Extraction of heavy metals from aqueous solutions in a modified rotating disc extractor

B. A. Amer, M. H. Abdel-Aziz, E.-S. Z. El-Ashtoukhy and N. K. Amin

ABSTRACT

Extraction of Cu^{+2} from dilute aqueous solutions as a case study by liquid cation exchanger in a modified rotating disc extractor was studied. The liquid cation exchanger consisted of naphthenic acid dissolved in an inert carrier kerosene. Variables studied were: initial concentration of Cu^{+2}, disc rotational speed, concentration of naphthenic acid, flow rates of continuous and dispersed phase, degree of roughness, and number of rotating discs. The rate of extraction increased with increasing rotational speed, concentration of naphthenic acid, flow rate of dispersed phase, degree of roughness and number of discs to a certain number, while increasing Cu^{+2} concentration and flow rate of continuous phase decreased the rate of extraction. All variables were correlated by the dimensionless mass transfer equation. Possible practical applications of the present data in treating aqueous waste from different sources such as petrochemical industries, electroplating, and hydrometallurgical processes was highlighted.

Key words | extraction, heavy metals, modified disc, naphthenic acid, solvent extraction, wastewater

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NOMENCLATURE

- A Mass transfer area, (cm^2)
- C Concentration of Cu^{+2} at time t, (mol/cm^3)
- C_e Equilibrium concentration of Cu^{+2}, (mol/cm^3)
- Co Initial concentration of Cu^{++}, (mol/cm^3)
- D Diffusivity of Cu^{+2}, (cm^2/s)
- dp Diameter of pipe, (cm)
- d_T Column diameter, (cm)
- d_R Rotor disc diameter, (cm)
- K Mass transfer coefficient, (cm/sec)
- k' Volumetric mass transfer, (cm^3/sec)
- L Height of the extractor, (cm)
- P Power consumption, (Watt)
- S Degree of roughness, (mm)
- t Time, (s)
- V_c Continuous phase velocity, (cm/s)
- V_d Dispersed phase velocity, (cm/s)
- V_s Solution volume, (cm^3)

DIMENSIONLESS TERMS

- Re_s Reynolds number of the rotating disc ($\rho_c \omega d_R^2/\mu_c$)
- Re_c Reynolds number of the continuous phase flow ($\rho_c V_c d_p/\mu_c$)
- Re_d Reynolds number of the dispersed phase ($\rho_d V_d d_p/\mu_d$)
- Sc Schmidt number of the continuous phase ($\mu_c/\rho_c.D$)
- Sh Sherwood number (Kd_R/D)
- Sh_m Modified Sherwood number ($K/d_R D$)

GREEK SYMBOLS

- $\propto,\beta,\nu,\tau,\varepsilon$ Constants
- δ Diffusion layer thickness, cm

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μ	Viscosity of the solution (Poise)
μ_c	Viscosity of the continuous phase (Poise)
μ_d	Viscosity of the dispersed phase (Poise)
ρ	Solution density (g/cm^3)
ρ_d	Density of the dispersed phase (g/cm^3)
ρ_c	Density of the continuous phase (g/cm^3)
ω	Disc rotational speed (rps)

INTRODUCTION

Liquid extraction has received considerable attention as an important separation process, especially in view of its applicability in such processes where vaporization methods are not practicable. For the successful development of liquid extraction processes and the design of extraction equipment, it is necessary to have a knowledge of the phase equilibrium relations of ternary liquid systems.

The presence of heavy metals in wastewater is considered to be a concern for living organisms and the environment. Copper is one of these metals that presents in several industrial applications such as catalyst manufacturing, metal plating, electronic applications, hydrometallurgy and mining processes, fertilizers industry, and petroleum refining (Barakat 2011; Fu & Wang 2011). A great deal of work has been carried out on industrial waste streams with two objectives in mind: (i) to recover heavy metals and conserving them and (ii) to free waste streams from toxic metal ions. Various techniques can be used to remove copper ions from aqueous solution as adsorption on activated carbon (Li *et al.* 2010) chemical precipitation (Feng *et al.* 2000), membrane filtration technologies with different types of membranes (ultrafiltration) (Petrov & Nenov 2004), reverse osmosis (Shahalam *et al.* 2002), nanofiltration (Csefalvay *et al.* 2009) and solvent extraction, which is the main goal of the present work. Solvent extraction is a mass transfer process and its success depends mainly on selection of the extracting agent to be immiscible with feed solution and its ability to pick up the desired solute. Different functional groups of extracting organic solvents can perform this target, such as like ethers (Saito *et al.* 1990), amines (Magwa *et al.* 2012), esters (Soldenhoff 1987) and carboxylic acid (Preston 1985).

