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AXIAL DISPERSION MODEL IN PREDICTIVE MASS TRANSFER CORRELATION FOR RANDOM PULSED PACKED COLUMN

Article Highlights

- Mass transfer in a ceramic intalox saddle PPC using axial mixing model was studied
- The effects of Q_c, Q_d and Af on $K_{Oc}a$ for two different systems were investigated
- The comparison of the predicted data with experimental results indicated 22.3% *AARE*

Abstract

The continuous phase volumetric overall mass transfer coefficient in a ceramic intalox saddle pulsed packed extraction column using axial dispersion model was studied for two different liquid-liquid systems containing water/acetone/toluene and water/acetone/n-butyl acetate. The effects of pulsation intensity, continuous and dispersed phase flow rates on mass transfer coefficient were investigated. The experimental results indicated that the mass transfer coefficient was enhanced by increasing pulsation intensity and dispersed and continuous phase flow rates. The utilization of nonlinear least square method provided a new predictive correlation for the continuous phase overall mass transfer coefficient. The developed mass transfer model was validated via the comparison of the modeling data with experimental results with 18.7% average absolute relative error (AARE). Furthermore, an empirical correlation was developed for prediction of the continuous phase overall mass transfer coefficient as the function of the aforementioned operating variables.

Keywords: pulsation intensity, packed extraction column, mass transfer correlation, non-ideal flow model.

The pulsed packed extraction column is commonly utilized for the separation of one component from a liquid mixture *via* a liquid solvent. Currently, the available modeling and experimental data for the performance of extraction column is not enough for industrial design and scale-up. The complexity of mass transfer and fluid mechanics in the pulsed extraction column is the main cause of such shortcomings. Thus, providing viable predictive and experimental data has been the subject of many studies in the last two decades [1-8]. The effect of non-ideal flow has been neglected in most of the studies in which the mass transfer trend was investigated for pulsed packed extraction column. Obviously, determination of overall mass transfer coefficient can lead to erroneous results without consideration of axial dispersion effects [9,10]. Several incentives have been indicated concerning the application of pulsed packed column such as leakage minimization, mass transfer enhancement, elimination of internal mechanical parts and the reduction of packed column height [11-13]. Therefore, many researches are needed in order to provide the neccesary design information for industrial application of liquid-liquid pulsed packed extraction columns in removal of toxic components from a liquid phase mixture such as radioactive or corrosive wastewaters. Many other applications of this specific extraction column have also been considered in petroleum, chemical and biochemical industries due to maintenance simplicity. The essential research task in this regard is the study of important effective variables such as pulsation intensity and flow rates on the column design. One of the most important research chal-

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lenges facing the investigators is the development of viable predictive correlation to determine the overall mass transfer coefficient in the design of column height. Experimental concentration profiles are usually used to obtain the mass transfer coefficient. Several models including tubular plug flow, back flow model, axial dispersion model and forward mixing model have been previously developed in order to reduce the cost of experimental studies [14-16]. In order to describe the mass transfer performance in extraction columns, different models, like the plug flow model, are developed. The overall flow pattern in extraction columns, however, is complex and based on experience it became evident that the description of mass transfer in column extractors with the plug flow was oversimplified. Therefore, in general, axial dispersion (back-mixing) of one or both phases needs to be included in the description, since it shows a major influence on the mass transfer performance. The type of packing is considered to be another important variable in the design of extraction column. Even though some studies including pulsed packed columns with random packing such as stainless steel supermini ring (SMR) [16], stainless steel Raschig ring [17] and ceramic Raschig ring [18] have been carried out, but the mass transfer reliable data for random pulsed packed column using ceramic intalox saddle packing is still lacking. Therefore, the development of a reliable mass transfer correlation is needed to predict the mass transfer behavior for the design of random pulsed packed extraction columns using ceramic intalox saddle packing.

The aim of this work was to determine both mass transfer and axial mixing parameters simultaneously from solute concentration profiles. For this purpose, a pilot scale pulsed packed column with intalox saddle packing was employed. The concentration profile of the solute along the column was measured and used to estimate the mass transfer and axial mixing parameters in terms of the axial diffusion model. The effects of important operating variables (pulsation intensity, dispersed and continuous phase flow rates) were experimentally investigated on the volumetric mass transfer coefficient. Finally, an optimization method such as nonlinear least square was used to develop an empirical correlation for the prediction of the volumetric overall continuous phase mass transfer coefficient as a function of dimensionless variables.

