

Experimental Study and Correlation for Mass Transfer Coefficient in the Pilot Plant Multistage Column with the Presence of Molybdenum

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ABSTRACT: *In the present work, multistage extraction column performance for reactive separation of molybdenum from the leach aqueous phase by using D₂EHPA and TBP in kerosene was investigated by using the axial diffusion model. The variation of volumetric overall mass transfer coefficients based on continuous phases at various operating conditions was obtained. By considering the chemical reaction conditions, the experimental data indicate that the volumetric overall mass transfer coefficients increase with an enhancement in rotor speed and continuous and dispersed phase flow rates. According to the sensitivity analysis, the rotor speed and flow rate of the dispersed phase have more influence on column performance. Also, the continuous flow rate has little effect on the volumetric overall mass transfer coefficients. After the steady-state condition was established, the concentration profile for the extraction of molybdenum along the column was obtained. The experimental data were studied and compared with the previous models. A new model based on dimensionless numbers was derived from the evaluation of overall mass transfer coefficients with consideration of the reactive extraction system.*

KEYWORDS: *Multistage column; Molybdenum; Mass transfer coefficients; Reactive extraction.*

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INTRODUCTION

Molybdenum is one of the precious and strategic elements in the world that is found with copper in nature. The application of Molybdenum is in the leading position in the iron and steel industry, and chemical industry. Furthermore, molybdenum has also been used in electrical and electronic technology, medicine, and agriculture. Solvent extraction processes are widely used on an industrial scale for the separation of molybdenum from impurity metals. In recent decades, many extractants such as Alamine336 [2], Alamine304-I [3], TOA [4], D2EHPA, TOPO [6], PC-88A [7], LIX63 [8], and HBL101 [9] have been utilized to extraction of molybdenum from an acidic aqueous phase in the bench experiment. In the continuous experiments, *Morís et al.* investigated the extraction of molybdenum and tungsten from the sulfate aqueous phase with D₂EHPA and Alamine336 in a rotating disc contactor. One of the diverse sorts of agitated extraction columns, multistage columns are used for many applications in the specialty of stainless steel solutions due to increasing mass transfer velocity by applying moving impellers inside the column. The design process of the extraction columns still are dependent on various factors including the industrial scale-up difficulties, types of the reactive extraction system and drops resistance time conditions. Thus, calculation of the mass transfer process is necessary. The mass transfer rate of a multistage column for the extraction of molybdenum has not been studied in the literature. Therefore, the experimental work illustrates the continuous phase overall mass transfer coefficients of the column using an axial diffusion model as a function of dimensionless groups with consideration of molybdenum extraction from leach liquor by a mixture of TBP and D₂EHPA in the kerosene. The influence of effective parameters including agitation speed, and dispersed and continuous phase flow rates on continuous phase overall mass transfer coefficients have been reported in this paper. A new empirical prediction model is derived for estimation of the Sherwood number as a subordinate of the holdup of dispersed phase and Reynolds and Froude numbers.

THEORETICAL SECTION

Mass transfer between droplets and the continuous phase is usually carried out by a combination of molecular diffusion, and natural and forced convection

in the continuous phase. Also, the continuous phase mass transfer coefficients depend on droplet mechanisms, including stagnant, circulating, and oscillating. Recently, many researchers have developed the rate of mass transfer in different types of extraction columns. Thus, several of the published models for the estimation of the mass transfer function in various types of liquid-liquid mass transfer devices are presented in Table 1.

EXPERIMENTAL SECTION

Definition of Extraction Column Components

This liquid-liquid mass transfer device employed in the present work was described elsewhere. A schematic diagram of the multistage extraction column set-up is shown in Fig.1.

As can be seen, the nitrate solution and the solvent containing D₂EHPA and kerosene were brought into the column from the top and bottom of the column, respectively. The column is filled with the aqueous phase until the allowable height, which was controlled by an optical sensor on top of the column. Once the location of the interface was going to change, the diaphragm opens the valve. Therefore, the heavy phase evacuates the column due to the received signal from the optical sensor. It should be noted that the light phase could leave the column by overflow. Both phase flow rates were controlled by two flow measurement devices. In addition, the accessory equipment such as storage tanks, cylindrical glass, pumps, and other types of valves used in this extraction column as previously were described by *Asadollahzadeh and co-workers*.