The extraction of metals using carboxylic acids can be described by the following reaction (Preez & Preston 1992):

$$\underset{aqueous}{Me^{n+}} + \underset{organic}{RCOOH} \rightarrow \underset{organic}{(RCOO)_n Me^{+}} + \underset{aqueous}{nH^{+}} \tag{1}$$

This equation shows that carboxylic acids behave as cation exchangers and extraction depends mainly on the acidity of the feed solution (Fletcher *et al.* 1964). The present work aims to use stable and cheap extracting agent, namely naphthenic acid, to extract copper ions from different concentrated solutions. This system has been studied by different authors (Flett & Spink 1974; Fadali 2004). Solvent extraction is not limited on choice of solvent and operating conditions but also the extraction equipment. The increasing use of rotating disc contactors in the petrochemical industry is due to its high efficiency, simple construction, low energy consumption and high throughput in comparison to a centrifugal extractor and perforated plate. Developments have been carried out on this equipment to overcome its weak points such as using a rotating perforated disc contactor (Wang *et al.* 2000) and horizontal screens instead of a solid flat disc (Shehata *et al.* 2011). The aim of the present work is to explore the possibility of using a rotating grooved disc contactor as a modified disc with different degrees of roughness to recover copper ions from copper sulfate using naphthenic acid at different operating conditions. Previous studies on a $CuSO_4$–naphthenic acid system revealed that the process is diffusion controlled (Abdel-Aziz *et al.* 2013).

EXPERIMENTAL TECHNIQUE

Materials

Stock solution of copper sulphate was prepared by dissolving $CuSO_4.5H_2O$ A.R. grade chemical in distilled water. Various concentrations of Cu^{2+} were obtained from the stock solution by dilution. Naphthenic acid was used as an extracting agent with acid number 180 and specific gravity 0.91, different molarities were dissolved in odorless kerosene as diluent. Ammonium sulphate and ammonia were used to adjust pH at 6.3 while the temperature was fixed at 23 ± 1 °C during experiments.

Apparatus

Figure 1 shows the experimental apparatus used to conduct the experiments. The apparatus consists of a cylindrical stainless steel column, two glass storage tanks, and rotating horizontal rough discs. The cylindrical column has an inner diameter of 15 cm, height of 50 cm, and 2 mm wall thickness. The column contents were agitated by means of a multirotating rough disc system connected to a stainless steel shaft and driven by a 0.33 hp digital motor. The column height was

