both phases (cm^3/s), V is the volume of the settler (cm^3), ΔZ is the initial thickness of the dispersion band (cm), ω is the rotational velocity (rad/s), and $\bar{r}$ is the average radius (cm) given by:

$$\bar{r} = \frac{2(r_{out}^3 - r_{in}^3)}{3(r_{out}^2 - r_{in}^2)} \tag{3}$$

where r_{out} is the radius to the outer edge of the dispersion band (cm) and r_{in} is the radius to the inner edge of the dispersion band (cm).

The dispersion number is proportional to the maximum throughput for solvent extraction equipment of a given size with a given accelerational force field. N_{Di} values for centrifugal units can be obtained for any extraction system, either from batch gravity settling test or by using a small centrifugal settler with interface control provision, so that the dispersion can be positioned to occupy the full separating zone. Leonard et al. present a standard test for measuring N_{Di}. In addition, they included some notes that addressed typical questions which a user might have on the standard test and variations on the standard test that might, in some circumstances, be appropriate and even necessary (14). After obtaining the experimental dispersion numbers for both aqueous and organic phase continuous operation, the maximum throughput is obtained from Equation 2.

The Choice of GFRP Material

GFRP composite materials are essentially composed of the glass fiber and a resin matrix. The glass fiber has a high tensile strength and a high modulus of elasticity and is the load bearing component. The matrix is the bonding material used to hold the fibers together so as to prevent shear between them, but also protect the fibers and to maintain the dimensional stability of the GFRP product (10–12).

Three types of the glass fiber most commonly used are alkali-free, moderate-alkali, and high-alkali glass fibers. The content of alkali metals in the alkali-free glass fiber is less than 2%, so the alkali-free glass fiber has some advantages, such as resistance to alkali corrosion, good water fastness, and excellent insulating properties, et al., while its disadvantage is bad acid fastness. The content of alkali metals in the moderate-alkali glass fiber is about 5%–12%, so advantages of the moderate-alkali glass fiber are good acid fastness and low cost, while its disadvantages are bad water fastness, bad resistance to alkali corrosion and bad insulating properties. The content of alkali metals in the high-alkali glass fiber is higher than 12%, so it is usually not used in the practical engineering.

The resin determines mainly some properties of GFRP, such as corrosion resistance, high temperature resistance, and fire resistance. There are three types of resins, namely the unsaturated polyester resin, the vinyl resin, and the epoxy resin. The main advantages of the unsaturated polyester resin for GFRP are low viscosity, fast cure time, dimensional stability, excellent chemical resistance, and moderate cost. Its disadvantage is high volumetric shrinkage during processing. The properties of the unsaturated polyester resin are lower than those of the epoxy resin. However, the combination of low cost, excellent properties, and processability make it the most widely used resin for GFRP. The vinyl resin has advantages over the unsaturated polyester resin in terms of chemical resistance and high temperature resistance. It is easier to handle during processing than either the unsaturated polyester and epoxy resins, and has better resilience than the unsaturated polyester resin. It has also high interfacial strength. The vinyl resin is well suited for the manufacture of GFRP due to its low viscosity and short sure time, but it has a high volumetric shrinkage during cure, and is more expensive than the unsaturated polyester resin. The epoxy resin has high strength and creep resistance, strong adhesion to fibers, chemical and solvent resistance, good electrical properties, high glass transition temperature, and low shrinkage and volatile emission during cure, and can be used in all GFRP manufacturing processes. However, the epoxy resin is also more expensive than the unsaturated polyester resin, and its viscosity is high therefore more difficult to process by hand than the unsaturated polyester resin. Typical physical and mechanical properties of commercial matrix resins are shown in Table 1 (12).

Because the extraction systems in the industrial scale test for separating rare earths to obtain the high purity yttrium at INET will contain H_2SO_4 or/and HCl, and more than 300 stages GFRP centrifugal contactors will be needed for the test. So we selected both the moderate-alkali glass fiber and the unsaturated polyester resin to process the GFRP for the centrifugal contactor to meet the requirements of the test and reduce costs. The main auxiliary materials were crosslinker, evocator, accelerant, and inhibitor, etc. Some properties of the GFRP are shown in Table 2.

