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Antero Laitinen[a]; Juha Kaunisto[a]
[a] VTT CHEMICAL TECHNOLOGY, VTT, FINLAND

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Oldshue–Rushton Column in Supercritical Fluid Extraction

ANTERO LAITINEN* and JUHA KAUNISTO
VTT CHEMICAL TECHNOLOGY
P.O. BOX 1401, FIN-02044 VTT, FINLAND

ABSTRACT

The performance of a mechanically agitated Oldshue–Rushton column using carbon dioxide solvent at 10 MPa and 313 K to extract ethanol from aqueous feed was investigated. The overall mass-transfer coefficient $K_{od}a$, the height equivalent to a theoretical stage (HETS), dispersed phase holdup, as well as the column capacity data were measured as a function of solvent-to-feed ratio and rotor speed. The values of overall mass transfer coefficient $K_{od}a$ generally ranged from 0.009 to 0.012 s^{-1}, and the values of the HETS ranged from 0.44 to 0.74 m. The total throughput of the Oldshue–Rushton column without agitation was approximately 68 $m^3 \cdot h^{-1} \cdot m^{-2}$, whereas at 300 rpm the total throughput was approximately 48 $m^3 \cdot h^{-1} \cdot m^{-2}$.

INTRODUCTION

In recent years, utilization of gases in the supercritical state as a solvent in countercurrent continuous extraction operations has received considerable interest, particularly in the food and pharmaceutical industries due to several reasons. Supercritical fluids, such as carbon dioxide, offer favorable solvent properties in many extractions. Carbon dioxide in the supercritical state has a liquidlike density, and its viscosity and diffusivity is intermediate between those of liquidlike and gas-like values. Carbon dioxide is environmentally acceptable, nontoxic, nonflammable, and the second least expensive solvent after water (1). Additionally, separation of a gas-like solvent from a solute after supercritical extraction is usually more simple and cost-effective than in conventional liquid–liquid extraction operations.

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* To whom correspondence should be addressed. FAX: +358 9 456 7026. E-mail: Antero.Laitinen@vtt.fi

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Several authors have tested their applications in spray, sieve tray, or packed countercurrent supercritical extraction columns (2–14). In these studies the columns, particularly packed supercritical extraction columns, have been shown to be very efficient compared to conventional liquid–liquid extraction columns (3). Spray columns have relatively high capacity, but the column efficiency is not as good as in packed columns. Although the packing increases the overall column efficiency, the capacity of a spray column is greatly reduced if the column is packed (3). In high-pressure engineering, small-diameter columns are desirable due to pressure vessel cost reasons. The diameter of a packed column needs to be larger than in a spray column to maintain the same capacity, which means that packed columns are usually more expensive to construct than spray columns. One solution combining the high capacity of spray columns and the high efficiency of packed columns could be a mechanically agitated extraction column.

There have been very few systematic studies concerning mechanically agitated extraction columns operated under supercritical conditions. However, liquid carbon dioxide has been used in a Scheibel column to extract volatile flavors from aqueous solutions (15). Further, a Karr-type reciprocating-plate column has been used to extract ethanol and furfural from aqueous solutions using supercritical carbon dioxide as a solvent (16). We recently tested a rotating disk supercritical extraction column by extracting ethanol from aqueous solution (17).

The purpose of this paper is to report mass-transfer efficiency and capacity of a high-pressure bench-scale mechanically agitated Oldshue–Rushton type supercritical extraction column.

EQUIPMENT AND EXPERIMENTAL PROCEDURES

The Oldshue-Rushton extraction column (Chematur Ecoplanning) used in our experiments has 35 mm inside diameter and 2 m height. The highest operating pressure of the column is 40 MPa, and the maximum rotor speed in this column is approximately 400 rpm. A special pressure compensating sealing system was developed to lead the electrically driven agitator shaft through the column head. The type of column introduced by Oldshue and Rushton (18) was applied in this work. The Oldshue–Rushton column consists of a vertical shell, in which the stator plates with a central opening are placed. Stator plates serve to separate each compartment from its neighbor in the vertical direction. The number of compartments in the column is 99. In each compartment there is an impeller connected to the centrally placed agitator shaft. The Oldshue–Rushton column geometry is summarized in Table 1. The calculation of the smallest cross-sectional area is based on the stator plate opening diameter. A simplified flow diagram of the bench-scale experimental equipment is seen in Fig. 1.

