

Recycling and Extraction of Zinc Ions in Disc-Donut Column Considering Forward Mixing Mass Transfer, Chemical Reaction and Effects of Pulsed and non-Pulsed Condition

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Abstract

The current study focuses on the recovery of zinc ions by solvent extraction in the pulsed contactor. The Zn(II) ions from chloride solution were extracted into the organic phase containing D2EHPA extractant. The resulting data were characterized for the relative amount of (a) pulsed and no-pulsed condition; and (b) flow rate of both phases. Based on the mass balance equations for the column performance description, numerical computations of mass transfer in a disc-donut column were conducted and validated the experimental data for zinc extraction. Four different models, such as plug flow, backflow, axial dispersion, and forward mixing were evaluated in this study. The results showed that the intensification of the process with the pulsed condition increased and achieved higher mass transfer rates. The forward mixing model findings based on the curve fitting approach validated well with the experimental data. The results showed that an increase in pulsation intensity, as well as the phase flow rates, have a positive impact on the performance of the extractor, whereas the enhancement of flow rate led to the reduction of the described model parameters for adverse phase.

1. Introduction

The find of many applications in various extraction processes such as chemical, and petroleum industries is one of the main advantages of pulsed solvent extraction columns^{1,2}. The main specific characteristic of this column is the utilization of pulsing condition for the intensified process, it is possible not only to increase its efficiency; but also to obtain compactness of equipment, to diminish maintenance efforts, and to improve the quality of extracted products³⁻⁵. However, these columns have grown well in the extraction stages from a primary source such as the leaching aqueous solution⁶⁻⁹. But, its path to secondary resource development and recovery has slowed in industries. Recovery of toxic metals from wastewaters is one of the needs of environmental refinement done by various processes¹⁰⁻¹⁴. The use of the solvent extraction process has expanded with environmentally friendly solvents^{15,16}. But the solvent type alone is not enough to achieve the desirability, and the selected equipment must have a good performance¹⁷.

Zinc is one of the heavy metals with adverse effects on water resources, soils, vegetables, and crops¹⁸. In some areas, this pollution is hazardous to human health¹⁹. The extraction and separation of which is critical from an environmental point of view. Jafari and co-workers' studies showed that zinc can be extracted from a synthetic solution with a high extraction percentage ~94% with D2EHPA extractant²⁰. The zinc recovery process from effluents such as spent chloride brass pickle liquors²¹, and zinc-plating mud²² has been investigated in the literature. Extraction species depend on different acidic conditions and type of extractant (D2EHPA, Cyanex301, Cyanex272, LIX 841C), and ionic liquids. The zinc recovery from the complex ore resources was investigated by a mixture of TOPO and Aliquate336 in the extraction stage and the ammoniacal chloride solution in the stripping stage.

The synergistic effects in the extraction stage and the antagonistic effects in the stripping stage are the advantages of using a mixture of extractants²³. The melting effluents are rich sources of heavy metals such as zinc, copper, nickel, and cadmium. Studies on the recovery of these ions with 2-hydroxy-5-nonylacetophenone oxime and Cyanex272 have shown that the solvent extraction process can play an influential role in their recovery and reduce environmental impact²⁴. The technology for extracting zinc from molten effluents has been extensively developed, but the issue of removing other ions has received little research. Song and co-workers reported the extraction of cobalt ions from these effluents for recovery due to the value of cobalt in various industries²⁵.

In addition to experimental work, it is possible to describe the process based on different models and mass transfer coefficients²⁶⁻³⁰. The mass balance induced by rising droplets or continuous phase can be crucial in the numerical modeling of the extraction column³¹. Using the mathematical models of the extraction process, it is possible to deduce the dependences of the mass transfer coefficient on the operating condition, and the dependence of the mass transfer coefficient on the physical properties^{32,33}. The evaluation of concentration profile curves is of paramount importance, especially for establishing the optimum operating conditions, determining parameters used for scale-up and process design. Mathematical modeling of the extraction column is fundamental due to the economic potential³⁴. It is important to develop models for the process when the extraction operations should be optimized for commercial applications. However, such predictions require the establishment of a model predicting column performance^{35,36}. The plug flow model (PFM) is a common model with simple assumptions in determining mass transfer coefficients. The back-flow model (BFM) and axial dispersion model (ADM) are described by determining the specific coefficients for the deviation from the ideal state³¹. The forward mixing model (FMM) is more superior to the three mentioned models. Applying droplet size distribution in the model helps to bring the extraction conditions closer to the real conditions^{37,38}. Mass balance equations illustrate these models, these equations are described in Table 1, and a schematic of the mass transfer equilibrium based on the PFM, BFM, ADM, and FMM models is given in Fig.1.

Several physical systems have been investigated in the pilot scale pulsed disc-donut column in the literature³⁹⁻⁴², but limited studies have been reported with this column to extract metal ions associated with the reaction condition^{4,43-45}. One of the new findings and innovations of this paper are the study of different mass transfer models in the extraction of zinc ions. So far, no studies have been reported in this field, and this finding is significant in the design of this pulsed column according to the standard chemical system for zinc extraction.

2. Experimental

2.1. Materials

A series of continuous experiments were carried out for the removal of zinc ions from an aqueous solution at room temperature (25 ± 1.5 °C). The extraction process was carried out by preparing two aqueous and organic solutions. The organic solution was obtained from the dissolution of an extractant from Aldrich company, tri-di(2-ethylhexyl) phosphoric acid/ D2EHPA (0.1 M) in kerosene. The aqueous solution was obtained from the dissolution of zinc chloride salt (Merck Company, with 99% purity) in distilled water with a concentration equivalent to 600 ppm at pH~6. The details of physical properties are shown in Table 2.

2.2. Pilot Plant Column

As shown in Fig.2, the experiments have been carried out in a 0.076 m diameter disc-donut column with a 0.74 m long glass extraction section. The average fractional free area has been maintained at 23.5%. The arrangement of discs and donuts inside the column was made so that 30 discs (0.067 cm O.D) and donuts (0.036 cm I.D) were placed one by one inside the active part.

The aqueous and organic phases are pumped into the column from two stainless steel tanks and the control of flows with two rotameters. The photos of droplets in the column were recorded by a video camera, which was installed at the center of the column section. The evaluation of drop sizes was obtained by the values of Sauter mean drop size (d_{32}) for each run. The composition of the aqueous solution in the feed and residual was determined by using UV-spectrophotometer (UNICO model). The shutdown method was used to measure of the value of holdup (φ) from the

separation of the organic phase in the interface location. The interfacial area for reaction between zinc ions and extractants in the organic phase was obtained by the following equation:

$$a = \frac{6\phi}{d_{32}} \quad (1)$$

The mass transfer coefficient for the organic-side phase was calculated using four models (PFM, BFM, ADM, and FMM models) in Table 1.

3. Results And Discussion

The zinc extraction process in the pulsed disc-donut column was continuously investigated in two steps. In the first step, the pulse conditions inside this column were not activated, and only the effects of phase flow rates were investigated in this column. In the second step, pulse conditions were applied inside the column. Different intensities of the pulse were investigated, and the variation in the phase flow rates at different pulse intensities was studied in this column. Four different types of models (PFM, BFM, ADM, FMM models) were evaluated in the performance recognition of this column, according to Table 1. The model analysis result showed that the column behavior using the FMM model is closer to the real values. The errors from the experimental data are much less with the FMM model results. The variation in X-value and Y-value errors with average absolute relative error (AARE) are presented in Table 3, which indicates that the pulsed disc-donut column is more consistent with the forward mixing model (10.74% for X-value, 13.28% for Y-value).

The main reason for reducing errors is related to the distribution of droplets inside the column, which applies the droplet distribution in the forward model and avoids simplifying assumptions that bring the column's behavior closer to the actual value. Table 3 shows the plug flow model with simple assumptions is associated with more errors. The back-flow and axial mixing models are related to significant errors that are acceptable to use in the study of column behavior. The back-mixing and axial dispersion coefficients defined in these models help the system approach the ideal conditions.

Figs.3, and 4 showed the examples of concentration profiles measured by the experimental method under different operating conditions and the concentration profiles predicted by the forward mixing model to extract zinc ions.