EXPERIMENTAL

Figure 1 shows the experimental apparatus used in this investigation. The stainless steel column with internal diameter of 0.1 m and height of 2 m was packed with 1/2 inch ceramic intalox saddle (bed voidage = 0.7). The column was pulsed by blowing air at the required amplitude and frequency into pulse leg. The air pressure was controlled by a regulator to provide pulses of the required amplitude in the column while the frequency of the pulses was controlled by using two solenoid valves. The following correlation was used for calculation of the pulse amplitude in the pulsed packed column:

$$A_1 S_1 = A_2 S_2 \varepsilon$$

where A_1 is the pulse amplitude in the air leg, A_2 is the pulse amplitude in the pulsed packed column, S_1 is the cross-sectional area of the air leg, S_2 is the cross-

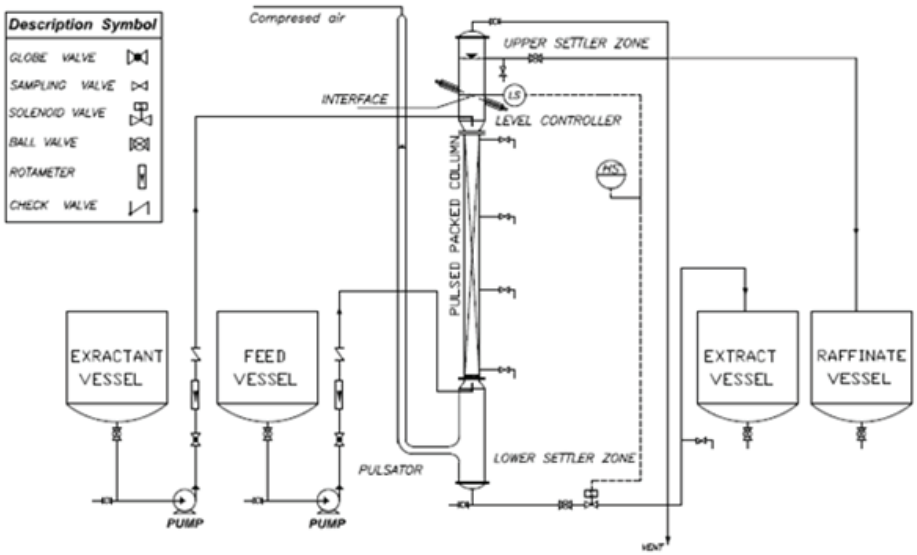

Figure 1. Schematic diagram of the structured pulsed packed extraction column [17].

-sectional area of the pulsed packed column, and ε is the packing porosity.

The column inlet and outlet were connected to four tanks of 180 L capacity. The flow rates of continuous and dispersed phases were measured by two rotameters. The interface location of two phases at the top of the column was automatically controlled via an optical sensor.

Two liquid systems of water/acetone/*n*-butyl acetate (medium interfacial tension) and water/acetone/toluene (high interfacial tension) were studied in this work. These two liquid systems were recommended by the European federation of chemical engineering (E.F.C.E.) as official standard testing for extraction studies [19]. The dilute solutions with 3.5 wt.% acetone in dispersed phase were used in all experiments. Distilled water, *n*-butyl acetate and toluene of 99.5 wt.% purity were used as continuous and dispersed phases, respectively. The physical properties of two liquid–liquid systems are shown in Table 1. The physical properties of two liquid systems in the continuous and dispersed phases were estimated by using the average values of acetone concentration in the inlet and outlet streams.

Table 1. Physical properties of liquid systems at 20 °C [19]

Physical property	Water/acetone/toluene	Water/acetone/*n*-butyl acetate
ρ_c / kg m^{-3}	994.4-995.7	994.3-995.8
ρ_d / kg m^{-3}	864.4-865.2	879.6-881.4
μ_c / mPa s	1.059-1.075	1.075-1.088
μ_d / mPa s	0.574-0.584	0.723-0.738
σ / mN m^{-1}	27.5-30.1	12.4-13.2

First, the column was filled with distilled water and the dispersed phase was introduced. Then the pulsation amplitude and frequency were fixed to the suitable values. The proper height of interface location was controlled and after the period of one hour the system reached the steady state condition. Eight sampling ports were situated at equal distances along the 2 m effective height of the column. Samples of the phases were taken from each port for concentration profile measurements under each given operation condition. The acetone concentration in the samples was measured by a UV/Vis spectrophotometer. The holdup measurements were carried out via shut down technique. The shutdown technique included closing the inlet and outlet valves and allowing the dispersed phase to coalesce to the interface at top of the column. A period of 10–15 min was allowed for the dispersed phase settlement. The drainage valve was used for the collection and measurement of dispersed phase volume in order to obtain the holdup. This technique was repeated until the interface was at its initial location. The flooding was avoided via selection of proper operating conditions.