TABLE 1
Oldshue–Rushton Column Characteristics

Height of the column, mm	2000
Total agitated height, mm	1400
Column diameter, mm	35
Compartment height, mm	20
Stator plate opening diameter, mm	24
Smallest cross-sectional area, mm^2	452
Number of compartments	99

A high-pressure compressor (Nova Werke AG) was used to feed carbon dioxide to the column, and a high-pressure piston pump (Lewa AG) was used to pump the aqueous feed stream to the column. Two high-pressure turbine flowmeters (EG&G Flow Technology, FTO-2NITWBLHC-5, maximum pressure 40 MPa) placed in solvent and extract streams were used to measure carbon dioxide flow rates. The pressure inside the column was automatically controlled by a micrometering valve placed on the solvent stream after the compressor, and the flow rate was controlled by another micrometering valve placed on the extract stream. The window at the top of the column allowed us the observation of the liquid level during the experiments.

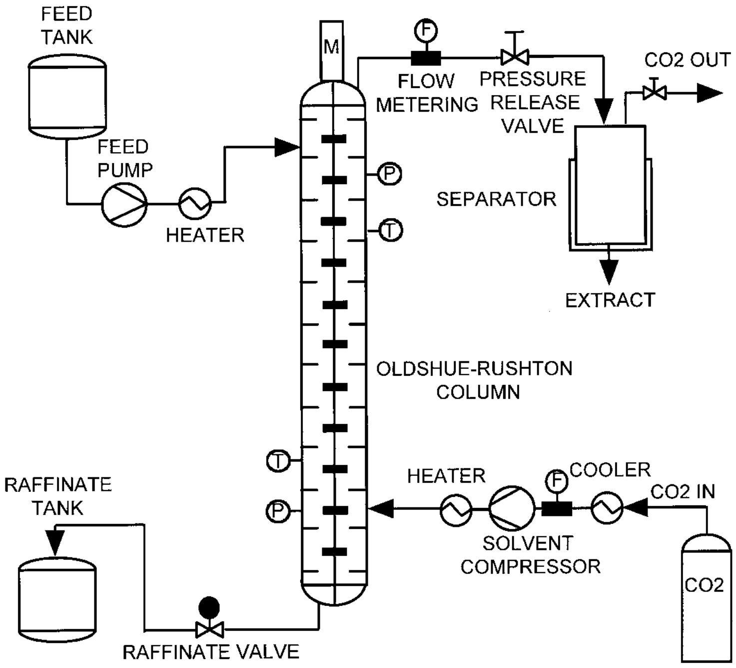

FIG. 1 Experimental apparatus.

The extraction temperature was 313 K and the pressure was 10 MPa in all experiments. Carbon dioxide was not recycled during the experiments to avoid the use of a preloaded solvent and to simplify the calculations. Consequently, fresh carbon dioxide from the container was used in all experiments. The column may be operated by using either feed or the solvent phase as a continuous phase. In this work, the carbon dioxide phase was dispersed into the continuous aqueous phase, which initially contained 10 wt% ethanol. The aqueous feed was pumped to Compartment 84, and carbon dioxide solvent flow was pumped to Compartment 14.