The results in this diagram showed that without applying a pulse inside the column, a large mass transfer driving force is established in the column that cannot reach the desired mass transfer and efficiency (see Fig.3a). But when the pulse is applied inside the column, the column conditions lead to better mass transfer and intensification of the process, which results in a reduction in the driving force of the mass transfer is shown in Fig.3b.

Increasing the continuous phase flow rates in Figs. 3b, 4a, and 4b, increasing the phase flow ratio (Q_c/Q_d) causes a change in the mass transfer's driving force. The decrease in driving force indicates that the phase change curves of the phases are getting closer to each other. The system is closer to ideal conditions and optimal extraction. Therefore, creating pulse conditions and increasing the inlet aqueous phase flow rates intensify the process and reach equilibrium concentrations. Another finding in these two graphs is the correspondence between the forward model and the laboratory data, which shows that the descriptive model can be used with high accuracy in predicting the results.

The changes in back-flow coefficients for aqueous and organic-side phases are shown in Figs. 5, and 6 by examining the effect of operating parameters. These figures showed that increasing the pulse intensity inside the column leads to increasing the back-flow coefficients. Further stress due to the pulse causes the increase in collision frequency of the droplet with the column's internal components. This factor increases the back-flow coefficients (α , and β).

The effect of increasing the continuous phase flow rate on the α , and β coefficients showed that increasing of Q_c increases the frequency of collisions with the internal parts, which increases the aqueous-side phase coefficients (α). But it has an adverse effect on the organic side-phase coefficients (β), because the higher continuous flow rate helps carry droplets. Increasing the dispersed phase flow rate also reduces α and increases β coefficients. Because with the presence of more droplets inside the column, the coalescence rate increases, which has the opposite effect on the α coefficient, but also helps increase the amount of collisions at a constant volume for a higher number of droplets and increase the β coefficient.

The results showed that the lack of pulse inside the column is associated with an increase in these coefficients and increases the deflection conditions of the plug flow. However, the use of pulses inside the column causes the necessary stresses to provide a better flow, which intensifies the column's process and better performance by describing the mass transfer coefficients.

The results in Fig.7 described that increasing the pulse intensity at constant phase flow rates increases the axial dispersion coefficient for the aqueous side-phase. With increasing turbulence in the system, the droplet aggregation and the vortex formation increase in a continuous phase. Therefore, the higher values for E_c are observed with an increment of A_f .

The variations in the dispersed and continuous phase flow on the axial dispersion coefficient of the continuous phase are shown in Figs.8 and 9. The increase in Q_c reveals a positive effect, and the increase in Q_d reveals a negative effect on the E_c . The decrement impact is due to uniform conditions with a reduction in collision frequency of droplets with the internal components. The incremental impact is due to the presence of droplets and participation in more turbulence through the column. These coefficients are much larger in non-pulsed conditions, where the deviations from the ideal current are much larger, and the driving force for mass transfer is high, and reaching the perfect conditions is not achieved by changing the phase flow rate alone.

The axial dispersion coefficients for the organic-side phase also change similar to continuous axial dispersion coefficients with operating parameters, as shown in Fig.10. The increase of this parameter is observed by the pulse intensity in Fig.10a and Fig.10c. Its decrease is observed by Q_c in Fig.10b. Changes in the droplet's behavior inside the column under operating parameters help to significantly affect these coefficients, and the variation in flow deflection becomes apparent.

Mass transfer coefficients were evaluated by the forward mixing model, the results of which are shown in Fig.11. The observation indicated that as the pulse intensity increases, the droplet diameter decreases, and the dispersed phase holdup increases, increasing the effective interfacial area for the reaction. The reduction in mass transfer occurs due to the droplet behavior approaching the rigid state and reducing droplet sizes. But the conditions with increasing the required area for the reaction help provide larger volumetric overall mass transfer coefficients. Increasing the phases' flow rate helps to shift the mass transfer rates within the droplets toward the internal circulation, as larger droplets are formed when the coalescence rate increases. In addition to increasing the required interfacial area for the reaction, the mass transfer coefficients for the organic-side phase increase along this column. The results in these figures showed that the pulse conditions are an essential factor in intensifying the column process and providing the

requirements for mass transfer. Without this factor inside the column, very small mass transfer coefficients are obtained, indicating the column's poor performance in non-pulsed condition.

The results of the percentage of zinc extraction with Q_c and Q_d are shown in Fig.12. The results illustrated that the zinc ions' extraction is a function of the inlet phase rates and pulsed condition. Increasing the flow rate of the phases helps to establish the higher mass transfer rate in the pulsed disc-donut column and the result is an increase in the percentage of extraction of zinc ions from the aqueous to the organic phase. Intensification of the process by applying pulses in the column and performing the extraction reaction and increasing the efficiency are also observed in these diagrams.

4. Conclusion

In this study, the extraction of zinc ions from aqueous to the organic solution was investigated as a standard reaction system in the pulsed disc-donut column. The operating conditions of the pilot plant column were checked by changing the flow rate of the inlet phases and applying pulses inside the column. The extraction process description was investigated by examining the mass transfer models (plug flow, back-flow, axial dispersion, and forward mixing models). The results of variation in the operating parameters showed that the optimal extraction conditions are obtained at equal flow rates from the organic and aqueous phases ($Q_c=Q_d=28$ L/h) and the maximum pulse intensity ($A_f = 4.4$ cm/s) in which the volumetric mass transfer coefficient is equal to 0.00639 1/s $K_{od} \times a$, and the extraction efficiency is equivalent to 93.34%. The models' evaluation showed that the error of the forward mixing model is less than other models in predicting the results. Deformation of the concentration profile inside the column by examining the effect of pulse intensity (A_f) and flow rate ratio (Q_c/Q_d) showed that the reduction in the driving force and achieving equilibrium are the effects of the pulsed condition and the increment in continuous phase flow rate. These parameters help to exchange and transfer more ions between both phases with a higher extraction efficiency.

Nomenclature

Af	Pulse intensity (cm/s)
a	Specific surface area per unit volume ($6\phi/d_{32}$) (1/m)
d	Drop diameter (m or mm)
d_{32}	Sauter mean drop diameter (mm)
d_{43}	Mean diameter (m)
E	Axial dispersion coefficient (cm^2/s)
f	Volumetric drops size distribution (-)
g	Dynamic drop size distribution (-)
H	Height of column (m)
K	Overall mass transfer coefficient (m/s)
n	Number of drops (-)
N_d	Number of drop classes (-)
Q	Volume flow rate (L/h)
S	Cross-section area of the column (m^2)
t	Residence time of drops (s)
u	Drop velocity (m/s)
U	Superficial velocity (Q/S) (m/s)
U_{slip}	Slip velocity ($U_c/(1-\phi)+U_d/\phi$) (m/s)
V	Volume of phases (m^3)
x	Mass fraction of solute in the continuous phase (-)
y	Mass fraction of solute in the dispersed phase (-)
z	Height variable (m)
Dimensionless symbols	
NTU	Number of transfer unit ($K_{od}aH/U_d$)
P	Slope of the concentration equilibrium equation ($y=p^*x+q$)
Pe	Peclet number (HU/E)
q	Constant of the concentration equilibrium equation ($y=p^*x+q$)
X	Continuous phase concentration ($(x-x^*_{out})/(x_{in}-x^*_{out})$)
Y	Dispersed phase concentration ($(y-y_{in})/(y^*_{out}-y_{in})$)
Z	Height ($Z=z/H$)

Ω	Constant ($pU_d\rho_d/U_c\rho_c$)
Greek symbols	
μ	Viscosity (kg/m.s)
ρ	Density (kg/m ³)
σ	Interfacial tension between two phases (N/m)
φ	Dispersed phase holdup (-)
Subscripts/Superscripts	
*	Equilibrium concentration
c	Continuous phase
d	Dispersed phase
i	Drop class
in	Inlet to the column
j	Cross-section
out	Outlet from the column

Abbreviations

AARE	Average Absolute Relative Error
ADM	Axial Dispersion Model
BFM	Back Flow Model
PFM	Plug Flow Model
FMM	Forward Mixing Model

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Tables

Due to technical limitations, Table1 is only available as a download in the Supplemental Files section.