Reagents and Solution Preparation

All of the materials applied in the experimental works are listed in Table 2. A digital pH meter (Sartorius PB-11) and an ICP-AES instrument (PerkinElmer, Optima 2000 DV) were used the determination the initial aqueous pH and measurement of the molybdenum ion concentration in the aqueous phase, respectively. Furthermore, in the batch experiments, contact of two phases was carried out by a mechanical shaker (CH- 4103 Bottmingen).

Measurement of Physical Properties

All components' physical properties are given in Table 3. The interfacial tension, densities of the two phases, and viscosity of the aqueous and organic phases have

Table 1: Previous models for prediction of mass transfer coefficients in single drop system and extraction column.

Reference	Models	Comments	Equation
[11]	$Sh_c = 0.7Re^{1/2}Sc_c^{1/3}$	High Reynolds number	(1)
[12]	$Sh_c = 2 + \left[\frac{0.66}{1 + Sc_c} + \frac{Sc_c}{2.4 + Sc_c} \left(\frac{0.79}{Sc_c^{1/6}} \right) \right] \frac{Pe_c^{1.7}}{1 + Pe_c^{1.2}}$	$1 < Re < 100$	(2)
[13]	$Sh_c = 1 + 0.72Re^{0.48}Sc_c^{1/3}$	$100 < Re < 2000$ $Sc_c > 200$	(3)
[12]	$Sh_c = 2 + 0.0511Re^{0.724}Sc_c^{0.7}$	$4 < Re < 1000$ $130 < Sc_c < 23600$	(4)
[14]	$Sh_c = 2 \left(\frac{Pe_c}{\pi} \right)^{1/2} \left[1 - \frac{1}{Re^{1/2}} (2.89 + 2.15K^{0.64}) \right]^{1/2}$	$K \leq 2$ & $\frac{\rho_d}{\rho_c} \leq 4$ $Re \geq 70$	(5)
[15]	$\frac{Sh_c - Sh_{c,rigid}}{Sh_{c,circ} - Sh_{c,rigid}} = 1 - \exp[-4.18 \times 10^{-3} Pe_c^{0.42}]$	$10 < Re < 1200$ $190 < Sc_c < 241000$	(6)
[16]	$Sh_{oc} = -40 + 23Re^{1/3}$ $Sh_{oc} = 200 + 0.047Re^{1.44}$	$Re > 40$ (c → d) $50 < Re < 1000$ (d → c)	(7)
[17]	$Sh_{oc} = 12.34 + 0.116Re^{1.389}$ $Sh_{oc} = 2.586 + 0.000217Re^{4.86}$	$Re > 10$ $10 < Re$	(8)
[18]	$Sh_{oc} = -11.95 + 10.96Re^{0.54}(1 - \varphi)$ $Sh_{oc} = -30.19 + 21.59Re^{0.46}(1 - \varphi)$	$5 < Re < 23$ (c → d) $6 < Re < 26$ (d → c)	(9)
[19]	$Sh_{oc} = -49.76 + 14.8Re^{0.64}$ $Sh_{oc} = -59.35 + 27.27Re^{0.45}$	$10 < Re < 150$ (d → c) $9 < Re < 105$ (c → d)	(10)
[20]	$Sh_{oc} = -37.31 + 22.94Re^{0.4}(1 - \varphi)$ $Sh_{oc} = -120.72 + 117.01Re^{0.12}(1 - \varphi)$	(d → c) (c → d)	(11)
[21]	$Sh_{oc} = -121.56 + 103.62Re^{0.16}(1 - \varphi)$ $Sh_{oc} = -119.50 + 113.30Re^{0.12}(1 - \varphi)$	$11.73 < Re < 69.43$ (d → c) $9.45 < Re < 57.08$ (c → d)	(12)