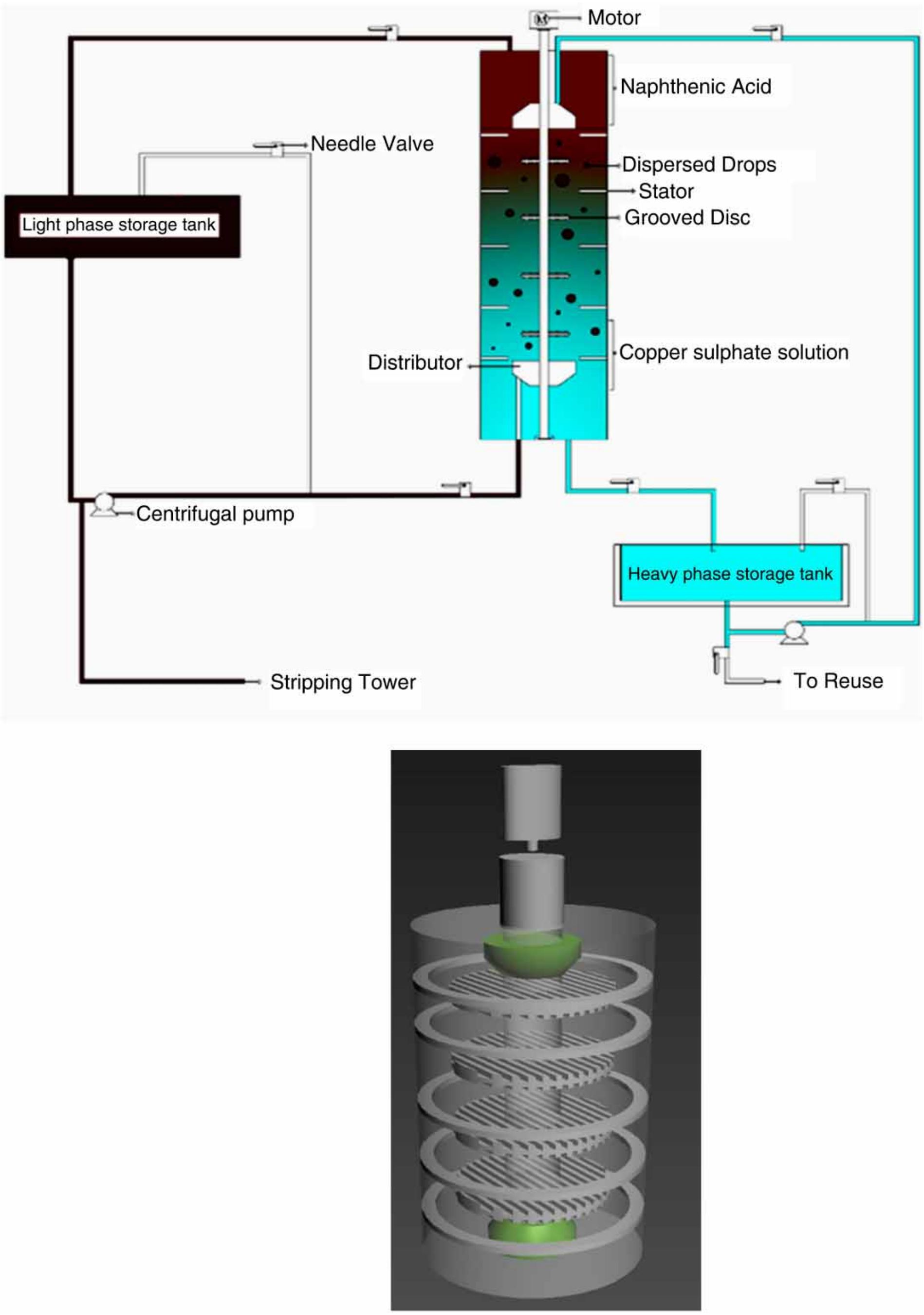

Figure 1 | Experimental apparatus.

divided into four compartments formed by a series of stator rings connected to the column walls. The stator rings were made of stainless steel sheets of 2 mm thickness. Rotating rough discs were centered in each compartment. Each disc had rectangular grooves on both sides with different degree of roughness (1, 2, 3 and 4 mm), as shown in Figure 2. The discs were made of aluminum mounted on the central stainless steel shaft through a two holes guide. All the discs and the stainless steel shaft were isolated by epoxy. The dimensions of the column used in the present work conform to the standard dimensions usually employed in designing extraction columns (Philip 1997). The column end cover plates were provided with the necessary pipe connections for the inlet and outlet flow lines of the dispersed and continuous phase solutions. The flow lines were made of half inch inner diameter stainless steel pipes. Each phase was introduced into the column through a distributor close to the first stator ring from the respective column ends. The exit pipes were flushed with the cover plates on the inside of the column. Each flow circuit of both heavy and light phase consisted of a 20 L glass storage tank and a 0.33 hp plastic centrifugal pump which circulated the solution between the agitated column and the storage tank. The flow rates of both feed and solvent were adjusted by means of a bypass line and a plastic needle valve and were measured by a calibrated rotameter.

Procedure

Before operation was started, the column was first filled with $CuSO_4$ solution (the heavy phase) and set at the desired flow rate, and then the speed of the rotor shaft was adjusted to the desired value, which ranged from 100 to 500 rpm. The light phase (naphthenic acid dissolved in kerosene) was then introduced at the bottom end of the column and the flow rate was set at the desired value. All parameters studied and their values are listed in Table 1. The rate of extraction of copper ion from copper sulfate solution was followed by measuring the change in copper ions concentrations in the aqueous phase with time. Samples of 5 cm^3 were withdrawn from the aqueous (heavy) phase tank at 2 minute intervals for analysis by atomic absorption method using a Perkin-Elmer 2380 atomic absorption spectrophotometer. Ostwald viscometer and a density bottle were used to measure solution viscosity and density, respectively (Findlay & Kitchener 1965) while the diffusivity of copper ions were taken from the literature (Abdel-Aziz *et al.* 2013) and corrected for change in temperature and viscosity by using the Stokes–Einstein equation (Welty *et al.* 2008). All experiments were carried out at $23 \pm 1\,^{\circ}C$.

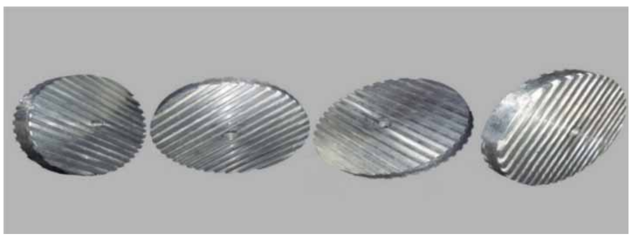

Figure 2 | Grooved disc with different degree of roughness.