A typical experiment lasted approximately 4 hours. Measurements were taken after steady state had been achieved as determined by constant raffinate composition and steady flows. Samples were taken from the feed, raffinate, and extract streams once every 20 minutes, beginning 2 hours after the start of the experiment. Feed stream samples were taken from the pipeline after the feed pump by opening the sample valve, and raffinate samples were taken from the bottom of the column by opening the sample valve. According to the phase equilibrium measurements by Lim et al. (11), raffinate stream contained approximately 7.5 wt% carbon dioxide. This carbon dioxide was not collected, instead it was allowed to evaporate before the sample bottles were sealed. Extract stream samples were taken with a loop system placed on the extract stream line. This system consists of a pipe with a known volume (1.5 mL) and valves at both ends of the pipe. When sampling, the loop valves were opened so that the extract stream can flow to the loop. After a short period (3–5 minutes) the loop valves were closed. The sample inside the loop was slowly bubbled to water through a needle valve, and the loop was washed with a small amount of water. The extract from the separator was also continuously collected. The final extract samples were taken from the bottle containing all the collected extract. Typical liquid sample volume was 10 mL. The concentration deviations in the consecutive feed and raffinate samples were typically within 2%. More information about the experimental procedure is available in a recent paper (17).

The feed and raffinate flow rates were measured by weighing the feed and raffinate tanks. The readings of the feed, raffinate, and carbon dioxide flow rates were taken every 20 minutes. Calculated extract stream ethanol concentration values based on the overall material balance were used to calculate $K_{od}a$ and HETS values. Material balances were checked after each run. The relative deviation of overall material balance around the column was within 1.5%. The relative deviations of ethanol balances around the column were typically within 9%.

The holdup was measured by closing the column inlet and outlet valves and measuring the time required for the carbon dioxide drops that entered the bottom of the column to rise to the top of the column. This information combined with the solvent volumetric flow rate was used to calculate the dispersed phase holdup. The method was adopted from Rathkamp et al. (3). Flooding was not observed during the experiments because the automatically controlled raffinate stream valve opened when the liquid level at the top of the column started to rise. However, when the feed and solvent flow rates reached certain values, the raffinate stream valve opened so much that the major part of the solvent flow came out from the bottom of the column. At this point the column pressure decreased rapidly and the column was no longer operable. This point was used to describe the capacity of the column.

The aqueous ethanol samples were analyzed by a Perkin-Elmer 900 gas chromatograph (GC) equipped with flame ionization detector (FID) and Porapak Q column using *n*-butanol as an internal standard. One gram of liquid sample was weighed (Precisa 240 A), and a known amount of the internal standard was added to the sample. Aqueous reference solutions containing different ethanol concentrations and internal standard were made and run with the GC to determine the response factors. The GC injections (0.5–1 μL) of liquid samples were repeated 2 or 3 times. The amount of ethanol in the samples was calculated on the basis of the valid response factor.

Carbon dioxide (99.7%) was obtained from AGA, ethanol (99.5%) from Primalco, and *n*-butanol (99.5%) from Merck. Ion-exchanged water was used in the experiments.

CALCULATIONS

According to recent measurements (11), the distribution coefficient of ethanol between supercritical CO_2 and water measured at 10.1 MPa and 313 K for the dilute ternary carbon dioxide–ethanol–water system is approximately 0.12. This value was used in our calculations. The concept based on overall transfer units was used to estimate the column efficiency and to calculate the mass transfer coefficient. The calculation was based on the dispersed carbon dioxide phase. The total agitated height Z of the differential extractor is given by

$$Z = \mathrm{HTU_{od}} \mathrm{NTU_{od}} \tag{1}$$

The number of transfer units ($\mathrm{NTU_{od}}$) and the height of a transfer unit ($\mathrm{HTU_{od}}$) for the dispersed phase in the case of dilute solutions and an immiscible solvent can be written as

$$\mathrm{NTU_{od}} = \int_{y_2}^{y_1} \frac{dy}{y^* - y} \tag{2}$$

$$\mathrm{HTU_{od}} = V_d / K_{od} a \tag{3}$$

where y is the weight fraction of solute in the dispersed extract phase, y^* is the equilibrium value, V_d is the superficial velocity of the dispersed phase, K_{od} is the overall mass transfer coefficient based on the dispersed phase, and a is the interfacial area. As a matter in fact, the assumption of an immiscible solvent is not strictly valid, but for simplicity this assumption was made. The following relationship between the height equivalent of a theoretical stage (HETS) and height of a transfer unit (HTU_{od}) can be written (19):