Table 2. Characteristics of chemical system used in the pilot-plant column

Phases		Solute	ρ_c	ρ_d	μ_c	μ_d	σ
Continuous	Dispersed		(kg/m ³)	(kg/m ³)	(10 ⁻³ kg/m.s)	(10 ⁻³ kg/m.s)	(10 ⁻³ N/m)
Aqueous nitrate solution of Zn(II)	D2EHPA extractant diluted in kerosene	Zinc ions	1011	824	0.975	1.615	18.7

Table 3. Comparison of the AARE values between PFM, BFM, ADM, and FMM models.

Conditions	PFM		BFM		ADM		FMM	
	AAREX (%)	AAREY (%)						
Without pulsation	10.93	38.63	5.76	22.35	3.92	11.37	2.38	7.87
With pulsation	13.66	22.48	11.44	17.12	12.88	16.37	10.74	13.28

Figures

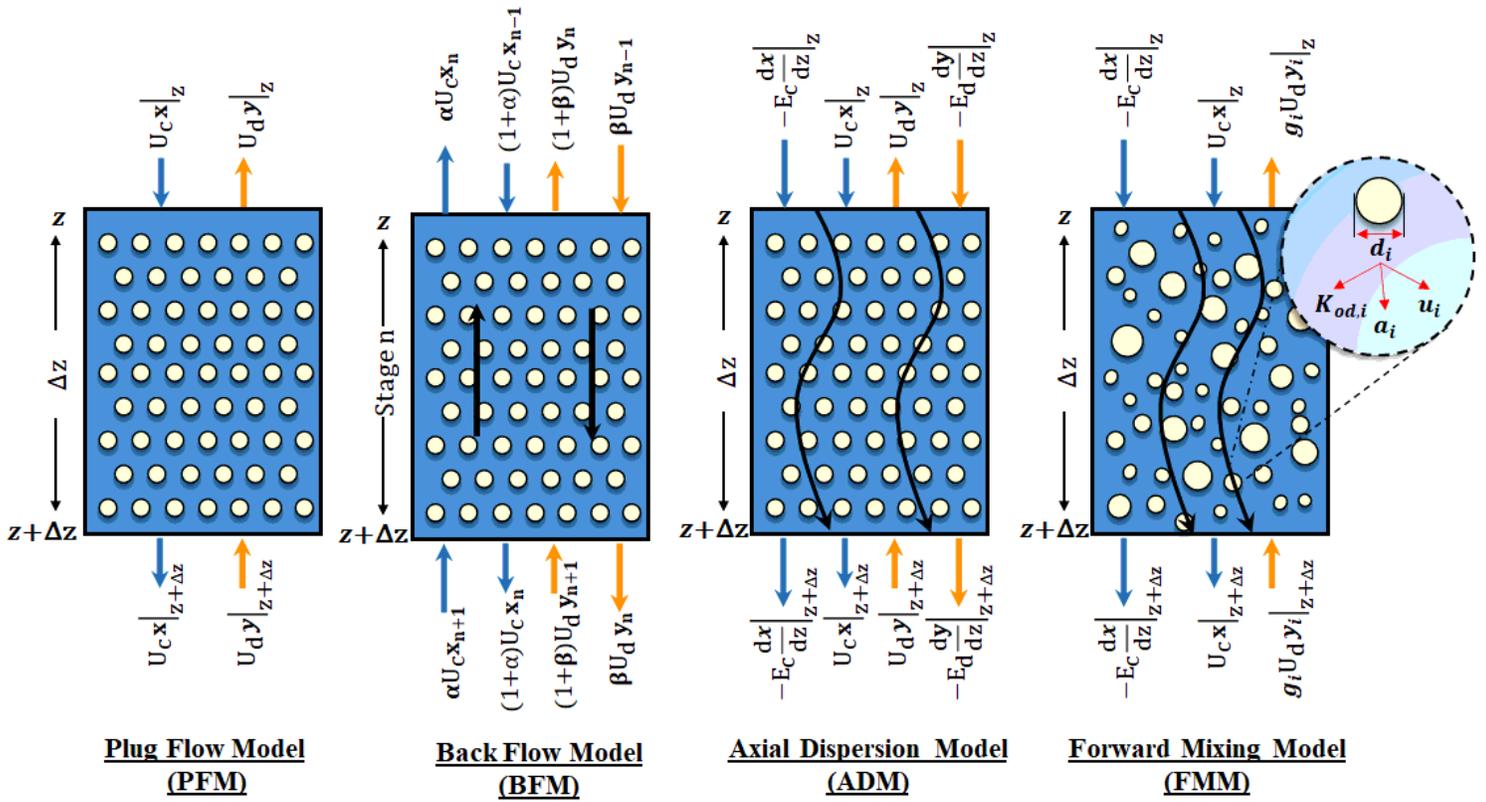


Figure 1

Mass balance over a volumetric element based on the plug flow, back-flow, axial dispersion and forward mixing models