RESULTS AND DISCUSSION

Mass transfer at rotating disc contactor

Extraction of copper from $CuSO_4$ by using naphthenic acid was expressed in terms of the volumetric mass transfer (kA). The volumetric mass transfer coefficient (kA) coefficient was obtained under different operating conditions (as listed in Table 1) using the equation:

$$-\frac{dC}{dt} = \frac{kA}{V}(C_e - C) \qquad (2)$$

which integrates to:

$$\ln\left(\frac{C_o - C_e}{C_e - C}\right) = \left(\frac{kA}{V}\right)t \qquad (3)$$

Table 1 | List of all parameters studied and their values

Parameter	Value
Speed of rotating disc, rpm	100, 200, 300, 400, 500
Initial concentration of copper sulphate, M	0.01, 0.02, 0.03, 0.04
Concentration of extracting agent, M	0.05, 0.1, 0.15, 0.2
Flow rate of continuous phase, cm^3/s	100, 150, 200, 280, 330
Flow rate of dispersed phase, cm^3/s	120, 180, 230, 300
Degree of roughness, mm	1, 2, 3, 4
Number of discs	1, 2, 3, 4, 5

Calculated from the slope of the straight line obtained by plotting ln $((C_o–C_e)/(C–C_e))$ vs. t. Figure 3 shows that the present data fit Equation (2)' well under various conditions.

Effect of operating variables

Figure 4 shows the effect of disc rotation speed on the volumetric mass transfer coefficient at various operating conditions. Figure 4 shows that the volumetric mass transfer coefficient increases with increasing rotation speed of the discs, the data fit the equation:

$$k \propto N^b \quad (4)$$

where b is constant, according to Figure 4(a)–4(e)), ranging from 0.32 to 0.38. The increase in the volumetric mass transfer coefficient with increasing rotational speed of the discs may be attributed to the increase in the degree of turbulence generated by the rotating discs. This turbulence enhances the rate of mass transfer between the organic phase and the aqueous phase via the following effects:

(i) Turbulence reduces the thickness (δ) of both the organic phase and aqueous phase diffusion layers around the drop with a consequent increase in mass transfer coefficient ($K = D/\delta$).

(ii) The high shear stress arising from turbulence leads to rapid breakup and coalescence of the dispersed phase drops, the repeated coalescence and re-dispersion of drops enhances the rate of mass transfer through surface renewal (Glasser et al. 1976).

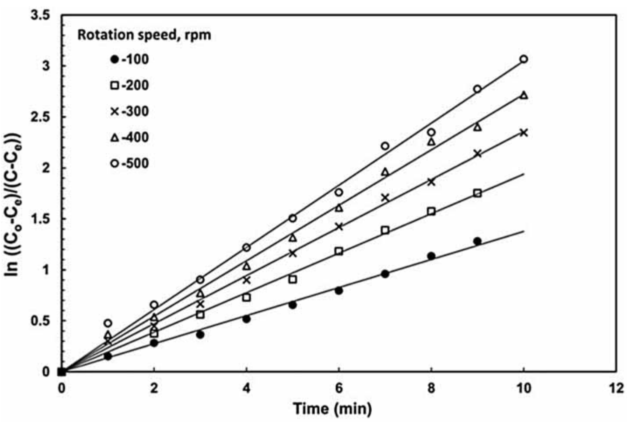

Figure 3 | Typical plots of concentration-time at different rotational speed ($CuSO_4$ conc. = 0.01 M, naphthenic acid conc. = 0.1 M, continuous phase flow rate = 200 cm^3/sec, dispersed phase flow rate = 180 cm^3/sec, degree of roughness = 1 mm.

Effect of concentration of copper sulphate

Figure 4(a) also shows that for a given rotation speed, the volumetric mass transfer coefficient decreases with increasing the initial $CuSO_4$ concentration from 0.5 to 0.2 M. This may be attributed to the fact that: (i) on increasing the initial $CuSO_4$ concentration, solution viscosity and interionic attraction increase with a subsequent decrease in the diffusion coefficient of Cu^{+2} and decrease in the mass transfer coefficient ($k = D/\delta$); (ii) as the initial $CuSO_4$ concentration increases, the concentration of Cu^{+2} will increase in the organic phase (naphthenic acid) which leads to an increase in its viscosity (Fletcher & Wilson 1961) and the resistance to mass transfer.

Effect of continuous phase flow rate

Figure 4(b) shows that the volumetric mass transfer coefficient decreases by increasing the continuous phase flow rate and this may be attributed to the following effects:

(i) The dispersed phase hold up decreases and the diameter of dispersed phase drop enlarges on account of the fact that residence time reduces by increasing the continuous phase flow rate. As a result, the interfacial area decreases and mass transfer coefficient will decrease (Torab-Mostaedi et al. 2008).

