



Article A Newly Developed Empirical Predictive Model for the Dispersed Phase (DP) Holdup in Rotating Disc Contactors

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Abstract: Newly novel developed correlations were derived to predict the dispersed phase (DP) holdup in a rotating disc contactor (RDC) extraction column. DP holdup is one of the significant parameters in the design of liquid-liquid contactors and for calculating their production capacity. Despite the availability of quite a large number of holdup prediction correlations for the RDC, most of these correlations are either general in nature or valid for a limited range of operating conditions. This study conducted an experimental and theoretical investigation of the RDC holdup under the influence of varying geometries, including variations in the dispersed phase distributor, speed of the disc, flow rate, and physical characteristics of the system. The analysis revealed that the holdup decreased with an increasing distributor hole diameter and increased with an increasing disc speed and total flow rate. The effect of the physical properties on the holdup was larger than the effect of the disc speed. Using the measurements of over 150 runs, two RDC column holdup predictive models were proposed and evaluated. The first correlation was derived in terms of the distributor hole diameter, operating parameters, system physical properties, and column geometry. The second correlation excluded the column geometry. These correlations, which consider the distributor hole inlet diameter in predicting the DP holdup for an RDC column, were presented for the first time in this study. The predictive capability of these correlations was evaluated via their standard deviation (SD) and mean average percentage error (MAPE). The respective SD and MAPE of the two correlations were 1.7 and 5.2% for the first correlation and 1.6 and 11.4% for the second.

Keywords: dispersed phase; extraction; holdup; mathematical modeling; rotating disc contactor

1. Introduction

Liquid–liquid extraction is increasingly utilized in chemical, pharmaceutical and petrochemical applications for recovering of large- and medium-sized solutes, which are difficult to separate by other techniques. The state of the art for the extraction columns design is to predict the dispersed phase holdup (DP) and include it in the forward or back mixing models, where this parameter accounts for deviations from the ideal plug flow behavior. Acquiring highly efficient counter-current liquid–liquid extraction columns design depends mainly on an excellent mixing in the continuous phase and perfect dispersed drop size distribution. This is challenged by the nature of the dispersed phase holdup which is an essential parameter in the design of extraction columns was reported by Al Rahawi and Al Dawery [1]. They reported the importance of dispersed phase inlet distributor on controlling the droplet size and holdup, which finally affect quantitatively the mass transfer rate inside the column. Al-Aswad et al. [2], checked the application of drop size



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). distribution and mass transfer on column performance, using a large, 45 cm-diameter rotating disc contactor (RDC) column, on the column performance. Kirou et al. [3] studied the hydrodynamics of a column extractor using a toluene/water system, to monitor the holdup profile in the proximity of flooding. This leads to formulate criteria for the prediction of flooding in the extractor. Slater M. J. [4] reported that the holdup is a main factor required to determine the relationships between the phase flow rates, the interfacial area and the internal capacity of the columns. Kalaichelvi and Murugesan [5], used a large bank of published literature, from 13 different sources with a wide range of variables, to estimate the different regions of effective operation in extraction column, in terms of rotor critical speed, along with the effect of the geometry factor and no holdup and solute transfer in the column. Korchinsky and Ismail [6] used experimental data from two rotating-disc contactors to study the influence of different variables on the generating of the dispersed phase and continuous-phase mass-transfer coefficients, using the forward-mixing model. Only disc speed was proved to have a significant influence on the interfacial area and the hydrodynamics of the columns in the small column, whereas the larger column mainly depended on flow rate and the distributor used to control drop size. The flow capacity and nature of the mixing of the two phases in the extraction column plays a role in determining the column's size. Increasing the mixing level results in an increase in the residence time of droplets inside the column due to the radial non-uniformity flow of the droplets [7]. Significant errors occur when conventional design correlations derived for the homogenous flow model are used to describe a non-uniform flow pattern with circulating droplets in heterogeneous flow contactors. Therefore, new models are needed to describe the non-uniform flow patterns in the liquid–liquid extractors [8].

Kumar and Hartland [9] used 611 experimental results for 29 liquid–liquid systems from 15 published data sources to derive four correlations to estimate DP holdup in rotating disc extractors in the presence and absence of mass transfer. The suggested correlations take the following general form shown in Equation (1):

$$\varnothing = [k_1 + k_2 (\frac{DN^2}{g})^{n_1}] (\frac{D_s^2 C_h \rho_c g}{D_c^2 \sigma})^{n_2} (\frac{\Delta \rho}{\rho_c})^{n_3} (\frac{\mu_c g^{0.25}}{\rho_c^{0.25} \sigma^{0.75}})^{n_4} (\frac{V_d^4 \rho_c}{g \sigma})^{0.22} (1 + \frac{V_c}{V_d})^{0.35}$$
(1)

The values of k_1 , k_2 , n_1 , n_2 , n_3 and n_4 constants are given in Table 1, for three cases of; (1) no solute transfer, (2) solute transfer from the continuous to the dispersed phase (c–d) and (3) in the opposite direction (d–c). In addition, the authors also proposed a single general equation to be utilized to depict the cases mentioned above in the presence and absence of mass transfer. They claimed that the equations were more precise compared with those previously reported based on the concept of characteristic velocity.

