Deacidification of Corn Oil by Solvent Extraction in a Perforated Rotating Disc Column

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ABSTRACT: The deacidification of corn oil by continuous liquid–liquid extraction was investigated in a rotating disc column. The solvent was ethanol containing approximately 6% water. The influence of rotor speed, oil phase flow, and column geometry upon the dispersed phase holdup and the mass transfer efficiency was studied. The dispersed phase holdup increased with the increase of rotor speed and oil phase flow. Pratt's equation was used for calculating the characteristic velocity. An inverse relation was observed between the characteristic velocity and rotor speed, which is different from data previously reported in the literature. The estimated volumetric mass transfer coefficients increased as rotor speed and oil phase flow increased. The experimental results proved that it is feasible to obtain a refined oil with an oleic acid content less than 0.3 wt% by continuous solvent extraction. They also indicated that the corresponding loss of neutral oil was less than 5 wt%. Such value for the loss of neutral oil is significantly lower than the results reported in the literature for alkali or physical refining of corn oil.

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KEY WORDS: Corn oil, deacidification, ethanol, extraction column, holdup, liquid–liquid extraction, mass transfer, mass transfer coefficients, oleic acid, rotating disc contactor.

Deacidification of edible oils can be accomplished by alkali refining, physical refining, or liquid–liquid extraction. Alkali refining is performed at temperatures in the range of 70 to 90°C and requires centrifuges for neutralization and washing, resulting in higher investments and effluent treatment costs. For oils with high acidity, alkali refining causes high losses of neutral oil due to saponification and entrainment by the soapstock. Physical refining is performed at high temperatures (200–250°C) and very low pressures (5–10 mm Hg) (1). It involves high energy inputs and costly machinery. Liquid–liquid extraction can be carried out at room temperature and atmospheric pressure, reducing the energy consumption for oil refining without losses of natural components. Because of the high difference between the boiling points of the solvent and fatty compounds, solvent stripping from refined oil and solvent recovery from extract stream can be easily carried out. In fact, both separations can be accomplished by evaporation or distillation at relatively low temperatures, in most cases lower than 80°C. By using a moderate vacuum, for instance an absolute pressure of 250 mm Hg, this temperature can be further reduced to 55°C. Especially in the case of oils having high acidity, the literature reports that solvent extraction offers a promising alternative route for partial deacidification (2,3).

Oils from corn and rice bran have high acidity due to enzymatic activity in the raw material. They need special care during refining by any process. According to Leibovitz and Ruckenstein (4), alkali refining of crude corn oil with a free acid content between 8 and 14% results in neutral oil loss of 15 to 25%, while for physical refining the loss of neutral oil varies between 11 and 20%. In the Brazilian oil industry, the neutralization process results in a neutral oil loss of 14% for corn oil containing 4% of free fatty acids (1).

Shah and Venkatesan (3) studied oil refining by aqueous isopropryl alcohol extraction and Antoniassi *et al.* (5) studied oil deacidification by liquid–liquid extraction, including the required pretreatment. Batista *et al.* (6,7) determined, correlated, and predicted the liquid–liquid equilibrium for triglycerides, fatty acids, and short-chain alcohols. However, there is little information available about the performance of extractors in the deacidification of edible oils. To study such performance, it is necessary to analyze the hydrodynamic behavior and mass transfer phenomena inside the equipment under different operational conditions.

With this purpose, we investigated the dispersed phase holdup and mass transfer in a rotating disc column for the corn oil/oleic acid/aqueous ethanol system. A specific feature of this system is the high viscosity of the oil phase (8,9), which is unusual as compared with most systems used for studying liquid–liquid extraction. Our results proved the feasibility of the deacidification of edible oils by continuous solvent extraction and indicated that the neutral oil loss was less than 5%.