$$\mathrm{HETS} = \frac{\ln E}{E - 1}\,\mathrm{HTU}_{\mathrm{od}} \tag{4}$$

where E is the extraction coefficient, defined as

$$E = m\frac{S}{F} \tag{5}$$

where m is the distribution coefficient, S is the solvent mass flow rate, and F is the feed mass flow rate. Slip velocity $\overline{V}_s$ describes the relative velocity of the phases, and is defined as

$$\overline{V}_{\mathrm{s}} = \frac{\overline{V}_{\mathrm{d}}}{h} + \frac{\overline{V}_{\mathrm{c}}}{1 - h} \tag{6}$$

where $\overline{V}_d$ and $\overline{V}_c$ are the relative velocities of the dispersed and continuous phases, respectively, through the smallest cross-sectional area, and h is the dispersed phase holdup.

RESULTS AND DISCUSSION

Extraction Results

The extraction results are seen in Table 2. The aqueous feed initially contained 10 wt% ethanol, and the raffinate stream contained 0.9–4.2 wt% ethanol depending mainly on the solvent-to-feed ratio. Agitation slightly decreased the ethanol concentration in the raffinate from 1.3 to 0.9 wt% when the highest solvent-to-feed ratio ($S/F = 14/1$) was applied, whereas in the case of a lower solvent-to-feed ratio agitation did not seem to decrease the ethanol concentration in the raffinate stream. The ethanol concentration in the extract was 82–91 wt%. Due to practical reasons the separator was operated at approximately 3.8 MPa and 279 K.

TABLE 2

Oldshue–Rushton Column Mass Flow Rates and Ethanol Concentrations

No.	Feed stream flow rate (kg/h)	Raffinate stream flow rate (kg/h)	Solvent stream flow rate (kg/h)	Extract stream flow rate (kg/h)	Extract collected (kg/h)	Ethanol concentration in feed stream (wt%)	Ethanol concentration in raffinate stream (wt%)	Ethanol concentration in solvent stream (wt%)	Ethanol concentration in extract stream (wt%)	Ethanol concentration in extract (wt%)
1	0.97	0.90	14.0	13.9	0.09	10	1.27	0	0.62	90
2	1.0	0.96	13.9	13.8	0.08		1.15		0.68	85
3	0.98	0.90	14.0	13.9	0.08		0.87		0.56	87
4	0.98	0.92	14.0	13.9	0.08		0.92		0.67	91
5	0.98	0.92	14.0	13.9	0.08		1.03		0.58	86
6	1.54	1.51	14.1	14.1	0.11	10	2.47	0	0.69	84
7	1.59	1.55	14.1	14.0	0.11		2.55		0.71	82
8	1.53	1.50	13.9	13.8	0.11		2.56		0.73	85
9	1.55	1.52	14.1	14.1	0.11		2.56		0.7	86
10	2.0	2.0	14.1	14.0	0.08	10	4.00	0	0.78	90
11	2.15	2.14	14.0	14.0	0.11		4.13		0.86	91
12	2.1	2.1	14.0	14.0	0.11		4.19		0.8	86
13	2.0	2.0	14.0	13.9	0.11		3.98		0.91	90

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TABLE 3
Mass Transfer in Oldshue–Rushton Column

No.	S/F	Rotor speed (rpm)	$K_{od}a$ ($1/s \times 10^3$)	HETS (m)
1	14/1	0	8.7	0.55
2		45	9.8	0.50
3		120	10.9	0.44
4		227	10.6	0.45
5		324	10.0	0.48
6	14/1.5	0	11.5	0.53
7		100	11.9	0.52
8		199	11.0	0.55
9		303	11.3	0.54
10	14/2	0	9.7	0.72
11		103	10.6	0.68
12		201	9.8	0.74
13		311	10.3	0.69

Mass Transfer Efficiency

Mass transfer efficiency of the Olshue–Rushton column as a function of rotor speed is seen in Table 3 and in Figs. 2 and 3. The values of $K_{od}a$ generally range from 0.009 to 0.012 s^{-1}, and the values of HETS range from 0.44 to 0.74 m. Agitation increased column efficiency slightly at the highest solvent-to-feed ratio ($S/F = 14/1$), whereas at a lower solvent-to-feed ratio agitation did not enhance the mass transfer. According to Rathkamp et al. (3), in the carbon dioxide–ethanol–water system equilibrium is approached relatively easily in a column at low solvent-to-feed ratios. We therefore assume that this was the reason why agitation did not enhance mass transfer in this work at low solvent-to-feed ratios. Also, the relatively high solvent flow rate necessary to extract ethanol from aqueous solutions already creates agitation inside the small diameter column.