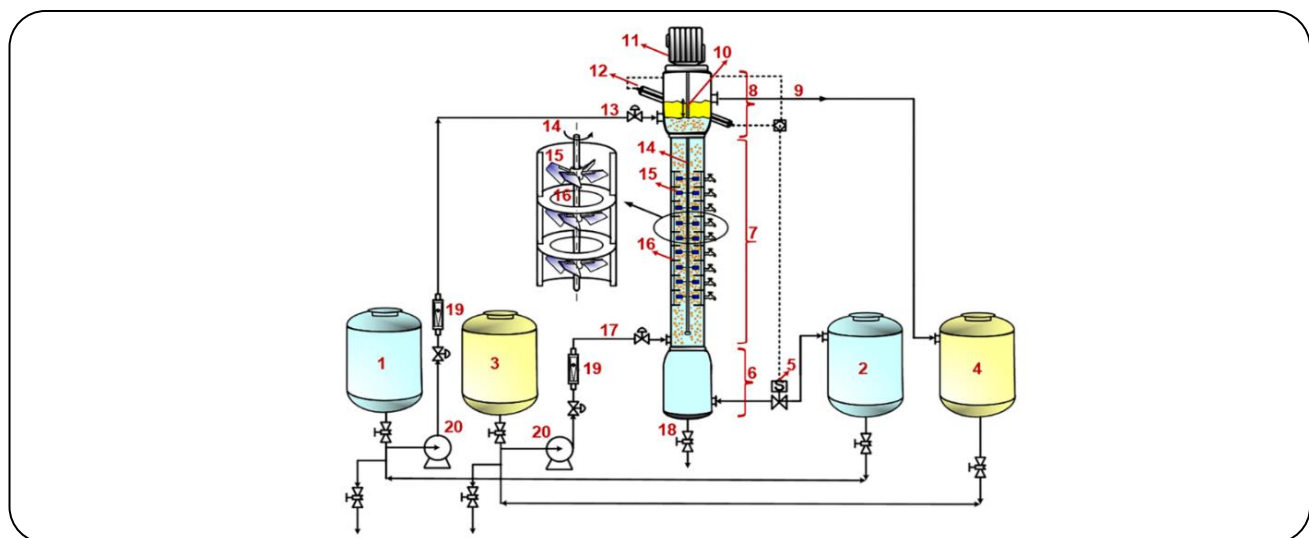


Fig.1. Multistage column with schematic illustration: (1) & (2) aqueous phase reservoirs; (3) & (4) organic phase reservoirs; (5) solenoid valve; (6) bottom separation region; (7) extraction region; (8) top separation region; (9) extract stream output; (10) extract and raffinate streams interface height; (11) electric motor; (12) optical sensor; (13) continuous phase input; (14) rotors haft; (15) 6-blade impellers; (16) stator; (17) organic phase input; (18) raffinate stream output; (19) flow meter; (20) pump. [23].

Table 2: Materials, stated purities, suppliers and utilization.

Chemical	Purity percentage	Supplier	Application
D ₂ EHPA	97%	Sigma-Aldrich	Extractant
TBP	≥99%	Merck	Extractant and phase modifier
Kerosene	≥99%	Tehran Petroleum Refinery	Dilute
Na ₂ MoO ₄ ·2H ₂ O	99%	Sigma-Aldrich	Mo (VI) metal source
H ₂ SO ₄	98%	Merck	pH adjuster
NaOH	97%	Sigma-Aldrich	pH adjuster

Table 3: Physical properties of liquid system used.

	ρ / kgm^{-3}	$\mu / \text{pa.s}$	$D / \text{m}^2\text{s}$	σ / Nm^{-1}
Aqueous phase	998.2	0.963×10^{-3}	2.371×10^{-9}	14.2×10^{-3}
Organic phase	804	1.66×10^{-3}	1.285×10^{-9}	

measured at 25 °C by Krüss tensiometer, pycnometer method, and DVI-Prime viscometer, respectively.

Determination of Dispersed Phase Holdup and Drop Size

After each experiment was completed, the shutdown method was utilized to measure the holdup of the dispersed phase. When the system occurred in a steady state, the outlet and inlet valves of the continuous and dispersed phase were closed, then, the light phase was allowed to settle and the fractional volumetric holdup (φ) was defined as follows:

$$\varphi = \frac{\vartheta_d}{\vartheta_d + \vartheta_c} \quad (13)$$

Drop diameters were measured by photographing the extraction column contents using a Nikon D5000 digital camera. To determine the diameter of drops, ImageJ software (Image Processing and Analysis in Java) were used for analyzing the recorded photographs. Also, the drops were arranged into 0.1 mm size diameter and nearly 1000 drops were analyzed in any photo to investigate the importance of the determined average diameter of droplets. Then, d_{32} is calculated by the following equation:

$$d_{32} = \frac{\sum_{i=1}^n n_i d_i^3}{\sum_{i=1}^n n_i d_i^2} \quad (14)$$

Where n_i and d_i assume the number of droplets and diameter of drops, respectively, and the calculation of the

equivalent diameter for non-spherical droplets is as follows:

$$d_e = (d_1^2 d_2)^{1/3} \quad (15)$$

In addition, the specific interfacial area (a) calculated as follows:

$$a = 6 \frac{\varphi}{d_{32}} \quad (16)$$

Modeling

An axial diffusion model was utilized for the evaluation of mass transfer coefficients in this column. In this paper, the experimental data are interpreted in terms of the axial diffusion model as previously depicted by Pratt and Stevens [25]. The steady-state process was established with the consideration of this model and mass balance in the extraction column for the differential components of the column. The equation set for this process with constant superficial velocities and any agitation speed can be defined as follows:

$$\frac{\partial}{\partial Z} \left(x - \frac{1}{P_c} \frac{\partial X}{\partial Z} \right) + N_{oc} (x - x^*) = 0 \quad (17)$$

$$\frac{\partial}{\partial Z} \left(y + \frac{1}{P_d} \frac{\partial y}{\partial Z} \right) - N_{oc} \frac{V_c}{V_d} (x - x^*) = 0 \quad (18)$$

$$\text{Where, } P_c = H \frac{V_c}{E_c}, P_d = H \frac{V_d}{E_d}, Z = \frac{h}{H}, N_{oc} = K_{oc} a \frac{H}{V_c}.$$

Also, the boundary conditions are as follows:

$$-dx/dZ = Pe_c (x^0 - x) \quad (19)$$

$$dx/dZ = 0 \quad (20)$$

$$y = y^0 \quad (21)$$

Matlab 2014 software with consideration of Eqs. (17) and (18) are used for the computation of overall mass transfer coefficients. The axial mixing coefficient of the dispersed phase at the mass transfer performance is neglected. Tsouris described the following equation for the calculation of the continuous phase axial mixing coefficient in a multistage column contactor.

$$\frac{(1-\phi)E_c}{V_c h_c} = -0.0093 + 0.0144 \frac{D_i N (1-\phi)}{V_c} \quad (22)$$

In the present work, the above equation was used for the prediction of the axial mixing coefficient of the dispersed phase.

RESULTS AND DISCUSSION

In this study, the concentration of molybdenum in sulfate solution was kept at 500 ppm. The optimum operating parameters for the extraction of molybdenum have been carried out at the bench scale. An organic phase with 0.3 M D2EHPA and 0.36 M TBP in kerosene was selected to examine the mass transfer performance of the extraction column. In the continuous experiments, the extraction column has operated under different operating parameters. By considering the molybdenum extraction, all of the experiments were run far from flooding conditions at room temperature ($25^\circ\text{C} \pm 1^\circ\text{C}$). After the steady-state process is established in the present column, and the concentration profile of the aqueous phase for molybdenum extraction from aqueous sulfate solution to the organic phase in each stage is depicted in Fig.2.

The Variation in the Column Performance with the Variation in Rotor Speed

The overall mass transfer coefficient and the interfacial area can be affected the overall performance of the device for the mass transfer process. Fig.3 illustrated the impact of rotation speed on the volumetric overall mass transfer coefficient for the molybdenum reaction with D₂EHPA and TBP in the extraction stage.

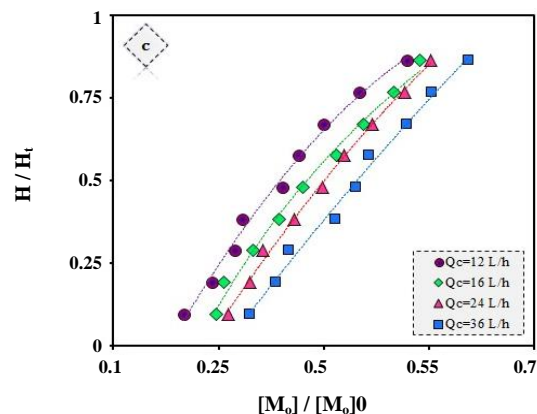
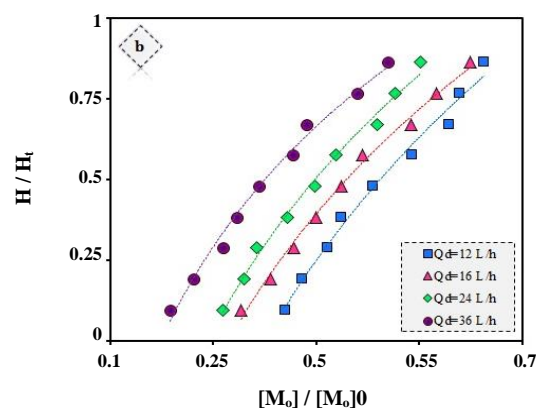
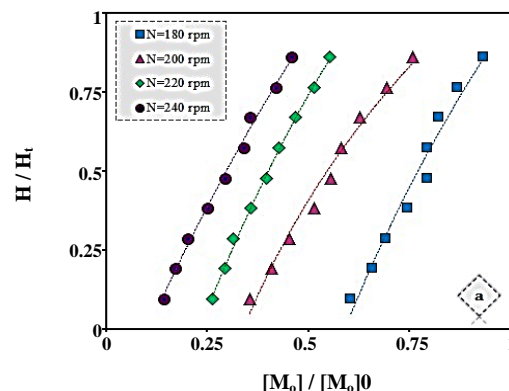