EXPERIMENTAL PROCEDURES

Apparatus and measuring procedures. The original rotating disc contactor (RDC) consists of a column equipped with a

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central rotating shaft carrying equally spaced discs, which are positioned at the center of each compartment made of fixed stator rings. As the discs rotate, they generate torroidal vortices in each compartment causing turbulence in the liquid in proportion to their rotating speed. The perforated rotating disc contactor (PRDC) is a modified RDC equipped with perforated discs (10).

The dimensions (in cm) of the equipment used in the present work are as follows: diameter: column inside, 5; stator, 4; RDC/perforated rotating disc contactor with stator rings (PRDC I) disc, 3.0; perforated rotating disc contactor without stator rings (PRDC II) disc, 4.7; height: column, 130; extraction zone, 100; column compartments with 15 discs, 6; column compartments with 33 discs, 2.5. Three different versions were used: the original RDC, a PRDC I, and a PRDC II, whose discs have a larger diameter in comparison to the discs employed in the other versions. All versions were equipped with 33 discs, although the PRDC II was additionally tested in a version equipped with only 15 discs. The net free area in the discs used in the PRDC versions was 20%, containing holes of 3 mm diameter. Schematic drawings of the RDC and PRDC versions used in the present work are shown in Scheme 1.

All experiments were performed at room temperature (25°C) and local atmospheric pressure (727 mm Hg). For the holdup experiments the operational variables were rotor speed (50–350 rpm) and phase ratio ($0.25 < V_d/V_c < 4.00$), where V_d and V_c are the superficial velocity of the dispersed and continuous phases, respectively. Before each experiment of holdup measurement, the liquids, refined corn oil, and aqueous ethanol (containing approximately 6% water), were saturated with each other by intensive mixing and phase separation by gravity overnight. Subsequently, the equipment was filled with the continuous, alcoholic phase through the bottom of the column and its flow rate was maintained at the desired constant value. The rotor was started and the rotating speed was measured by a digital tachometer 1726 (Ametek, Largo, FL). The dispersed, oil phase was fed to the top of the column with its flow rate at the desired value. Both phases were pumped into the column by peristaltic pumps (Cole Parmer, Chicago, IL).

Inside the column, steady state was achieved after approximately 20 min of continuous operation. This was verified by the constant position of the interface level at the bottom of the equipment. After another 20 min of steady-state operation the rotor and all inlet and outlet flows were stopped simultaneously. The dispersed phase was allowed to settle and the holdup was determined according to the displacement method (10). The displaced dispersed phase volume was recorded, and the fractional holdup (x) was calculated by Equation 1:

$$x = \varepsilon_d / \varepsilon_t \qquad [1]$$

where ε_d is the volume of the dispersed phase, and ε_t is the total volume of the extraction zone.

Each set of experiments was performed under conditions of constant continuous phase mass flow rate (3.35×10^{-4} kg/s) and rotor speed. The dispersed phase flow rate was varied until flooding was reached. This flooding state was characterized by the formation of a second interface at the top and an unsteady interface at the bottom of the column.

The physical properties of the liquids at 25°C, when mutually saturated, are given in Table 1. The density measurements were performed using a DMA 58 Density Meter (Anton Paar, Graz, Austria). The viscosity data were obtained by an AMV 200 Viscometer (Anton Paar) and the interfacial tension data were measured by a K10ST Digital Tensiometer (Krüss, Kirchheim, Germany).