(ii) Entrainment of the continuous phase by dispersed phase droplets and formation of eddies below the stator ring. Consequently, axial mixing coefficient in the continuous phase will increase. As a result of axial mixing, a reduction in the concentration driving force for the interphase mass transfer will occur (Murugesan & Regupathi 2004).

Effect of dispersed phase flow rate

Figure 4(c) shows that the volumetric mass transfer coefficient increases by increasing the dispersed phase flow rate, which may be attributed to the turbulence generated

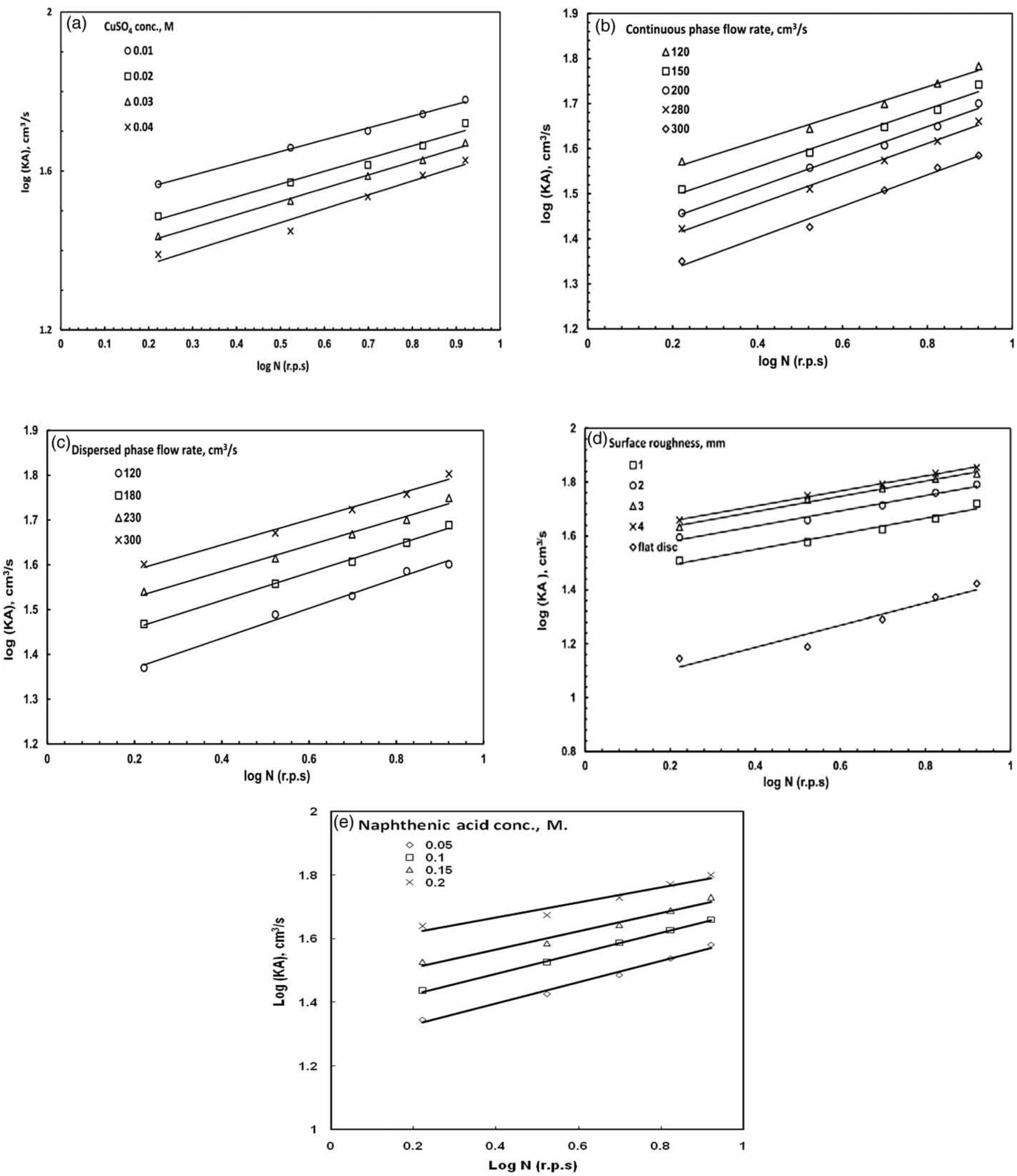

Figure 4 | Effect of rotational speed on the volumetric mass transfer coefficient ((a) at different initial $CuSO_4$ concentrations, (b) at different continuous phase flow rate, cm^3/ s, (c) at different dispersed phase flow rate, (d) at different degree of roughness, (e) at different concentration of naphthenic acid).

downstream of the rotating disc as the axial solution passes through the column and this fits with previous studies shown in Bourne & Lips (1991) and Villermaux *et al.* (1991). The degree of turbulence generated downstream of the disc increases with increasing axial solution flow rate with a consequent increase in the mass transfer coefficient *k* and the interfacial area *A*. Also, roughness elements act as active turbulence promoters where disc rotation turns axial flow to radial flow at the rotating disc surface across the roughness elements.