Mass Transfer Direction \mathbf{k}_1 \mathbf{k}_2 n₁ n_2 n_3 n_4 (case 1) No solute transfer 570.53 747.78 1.28 -0.45-0.580.85 (case 2) $c \rightarrow d$ transfer 6.4 14.08 1.28 -0.16-0.680.29 -0.78(case 3) $d \rightarrow c$ transfer 19.68 60.00 0.68 -0.170.64 (case 4) General for above three cases 65.73 74.2 1.24 -0.34-0.490.53

Table 1. Values of dispersed phase (DP) holdup of Kumar and Hartland correlation under various mass transfer methods.

The same authors [10] also reported that distributor hole size and continuous phase density were not included in the analysis, since they did not vary appreciably in the experiments. The effect of interfacial tension was investigated and found to be insignificant, even over a wider range of operating conditions.

In another related study, Kumar and Hartland [11] used published experimental results for eight different types of extraction column. Namely the rotating disk, asymmetric rotating disk, Wirz-11, pulsed perforated-plate, Karr reciprocating-plate, packed, and spray columns, in order to derive a unified correlation for the determination of dispersed-phase

holdup. Analysis of the data shows that the holdup can be expressed in terms of the mechanical power dissipation, phase flow rates, physical properties, and column geometry. The average error on the entire data set is 18.1%, which is better than that achieved by most researchers in attempting to correlate their own experimental results. The highest error recorded, was 22.7%, for rotating disk and asymmetric rotating disk columns. The lowest error of 14.1% was achieved for the spray columns. Korchinsky [12] reports that the accuracies of the literature correlations used in the "axial dispersion model" and the more complex "forwarding mixing model models, for prediction of the hydrodynamic and mass transfer parameter in an RDC column, includes holdups that are generally poor, especially for the low interfacial tension system of n-butanol–succinic acid–water. Murakami et. al. [13] proposed a correlation to predict dispersed phase holdup in a rotating disc extraction column which is primarily based on the system physical properties and column geometry:

$$\varnothing = 3.3 \left(\frac{\Delta\rho}{\rho_c}\right)^{-0.13} \left(\frac{DN^2}{V_c}\right)^{0.55} \left(\frac{V_d}{V_c}\right)^{0.8} \left(\frac{D_c V_c^2 \rho_c}{\sigma}\right)^{0.18} \left(\frac{V_c^2}{g D_c}\right)^{0.6} \left(\frac{D}{D_c}\right)^{0.4} \left(\frac{C_h}{D_c}\right)^{-0.6} \left(\frac{D_c^2 - D^2}{D_c^2}\right)^{-0.3}$$
(2)

Hemmati, et al., [14] investigated the behavior of dispersed phase holdup and flooding point in a pilot plant scale, perforated rotating disc contactor (PRDC), using three different liquid–liquid systems. They derived empirical correlation for a dispersed phase holdup based on operating variables as follows:

$$\varnothing = 0.089 \left(\frac{N^2 D_r}{g}\right)^{0.254} \left(\frac{D_r}{C_h}\right)^{0.256} \left(\frac{\mu_d}{\mu_c}\right)^{0.272} \left(1 + \frac{V_c}{V_d}\right)^{-1.04}$$
(3)

The proposed empirical correlation was validated by comparing it with experimental data. It was reported that the predictive correlation can be utilized for the perforated rotating disc contactors design [13]. Al Rahawi, A.M. [15] insisted that besides the calculation of the holdup, the calculation of the droplet size along the column height is decisive to describe the nature and behaviour of mass transfer and hydrodynamics parameters in liquid extraction column. Sá et al. [16] measured the dispersed phase holdup in a liquid–liquid extraction column using two columns with the same diameter (0.092 m) but with different lengths. The first had a height of 0.70 m with three stages, while the second had a height of 0.90 m with four stages. An empirical correlation was proposed for calculating the DP holdup in terms of column height and operating conditions. Shahrokh et al. [17] investigated the impact of operating parameters on the dispersed phase holdup in a liquid-liquid Scheibel extraction column. Three chemical systems (butyl acetate/ H_2O , toluene/ H_2O , and n-butanol/H₂O) were used to study the holdup characteristics. Another empirical correlation calculated the DP holdup with an average absolute relative error of 8.72%. Mirmohammadi et al., [18] investigated the effects of operational parameters on the mean drop diameter, the dispersed phase holdup and the slip velocity a horizontal pulsed sieve plate column. It was found that an increase in the pulsation intensity led to a decrease in holdup and d_{32} , and an increase in the phase flowrates cause an increase in both holdup and d_{32} , although the impact of the flow rates on the mean drop diameter was much milder than the pulsation intensity. Also, it was observed that the slip velocity increases by increasing the pulsation intensity and the phase flowrates. They proposed three experimental-theoretical correlations for predicting mean drop diameter, the dispersed phase holdup and the slip velocity based on the experimental results and dimensional analysis. The average absolute relative errors (AARE) of these parameters were 9.90%, 14.45%, and 8.58%, respectively, signifying their accuracy and usefulness for scaling up a horizontal pulsed sieve plate column. Moreover, the hydrodynamic behavior of the reactive extraction system in a horizontal pulsed sieve-plated column was also studied by Khanramakia et al. [19]. The effects of operating parameters and reactive condition were investigated. It was showed that an increase in pulse intensity caused the decrease of dispersed phase holdup and increase slip velocity. They demonstrated from the comparison between their experimental