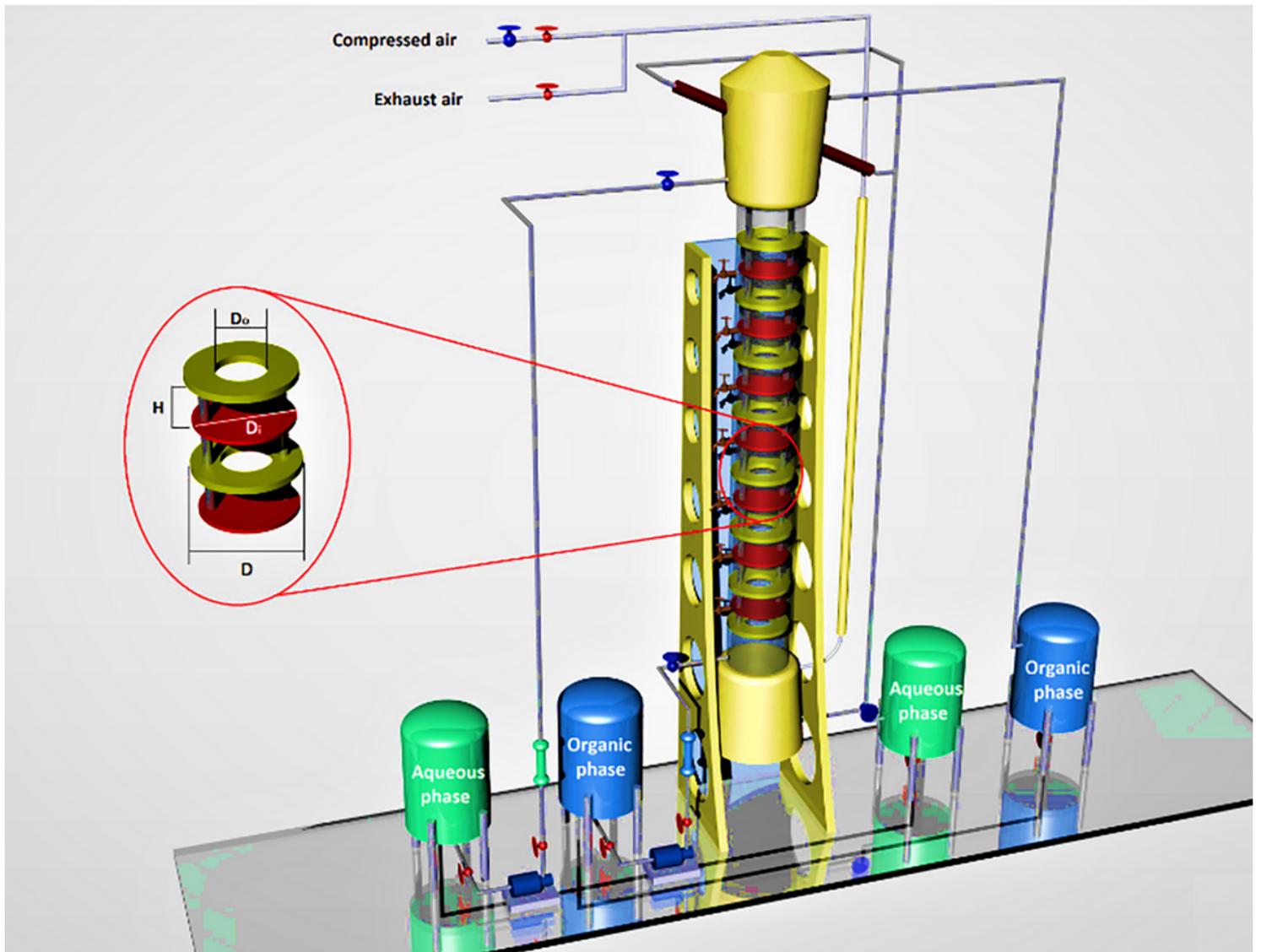


Figure 2

Schematic view of a pilot plant pulsed disc-donut column

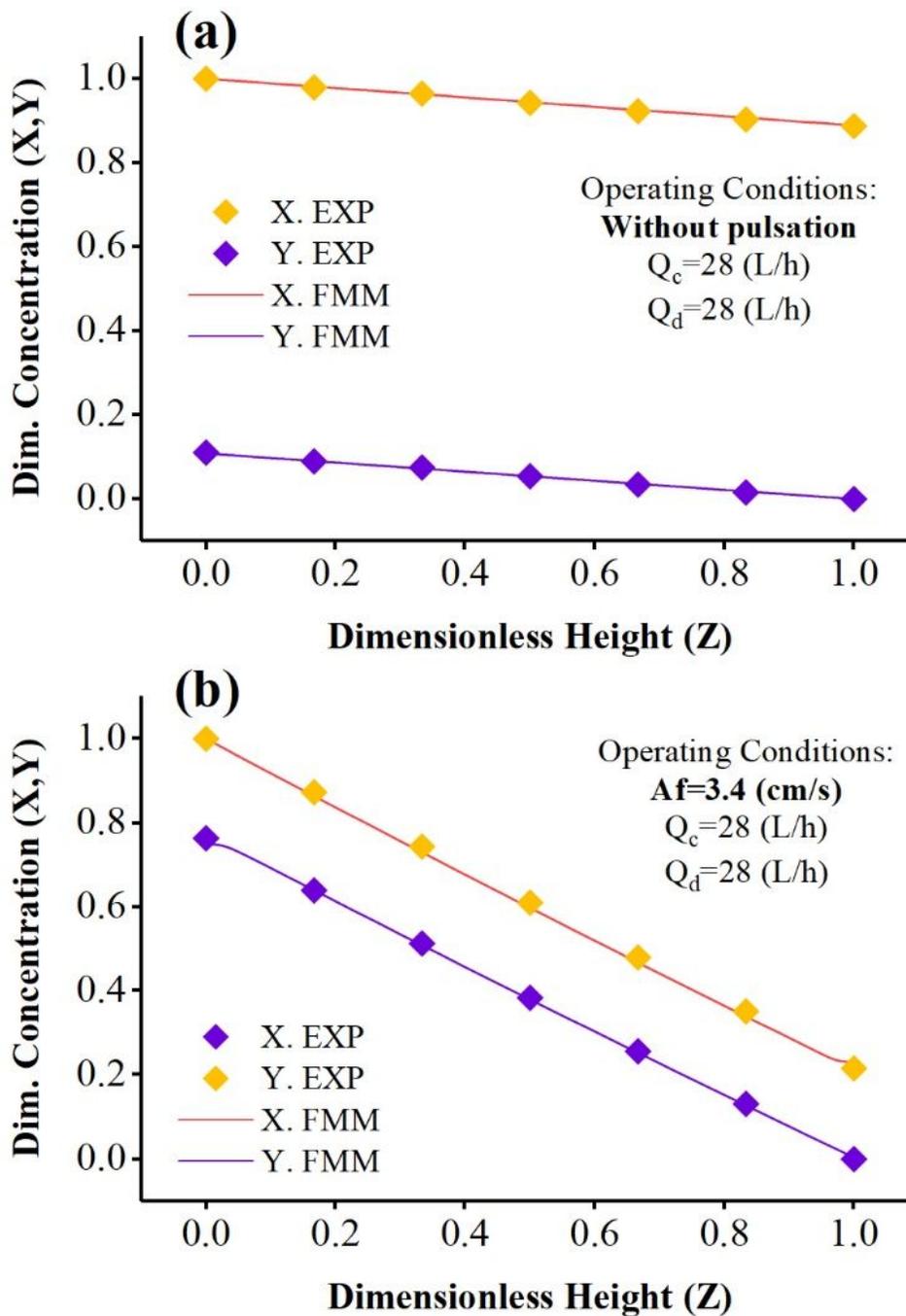


Figure 3

Comparison of laboratory concentration profiles with predicted by the forward mixing model under $Q_c=Q_d=28$ (L/h) and a. without pulsation, b. $Af=3.4$ (cm/s).

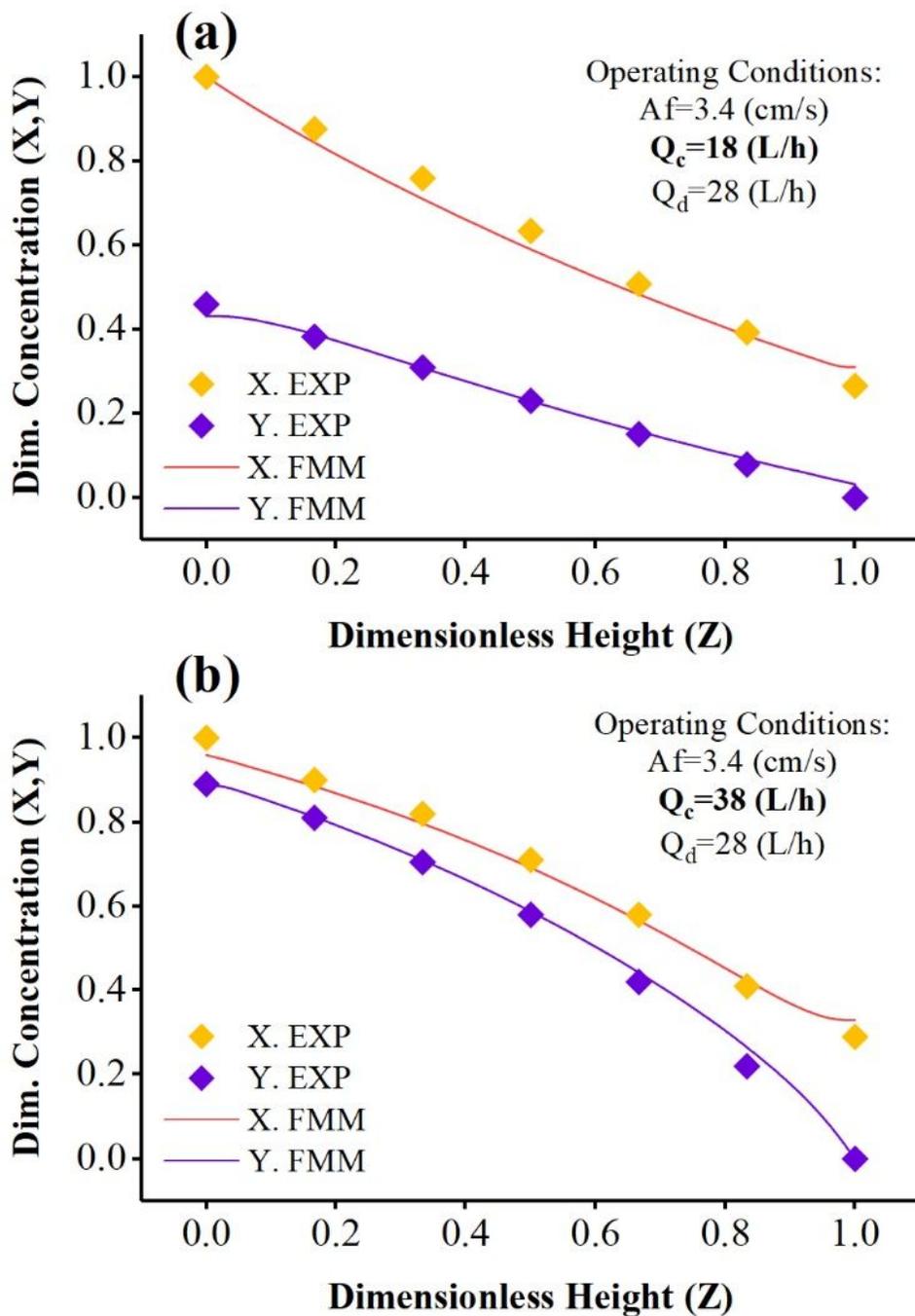


Figure 4

Comparison of laboratory concentration profiles with predicted by the forward mixing model under $Q_d=28$ (L/h) and $Af=3.4$ (cm/s) and a. $Q_c=18$ and b. $Q_c=38$ (L/h).