For the mass transfer experiments the operational variables were rotor speed (50–350 rpm), phase ratio ($V_d/V_c = 0.5$ or 1.0), and fatty acid content in the oil feed stream [1.4 wt% < weight fraction of fatty acid (w_{Fa}) < 4.3 wt%]. The desired fatty acid content in oil feed stream was obtained by adding the commercial oleic acid to the oil phase previously saturated with aqueous ethanol. For this reason, the fatty acids were the main compounds that moved away from the oil phase to the alcoholic phase during the mass transfer experi-
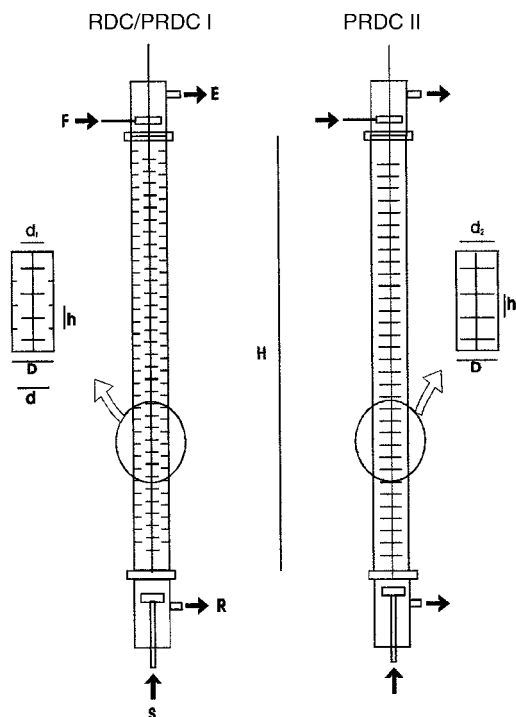
SCHEME 1

TABLE 1
Physical Properties of the Phases at 25°C

Phase	Density (kg/m³)	Viscosity (mPa·s)	Interfacial tension (mN/m)
Alcohol rich[a]	804.4	1.28	2.15
Oil rich[b]	904.8	31.8	

[a]Alcoholic phase: ethanol = 92.4 wt%, water = 6.0 wt%, and oil = 1.6 wt%.
[b]Oil phase: ethanol = 8.1 wt%, water = 0.3 wt%, and oil = 91.6 wt%.

ments. In the case of these experiments, the time necessary for attaining the steady state was approximately 75 min. After another 75 min of steady-state operation, samples of both the extract and raffinate were taken and analyzed to determine the alcohol, water, and acid concentrations of each phase.

Materials and methods. The refined corn oil was purchased from Refinações de Milho Brasil (São Paulo, Brazil). The fatty acid source was a commercial oleic acid (Riedel de Häen, Salze, Germany). Aqueous ethanol extra pure was obtained from Merck (Darmstadt, Germany). The reagents used in the Karl Fischer titration (methanol p.a. and Karl Fischer's solution) were also purchased from Merck.

Oleic acid and corn oil were analyzed by gas chromatography of fatty acid methyl esters according to official method 1-62 of the American Oil Chemists' Society (AOCS) (11). The fatty acid composition of the commercial oleic acid and of corn oil is given by Batista *et al.* (6). The estimated trygliceride composition of the oil is also presented in Batista *et al.* (6).

The fatty acid content was determined by titration with NaOH, according to AOCS method Ca 5a-40 (12). The titration cloud point was assessed by a Chemcadet 5986-50 pH/ion/mV meter (Cole Parmer). Solvent concentration was measured by evaporation in a 320-SE oven (Fanem, São Paulo, Brazil) at 60°C. The water content was determined by Karl Fischer titration (Metrohm, Herisau, Switzerland), according to AOCS method Ca 2e-55 (12). The oil concentration was calculated by difference.

RESULTS AND DISCUSSION

Holdup experiments. A typical set of experimental curves showing the variation of dispersed phase holdup with phase flow is presented in Figure 1. In the case of PRDC I and PRDC II, it was noticed that the holdup increased with phase flow ratio and with rotor speed, and flooding was reached at lower values of the phase flow ratio as the rotor speed increased. By contrast, the original RDC exhibited only a very small holdup increase with an increase in rotor speed.

Our holdup experimental results emphasize the advantage of perforated discs for the system studied. In the original RDC the dispersed phase drops were bigger and settled faster and, hence, resulted in lower equipment residence times. Consequently, the holdup data were lower than those obtained with perforated discs. In fact, the perforated discs improved the breakup of drops, increasing the number of droplets in the extraction zone. In the absence of stator rings, it was observed that the holdup data increased considerably. This must be due to the size of the discs, because without stator rings the discs were bigger and closer to the column wall (see Scheme 1). In this case the obstacles to phase flow were greater and they diminished the velocity of the droplets.