holdup results with the proposed correlations obtained from the previous studies that the previous correlations have reasonable accuracy to prognosticate the holdup as well as the characteristic and slip velocities in this column. The novel correlations were offered to prognosticate the holdup of the dispersed phase as well as to the characteristic and slip velocities for the reactive uranium extraction situations in the horizontal column, as functions of the physical properties of systems and operating conditions. They found that the AAREs of these parameters were 9.87%.

According to the aforementioned, comparison between the operating conditions of the RDC of the present study and various studies found in the literature are summarized in Table 2.

Table 2. Comparison between the operating conditions of the present study and various studies found in the literature.

Continuous Phase Flow (cm ³ /s)	Dispersed Phase Flow (cm ³ /s)	Disc Speed (r.p.s)	Ref.
3.45-9.45	4.15–10.59	3.5-11.86	[1]
19.5–52.3	23.456-95.7	2.9-6.8	[8]
11.6-231	5.8-126.7	3–32	[9]
32–45	25.9-43.27	3.67-13.33	[13]
38.29-95.82	72.07-92.54	5–7	[14]
2.57-57.2	3.38-80.0	1.3-9.7	[20]
20.2-48.2	26.7–90.0	4.0-6.3	[21]
2.76-9.08	4.3–11.1	4.8–11.83	This work

From the above, the available reported correlations for DP holdup are specific to the column type and geometry. More studies need to be conducted for better holdup modeling. Therefore, an experimental and theoretical investigation was conducted to study the holdup in an RDC under the influence of varying geometries, including the following: DP distributor, disc speed, flow rate, and physical characteristics of the system. Two RDC column holdup predictive models were proposed and evaluated. In this study, new correlations for calculating the DP holdup in the RDC column were developed and presented.

2. Experimental Method

The objective of the present work is to develop a correlation that predicts the DP holdup in RDC columns. To generate the data required to determine the most significant variables, experiments were designed that controlled the holdup in an RDC, so that these variables could be used to examine the validity of the proposed correlations. In this work, over 150 experimental runs were conducted. Most of these runs were performed in both the presence and also the absence of mass transport. Most runs used a toluene–water–acetone system. For experiments with mass transfer, acetone as a solute was transported from a continuous water phase to the dispersed phase of toluene. However, for the experiments without mass transfer, the toluene was dispersed into water (continuous phase). For both cases, toluene was dispersed into water to prevent the adhesion of large or small drops dispersed in the aqueous phase. A sample of the data is provided in the supplementary file (Appendix A).

2.1. Experimental Setup

Two pilot plant liquid–liquid extraction RDC columns with diameters of $D_c = 7.62$ cm and 21.9 cm for small and large columns, respectively, were used to collect data. The dimensions of the small column were: stator ring diameter (D_s) of 4.5 cm, disc diameter (D) of 4.0 cm, compartment height (h_c) of 2.54 cm, and column working height (Z) of 73 cm, whereas the large column dimensions were: stator ring diameter (D_s) of 12.934 cm, disc diameter (D) of 11.496 cm, compartment height (h_c) of 7.3 cm, and column working height (Z) of 150 cm. Each column consisted of a QVF[®] glass column shell equipped with two cribriform stainless steel distributors for the continuous and dispersed phases, placed at



opposite ends of the column. The flow diagram of the equipment and the dimension of the RDC column are given in Figure 1.

Figure 1. Schematic diagram of the rotating disc contactor (RDC) layout.

The small column was fitted with two stainless steel perforated-distributor plates placed at opposite ends of the column, one for the dispersed phase and the other for the continuous phase A description of the four various distributors used in this effort is presented in Table 3.

Table 3. Details of dispersed phase distributors.

Distributor No.	Α	В	С	D
Average hole diameter D _h (mm)	0.80	1.00	1.22	1.50
Holes number	19	12	8	5
Hole Area (mm ²)	0.503	0.786	1.17	1.77
Total holes area (mm ²)	9.55	9.43	9.36	8.84

2.2. Experimental Procedure

The DP holdup was measured by shutting off all valves around the column simultaneously; therefore, water and toluene were trapped in the column. The displaced dispersed phase fraction was collected and we measured its volume.