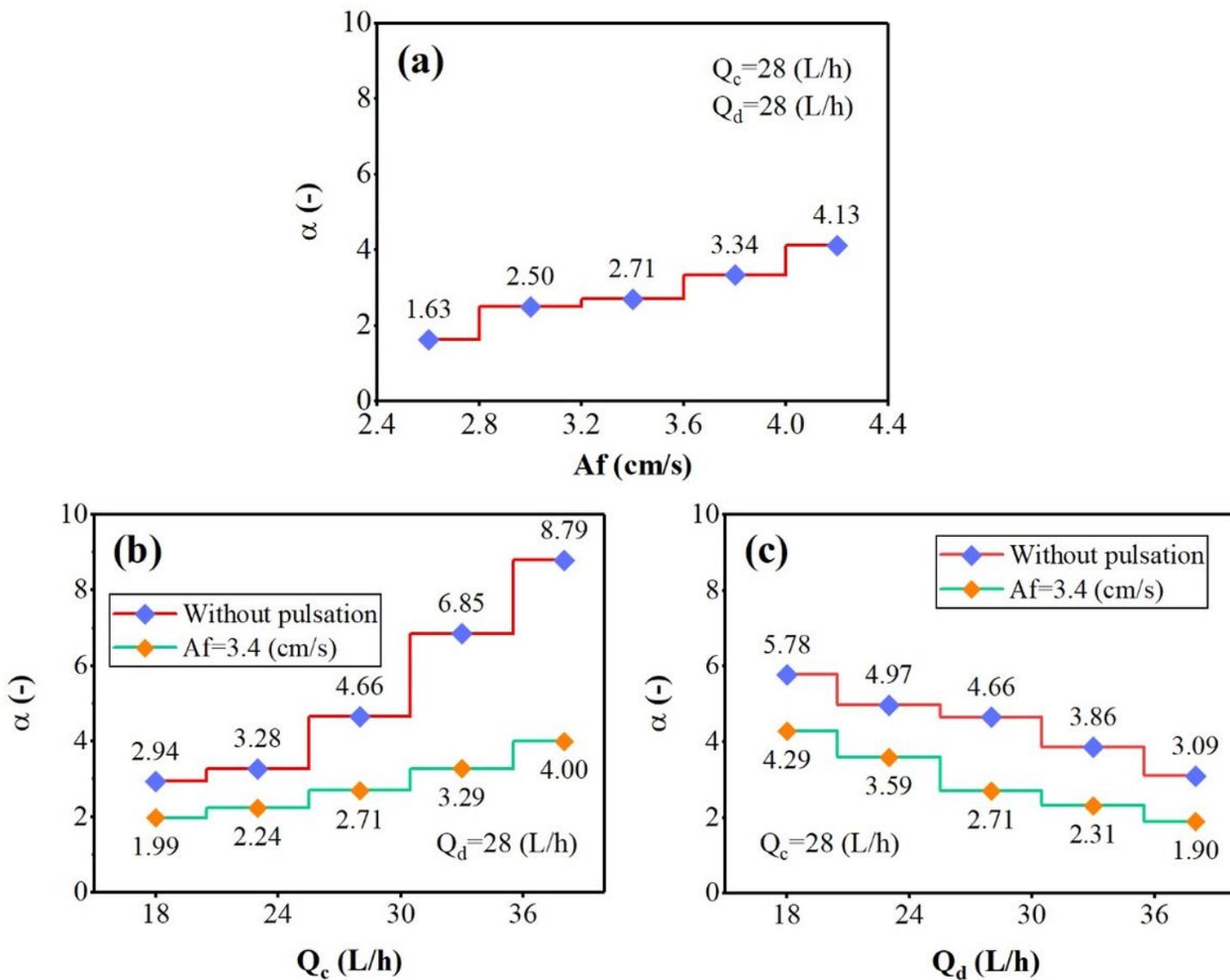


Figure 5

The effect of operating parameters (a. pulsation intensity, b. aqueous phase flow rate, and c. organic phase flow rate) on the continuous phase backflow coefficient.

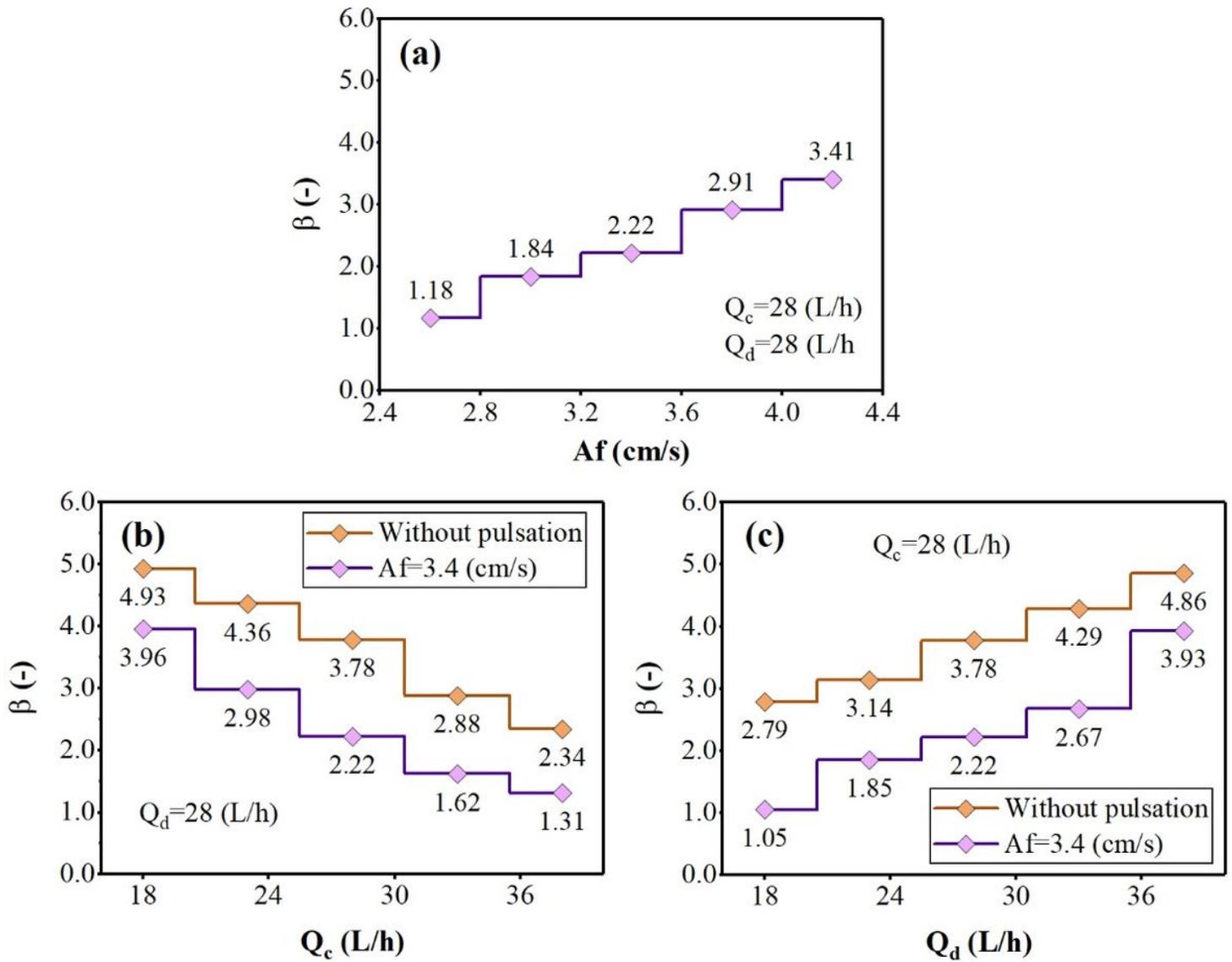


Figure 6

The effect of operating parameters (a. pulsation intensity, b. aqueous phase flow rate, and c. organic phase flow rate) on the dispersed phase backflow coefficient.