Modeling

The backflow and the axial diffusion models are the most important practical approaches for considering the influence of the axial mixing on the mass transfer performance of an extraction column. In the diffusion model, applicable to differential-type extractors, non-ideality is expressed in terms of an axial dispersion coefficient, E. In contrast, the backflow model is basically a stage-wise model, in which the total mixing is assumed within each stage, and the deviation from plug flow is presented by a back-mixing coefficient, α, between adjacent stages. The following differential mass balance equations at steady state were obtained based on the actual physical conditions of this study:

$$-\frac{1}{Pe_c}\frac{\mathrm{d}^2x}{\mathrm{d}Z^2}+\frac{\mathrm{d}x}{\mathrm{d}Z}+N_{Oc}(x-x^*)=0 \quad (1)$$

$$-\frac{1}{Pe_d}\frac{\mathrm{d}^2y}{\mathrm{d}Z^2}-\frac{\mathrm{d}y}{\mathrm{d}Z}-\frac{\rho_c Q_c}{\rho_d Q_d}N_{Oc}(x-x^*)=0 \quad (2)$$

where:

$$Pe_c=\frac{Q_c H}{s\varepsilon(1-\varphi)E_c},\ Pe_d=\frac{Q_d H}{s\varepsilon\varphi E_d},$$

$$Z=\frac{h}{H}\ \text{and}\ N_{Oc}=\frac{Hs\varepsilon(1-\varphi)K_{Oc}a}{Q_c}$$

The applied boundary conditions are:

$$Z=0:\ \frac{\mathrm{d}y}{\mathrm{d}Z}=0\ \text{and}\ -\frac{\mathrm{d}x}{\mathrm{d}Z}=Pe_c(x_0-x_{Z=0^+})$$

$$Z=1:\ \frac{\mathrm{d}x}{\mathrm{d}Z}=0\ \text{and}\ \frac{\mathrm{d}y}{\mathrm{d}Z}=Pe_d(y_1-y_{Z=1^-})$$

The measured concentration profiles were used to evaluate the parameters Pe_c, Pe_d and N_{Oc}. The coupled differential Eqs. (1) and (2) were simultaneously solved by the modified 4th-order Runge-Kutta numerical technique along with genetic algorithm to obtain the three parameters.

RESULTS AND DISCUSSION

One of the main effective operating variables on the mass transfer coefficient in the pulsed packed column is pulsation intensity. The behavior of volumetric overall mass transfer coefficient is shown in Figure 2 as a function of pulsation intensity. As exp-

ected, a higher pulsation intensity results in a lower value of the average droplet diameter and a higher value of the dispersed phase holdup. The interfacial area increases with both effects. However, the reduction of droplet size results in a reduction of internal circulation and turbulence in the droplets and, consequently, the overall mass transfer coefficient decreases with an increase in pulsation intensity. At low and medium values of the pulsation intensity, the effect of the interfacial area is larger than that of the overall mass transfer coefficient and, consequently, the overall column performance increases with an increase in pulsation intensity. At high values of the pulsation intensity, however, the overall mass transfer starts to fall significantly with the formation of rigid droplets. The column performance is also largely affected by the axial mixing at high values of the pulsation intensity. On this basis, the effect of pulsation intensity on the volumetric overall mass transfer becomes limited at high pulsation intensities.