Aiming to compare our holdup data with those predicted by correlations developed for conventional liquid–liquid systems, we have used the Kumar and Hartland (13) equation for dispersed phase holdup with no mass transfer in an RDC. The
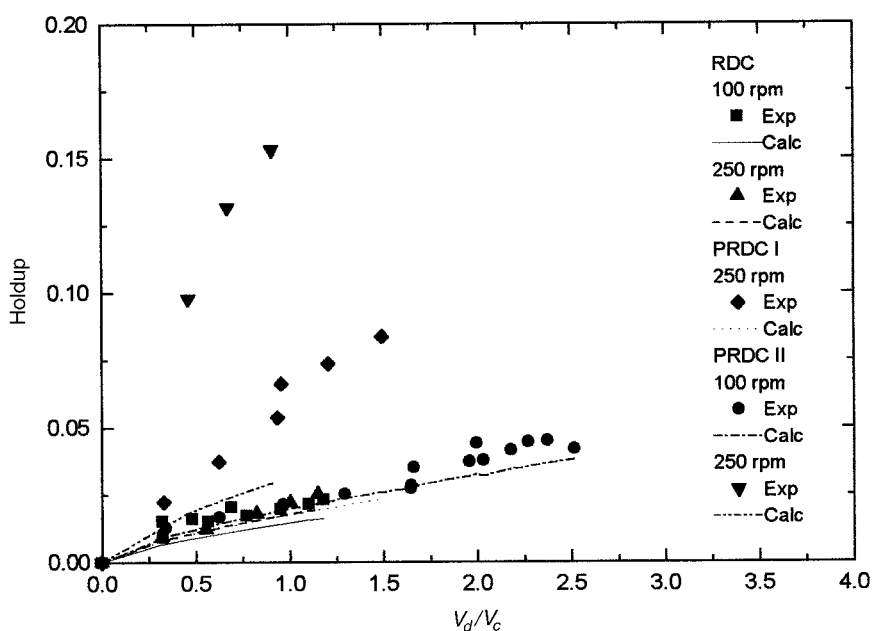

FIG. 1. Experimental and predicted holdup data. Abbreviations: V_d/V_c, phase ratio, where V_d and V_c are the superficial velocities of the dispersed and continuous phases, respectively; RDC, rotating disc contactor; PRDC I, perforated rotating disc contactor with stator rings; PRDC II, perforated rotating disc contactor without stator rings.

$$x = \left[0.19 + \left[\frac{\sigma}{g}\left(\frac{\rho_c}{g\gamma}\right)^{1/4} \right]^{0.67} \right]\left[\left[V_d\left(\frac{\rho_c}{g\gamma}\right)^{1/4} \right]^{0.69} \exp\left[7.13V_c\left(\frac{\rho_c}{g\gamma}\right)^{1/4} \right] \right]\left[\left(\frac{\Delta\rho}{\rho_c}\right)^{-0.65}\left(\frac{\mu_d}{\mu_w}\right)^{0.14} \right]\left[\left(\frac{d_1}{h}\right)^{0.62}\left(\frac{d^2}{D^2}\right)^{-0.26}\left[h\left(\frac{\rho_c g}{\gamma}\right)^{1/2} \right]^{-0.10} \right]$$

where

$$\sigma = \frac{4N^3 d_1^5 \rho_c}{\pi D^2 h \rho_c}\left[\frac{109.36}{Re_R} + 0.74\left[\frac{1000 + 1.2\,Re_R^{0.72}}{1000 + 3.2\,Re_R^{0.72}} \right]^{3.30} \right] \qquad [2]$$

predicted curves for some of our experimental data are shown in Figure 1. The expression used to derive this relationship was formulated by Kumar and Hartland (13), as presented in Equation 2.