Structure Design

The GFRP centrifugal contactor has been developed on the basis of the former metallic centrifugal contactor at INET, which is shown in Fig. 1. The rotor

Table 1. Typical physical and mechanical properties of commercial matrix resins

	Polyester	Epoxy	Vinyl ester
Tensile strength (MPa)	20–100	55–130	70–80
Modulus of elasticity (GPa)	2.1–4.1	2.5–4.1	3.0–3.5
Ultimate strain (%)	1–6	1–9	3.5–5.5
Density (g/cm^3)	1.0–1.45	1.1–1.3	1.1–1.3
The maximum working temperature (°C)	100–140	50–260	90–140
Cure shrinkage (%)	5–12	1–5	5.4–10.3

Table 2. Properties of the GFRP

Density (g/cm^3)	Tensile strength (MPa)	Flexural strength (MPa)	Modulus of Elasticity (GPa)	Cohesional strength with carbon steel (MPa)	The highest temperature of application (°C)
$1.7 \sim 1.8$	>100	>150	>12.0	10	100

diameter is 70 mm, and the hold-up volume of both the mixing zone and the rotor is about 1 L. Because both properties and processability of GFRP were different to metal, many changes were made for the development of the GFRP centrifugal contactor. To meet the requirements of both the mechanical performance and the working accuracy, many parts of the GFRP centrifugal contactor were made through which GFRP was cohered with the carbon steel.

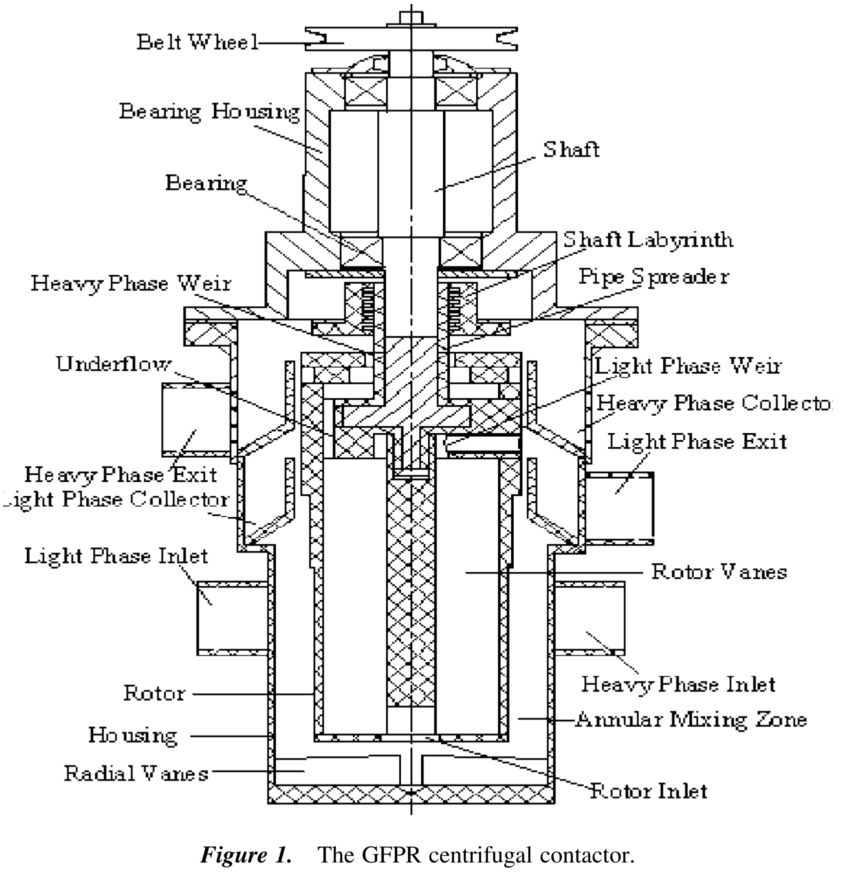

Figure 1. The GFPR centrifugal contactor.

The rotor was the most important part of the GFRP centrifugal contactor. Its upper section was the weirs including the light phase weir and the heavy phase weir, and its lower section was the settling chamber. In order to control the geometrical dimensions, these two sections were separately made through moulding, and then were bonded to the rotor. Since the concentricity of the rotor had directly affected the mechanical performance of the GFRP centrifugal contactor, these two sections were fixed in the mould to be bonded. The rotor speed was higher than 2000 r/min, so the bonded section must be longer and thicker to increase the cohesional strength.

The shaft made of the carbon steel (40Cr) was jointed with the upper section of the rotor by the mating cone. In order to ensure strength of the connected structure, the carbon steel casing was embedded in the upper section of the rotor (see Fig. 2). The pipe spreader that its material was GFRP was used to prevent the shaft from being corroded by acids. The material of the bearing housing was the carbon steel, but its bottom face was covered by GFRP, and its the thickness was about 5 mm. The material of the shaft labyrinth was polypropylene. The housing as well as vanes, inlets, collectors, and exits were made by hand with GFRP.