To validate the proposed correlations, the results of this study were compared with data from two previous works that employed a similar experimental operating conditions, procedure, and technique. The first set of data was reported by Korchinsky and Bastani [20], who used an n-butanol–succinic acid–water system with a low surface tension value. These data were chosen to investigate the impact of the physical characteristics on the holdup. At the same time, the data of Korchinsky and Al-Husseini [21], who used a bigger column of 219 mm diameter, were chosen to test the impact of the column diameter on the holdup.

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3. Results and Discussion

Quantifying the holdup in the RDC column is a key step in the design of the contactor. This is because holdup represents the active portion of the dispersed phase in contact with the continuous phase.

One of the study's goals was to examine the influence of the column geometry, operating conditions, and physical properties on the dispersed phase holdup. Two new correlations to calculate the DP holdup in the RDC liquid–liquid extraction column were developed and tested with two-column geometries for the two systems.

To obtain a uniform initial drop size and size distribution in the continuous countercurrent extraction columns, the phase being dispersed is invariably introduced as the continuous phase via a distributor plate unit. These distributors were reported to mainly control the hydrodynamics and mass transfer parameters, including the holdup in the RDC [1,15].

3.1. Correlations Proposed

It is obvious that measuring the DP holdup in a liquid–liquid extraction column at an industrial scale is challenging, costly, and burdensome. There are many reasons for the lack of a fundamental theoretical correlation to predict the holdup. One of the reasons is the complex nature of the multiphase flow within the RDC column resulting from the many interactions between the phases. Most of the available holdup correlations ignored the distributor hole diameter or the drop sizes, and some other correlations were based on the characteristic velocity, which can only be evaluated by trial and error. This forced the designer to expend extra effort to minimize the prediction error.

The objective of this study was to obtain a reliable correlation for the calculations of DP holdup based on the distributor hole diameter, geometry of the column, disc speed, flow nature, and physical characteristics of the system.

Different statistical methods were used to derive the new correlations. The matrix method was used to obtain the values of the correlation's constant and exponents. Meanwhile, to determine the essential parameters which influence the holdup in the RDC column, the upper triangle of Spearman's method and lower triangle of Kendall's method were utilized. The DP hole distributor, flow rate, disc speeds, and physical properties were mainly found to control the holdup values. Neglecting any one of these variables can cause serious prediction errors. The methods of regression analysis and the least square method were employed to determine the correlation's constant and exponents.

Multiple linear regression was used to generate prediction equation for DP holdup from the most effective independent variables pointed out in the statistical analysis.

The minimum sum of squared differences obtained, based on all data, was below 1×10^{-6} . This minimum was obtained with the number of iterations varying between 33 and 89.

Precise correlations were proposed for the prediction of the DP holdup in the RDC extraction columns. The first was derived in terms of the distributor hole diameter, column geometry, disc speed, physical properties of the system, and operating conditions. The proposed Equation (4) can be used to estimate the dispersed phase holdup data for existing RDC columns. It was validated for RDC columns up to 22 cm in diameter.

$$\phi = 0.037 \left(\frac{U_d^{1.05}}{U_c^{0.2}}\right) \left(\frac{N}{h_d}\right)^{0.325} \left(Q_d + Q_c\right)^{0.175} \left(\frac{\sigma}{g\Delta\rho}\right)^{0.25} Q_D^{0.028} Q_C^{0.001} \frac{C_h^{0.04}}{\left(D_c - D\right)^{0.3}} \tag{4}$$

Similar variables were considered in the second correlation Equation (5), except that the column dimensional terms are lacking. This correlation can be used for design purposes as it is based on variables that are known in advance, such as flow rate and physical properties:

$$\phi = 0.0038 \left(\frac{Q_d^{1.065}}{Q_c^{0.901}}\right) \left(\frac{N^{0.5}}{h_d^{0.25}}\right) \frac{\left(\frac{\sigma}{g\Delta\rho}\right)^{0.205}}{\left(Q_d + Q_c\right)^{0.01}}$$
(5)

The proposed correlations are the first of their kind that account for the distributor inlet hole diameter in the RDC column. The distributor hole diameter proved to primarily control the drop size but also the holdup [15]. This parameter was neglected by previous investigators.

The first term in the new correlation $(V_d^{1.05}/V_c^{0.2})$ represents the ratio of the superficial velocities of the two phases. The dispersed phase velocity V_d provides information about the time the drops need to travel through the continuous phase. The continuous phase will increase the drag force on the dispersed phase droplets and slightly decrease their velocity, which consequently increases the dispersed phase holdup.