Fig. 2: Molybdenum aqueous phase concentration profiles along the column for the extraction of molybdenum by the mixture of D2EHPA and TBP (a) effect of rotor speed (Experimental conditions: $Q_c=24$ L/h; $Q_d=24$ L/h), (b) effect of dispersed phase flow rate (Experimental conditions: $N=220$ rpm and $Q_c=24$ L/h) (c) effect of continuous phase flow rate (Experimental conditions: $N=220$ rpm and $Q_d=24$ L/h).

As expected, with enhancement in the rotor speed, the value of the dispersed phase holdup and the value of the average drop diameter are increased and decreased, respectively. Therefore, it can be concluded that the value of interfacial area increments with expanding the agitation speed. The higher rotor speed results in the formation of rigid droplets, which reduces the mass transfer coefficient. According to Fig.3, the continuous phase volumetric overall mass transfer coefficient increased with the enhancement of rotor speed. Because of that, the impact of interfacial area (a) on the volumetric overall mass transfer performance is bigger than the overall mass transfer coefficient (K_{oc}).

The Variation in the Column Performance with the Variation in Dispersed Phase Flow Rate

The change of dispersed phase flow rate on the continuous phase overall mass transfer coefficient is shown in Fig.4a. As the dispersed phase flow rate increases, the holdup, and average drop diameter increase, but the effect of the holdup is greater than the average diameter of the droplets. As a result of this process, the interfacial area slightly increases. On the other side, a higher flow rate of the solvent phase causes an increment in the turbulence within the droplets, consequently, the overall mass transfer coefficient increments by enhancing of dispersed phase flow rate. As can be seen from Fig. 4a, the volumetric overall mass transfer increments *via* an enhancement in the dispersed phase flow rate for the molybdenum reaction condition in the extraction stage.

The variation in the column performance with the variation in continuous phase flow rate

Fig.4b illustrates the variation in the column performance as a function of the change in the continuous phase flow rate at different rotor speeds. Besides, it is concluded that the continuous phase overall mass transfer coefficient slightly increases with consideration of an increase in the continuous phase flow rate. Moreover, the continuous phase flow rate has little change in average drop diameter and the increment on the dispersed phase holdup. Consequently, the interfacial area slightly increases with an increment in the continuous phase flow rate.

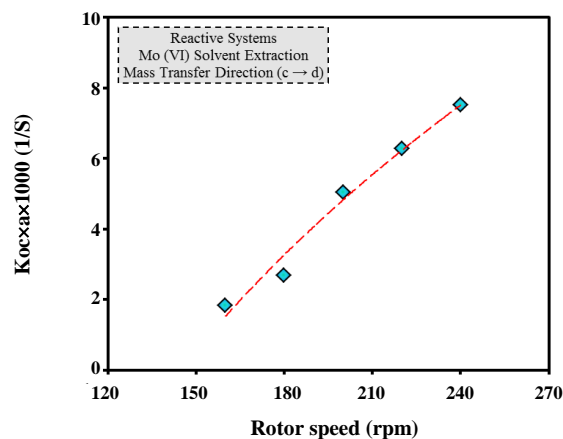


Fig. 3: Effect of rotor speed on volumetric overall mass transfer coefficient at constant flow rates (Experimental conditions: $Q_c=24$ L/h; $Q_d=24$ L/h).