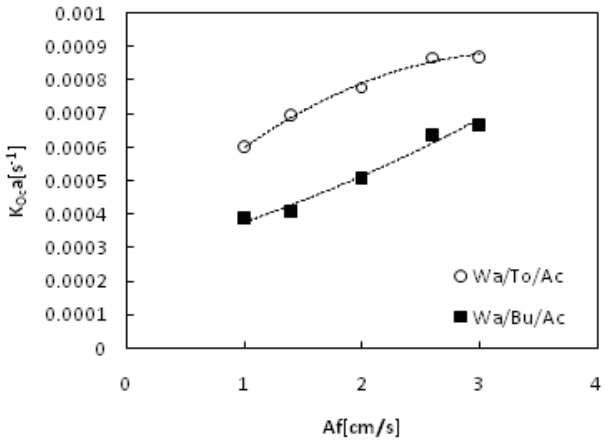

Figure 2. Effect of pulsation intensity on volumetric overall mass transfer coefficient at constant flow rates (Q_d = 17 l/h, Q_c = 14 l/h).

Another important effective operating variable on the mass transfer coefficient in the pulsed packed column is dispersed phase flow rate. The trend of volumetric overall mass transfer coefficient as a function of dispersed phase flow rate is shown in Figure 3. It is observed from the experimental data that the volumetric overall mass transfer coefficient is improved by increasing dispersed phase flow rates of both systems. In the observed phenomenon two countereffect variables of holdup and droplet size create the trend of volumetric overall mass transfer coefficient. Other researchers [20,21] have found that higher dispersed phase flow rate increases both holdup and droplet size. The obtained results of Figure 2 indicated that higher holdup and lower droplet size enhances the mass transfer coefficient. However the simultaneous higher holdup and droplet size are obtained in Figure 3 in which two countereffect phenomena of positive effect of higher holdup and negative effect of higher droplet size on mass transfer coefficient are observed. However, as it is observed in Figure 3, the overall effect of holdup and droplet size has caused an increase in the interfacial area and mass transfer coefficient. This observation may be explained by the effect of holdup on interfacial area, which is more significant than droplet size. Therefore, higher dispersed phase flow rate improves the interfacial area and consequently the mass transfer coefficient.

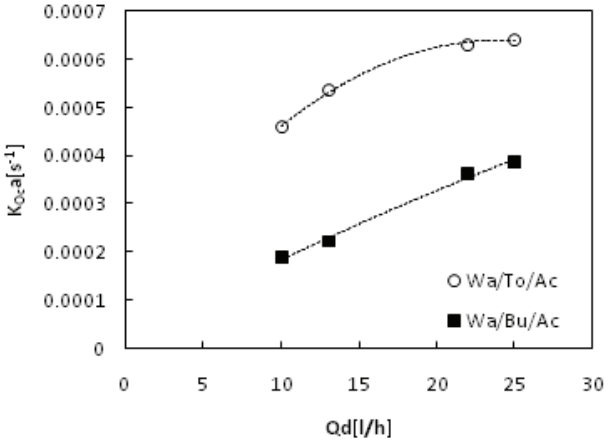

Figure 3. Effect of dispersed phase flow rate on volumetric overall mass transfer coefficient at constant continuous phase flow rate and pulsation intensity (Q_c = 14 l/h, A_f = 2 cm/s).

Figure 4 shows the effect of continuous phase flow rate on volumetric overall mass transfer coefficient. The higher continuous phase flow rate enhances the overall mass transfer coefficient for both systems. The observed mass transfer behavior in Figure 4 is very similar to Figure 3 trend in terms of two countereffect variables (holdup and droplet size). The aforementioned similarity exists in which the positive effect of higher holdup is more significant than the negative effect of higher holdup and consequently it is concluded that higher continuous phase flow rate enhances the interfacial area and mass transfer coefficient. As shown in Figure 4, higher mass transfer coefficient is obtained for the system of water/acetone/toluene with respect to water/acetone/*n*-butyl acetate. All the obtained experimental data are shown in Table 2. These experimental data are the average results of triplicate runs with standard deviation of ±1.7%.

The development of a viable mass transfer correlation for the design of random pulsed packed extraction column was the main objective of this study.