OPERATION OF THE GFRP CENTRIFUGAL CONTACTOR

Two immiscible liquids with different density are fed from the opposite sides into the annular mixing zone between the spinning rotor and the stationary housing. The liquid-liquid dispersion created by turbulent Couette flow in the annular mixing zone flows by gravity to the inlet in the bottom face of the rotor and thus into the centrifugal separating zone inside the rotor. Here the dispersion breaks and separates rapidly under the high centrifugal force. The separated phases flow separately through the heavy phase weir and the light phase weir of the rotor into their collector rings in the housing. Then each liquid leaves its collector ring through a tangential exit, and flows into an adjacent stage respectively. The extraction cascade is formed by linking

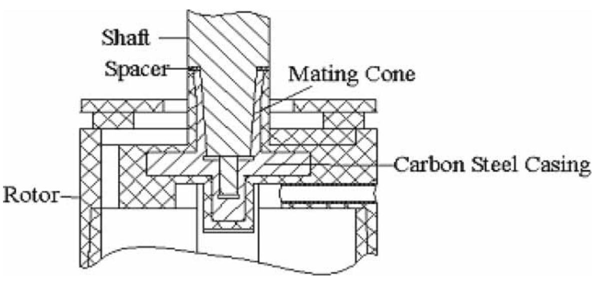

Figure 2. The upper section of the rotor.

the exit to its corresponding inlet of the neighbor extractors in the opposite direction.

PERFORMANCE OF THE GFRP CENTRIFUGAL CONTACTOR

Mechanical Performance

In order to know whether design and processing were reasonable, and whether the main parts of the GFRP centrifugal contactor such as the rotor, and the housing etc. had enough mechanical performance. In addition, the GFRP centrifugal contactors will be continuously operated for about 30 days in the industrial scale test for separating rare earths to obtain the high purity yttrium at INET. So the mechanical test that the GFRP centrifugal contactor was continuously operated for 700 h was carried out. The test conditions were that the rotor speed was 2800 r/min, the flow rate of water was 100 L/h, and the flow rate of the organic phase was 0 L/h. It was shown that the mechanical damage did not happen in all of the GFRP centrifugal contactor's parts, and all of the bonded sections between the GFRP part and the GFRP part, and between the GFRP part and the carbon steel part did not arise fracture too. Meanwhile, the temperature of the GFRP centrifugal contactor was lower than 50°C after 700 h of operation (25°C of ambient temperature). Both the amplitude and the noise intensity were lower than 10 μm and 80 dB respectively when the GFRP centrifugal contactor was being operated. For the GFRP centrifugal contactors, the lower the temperature is and the less both the amplitude and the noise intensity are, the better the performance of the contactors is. Both the low temperature and the little amplitude are useful to maintain the normal lifetime of the contactors. Moreover, the highest temperature of application for the GFRP centrifugal contactors is 100°C (see Table 2). And when the noise intensity is more than 80 dB, the health of the operators will be suffered.

Hydraulic Performance

The hydraulic performance of the single-stage GFRP centrifugal contactor was tested with H_2O-kerosene system. The experimental flowsheet is shown in Fig. 3. Each phase liquid was pumped through a rotameter to its inlet pipes of the contactor, and effluent streams were returned to proper tanks. To determine the amount of the opposite phase entrained, effluents were collected in a graduate; and after two phases separated, the entrainment was determined by volume measurement. The experimental results are shown in Table 3. It was shown that the hydraulic performance of the GFRP centrifugal contactor satisfied the expected design goal. Moreover, the different operating

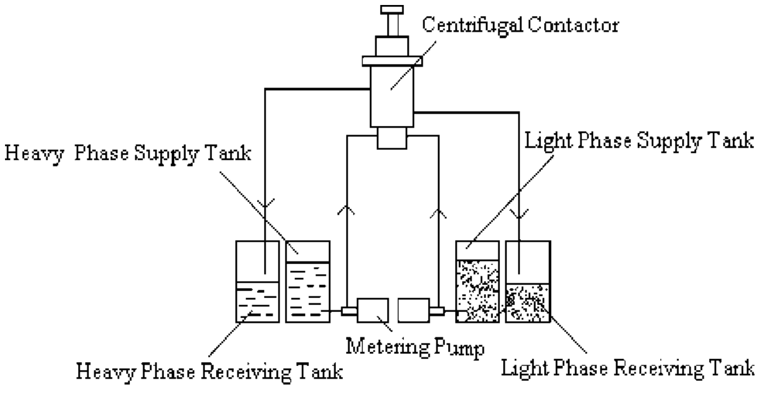

Figure 3. Flowsheet of the single-stage extractor test.

range could be changed by which the different radius of the heavy phase weir was selected, such as 1.7 cm, 1.8 cm, or 1.9 cm.