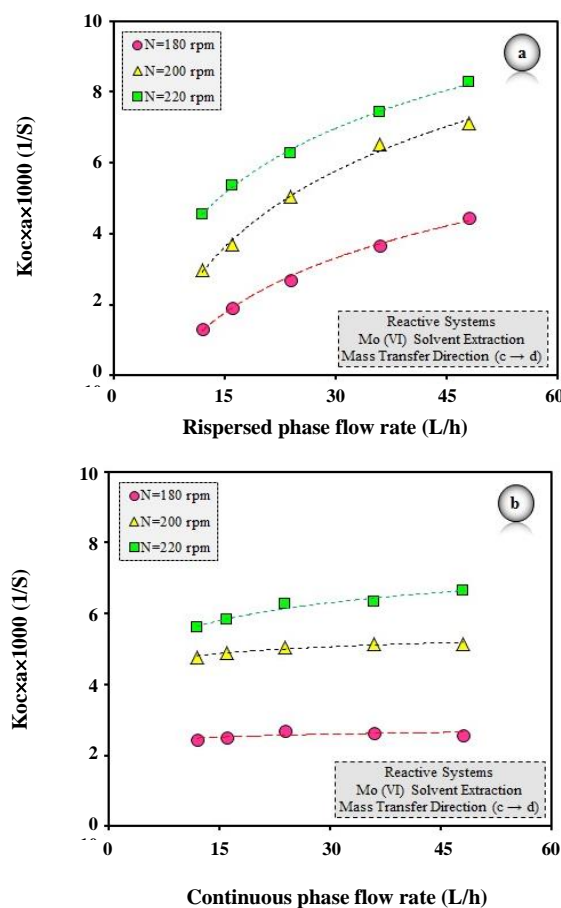


Fig. 4: (a) Effect of dispersed phase flow rate (Experimental conditions: $Q_c=24$ L/h); (b) Effect of continuous phase flow rate (Experimental conditions: $Q_d=24$ L/h) on volumetric overall mass transfer coefficient at various rotor speeds.

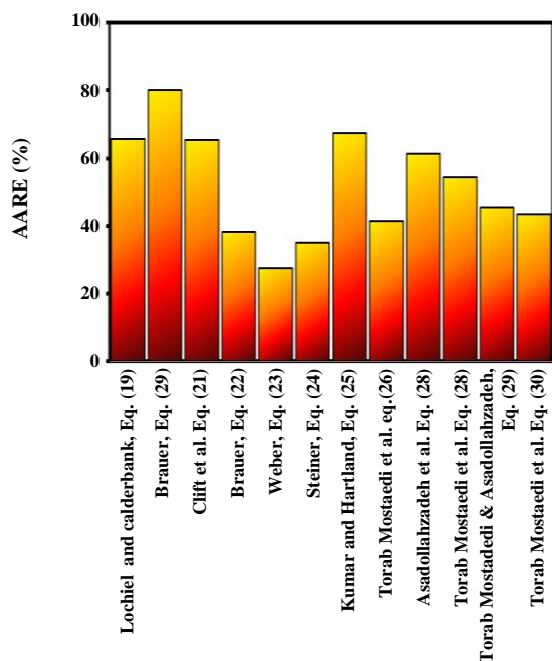


Fig. 5: AARE of Sherwood number resulted from previous models.

As a result of this process, column performance increase with the increment in the continuous phase flow rate.

Comparison of the two data (Experimental results and previous models)

In this section, each experimental data was compared by the predicted correlations and the Absolute Average Relative Errors (AARE) were calculated as follows:

$$AARE = \frac{1}{n} \sum_{i=1}^n \left| \frac{\text{Predicted value} - \text{Experimental value}}{\text{Experimental value}} \right| \quad (23)$$

In this equation, n denotes the number of data points. The values of AARE for previous models as a function of Sherwood numbers from experimental data are presented in Fig.5. Newman [27] proposed the following equation for very small drops that are assumed to be stagnant:

$$k_d = - \left(\frac{d}{6t} \right) \ln \left[\left(\frac{d}{\pi^2} \right) \sum_{n=1}^{\infty} \left(\frac{1}{n^2} \right) \exp \left(- \frac{4D_d \pi^2 n^2 t}{d^2} \right) \right] \quad (24)$$

Kronig and Brink [28] proposed a correlation with consideration of circulating droplets that these circulations are laminar. This equation can be written as follows:

$$k_d = - \left(\frac{d}{6t} \right) \ln \left[\left(\frac{3}{8} \right) \sum_{n=1}^{\infty} B_n^2 \exp \left(- \frac{64 \lambda_n D_d t}{d^2} \right) \right] \quad (25)$$

A comparison between the continuous phase overall mass transfer coefficients and previous works is presented in Fig. 6. The overall mass transfer coefficient of the continuous phase can be determined by using the relation between the overall and the individual mass transfer coefficients as follows:

$$\frac{1}{K_{oc}} = \frac{1}{k_c} + \frac{1}{m k_d} \quad (26)$$

According to the comparison done, the previous models were not able to give a reasonable estimation of the continuous phase overall mass transfer coefficients in the multistage column for extraction of molybdenum. Because of that, the reactive extraction occurs between aqueous and organic phase droplets at the interface. Also, mass transfer performance for this column, with the consideration of the reaction condition was not investigated in the previous correlations.