Effect of degree of roughness

Figure 4(d) shows that the volumetric mass transfer coefficient increases when the degree of roughness of the rotating disc is increased, as represented by the peak to the valley height. The increase in the rate of mass transfer with increasing surface roughness may be attributed to the fact that roughness elements act as active turbulence promoters where turbulence is generated downstream of the roughness elements by virtue of hydrodynamic boundary layer separation (Incropera & Dewitt 1990) with a consequent decrease in the diffusion layer thickness accordingly, the mass transfer coefficient increases. Also, the flow pattern on a rough rotating disc plays an important role where disc rotation induces axial flow, which turns to radial flow at the rotating disc surface across the roughness elements.

Effect of concentration of naphthenic acid

Figure 4(e) reveals that the volumetric mass transfer coefficient increases with increasing naphthenic acid concentration. As a result of varying microscopic hydrodynamic conditions around the dispersed phase drop, the removal rate of Cu^{++} at the drop surface is non-uniform all over the drop surface. Accordingly, the surface tension (Sherwood *et al.* 1975) which depends on dispersed phase concentration will be high at locations with low Cu^{+2} and low with high Cu^{+2}. This surface tension gradient gives rise to strong eruptions and interfacial turbulence at the drop surface (Marangoni effect) (Mao & Chen 2004), which enhances the rate of extraction.

Effect of number of discs

Figure 5 shows the effect of number of discs on the % extraction. The % extraction increases with an increasing number of discs to four discs, then it decreases when the number of discs reaches 5. The same result was obtained in previous studies but with different types of discs, as shown in Sarubbo *et al.* (2005) and Behzad *et al.* (2015). The increase in the % extraction with increasing number of discs may be attributed to the turbulence promoting ability of the discs compared to one rotating disc. A further increase in the number of rotating discs decreased the separating distance between discs and decreases the % extraction. Based on the flow pattern of two parallel rotating discs (Figure 6) each disc is trying to withdraw the solution located between the two discs to its surface, i.e. the solution in between the two discs is subjected to two opposing forces which decreases the axial and radial flow velocity at the lower surface of the upper disc and the upper surface of the lower disc. As a result, the rate of mass transfer decreases at each disc. On the other hand, with increasing the distance between the two discs the negative interaction between the two flow

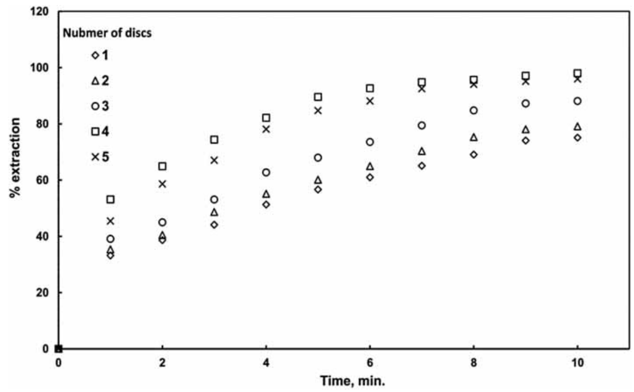

Figure 5 | Effect of number of discs on % extraction ($CuSO_4$ conc. = 0.01 M; Naphthenic acid conc. = 0.1 M; rotation speed = 500 rpm; Continuous phase flow rate = 200 cm^3/s; Dispersed phase flow rate = 180 cm^3/s; degree of roughness = 2 mm).

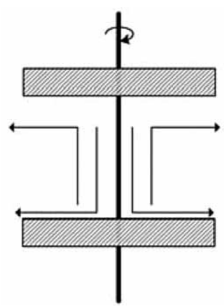

Figure 6 | Flow pattern at two parallel rotating discs.

patterns decreases with a consequent increase in the rate of mass transfer. Sedahmed *et al.* (2000) studied the effect of distance between two rotating screen discs mounted on the same shaft and found that the rate of mass transfer decreases with decreasing screen separation. This result agrees with the present finding.