Mass Transfer Performance

The experimental system of the mass transfer tests of the single-stage GFRP centrifugal contactor was the same as that of the hydraulic test (see Fig. 3). The extraction stage efficiency of the centrifugal contactor was determined by both extracting Co^{2+} with 35% N_{235}-20% iso-octyl alcohol-45% kerosene from $CoCl_2$ solution and stripping Co^{2+} with 0.1 mol/L HCl solution from the organic phase (35% N_{235}-20% iso-octyl alcohol-45% kerosene) containing Co^{2+}. The concentration of Co^{2+} in the aqueous phase was analyzed with ICP-AES (Inductive Coupled Plasma). The concentration of Co^{2+} in the organic phase was determined first by stripping with 0.1 mol/L HCl three times and then by analyzing the stripping solution with

Table 3. Results of the hydraulic test

Radius of the heavy phase weir (cm)	The flow ratio (A/O)	The maximum total flow (L/h)	Entrainment (%)
1.8	$10/1 \sim 1/10$	200	< 0.5

Table 4. Results of extracting Co from the aqueous phase

Rotor speed (r/min)	Total flow (L/h)	Flow ratio (A/O)	$C_{A,in}$ (g/L)	$C_{A,out}$ (g/L)	$C_{A,eq}$ (g/L)	Extraction stage efficiency (%)
2800	31.9	1/1	4.622	0.237	0.142	97.9
2800	36.8	1/1	4.622	0.257	0.142	97.4
2800	51.2	1/1	4.622	0.301	0.142	96.5
2800	60	1/1	4.622	0.317	0.142	96.1

ICP-AES. The extraction stage efficiency was calculated by Murphree equation as follows (15, 16):

$$E_A = \frac{(C_{A,in} - C_{A,out})}{(C_{A,in} - C_{A,eq})} \times 100\%(\text{ for the aqueous phase})$$

$$E_O = \frac{(C_{O,out} - C_{O,in})}{(C_{O,eq} - C_{O,in})} \times 100\%(\text{ for the organic phase})$$

where E_A and E_O are the extraction stage efficiency of the aqueous phase and the organic phase respectively (%), $C_{A,\mathrm{in}}$ and $C_{A,\mathrm{out}}$ are the inlet and the outlet concentrations of Co^{2+} in the aqueous phase respectively (g/L), $C_{O,\mathrm{in}}$ and $C_{O,\mathrm{out}}$ are the inlet and the outlet concentrations of Co^{2+} in the organic phase respectively (g/L), $C_{A,\mathrm{eq}}$ and $C_{O,\mathrm{eq}}$ are the equilibrium concentrations of Co^{2+} in the aqueous phase and in the organic phase respectively (g/L).

Results of the extracting test are presented in Table 4. It was seen that the extraction stage efficiency for aqueous phase could reach 96% when the rotor speed was 2800 r/min, the total flow of two phases was 31.9 ~ 60.0 L/h, and the flow ratio (A/O) was 1/1.

Results of the stripping test are presented in Table 5. It was also seen that the E for the organic phase was greater than 95% when the rotor speed was 2800 r/min, the total flow of two phases was 27.7 ~ 66.6 L/h, and the flow ratio (A/O) was 1/7.4 ~ 1/17.5.

Table 5. Results of stripping Co from the organic phase

Rotor speed (r/min)	Total flow (L/h)	Flow ratio (A/O)	$C_{O,in}$ (g/L)	$C_{O,out}$ (g/L)	$C_{O,eq}$ (g/L)	Extraction stage efficiency (%)
2800	27.7	1/7.4	1.1	0.061	0.026	96.7
2800	31.7	1/8.1	1.1	0.067	0.034	96.9
2800	64.3	1/10.2	1.1	0.078	0.043	96.7
2800	66.6	1/17.5	1.1	0.315	0.078	95.5

CONCLUSION

The GFRP centrifugal contactor with 70 mm of the rotor diameter has been developed. Results of a series of tests proved that the GFRP centrifugal contactor had good mechanical, hydraulic, and mass transfer performances, and satisfied the expected design goal. GFRP has many desirable properties over conventional metallic materials including high corrosion resistance, high strength and stiffness to weight ratio, low cost, and can easily be optimized to suit any applied load, so the GFRP centrifugal contactor has good applied prospect in the hydrometallurgical field.

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