In Equation 2, g is acceleration due to gravity (m/s^2), ρ_c is the continuous phase density (kg/m^3); γ is the interfacial tension (N/m); V_d and V_c are the superficial velocities of dispersed phase and continuous phase (m/s), respectively; $\Delta\rho$ is the density difference between phases (kg/m^3); μ_d and μ_w are the viscosities of the dispersed phase and water at 20°C (Pa·s), respectively; d_1 is the disc diameter (m); h is the compartment height (m); d is the stator opening diameter (m); D is the column diameter (m); N is the rotor speed (RPS); and Re_R is the rotor Reynolds number. The Reynolds number is calculated according to $Re_R = Nd_1^2\rho_c/\mu_c$, where μ_c is the continuous phase viscosity (Pa·s). In the case of PRDC II, which has no stator rings, the stator opening diameter d was equal to the column inside diameter D.

Kumar and Hartland (13) reported an average deviation (AD) between calculated and experimental holdup values of 22.7% for symmetric and asymmetric rotating disc columns. For our data, Kumar and Hartland correlation gives the following AD values: 24.9% for the RDC, 56.3% for the PRDC I, 44.3% for the PRDC II with 33 discs, and 81.7% for the PRDC II with 15 discs. Despite the unusual physical properties of our system, the Kumar and Hartland equation describes the observed behavior relatively well for the RDC. However, for both PRDC I and II the equation gives a good prediction only at low rotor speeds. As can be seen in Figure 1, at higher rotor speeds the experimental holdup data are much higher than the calculated ones. This is a consequence of the fact that the Kumar and Hartland equation was not developed for PRDC, and the differences between RDC and PRDC become more significant at higher rotor speeds. Such results confirm the advantage of using the perforated discs to improve the residence time of the dispersed phase in the extraction column.

The description of the hydrodynamics in liquid–liquid extraction columns has generally been based on correlations of the slip velocity (V_{slip}) with the dispersed phase holdup (x). The slip velocity under conditions of countercurrent flow is defined as the sum of the linear velocities of the dispersed (V_d) and continuous (V_c) phases.

$$V_{slip} = \frac{V_c}{1 - x} + \frac{V_d}{x} \qquad [3]$$

The slip velocity under the limiting condition of substantially zero dispersed phase holdup is the characteristic velocity V_k. There are a wide variety of equations for relating slip velocity to the continuous phase holdup $(1 - x)$. The first and simplest equation was that suggested by Gayler and Pratt (see Ref. 14) and is given by

$$V_{slip} = V_k (1 - x) \qquad [4]$$

By using Equation 4, characteristic velocities were calculated from our experimental holdup data. The characteristic velocity is the slope of the straight line obtained by plotting the group $[V_d + V_c x/(1 - x)]$ against $[x (1 - x)]$ for each set of experimental data. Pratt's equation was able to describe our experimental data with good accuracy. The following correlation coefficients were obtained: for 50% of the set of experimental data the correlation coefficient was higher than 0.99, for 33% it varied in the range of 0.96 to 0.98, and for 17% it had a value in the range of 0.86 to 0.92. Other equations for slip velocity, such as those suggested by Richardson and Zaki, by Misek, and by Godfrey and Slater (14), were also used but they did not fit well to our experimental data.

Figure 2 shows the dependence of the characteristic velocity on the rotor speed. For the original RDC, Kung and Beckman (15) observed the presence of two extraction regions: region I shows that the characteristic velocity is not dependent on the rotor speed until 400 rpm; in region II (above 400 rpm) the characteristic velocity is inversely proportional to the rotor speed. Our data for the RDC are not in disagreement with this observed behavior; only a very slight dependence on rotor speed was found. By contrast, for the PRDC an inverse relation between the characteristic velocity and the rotor speed was observed, even for low rotor speeds.