The term $(N/h_d)^{0.325}$ in the first correlation represents the ratio of the disc speed to the dispersed phase hole of the distributor. In the literature [1], there have been few attempts to quantify the importance of the DP inlet distributor, which was reported to control the DP drop sizes [14]. The disc speed is known to decrease the drop size and increase the drop rotation within each compartment of the column, which leads to an increase in the drop residence time and eventually increases the holdup. The smaller inlet distributor hole generates smaller drops with lower velocities.

In this study, the influence of the physical properties was represented by the third term in the derived equations, $(\sigma/g\Delta\rho)^{0.05}$. This term correlates the system properties to the disc speed and column geometry. Furthermore, it reflects the importance of the physical properties on the drop size, drop size distribution and finally on the nature of the two phases flow within the column. Small density differences would result in a very high holdup as well as low drop velocities; such a situation overestimates the needed column volume, causing a design flaw. Nonetheless, experimentally, Rincon-Rubio et al. [22] reported that there is an insignificant impact of the difference in density ($\Delta\rho$) on the size distribution. The effect of integrating physical property relationships, mainly surface tension (σ) and the difference in density ($\Delta\rho$), into the equations is to increase the reliability and accuracy of the correlation.

There was a limited variation in the effect of the magnitude of the last term $(C_h^{0.04}/(D_c - D)^{0.3})$ on the geometry of the column. As seen in the second correlation, Equation (5), it seems that this effect can be ignored to simplify the equation in the design phase. This will prevent the designer from assuming the column diameter and having to follow a tiresome iteration process. Korchinsky and Ismail [6] and Al-Rahawi [15] showed that the impact of column diameter on d₃₂ was trivial.

The mean absolute percentage error (*MAPE*) is employed in this work to measure the prediction accuracy of the proposed holdup models. The MAPE is a statistical validation method for trend estimation. It expresses accuracy as a percentage and is defined by the following formula:

$$MAPE = \frac{100}{n} \sum_{i=1}^{n} \left| \frac{A_i - F_i}{A_i} \right|$$
(6)

where A_i is the measured holdup value, and F_i is the predicted value [20].

Finally, the data for the regression analysis were the confidence index for Equations (4) and (5) which was 95 and 85, respectively. The correlation coefficient was +9 and +0.85, for Equations (4) and (5), respectively, and the regression coefficient was 0.9 and 0.8, for Equations (4) and (5), respectively.

3.2. Applicability of the Proposed Correlations

One of the criteria used in this work to check the reliability of the present correlations depended on the ability of the correlation to reproduce the experimental results with a minimum relative deviation of less than 5% under all conditions. To check the applicability and reliability of the proposed correlations, they were examined with four various distributors, using two column sizes and systems.

3.2.1. Effect of Dispersed Phase Distributor

The results demonstrate that the holdup was drastically affected by the diameter of the DP hole distributor, phase flow rates, disc speed, and interfacial tension. The holdup was also affected in a significant but weaker manner by the continuous phase flow rates.

The influence of the inlet hole distributor on the DP holdup fractions was tested. In runs represented in Figure 2, the DP holdup produced in the RDC column was significantly affected by the distributor devices employed. A higher dispersed holdup was achieved with smaller diameters of the distributor hole. This is because a larger drop size was produced by hole distributors with larger values [1,15]. Large drops have a higher velocity and shorter residence time, which results in lower holdup. As seen in Figure 2, the proposed Equation (4) responded well to the change in the distributor diameter and generated holdup values close to those measured. The values reproduced by the proposed equation showed a reasonable match between the measured and predicted holdup values.



Figure 2. Measured and predicted holdup influence of distributor hole diameter. Measured holdup (symbols) and predicted holdup (lines).

3.2.2. Effect of Flow Rate and Disc Speed

Figure 3 presents the measured and predicted holdup drawn as a function of the total flow. This figure illustrates that the experimental holdup increased as the flow rate increased. This increase in the holdup could be attributed to the increase in the dispersed phase fed to the column and the reduction in drop sizes balanced by the drag force exhibited by the continuous phase. The reproduced values of the experimental data generated by the new correlation were drawn as a function of the total flow rate. The overall results showed a high match between the measured and predicted values.

The impact of the continuous phase on the holdup was tested, and the results for the two distributors are illustrated in Figure 4. The measured and predicted results displayed in the figure indicate that the holdup was weakly affected by the increasing continuous phase flow rate. The proposed correlation reproduced the same trend and was in good agreement with the measured one.



Figure 3. Dispersed phase holdup versus total flow rate, for the values experimentally measured and the theoretically predicted, for all distributors.



Figure 4. Dispersed phase holdup, effect of continuous phase flowrate at a disc speed of 9.666 rps. Measured holdup (symbols) and predicted holdup (lines).