The values of $K_{od}a$ (3, 11) measured for spray and packed continuous countercurrent supercritical fluid extraction columns generally range from 0.006 to 0.04 s^{-1} depending on the type of column, packing, and solvent-to-feed ratio. Values of HETS for small-scale spray, packed, and sieve tray supercritical fluid extraction columns have been measured (3–5, 9, 11). The reported HETS values for spray, packed, and sieve tray columns are typically in the 0.2–0.5 m range. The values measured in our work for the Oldshue–Rushton column are of the same order of magnitude as those measured for small-scale spray and packed columns.

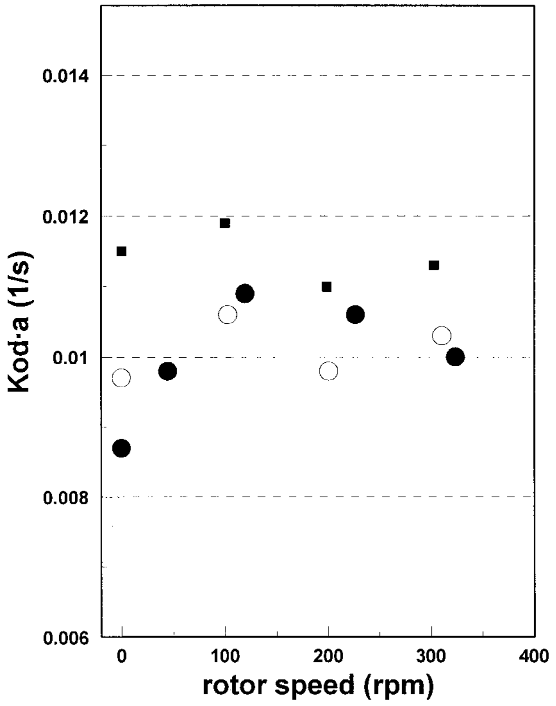

FIG. 2 Effect of agitation on overall mass-transfer coefficient K_{od} *a*. (○) *S*/*F* = 14/2; (■) *S*/*F* = 14/1.5; (●) *S*/*F* = 14/1.

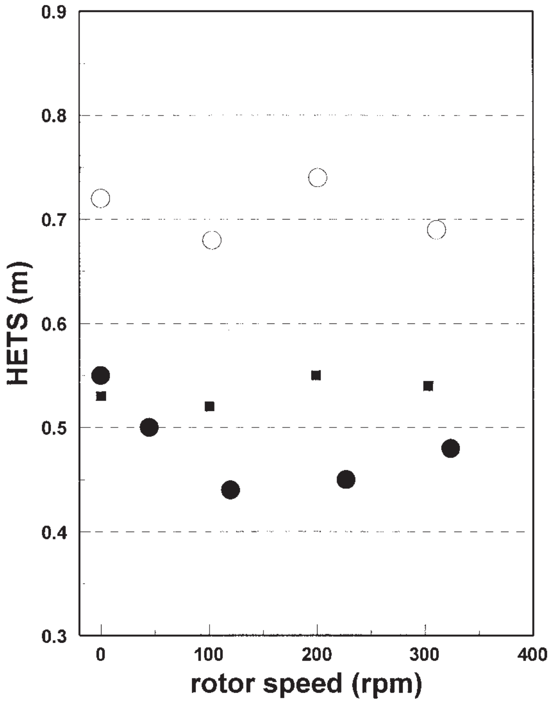

FIG. 3 Effect of agitation on HETS. (○) *S*/*F* = 14/2; (■) *S*/*F* = 14/1.5; (●) *S*/*F* = 14/1.