New proposed correlation for overall mass transfer coefficient

For these reasons, the amounts of errors for available empirical models are not acceptable with reactive extraction of molybdenum for the design of multistage columns. Thus, a novel model is proposed for the prediction of the overall continuous phase mass transfer coefficient for solvent extraction of Mo (VI) from sulfate solution. Consequently, EViews 10 software was used to derive the following equation from 29 experimental data in terms of dimension numbers by using regression analysis:

$$Sh_{oc} = -1.327 + 0.577 Re^{0.724} (1-\phi)^{-2.41} E_o^{-0.196} \quad (27)$$

Where

$$Re = \frac{\rho V_{slip} d_{32}}{\mu} \quad (28)$$

$$V_{slip} = \frac{V_d}{\phi} + \frac{V_c}{1-\phi} \quad (29)$$

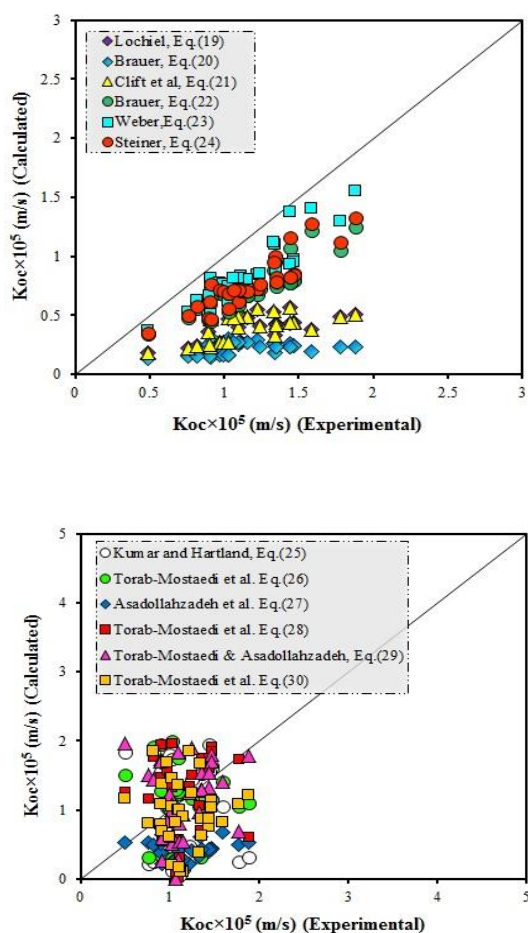


Fig. 6: Comparison of experimental values of volumetric overall mass transfer with previous models.

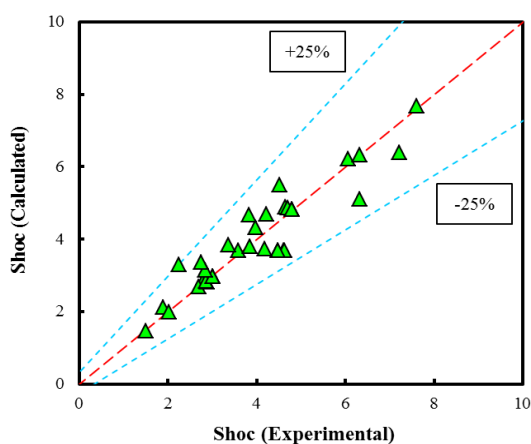


Fig. 7: Comparison of experimental data with calculated Eq. (27).

$$E_{o0} = \frac{g \Lambda \rho d_{32}^2}{\sigma} \quad (30)$$

The above equation predicts the experimental results by the values of AARE of 9.08%. The agreement between experimental data and calculated correlation is portrayed in Fig.7. It can be seen in this figure that the proposed correlation can predict the continuous phase overall mass transfer coefficient with great accuracy for the reaction between molybdenum in the leach solution and a mixture of D₂EHPA and TBP in the kerosene.

Sensitivity Analysis

The effect of changing input variables on the final output of a model could be determined by the sensitivity analysis process. In this research, operating conditions are considered for the prediction of column performance. Sensitivity analysis of affective variables on column performance was performed by using SPSS software with consideration of multivariate regression analysis. Fig.8 is a tornado diagram, which is shown the results of uncertainty analysis of the column performance in terms of input variables by an expected value of 6.86 and according to the sensitivity analysis, the important source of uncertainty in the modeling of column performance is rotor speed, dispersed phase, and continuous phase flow rates, respectively.