Meanwhile, the effect of the disc speed on the holdup was examined, and the results are illustrated in Figure 4. The measurements were performed using a small column for four distributors using three levels of flow rate (low: 7.759 cm³/s; medium: 13.13 cm³/s; and high: 20.03 cm³/s). The experimentally measured holdup increased with increasing disc speed. The increase in the holdup can be attributed to the reduction in the drop

diameter and to an increase in the turbulence level arising from the resistance to flow that originated from the disc action.

These results were reproduced efficiently by the new correlation, as depicted in Figure 5. The comparison between the experimental and theoretical holdup values clearly reflects the accuracy of the correlation to reproduce identical holdup values as a function of disc speed. There was a high degree of consistency and similar trends when comparing the theoretical values with the experimental holdup measured under variable operating conditions.



Figure 5. Dispersed phase holdup versus disc speed for the experimentally and theoretically predicted values. Low total flowrate (solid lines), medium total flowrate (long dash lines), high total flowrate (dotted lines).

The DP holdup was affected more by the flow rate changes than by the disc speed in spite of the minor change in the mean size of the drop with the former and the sharp change with the latter. This occurrence could be attributed to two primary parameters: (1) the drag forces caused by the counter-flow, and (2) the axial and radial mixing caused by the rotation of the disc. These two parameters led to an increase in the residence time spent by the drop fractions in the column, leading also to increased holdup.

3.2.3. Effect of Column Diameter

To check the applicability and validity of the suggested correlations, experimental values obtained from the small (7.62 cm diameter) and large column (21.9 cm diameter) were compared with predicted holdup values obtained from the new correlation. The results are illustrated in Figure 6, where the data from both columns is in good agreement for all distributors as observed around the 45° line. However, a relatively marked deviation of +7.5% was observed for the data obtained from the small distributor (0.1 cm hole diameter) at a high flow rate.

With the large column, quite a good agreement was noted at low flow rates only, and the predicted values deviated from one another as the flow rate increased. The extent of the variation in the holdup was not expected to be identical for the same variation in the d_{32} values because the latter does not exactly represent the reality of drop size distributions inside the extractor.

As clarified above, the last term in the proposed model equation involved the scale up factor which is represented as the ratio of compartment height to the difference in column and rotating disc diameter. The applicability and validity of the two new model correlations to the scale-up parameter were tested using different size columns using different systems from different workers; the holdup data predicted from all columns



were in good agreement with the experimental results with relatively marked deviation of +7.5% observed.

Figure 6. Comparison of the experimentally measured and predicted dispersed phase holdup values (using Equation ((4)), from the small and large column, for all four distributors, using the TAW (toluene–acetone–water) and BSW (butanol–succinic acid–water) system.

3.2.4. Effect of Chemical System

To check the capability of the physical characteristics term and the validity of the new correlation, two physical systems were used: BSW (butanol–succinic acid–water) and high interfacial tension TAW (toluene–acetone–water). Figure 7 shows the comparison between the values predicted by the new correlation with the experimental data from the two systems. The figure shows a remarkable overlap of the predicted values with those experimentally measured in the two columns, four various distributors, and under several operating conditions.

A sample of some runs was selected to compare the effect of the system properties on the column's dispersed phase holdup. The results are presented in Figure 8. As seen in Figure 8, higher dispersed phase holdup values were observed with the BSW system, which exhibited a lower interfacial tension than was obtained from the TWA system. This can be explained by the larger drops generated from the higher interfacial tension system (i.e., TWA) than what was produced by the lower interfacial tension system (i.e., BSW).

This variation in holdup is mainly attributed to the variation in the interfacial tension and distributor hole diameter. This is contrary to what was reported by Kumar and Hartland [11] who stated that the interfacial tension of the chemical system and the distributor hole size have an insignificant effect on the holdup.



Figure 7. Dispersed phase holdup experimentally measured and predicted in small (7.62 cm) and large (21.9 cm) RDC, effect of TAW and BSA systems' physical properties, (distributors I–IV).



Figure 8. Dispersed phase holdup experimentally measured in small (7.62 cm) and large (21.9 cm) RDC, using the BSW system (distributors I–IV).

3.2.5. Validating the Holdup Correlation with the Reported Data

The new correlation was tested to reproduce the reported holdup data, using the data of Korchinsky and Bastani [20] and Korchinsky and Al-Husseini [21] selected for that purpose. Both works employed the same experimental technique and columns in collecting their experimental data. The first work used the low interfacial tension system (BSW) as well as the high interfacial tension system (TAW). Figure 9 illustrates that the results were highly consistent. Additionally, trends for all data from the two correlations reproduced the holdup data from the two columns and two chemical systems with less than 7% difference, except for a few odd points; this might have occurred because the second correlation does not include the internal column dimensions.



Figure 9. Comparison of the predicted holdup from the two correlations (4 and 5) with the measured values of (Korchinsky and Bastani 1993) [20]; and (Korchinsky and Al-Husseini 1986) [21].