Column Capacity

The total throughput of the Oldshue–Rushton column calculated as feed plus solvent flow rate divided by the smallest cross-sectional area of the column is seen in Fig. 4. Agitation seems to decrease the column capacity significantly. Without agitation the total throughput is approximately 68 $m^3 \cdot h^{-1} \cdot m^{-2}$, whereas at 300 rpm the total throughput is approximately 48 $m^3 \cdot h^{-1} \cdot m^{-2}$. Increased agitation decreases dispersed phase drop size, which explains the decrease in column capacity. Flooding velocities for 25.4 mm diameter spray and packed columns operated under supercritical conditions for carbon dioxide–2-propanol–water system have been measured previously (3). According to the authors, the packed column flooded at $V_c = 0.4$ mm/s and $V_d = 3.0$ mm/s, whereas the spray column reached flooding at $V_c = 0.4$ mm/s and $V_d = 20.3$ mm/s. In this work the capacity of the Oldshue–Rushton column without agitation was approximately $V_c = 5.0$ mm/s and $V_d = 14.0$ mm/s, and at 150 rpm the capacity was approximately at $V_c = 4.9$ mm/s and $V_d = 11.6$ mm/s. Compared to conventional extraction systems, the capacity of continuous countercurrent supercritical fluid extraction columns is higher, most probably due to the larger density difference of the phases in supercritical fluid extraction. For example, the toluene/water system flooded in a me-

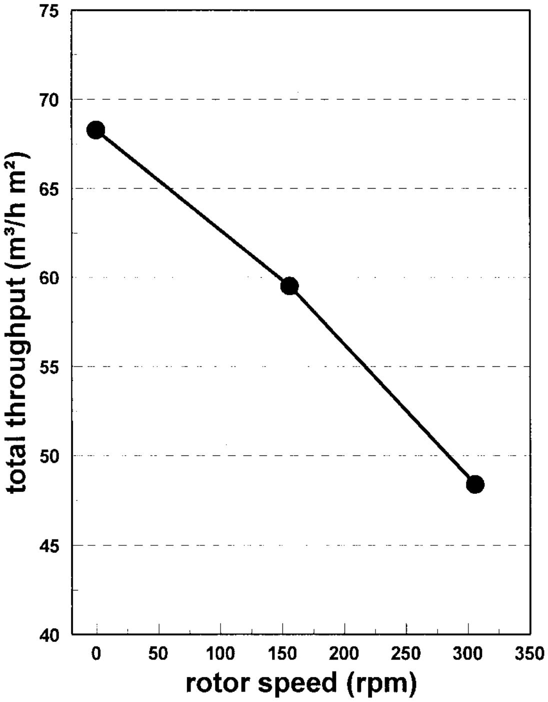

FIG. 4 Effect of agitation on column total throughput.

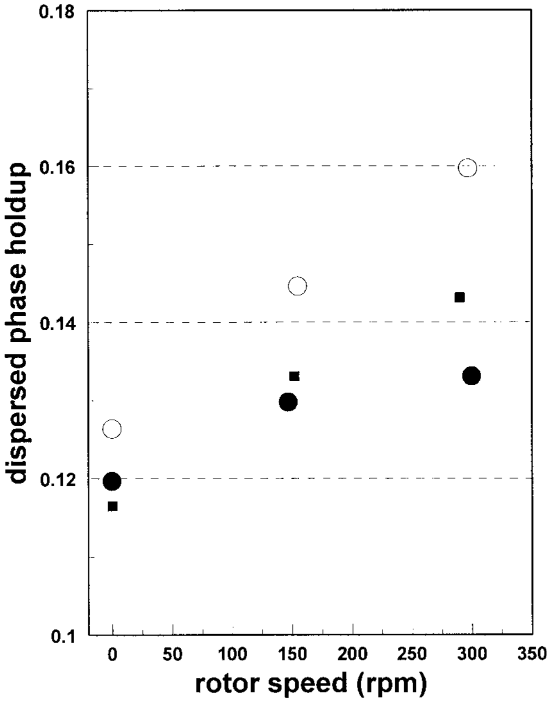

FIG. 5 Effect of agitation on dispersed phase holdup. (●) *S*/*F* = 14/1; (■) *S*/*F* = 14/1.5; (○) *S*/*F* = 14/2.