The same behavior was observed by Coimbra et al. (10) for a PRDC with features similar to those of the PRDC II used in the present work. For their experiments Coimbra et al. (10) used an aqueous two-phase (ATP) system composed of polyethylene glycol and potassium phosphate. Although both ATP systems and oil-alcohol systems have a high viscosity of the dispersed phase, their interfacial tensions are different. Whereas for the present oil-alcohol system the interfacial tension is 2.15 mN/m, most ATP systems have a much lower interfacial tension, in the range of 0.009 to 0.500 mN/m (10). Nevertheless, the interfacial tension for the present oil-alcohol system is still low if compared with most systems used in liquid–liquid extraction (13).
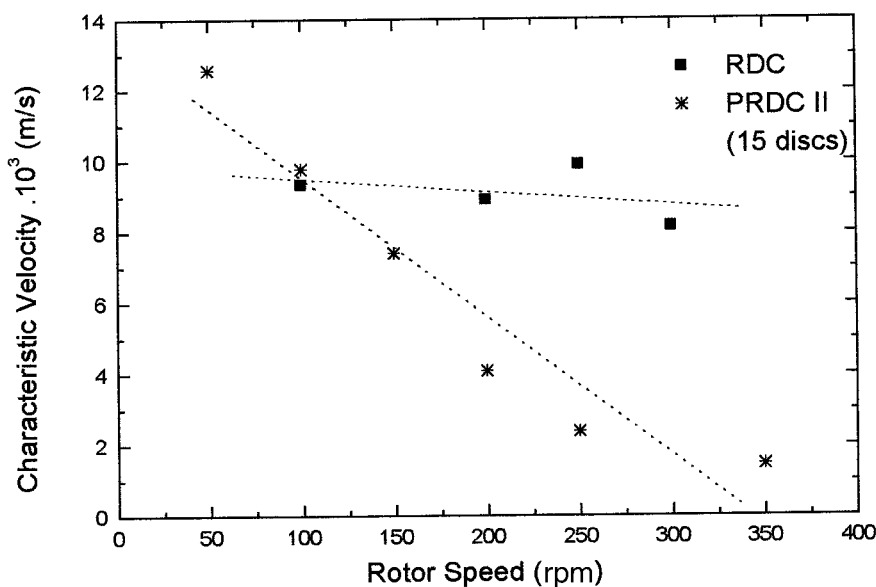
FIG. 2. Variation of characteristic velocity (Eq. 4) with rotor speed. For abbreviations see Figure 1.

As the same behavior was observed for both ATP and oil-alcohol systems, we can conclude that the utilization of perforated discs might explain the higher values for dispersed phase holdup and the inverse relation between the characteristic velocity and the rotor speed. The use of perforated discs makes it possible to obtain a more effective breakup of droplets at relatively low rotating speeds. The improvement in the droplets' breakup increases the mass transfer area and the residence time of the dispersed phase simultaneously.

Mass transfer experiments. For evaluating the mass transfer experiments we have estimated the volumetric overall mass transfer coefficients based on the units of concentration of the dispersed phase. Because the mass transfer coefficients for each phase are approximately constant for the entire apparatus and considering that the equilibrium curve is a straight line, it is possible to calculate an overall coefficient applicable to the entire device (16).

The equilibrium curve was obtained from equilibrium data determined according to the procedure described by Batista *et al.* (6) for the corn oil/oleic acid/aqueous ethanol system. The equilibrium curve is a straight line with a correlation coefficient of 0.9996 for a fatty acid concentration range of 0 to 20 wt%. This line is given by Equation 5:

$$w'_{ea} = 1.08014 w'_{oa} \qquad [5]$$

where w'_{ea} and w'_{oa} are the concentration of fatty acids in the alcoholic and oil phases, respectively. These concentrations were the weight fractions on a fatty acid-free basis, calculated using the following equation:

$$w'_a = \frac{w_a}{1 - w_a} \qquad [6]$$

where w_a is the weight fraction of fatty acids in each phase.