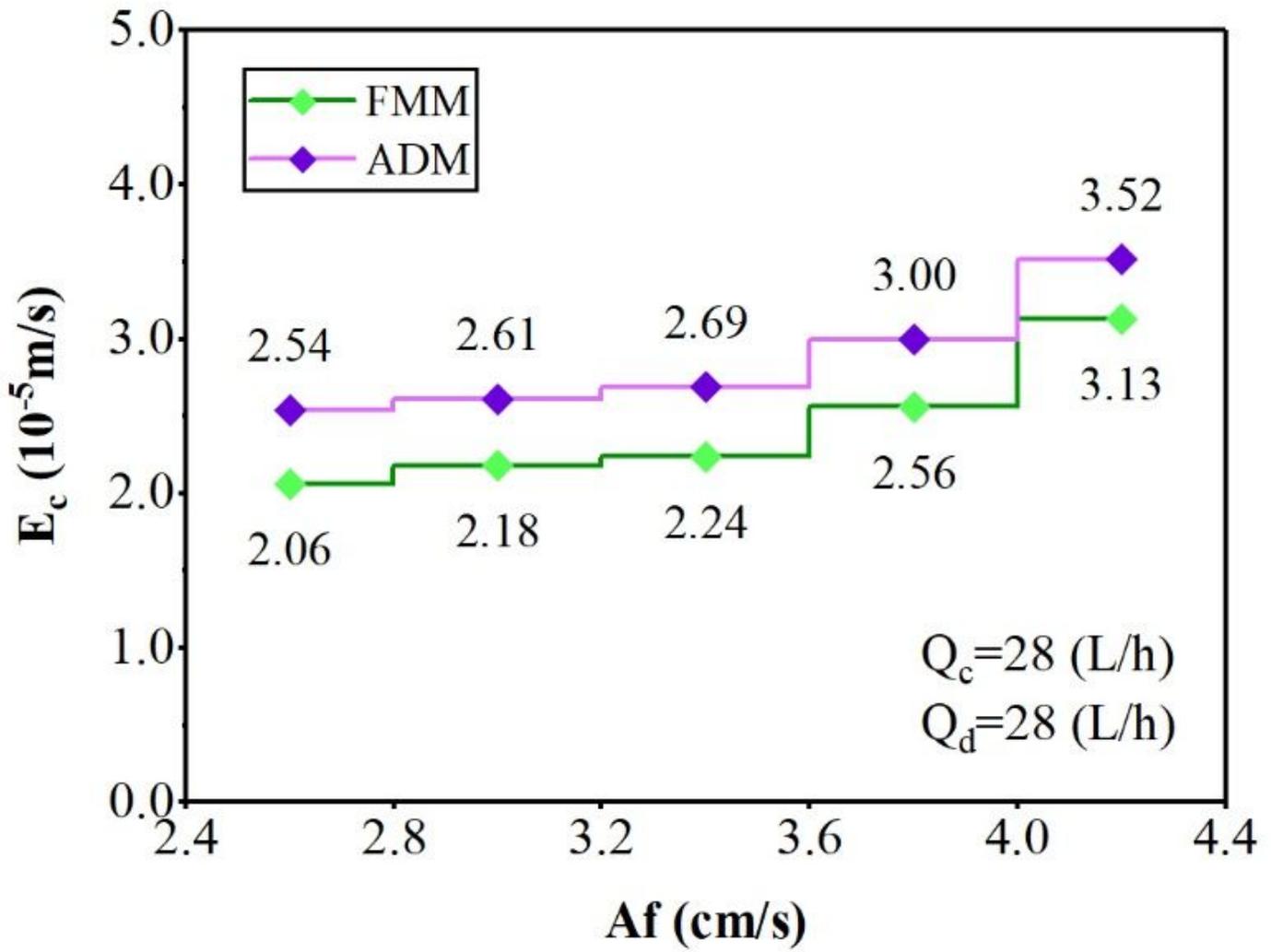


Figure 7

The effect of pulsation intensity on the continuous phase axial dispersion coefficient.

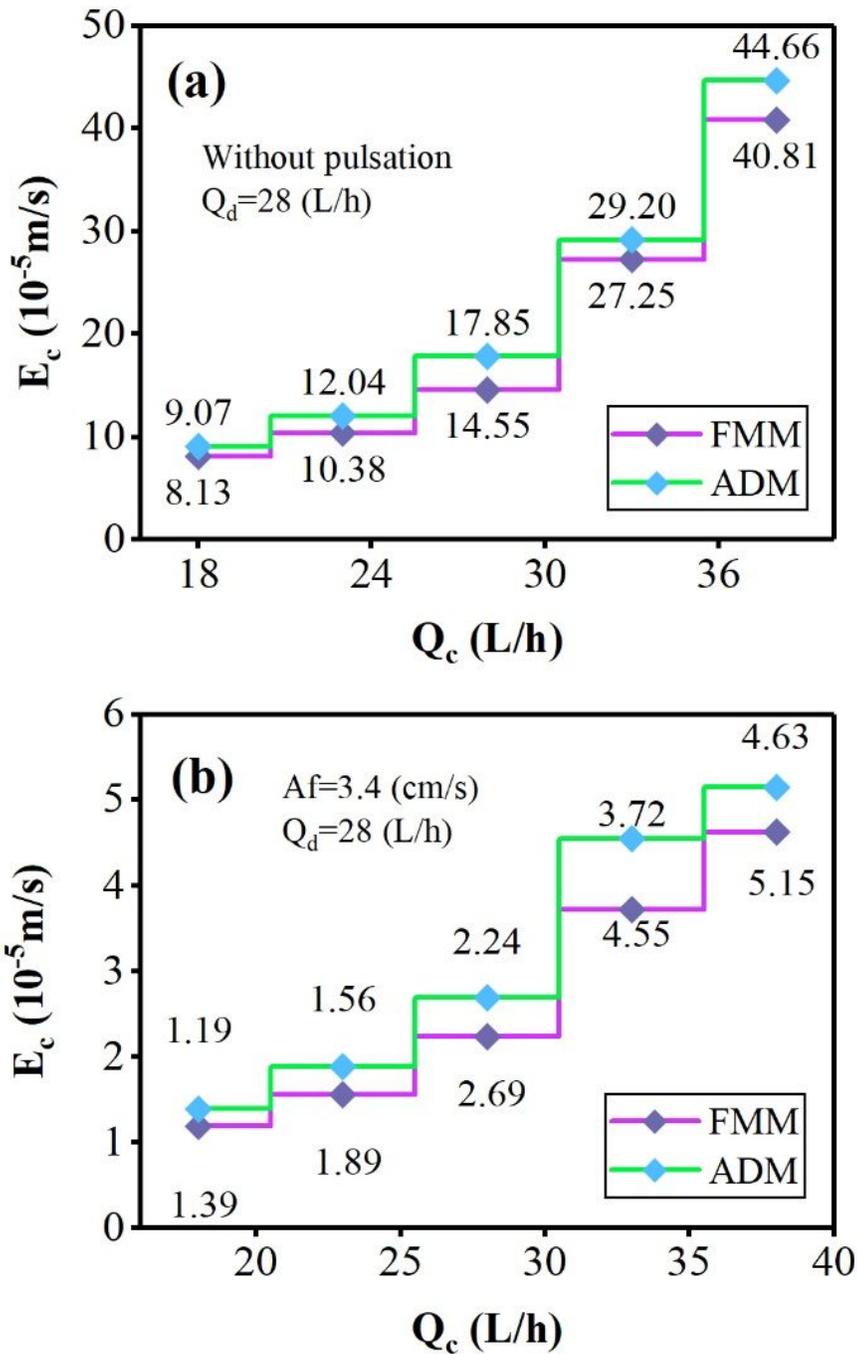


Figure 8

The effect of aqueous phase flow rate on the continuous phase axial dispersion coefficient a. without pulsation, b. $Af=3.4$ (cm/s).