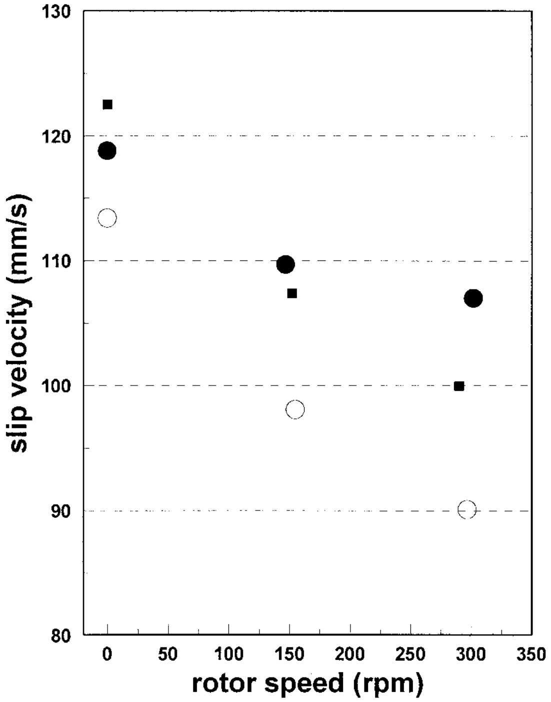

FIG. 6 Effect of agitation on slip velocity. (●) *S*/*F* = 14/1; (■) *S*/*F* = 14/1.5; (○) *S*/*F* = 14/2.

chanically agitated (200 rpm) Oldshue–Rushton column at approximately $V_c = 5.0$ mm/s and $V_d = 4.5$ mm/s (20).

The dispersed phase holdup in an Oldshue–Rushton column is seen in Fig. 5. The measured values are in the 0.12–0.16 range. As expected, all holdup values increase with increasing rotor speed. The operating slip velocities are seen in Fig. 6. All values decrease with increasing rotor speed because of increasing holdup.

CONCLUSION

The performance of the Oldshue–Rushton column in supercritical fluid extraction was investigated by using carbon dioxide at 10 MPa and 313 K as a solvent to extract ethanol from aqueous feed. The values of the overall mass transfer coefficient, $K_{od}a$, and the height equivalent to a theoretical stage (HETS) were in the same range as those measured for small-scale spray, packed, and sieve tray columns. The capacity of the Oldshue–Rushton column was significantly higher than measured for packed column, and approximately equal to spray column capacity.

In high-pressure engineering it is desirable to construct small-diameter columns due to equipment cost reasons. Mechanically agitated extraction columns, such as the Oldshue–Rushton column, seem to combine the advantages of high-capacity spray columns and high-efficiency packed columns, thus allowing the construction of efficient small-diameter columns. This type of column could potentially be used in other processes, such as industrial reactions, that take place continuously at high pressures and demand effective agitation.

NOTATION

a	interfacial area (m^2/m^3)
E	extraction coefficient = mS/F
F	flow rate of feed (kg/h)
h	dispersed phase holdup
HETS	height equivalent to a theoretical stage (m)
HTU_{od}	height of a transfer unit based on the dispersed phase (m)
K_{od}	overall mass transfer coefficient based on the dispersed phase (m/s)
m	distribution coefficient = concentration in solvent phase/concentration in feed phase
NTU_{od}	number of transfer units based on the dispersed phase
S	flow rate of the solvent (kg/h)
y	weight fraction of solute in the dispersed phase
V	superficial velocity (m/s)

V_s	slip velocity (m/s)
Z	total agitated height of the column (m)

Subscripts

1	at the top of the column
2	at the bottom of the column
c	continuous phase
d	dispersed phase

Superscripts

*	equilibrium value
–	through smallest cross-sectional area

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