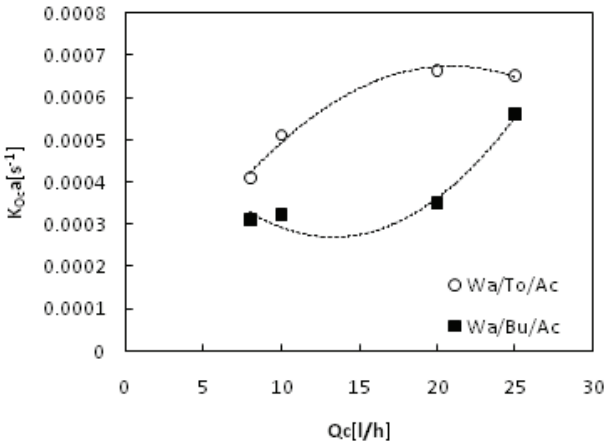

Figure 4. Effect of continuous phase flow rate on volumetric overall mass transfer coefficient at constant dispersed phase flow rate and pulsation intensity (Q_d = 17 l/h, A_f = 2 cm/s).

The numerical investigation of the experimental data of continuous phase overall mass transfer coefficient as a function of dimensionless variables led to the development of a general equation for the prediction of $K_{Oc}a$ at different operating conditions. The obtained correlation is economically a very valuable engineering tool in which high experimental expenses can be avoided in case the optimum design of extraction column is the ultimate aim of the study. The development of a correlation for the mass transfer coefficient was acomplished *via* the application of nonlinear least square method:

The above correlation was obtained by using the experimental data obtained in this study and that of Safari *et al*. [17]. The authentication of this correlation was carried out by the comparison of predicted data with experimental results. Thus the correlation of Eq. (3) was validated with the average absolute relaive error (*AARE*) of 18.7%. Figures 5 and 6 show an acceptable compatibility between experimental and predicted data. The process engineers whose objective is to optimize the operating conditions in the design of random pulsed packed extraction column using ceramic intalox saddle may use the reported correlation of this study in order to minimize the experimental expenses.

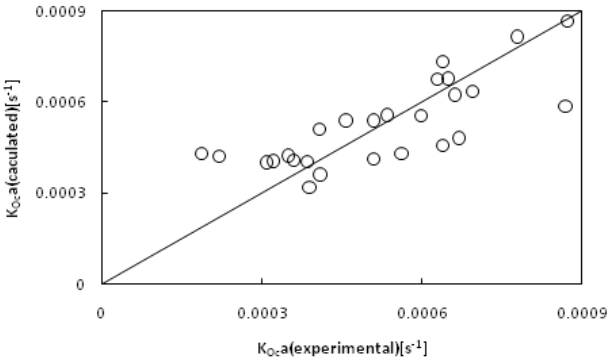

Figure 5. Comparison of experimental data with calculated values.

$$K_{Oc}a = 7.78\times10^{-5}\left[\frac{(A_f)^4\rho_c}{g\sigma}\right]^{-0.028}\left[1+\frac{V_c}{V_d}\right]^{-0.815}\left[\frac{\varepsilon^3 g}{a_p}\frac{\Delta\rho}{\rho_c}\right]^{-0.591}\left[\frac{\mu_c{}^4 g}{\Delta\rho\sigma^3}\right]^{0.171}\left[\frac{\mu_c}{\mu_d}\right]^{-2.511} \quad (3)$$

Table 2. Experimental values of axial dispersion model for ceramic intalox saddle packing

Ceramic intalox saddle packing			Water/acetone/toluene			Water/acetone/*n*-butyl acetate		
A_f / cm s^{-1}	Q_c / l h^{-1}	Q_d / l h^{-1}	$K_{Oc}a$ / 10^{-4} s^{-1}	E_c / 10^{-2} m^2 s^{-1}	E_d / 10^{-3} m^2 s^{-1}	$K_{Oc}a$ / 10^{-4} s^{-1}	E_c / 10^{-2} m^2 s^{-1}	E_d / 10^{-2} m^2 s^{-1}
2	14	10	4.58	2.11	4.96	1.87	1.21	1.01
2	14	13	5.35	2.02	3.87	2.20	1.63	0.824
2	14	17	8.70	2.73	3.8	5.10	2.41	1.41
2	14	22	6.30	1.48	6.14	3.60	3.35	3.00
2	14	25	6.40	1.34	2.94	3.86	2.64	1.38
1	14	17	6.00	3.85	1.69	3.90	2.80	1.4
1.4	14	17	6.96	1.05E	2.44	4.10	1.17	0.653
2.6	14	17	7.80	1.37	1.66	6.40	1.39	0.948
3	14	17	8.73	0.345	2.47	6.70	1.85	1.83
2	8	17	4.09	2.80	1.69	3.10	2.91	0.820
2	10	17	5.10	2.27	1.37	3.22	1.22	0.450
2	20	17	6.63	0.332	2.88	3.50	2.1	1.73
2	25	17	6.50	1.80	5.59	5.62	1.94	1.47