3.2.6. Effect of the Geometry Term

The effect of the geometry term on the holdup was tested using Equation (5). The same data from the large and small columns were used to test the Equation (5). However, as seen in Figure 10, some systematic deviation in the holdup was observed. The deviation was apparent with some experimental runs conducted at high flow rates. This reflects the need to include the geometry term in any design correlation.





For validating the new Equation (5), samples for some runs were drawn in a separate subplot, as seen in Figure 11. The trends measured and that predicted by Equation (5) were in good agreement.



Figure 11. The measured and predicted dispersed phase holdup values (from Equation (5)) obtained from the small and large columns, for different distributors and operating conditions.

3.3. Comparing the Predictions of the New Correlation with Those Reported in the Literature

The proposed new correlations were then validated by comparing their results with those previously proposed by other researchers. The measured data and the data predicted using Equations (1)–(5) were compared, as shown in Figure 12, where data predicted by the Murakami and Hemmati equations (Equations (2) and (3)) were ignored as the values produced were over predicted, with more than 100% error in comparison to the measured values. The comparison produced a good agreement, and close trends were observed between the measured holdup and that calculated by the new correlation. Only Kumar's equation (Equation (1) for case 4 shown in Table 1) gave a relatively reasonable agreement with the measured data. However, a large deviation was observed for the other three cases (i.e., no solute transfer; c to d transfer; and d to c transfer).

Using the new correlations, the MAPE between the measured and predicted holdup values was 5.2%. This compares to a MAPE of 25% for the Kumar general case correlation (Equation (1) with case 4 shown in Table 1). The MAPE for Kumar's correlations of Equation (1) with c to d transfer (case 2) and Equation (1) with d to c transfer (case 3); were 54% and 92.4%, respectively. The maximum MAPE of 154% was attained by the case of no solute transfer for Kumar's correlation (Equation (1) with no solute transfer (case 1) as shown in Table 1). Meanwhile, the Murakami and Hemmati correlations resulted in a MAPE of 120% and 198%, respectively.

For all data analyzed and used in this work, the new correlations (Equations (4) and (5)) reproduced the experimental results with a maximum standard deviation of ± 1.7 and ± 1.6 , respectively.

In summary, most of the previous dispersed phase holdup correlations that had been applied to determine the RDC failed to represent the actual holdup in an RDC column. This is due to the fact that they were primarily based on the physical properties, characteristics, or the superficial velocity and did not include the inlet distributor geometry effect.



Figure 12. Measured and predicted holdup data for the new and the reported correlations (dots represent the measured holdup). (**A**) For the small column (flowrate = $7.759 \text{ cm}^3/\text{s}$, distributor = 0.15 cm) and (**B**) the large column (flowrate = $80.3 \text{ cm}^3/\text{s}$, distributor = 0.12 cm).

4. Conclusions

The DP holdup in a pilot plant rotating disc contactor (RDC) extraction column has been experimentally investigated, using 150 data, from two column sizes and two systems. The results indicated that the holdup was drastically affected by the dispersed phase inlet size distributor and geometry, flow rate, disc speed, and physical properties of the system. The analysis also showed that the effect of the disc speed was less pronounced than those of the distributor hole diameter and interfacial tension. Higher holdup was obtained with the low interfacial surface tension system (BSW) and with increased dispersed phase holdup, but this significantly weakened the effect of the continuous phase flow rate. Two newly accurate correlations were proposed to predict DP holdup, incorporating for the first time the distributor hole diameter in addition to the flow rate, disc speed, superficial velocities, column geometry, and system physical properties. The predictive correlations were successfully checked to reproduce the experimental data for different column sizes and for different systems. Both new correlations reproduced the experimental results with a maximum standard deviation of 1.7 and 1.6, respectively. Additionally, the average standard error for the first and second correlation was 5.2% and 9.4%, respectively. The first correlation can be used for design purposes, while the second can be used to collect data about existing columns. The comparison of the new correlation with currently reported correlations showed that the new correlations were more accurate in predicting the holdup.

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Nomenclature

Symbols Used		
C _h	[cm]	Stage height, or compartment height
d ₃₂	[mm]	Average mean drop diameter
D _c	[cm]	Column diameter
D, D _R	[cm]	Rotating disc diameter
Ds	[cm]	stator opening diameter
h _d	[cm]	Dispersed phase distributor hole diameter
Ν	$[s^{-1}]$	Disc speed (revolution per second)
Q _d	$[cm^3 s^{-1}]$	Dispersed phase flow rate
Qc	$[cm^3 s^{-1}]$	Continuous phase flow rate
V _d	[cm/s]	dispersed phase superficial velocity
V _c	[cm/s]	continuous phase superficial velocity
Greek Letters		
ρ_c	$[g cm^{-3}]$	Continuous phase density
ρ_d	$[g cm^{-3}]$	Dispersed phase density
$\Delta \rho$	$[g cm^{-3}]$	Density difference $(\rho_c - \rho_d)$
μ_c	$[g s cm^{-1}]$	Continuous phase viscosity
μ_d	$[gscm^{-1}]$	Dispersed phase viscosity
σ	$[g s^{-2}]$	Interfacial surface tension
g	$[cm s^{-2}]$	Gravitational acceleration

Appendix A

Table A1. Toluene was dispersed into water to prevent the adhesion of large or small drops dispersed in the aqueous phase. A sample of the data is provided in Appendix A.