Data correlation

For the present data, dimensional analysis leads to the correlation (Shahalam *et al.* 2002):

$$Sh_m = a\, Sc^{\alpha}\, Re_s^{\beta}\, Re_c^{\tau}\, Re_d^{\gamma} \left(\frac{s}{d_R}\right)^{\varphi} \tag{5}$$

The volumetric mass transfer coefficient kA was used in obtaining the present correlation and Sh_m is the modified Sherwood number ($Sh_m = kA/d_R D = k'/d_R D$).

The constants a, α, β, τ, γ and φ were determined using the present experimental data. The value of α was fixed at 0.33 following previous theoretical and experimental mass transfer studies (Incropera & Dewitt 1990; Welty *et al.* 2008).

Figure 7 shows the effect of Re_s on Sh_m at different operating conditions, the data fit the equation:

$$\mathrm{Sh_m} \propto \mathrm{Re_s^{0.3}} \tag{6}$$

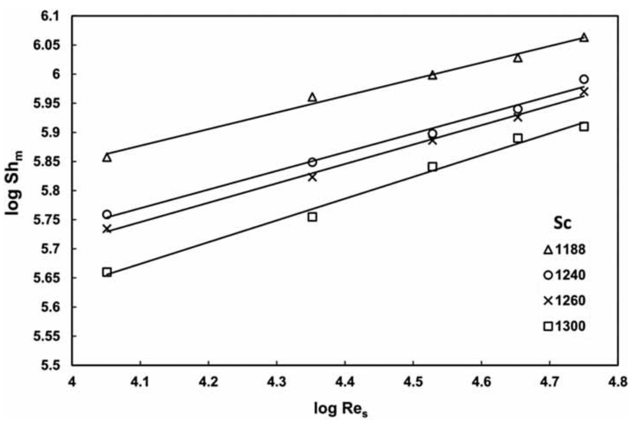

Figure 7 | Effect of Re_s on Sh_m at different Sc (continuous phase flow rate = 200 cm^3/s, dispersed phase flow rate = 180 cm^3/s, naphthenic acid concentration = 0.1 M, degree of roughness = 1 mm).

Figure 8 shows the effect of Re_c on Sh_m, the data fit the equation:

$$\mathrm{Sh_m} \propto \mathrm{Re_c^{-0.4}} \tag{7}$$

Figure 9 shows the effect of Re_d on Sh_m, the data fit the equation:

$$Sh_m \propto Re_d^{0.45} \tag{8}$$

Figure 10 shows the effect of the disc geometry dimensionless ratio $\left(\frac{S}{d_R}\right)$ on Sh_m, the data fit the equation:

$$Sh_m \alpha (S/d_R)^{-0.45} \tag{9}$$

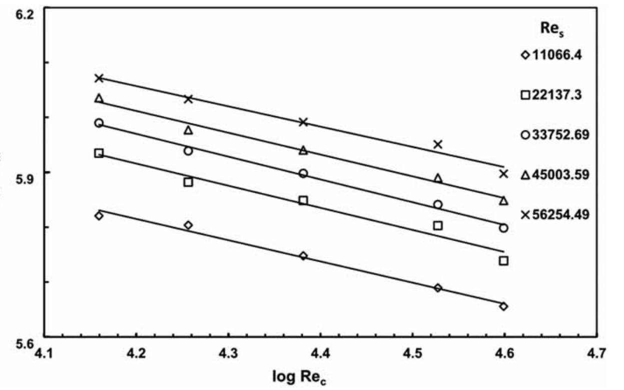

Figure 8 | Effect of Re_c on Sh_m at different Re_s (concentration of $CuSO_4$ = 0.01 M, concentration of naphthenic acid = 0.1 M, dispersed phase flow rate = 180 cm^3/s, degree of roughness = 1 mm).

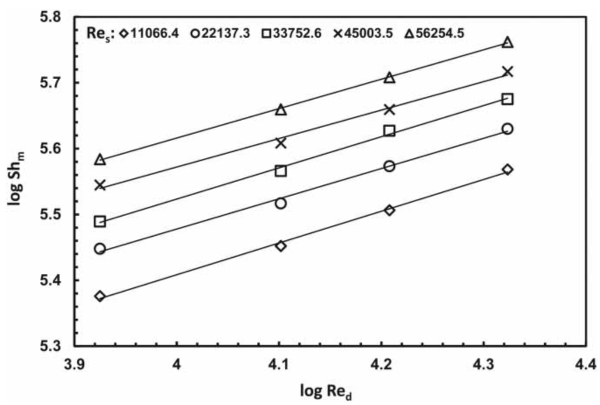

Figure 9 | Effect of Re_d on Sh_m at different Re_s (concentration of copper sulphate = 0.02 M, concentration of naphthenic acid = 0.1 M, continuous phase flow rate = 200 cm^3/s, degree of roughness = 1 mm).