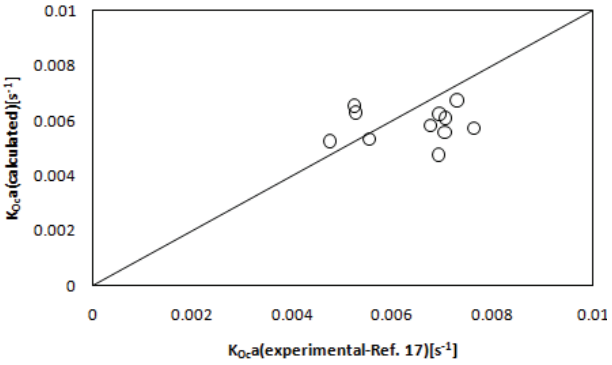

Figure 6. Comparison of calculated values and experimental data obtained from Safari et al. [17].

CONCLUSIONS

The lack of experimental data for the design of random pulsed packed extraction column using ceramic intalox saddle packing is considered to be the major research challenge facing the investigators. Therefore, an empirical correlation was developed in this study for the prediction of overall mass transfer coefficient. This correlation was validated in contrast to experimental data with 18.7% *AARE*. The effects of operating variables such as continuous and dispersed phase flow rates and pulsation intensity on volumetric overall mass transfer coefficient were investigated via the correlative parametric analysis. Both the obtained experimental and modeling data showed the enhancement of volumetric overall mass transfer coefficient by increasing pulsation intensity, dispersed and continuous phase flow rates. The proposed correlation may be used by process engineer for the optimal design of random pulsed packed extraction column (determination of mass transfer coefficient and column height) *via* parametric analysis with lower experimental efforts and costs.

Nomenclature

- a interfacial area (m^2/m^3)
- a_p surface area of packing (m^2/m^3)
- A pulse amplitude (m)
- E axial dispersion coefficient (m^2/s)
- f frequency (s^{-1})
- g acceleration due to gravity (9.81 m/s^2)
- H effective height of the column (m)
- K overall mass transfer coefficient (m/s)
- N_{Oc} Number of “true” transfer unit
- Pe Peclet number
- Q flow rate of the continuous or dispersed phase (m^3/s)
- x mass fraction of acetone in continuous phase
- y mass fraction of acetone in dispersed phase

Greek symbols

- α backflow coefficient
- ρ density (kg/m^3)
- $\Delta\rho$ density difference between phases (kg/m^3)
- μ viscosity (Pa s)
- ε packing porosity
- σ interfacial tension between two phases (N/m)

Subscripts

- c continuous phase
- d dispersed phase
- O overall value

Superscripts

- * equilibrium value

Acknowledgments

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NAUČNI RAD

KORELACIJA ZA PRENOS MASE U PULZACIONOJ PAKOVANOJ KOLONI NA OSNOVU MODELA AKSIJALNE DISPERZIJE

Zapreminski koeficijent prenosa mase u kontinualnoj fazi pulzacionoj ekstrakcionoj koloni punjenoj sa keramičkim sedlima Intalox je istraživan u dva sistema tečno-tečno (voda--aceton-toluen i voda-aceton-n-butil acetat) koristeći model aksijalne disperzije. Istraživani su uticaji intenziteta pulzacije i protoka kontinualne i dispergovane faze na koeficijent prenosa mase. Eksperimentalni rezultati su ukazali da se koeficijent prenosa mase povećava sa povećanjem intenziteta pulzacije i protoka kontinualne i dispergovane faze. Korišćenjem nelinearne metode najmanjih kvadrata dobijena je nova korelacija za zapreminski koeficijent prenosa mase u kontinualnoj fazi. Razvijeni model prenosa mase je potvrđen poređenjem izračunatih sa eksperimentalnim podacima sa srednjom relativnom apsolutnom greškom 18,7%. Takođe, razvijena je empirijska korelacija za izračunavanje zapreminskog koeficijenta prenosa mase u kontinualnoj fazi kao funkcija gore navedenih operativnih faktora.

Ključne reči: intenzitet pulzacije, ekstrakciona pakovana kolona, korelacija za prenos mase, model neidealnog strujanja.