The overall mass transfer coefficients can be calculated using Equation 7:

$$F'\left(w'_{Fa} - w'_{Ra}\right) = K_R\, a\, V\, \Delta w'_{aM} \qquad [7]$$

where F' is the dispersed phase mass flow on a fatty acid-free basis in kg/s; w'_{Fa} and w'_{Ra} are the fatty acid concentrations in the feed and raffinate streams, respectively; K_R is the overall mass transfer coefficient in kg fatty acids/[m^2·s·(kg fatty acids/kg oil phase)]; a is the mass transfer area per unit of extraction zone volume in m^2/m^3; V is the volume of the extraction zone in m^3; and $\Delta w'_{aM}$ is the logarithmic average of the concentration differences at the ends of the column based on the units of concentration of the oil phase. $\Delta w'_{aM}$ was calculated by taking into account the equilibrium concentrations given by Equation 5 (16). Note that by dividing the K values by the density of the oil phase, given in Table 1, we obtain the overall mass transfer coefficient in m/s, which is an entity frequently used in the literature (17).

We treated our system as a pseudo-quaternary one, composed of ethanol, water, a triglyceride equivalent to corn oil, and a fatty acid equivalent to commercial oleic acid. Such an approach was successfully used by Batista *et al.* (7), for modeling the phase equilibrium of systems composed by canola oil, commercial oleic acid, and short-chain alcohols. The equivalent fatty acid was the main pseudocompound that moved away from the oil phase to the alcoholic phase. In a system in which only this pseudocomponent was transferred from one phase to another, the use of concentrations and dispersed phase mass flow on a fatty acids-free basis guarantees that the equilibrium and the operating curves are straight lines (16).

The volumetric mass transfer coefficients are given in Figure 3. Their values increased as the rotor speed and the oil phase flow increased, as a consequence of the higher turbu-
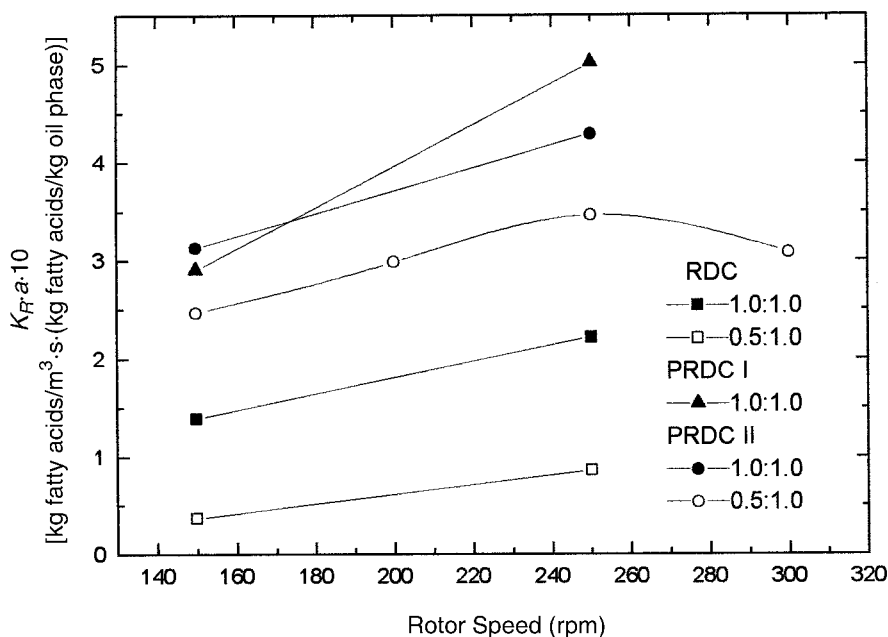
FIG. 3. Volumetric mass transfer coefficients (Eq. 7) as a function of rotor speed. K_R, overall mass transfer coefficient; a, mass transfer area per unit of extraction zone volume; for other abbreviations see Figure 1.

lence level in the apparatus. The volumetric coefficients for both PRDC versions are higher than the corresponding ones for the RDC, owing to the improvement of the mass transfer area in the columns equipped with perforated discs.