Run No	Disc Speed (rps)	Distributor Hole Diameter (cm)	Continuous Phase Flow Rate (cm ³ /s)	Dispersed Phase Flow Rate (cm ³ /s)	Measured Holdup
1	4.833	0.08	3.459	4.3	0.0443
2	6.666	0.08	3.459	4.3	0.049
3	9.666	0.08	3.459	4.3	0.0548
4	4.833	0.08	5.83	7.3	0.0704
5	6.666	0.08	5.83	7.3	0.0896
6	9.666	0.08	5.83	7.3	0.1116
7	4.833	0.08	7.787	9.68	0.1049
8	6.666	0.08	7.787	9.68	0.1184
9	5.9	0.08	3.87	4.3	0.0473
10	5.9	0.08	3.87	6.8	0.0693
11	5.9	0.08	3.87	8.1	0.0846
12	9.666	0.08	3.55	7.3	0.09983
13	9.666	0.08	4.915	7.3	0.1066
14	9.666	0.08	5.83	7.3	0.1116
15	9.666	0.08	9.08	7.3	0.11336
16	5.9	0.1	3.87	4.37	0.0433
17	9.666	0.1	3.87	4.37	0.054
18	11.833	0.1	3.87	4.37	0.0653
19	4.5	0.1	3.85	5.17	0.0443
20	5.9	0.1	3.85	5.17	0.047
21	8.4	0.1	3.85	5.17	0.0558
22	4.5	0.1	3.85	6.8	0.0598
23	5.9	0.1	3.85	6.8	0.0609
24	8.4	0.1	3.85	6.8	0.071
25	4.5	0.1	3.85	8.1	0.0744

Run No	Disc Speed (rps)	Distributor Hole Diameter (cm)	Continuous Phase Flow Rate (cm ³ /s)	Dispersed Phase Flow Rate (cm ³ /s)	Measured Holdup
26	5.9	0.1	3.85	8.1	0.0761
27	8.4	0.1	3.85	8.1	0.0863
28	9.666	0.1	3.87	6.11	0.0795
29	10.833	0.1	3.87	6.11	0.0856
30	11.833	0.1	3.87	6.11	0.0947
31	8.833	0.1	5.83	7.3	0.0829
32	9.666	0.1	5.83	7.3	0.0914
33	10.833	0.1	5.83	7.3	0.0947
34	11.833	0.1	5.83	7.3	0.0998
35	9.666	0.1	5.83	9.68	0.105
36	10.166	0.1	5.83	9.68	0.1245
37	8.833	0.1	5.83	11.1	0.1076
38	9.666	0.1	5.83	11.1	0.1201
39	8.4	0.1	3.85	5.17	0.0558
40	8.4	0.1	3.85	6.8	0.071
41	8.4	0.1	3.85	8.1	0.0863
42	9.666	0.1	5.83	7.3	0.09137
43	9.666	0.1	5.83	9.68	0.1049
44	9.666	0.1	5.83	11.1	0.1201
45	9.666	0.12	3.55	7.3	0.0765
46	9.666	0.12	4.915	7.3	0.0772
47	9.666	0.12	5.83	7.3	0.0778
48	4.833	0.12	3.459	4.3	0.0372
49	6.666	0.12	3.459	4.3	0.0413
50	9.666	0.12	3.459	4.3	0.0512
51	4.833	0.12	5.83	7.3	0.0609
52	6.666	0.12	5.83	7.3	0.069
53	9.666	0.12	5.83	7.3	0.0778
54	11.833	0.12	5.83	7.3	0.112
55	4.833	0.12	8.93	11.1	0.0913
56	6.666	0.12	8.93	11.1	0.1066
57	9.666	0.12	8.93	11.1	0.1218
58	5.9	0.15	3.87	4.3	0.0364
59	5.9	0.15	3.87	6.8	0.048
60	5.9	0.15	3.87	8.1	0.0534
61	4.833	0.15	3.459	4.3	0.0355
62	6.666	0.15	3.459	4.3	0.0379
63	9.666	0.15	3.459	4.3	0.0446
64	4.833	0.15	5.83	7.3	0.0602
65	6.666	0.15	5.83	7.3	0.0626
66	9.666	0.15	5.83	7.3	0.0727
67	4.833	0.15	8.93	11.1	0.0879
68	6.666	0.15	8.93	11.1	0.0896
69	9.666	0.15	8.93	11.1	0.0964

Table A1. Cont.

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