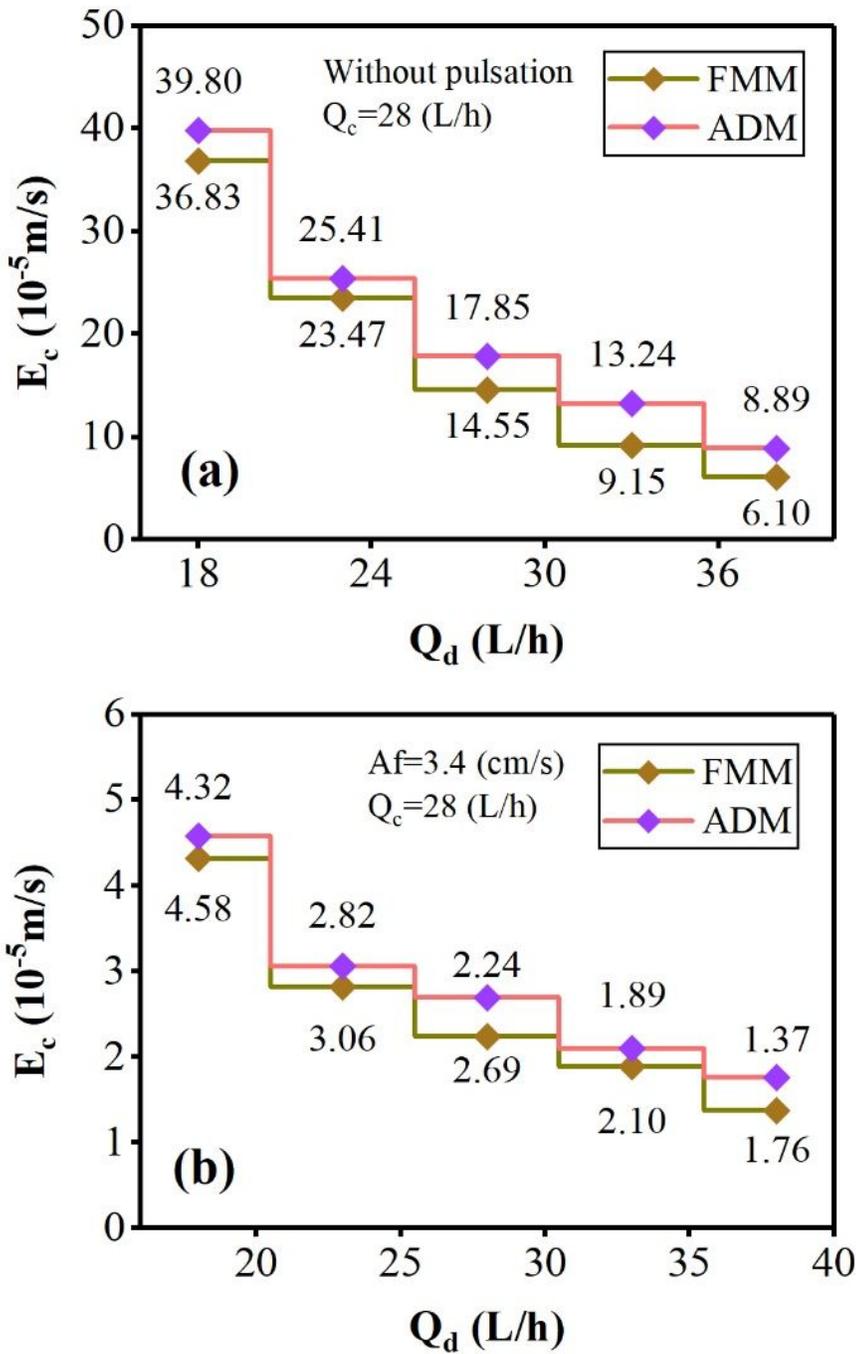


Figure 9

The effect of organic phase flow rate on the continuous phase axial dispersion coefficient a. without pulsation, b. $Af=3.4$ (cm/s).

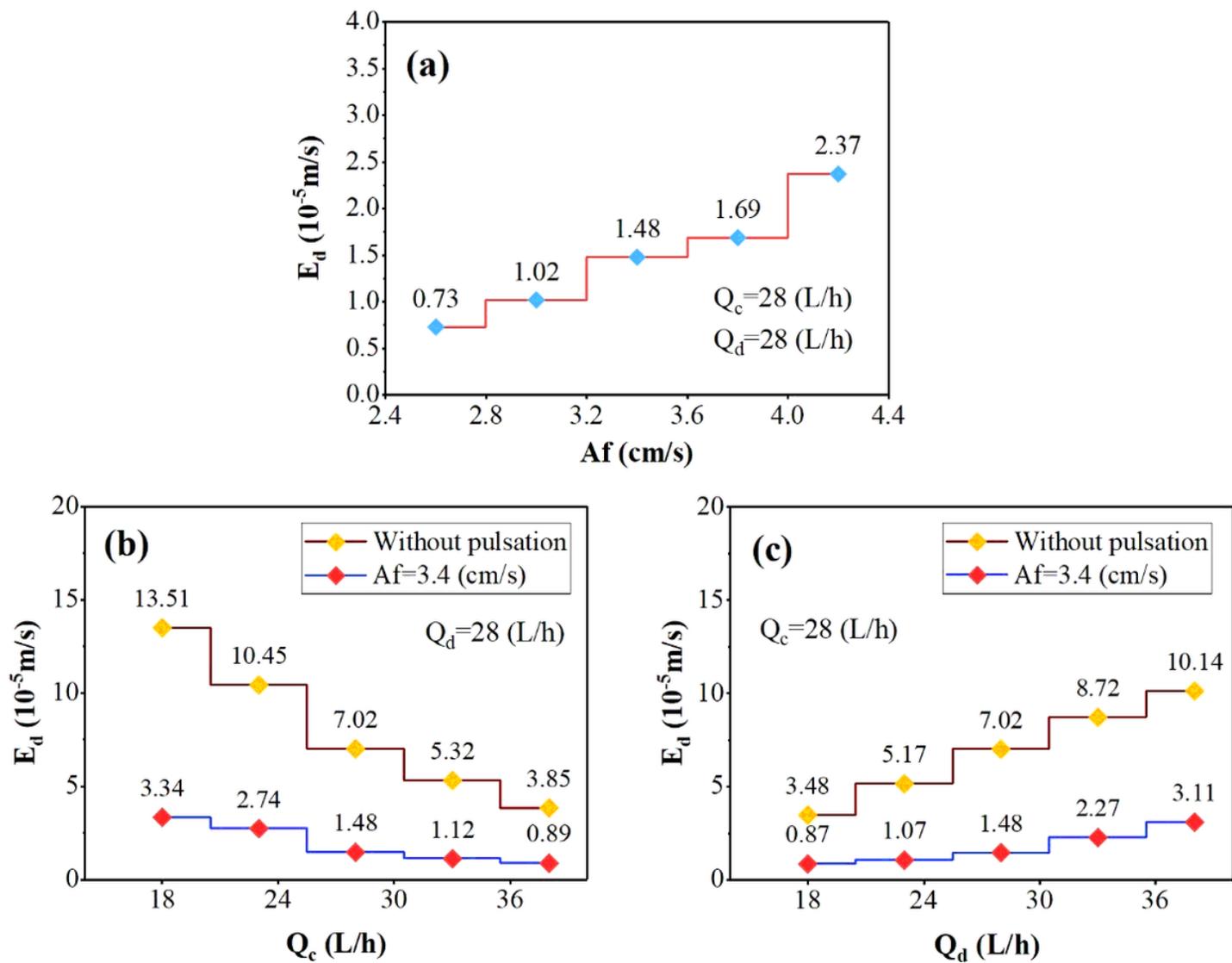


Figure 10

The effect of operating parameters (a. pulsation intensity, b. aqueous phase flow rate, and c. organic phase flow rate) on the dispersed phase axial dispersion coefficient.