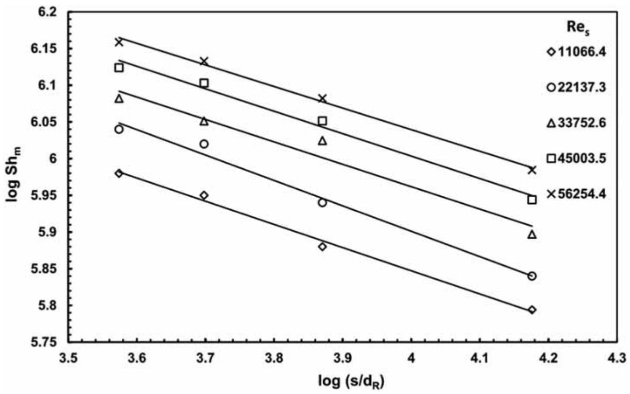

Figure 10 | Effect of the dimensionless ratio (S/d_R) disc geometry on Sh_m (concentration of copper sulphate = 0.02 M, concentration of naphthenic acid = 0.1 M, continuous phase flow rate = 200 cm^3/s, dispersed phase flow rate = 180 cm^3/s).

To obtain the value of the constant (a) in Equation (5), Sh_m was plotted against $Sc^{0.33}\ Re_s^{0.3}\ Re_c^{-0.4}\ Re_d^{0.45}\ (S/d_R)^{-0.45}$.

Figure 11 shows that the data for the conditions $11{,}000 < Re_s < 57{,}000$, $14{,}400 < Re_c < 40{,}000$, $8{,}000 < Re_d < 21{,}100$, $1 \times 10^{-4} < S/d_R < 3 \times 10^{-4}$ fit the equation:

$$Sh_m = 311 Sc^{0.33} Re_s^{0.3} Re_c^{-0.4} Re_d^{0.45} (S/d_R)^{-0.45} \quad (10)$$

with an average deviation of 4.8%.

Power consumption

In order to assist in the economic evaluation of the performance of the present work, mechanical power consumed in rotating discs was measured experimentally under different conditions by means of a watt meter. Table 2 shows the following results:

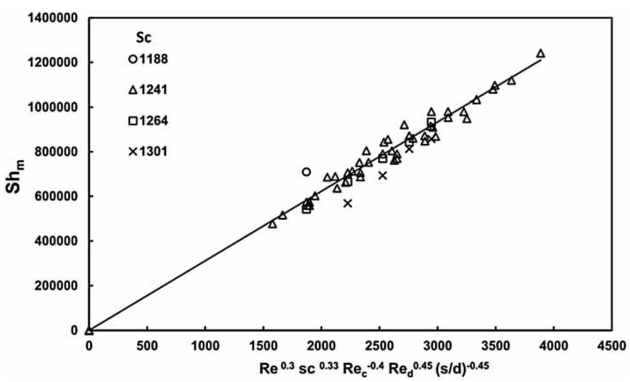

Figure 11 | Overall mass correlation at a rotating rough disc.

Table 2 | Mechanical power consumption at different degrees of roughness

	Degree of roughness, mm				
	1	2	3	4	
rpm	Power consumption, W				Flat disc
100	279	287.8	295	302	153
200	290	304	312.6	322.4	164
300	306	319	328.4	334.1	172
400	323	334	340.9	350.5	180
500	345	350	357	362.1	189

1. power consumption increases with increases disc rotational speed;
2. power consumption increases with degree of roughness.

The results of power consumption and mass transfer coefficient at a rotating disc are, in general, consistent with the finding of Calderbank & Moo-Young (1961) who found that mass transfer coefficient is proportional to power consumption.

CONCLUSIONS

A new extractor design consisting of multirotating rough discs with different degrees of roughness has been suggested. The mass transfer performance of the extractor in removing heavy metals from aqueous solutions was investigated. The study showed that within 10 minutes of operation the extraction ranged from 50 to 98%, depending on the number of rotating discs, rotation speed, and the disc roughness. The mechanical power consumed in rotating the discs ranged from 153 to 362.1 W depending on the rotation speed and degree of roughness. All operating parameters affecting the volumetric mass transfer coefficient were correlated in the form of a dimensionless equation which can be used in the design and operation of such an extractor. Future studies are needed to confirm the advantages of the present extractor and to validate its mass transfer behavior under a wider range of operating conditions such as using different solvent extraction system and different heavy metals.

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