It should also be mentioned that we have not taken into account the influence of the axial mixing in the mass transfer calculations. Axial mixing reduces the extraction efficiency, and its value, given by the coefficient of axial mixing, increases as the rotor speed increases. To include such an effect in our calculations, it would be necessary to perform special experiments for measuring the coefficient of axial mixing for each phase as reported in detail by Coimbra *et al.* (18).

From a technological point of view, it is also important to discuss the results of the mass transfer experiments in relation to the total amount of extracted fatty acids (q_a), the loss of neutral oil in the extract stream (l_o), and the concentration of fatty acids in the refined oil. The values for q_a and l_o were calculated by performing mass balances for both types of fatty compounds. In the case of fatty acids, the following equation can be derived:

$$q_a = 100\% \times \left(w_{Fa} \times F - w_{Ra} \times R\right)/\left(w_{Fa} \times F\right) \qquad [8]$$

where w_{Fa} and w_{Ra} are the weight fractions of fatty acids in the feed and raffinate, respectively. In the same way, F and R are the total mass flow rates of the feed and raffinate.

The values for q_a varied in the range of 30 to 94%, and the highest ones were obtained using both PRDC versions. The values increased with increasing rotor speed and decreasing phase ratio. The values for l_o showed the same behavior and varied in the range of 2.0 to 5.9%. For the best results concerning the recovery of free fatty acids (PRDC II, 33 discs,

flow ratio 0.5:1.0, and 250 rpm), the loss of neutral oil varied in the range of 4.5 to 4.9%. It should be emphasized that those values for loss of neutral oil are significantly lower than the results reported in the literature for alkali or physical refining of corn oil (1,4). In liquid–liquid extraction, the loss of neutral oil is mainly determined by the oil solubility in the solvent. According to the equilibrium data, the oil solubility in aqueous ethanol varies from 1.6 to 2.5 wt% as the fatty acid content changes from 0 to 5 wt%. In this case, using an oil/solvent flow ratio of 0.5:1.0, the maximal loss of neutral oil in the extract stream must be approximately 5%.

Table 2 gives the oleic acid concentration in the refined oil as a function of oleic acid content in the feed stream for the best results regarding free fatty acid recovery. Considering that our main concern is the fatty acid content in the refined oil, not in the raffinate stream, the concentrations given in Table 2 are expressed on a solvent (aqueous ethanol)-free basis, calculated according Equation 9:

$$w_a^* = \frac{w_a}{1 - w_s} \qquad [9]$$

TABLE 2
Oleic Acid Concentration (wt%) in the Refined Oil[a]

Oil feed stream (100% w_{Fa}^*)	Refined oil (100% w_{Ra}^*)
4.36	0.48
3.46	0.17
2.22	0.10
1.48	0.07

[a]Perforated rotating disc contactor II, without stator rings; flow ratio, 0.5:1.0; 33 discs; 250 rpm.

where w_a and w_s are the fatty acid and solvent weight fractions, respectively.

According to the *Codex Alimentarius* (19), a good edible oil refining process must achieve a concentration of free fatty acids in refined oil less than 0.3%. Batista *et al.* (20) simulated the deacidification of vegetable oils by liquid-liquid extraction and determined optimal operating conditions for obtaining a minimal concentration of free fatty acids in the refined oil with minimal loss of neutral oil. Their simulation results indicated the feasibility of the total deacidification of vegetable oils by liquid-liquid extraction.

Unfortunately, no experimental work concerning continuous extraction of fatty acids from edible oils has been reported in the literature to date. The experimental results of the present work confirm the simulation results obtained by Batista *et al.* (20) and reassert doubtlessly the feasibility of the total deacidification of edible oils by solvent extraction. For the experiments performed with a flow ratio of 0.5:1.0, the PRDC II, 33 discs, and 250 rpm, we have obtained a refined oil containing less than 0.3 wt% of fatty acids, provided that the fatty acid concentration in the feed stream was not higher than 3.5 wt%.

ACKNOWLEDGMENTS

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