CONCLUSIONS

Design methods for multistage column extractors depend on reaction conditions and more experimental data. Molybdenum is extracted into the solvent phase by chemical reaction with a mixture of D₂EHPA and TBP at the drop/aqueous phase interface. The formatted soluble complex then diffuses into the solvent phase. The mass transfer results indicate that the reactive extraction of the Mo (VI) ion with the mixture of extractant reagents is so rapid that the process may be classified as purely diffusion-controlled. At the first step, the values of initial pH and extractants concentration were optimized on the bench scale. In the second step, molybdenum concentration profiles in the aqueous phase for each stage of the mentioned column in terms of operating parameters were obtained. Volumetric overall mass transfer coefficients based on the continuous phase as a function of the Sherwood number for the extraction of

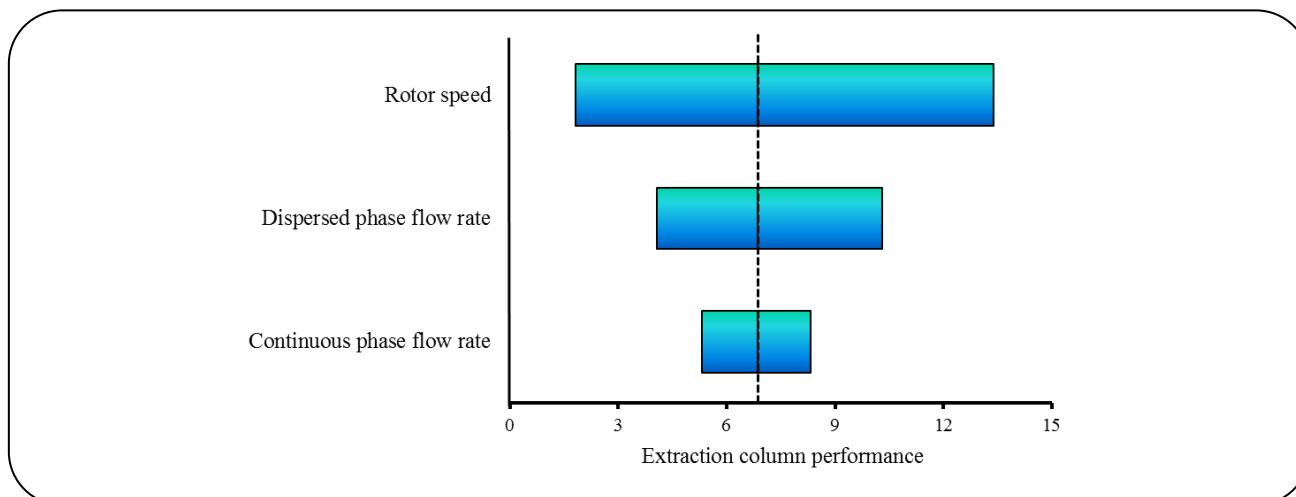


Fig. 8: Sensitivity analysis of the column performance.

molybdenum at various ranges of agitation speeds, continuous and dispersed phase flow rates was studied. In this study, the previous work results for continuous phase overall mass transfer coefficients were compared with the experimental results. The sensitivity analysis indicated that the agitation speed and flow rate of the dispersed phase have more influence on the mass transfer performance. The column performance with the presence of molybdenum reaction increases by increasing the operating parameters including the agitation speeds and inlet phase flow rates. By considering the reactive extraction conditions, a new correlation is presented for continuous phase volumetric overall mass transfer coefficients in a multistage extraction column.

Nomenclature

AARE	Average absolute relative error
a	Interfacial area, m^2/m^3
B_n	n th coefficient in Equation (13)
d_{32}	Sauter mean drop diameter, m
D	Molecular diffusivity, m^2/s
Sh	Sherwood number
E	Axial mixing coefficient, m^2/s
G	Acceleration due to gravity, m/s^2
H	Effective height of the column, m
h_c	Compartment height, m
K	Overall mass transfer coefficient, m/s
m	Distribution ratio
N	Rotor speed, rps
N_{ox}	Number of 'true' transfer unit
Pe	Peclet number

Re	Reynolds number
Eö	Eötvös number
t	Time, s
V_s	Slip velocity, m/s
x	Mass fraction of molybdenum in the continuous phase
x^*	Equilibrium mass fraction
y	Mass fraction of molybdenum in the dispersed phase

Greek letters

ρ_c	Density of continuous phase
ρ_d	Density of dispersed phase
μ_d	Viscosity of dispersed phase
μ_c	Viscosity of continuous phase
φ	Dispersed phase holdup
σ	Interfacial tension

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