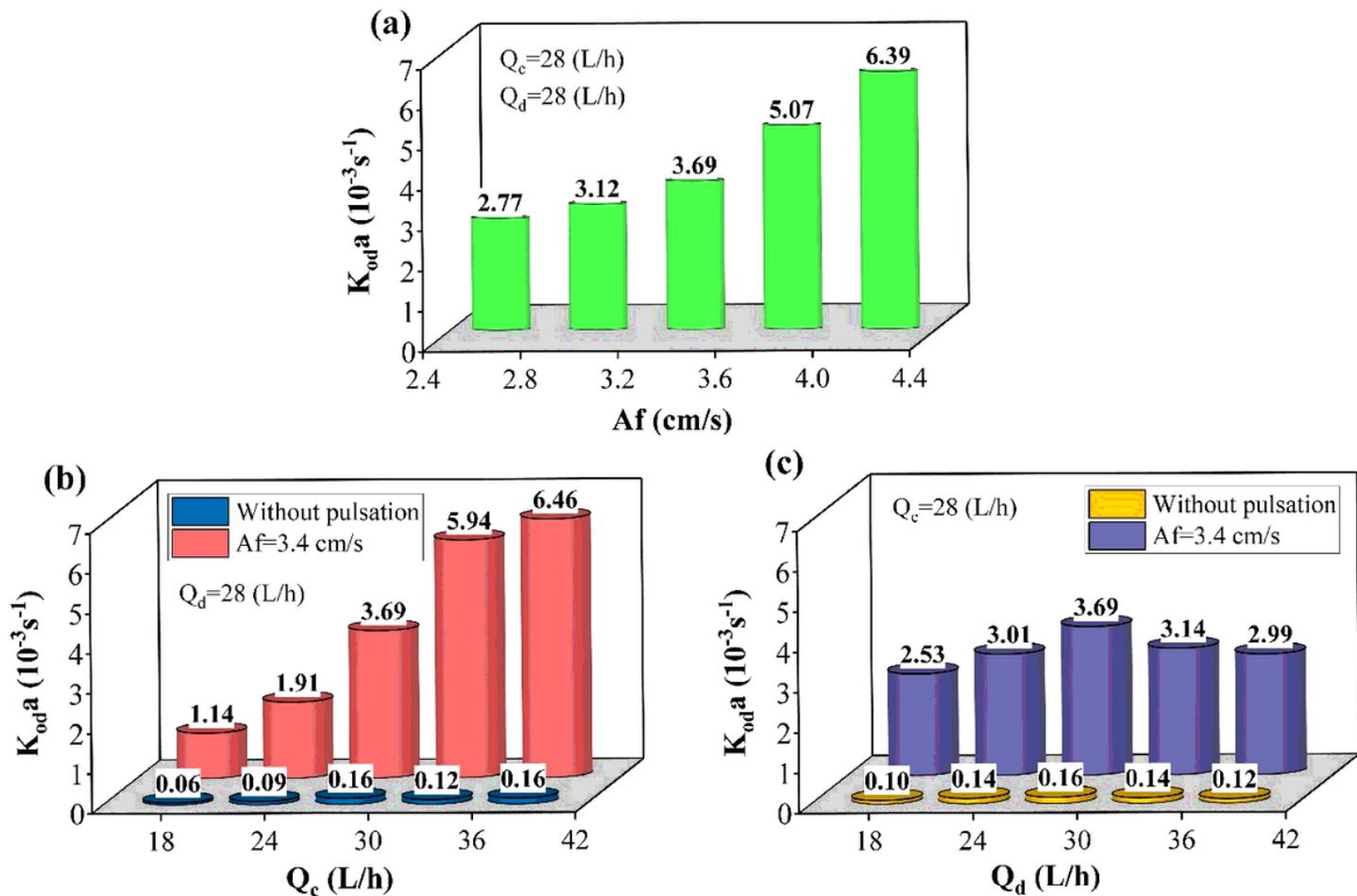


Figure 11

The effect of operating parameters (a. pulsation intensity, b. aqueous phase flow rate, and c. organic phase flow rate) on the volumetric overall mass transfer coefficient predicted by FMM.

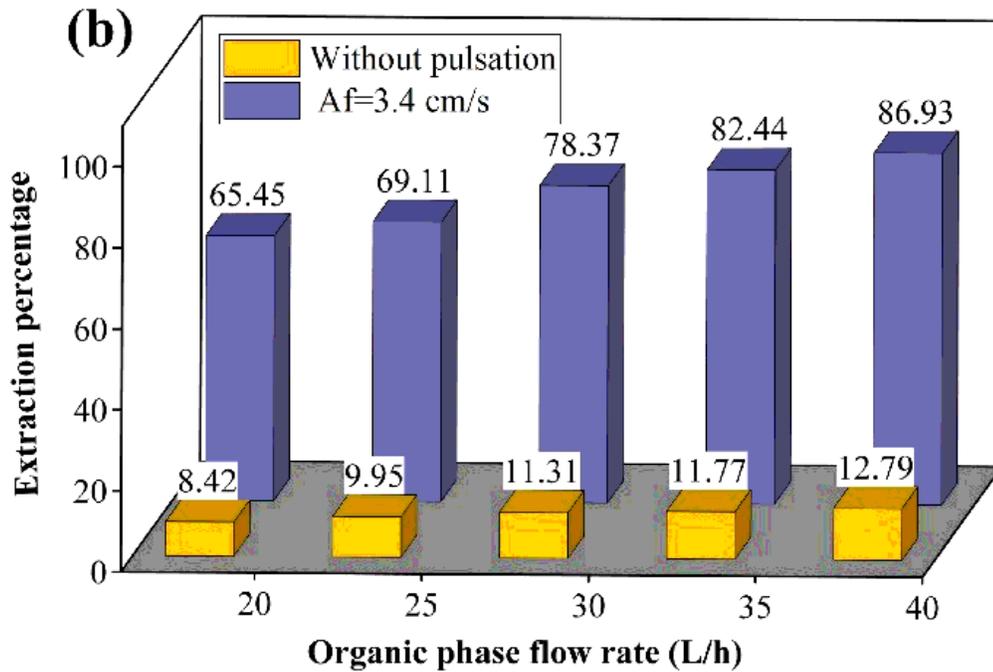
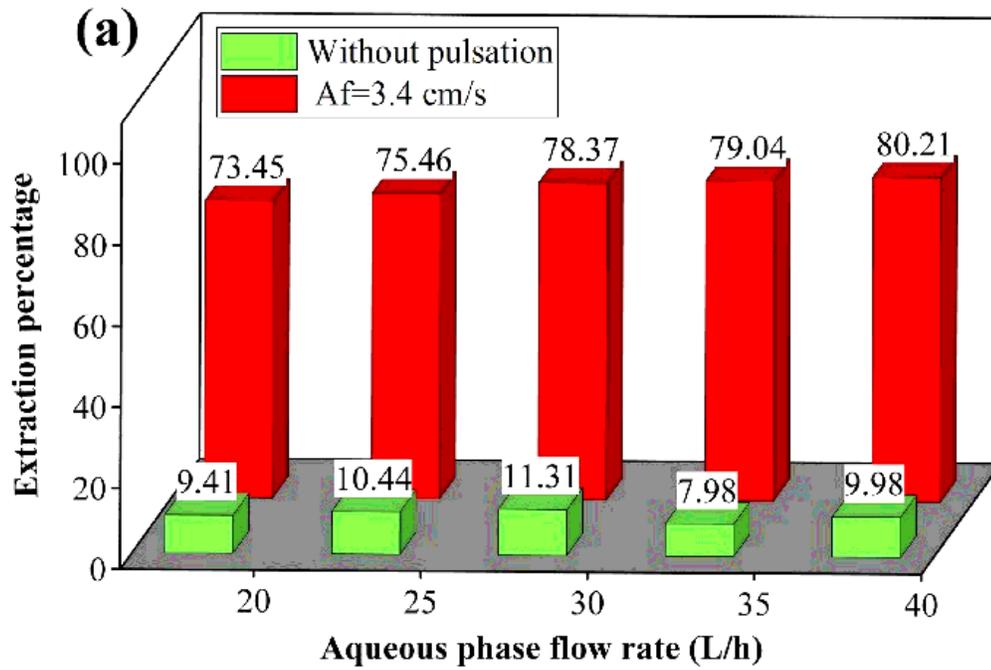


Figure 12

The influence of aqueous and organic phase flow rate on the extraction percentage

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- [Table1.docx](#)