

# Study on the effect of Cetyltrimethylammonium bromide surfactant on static holdup and mother drops' size in rotating disc columns

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## ABSTRACT

Local and average static holdup and also mother drops' size of dispersed phase in rotating disc column (RDC) and rotating sieved disc column (RSDC) have been investigated experimentally for water-kerosene chemical system. Experiments consist of five different CTAB concentrations in continuous phase and for each concentration five different rotor speeds. The presence of used surfactant in continuous phase not only reduced amounts of local and average static holdup in both columns, but it also greatly affected the variation process of local static holdup in the columns. Besides, CTAB reduced mother drops' size in both columns. As rotor speed went up in RDC, local and average static holdup decreased. In RSDC column, enhancement of rotor speed from zero to 150 rpm caused increase of static holdup but when the speed of rotors went up more, this hydrodynamic parameter decreased.

**Key words:** *Liquid-liquid extraction, Rotary disc contactor, Static holdup, Mother drops' size, Surfactant, Cetyltrimethylammonium bromide (CTAB)*

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## ABBREVIATIONS

C, Concentration of CTAB  
D<sub>r</sub>, Rotor diameter (m)  
Fr, Froude number (Fr=  $\rho_c N^2 D_r / g \Delta \rho$ )  
g, Gravity (m/s<sup>2</sup>)  
Mo, Morton number (Mo=  $g \mu_c^4 \Delta \rho / \rho_c^2 \gamma^3$ )  
n, Stage number  
N, Rotor speed (rpm)  
NE, Number of experiments  
We, Weber number (We=  $\rho_c N^2 D_r^3 / \gamma$ )  
 $Y_{i,cal}$ , Calculated value of parameter  
 $Y_{i,exp}$ , Experimental value of parameter  
*Greek letters*  
 $\gamma$ , Interfacial tension (N/m)

$\Delta\rho$ , Density difference (kg/m<sup>3</sup>)

$\mu_c$  , Viscosity of continuous phase (Pa.s)

$\mu_d$  , Viscosity of dispersed phase (Pa.s)

$\rho_c$ , Density of continuous phase (kg/m<sup>3</sup>)

$\rho_d$ , Density of dispersed phase (kg/m<sup>3</sup>)

$\varphi_{L_n}$ , Local static holdup for nth stage

$\varphi_{ave}$ , Average static holdup

## 1. INTRODUCTION

Among different liquid-liquid extraction equipment, rotary disc columns are taken into account due to high efficiency, high throughput, operational flexibility and also low energy consumption that cause low energy costs[1]. Information on hydrodynamic parameters such as holdup, axial dispersion and drop size can be helpful to achieve an appropriate design for rotary disc contactors [2].

Holdup as one of the most important hydrodynamic parameters is reported in three kinds of static, dynamic, and total holdup. Efficiency of an extraction column can be affected by dispersed phase holdup considerably [3]. Since this parameter has a great influence on drop coalescence, residence time of dispersed phase, and interfacial area[1].

Based on studies, geometry of extraction column (rotors, stators and column diameter, compartment height, and etc.), operational conditions (continuous and dispersed phase flow rate, rotor speed, and etc.) and also physical properties of phases (density, viscosity, interfacial tension, and etc.) have considerable influences on hydrodynamic parameters such as dispersed phase holdup [1,4-8]. According to the studies done on RDC column by Molavi and Bahmanyar [9] , when rotors are spinning, as continuous phase flow rate is not zero static holdup shows a decrement in comparison with the state of no continuous phase flow. Enhancement of interfacial tension causes an increment in static holdup[1] but in contrast to that, dynamic holdup decreases [2].

On the other side, the presence of surface active agents in industrial equipment even in inappreciable amount can influence on hydrodynamic behavior of rotary disc columns. Inevitable presence of surface active agents in extraction contactors can influence even on mass transfer rate in these equipment [10, 11]. Surface active agent, briefly called surfactant, consists of a hydrophobic hydrocarbon chain (tail) and a hydrophilic group (head) in its molecular structure. Surfactants reduce mass transfer efficiency of extraction systems by reducing and even eliminating internal circulation of drops, increasing drag force, decreasing terminal velocity and creating interfacial resistance [11].Therefore, investigating hydrodynamic and mass transfer performance of extraction systems that are contaminated by surfactants can be beneficial for better understanding of these processes and design of respective equipment scientifically.

Studies in the effects of surfactants on interfacial tension in extraction systems have been seen in literature of the subject mainly after 1980 [12-15]. Touhami et al. [16] studied the effects of the presence of sodium dodecyl sulfate in acidic oil/alkaline systems. Regarding to findings, the presence of the surfactant in the

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system with concentrations lower than CMC reduced interfacial tension of the system more in comparison with the condition of concentrations higher than CMC.

Variation of mass transfer rate and coefficient of single drops, considering the presence of surfactants in system, has been examined experimentally by researchers [10, 11, 17, 18]. Li et al. [11] realized that by increasing concentration of surface active agents, mass transfer rate will reduce. Some other researchers tried to simulate the motion of a single bubble contaminated by surfactants [19-21].

Respecting to the importance of static holdup and also effects of surfactants in liquid-liquid extraction systems, the necessity of studying this parameter in the presence of surface active agents can be understood. However, by investigating previous researches on hydrodynamic of rotary disc columns, it can be realized that there has been almost no study on the influence of surfactants on static holdup in these contactors. Nevertheless, many researchers have conducted studies on dynamic and total holdup in RDC columns without presence of surfactants [2, 3, 6, 8, 22-24].

In this paper, local and average static holdup and also mother drops' size of dispersed phase in rotating disc column (RDC) and rotating sieved disc column (RSDC) have been studied experimentally in the presence of Cetyltrimethylammonium bromide (CTAB) as a cationic-hydrophilic surfactant. Experiments consist of five different concentrations of CTAB and for each concentration five different rotor speeds. Water and kerosene were used as continuous and dispersed phase, respectively.

## **2. EXPERIMENTAL WORK**

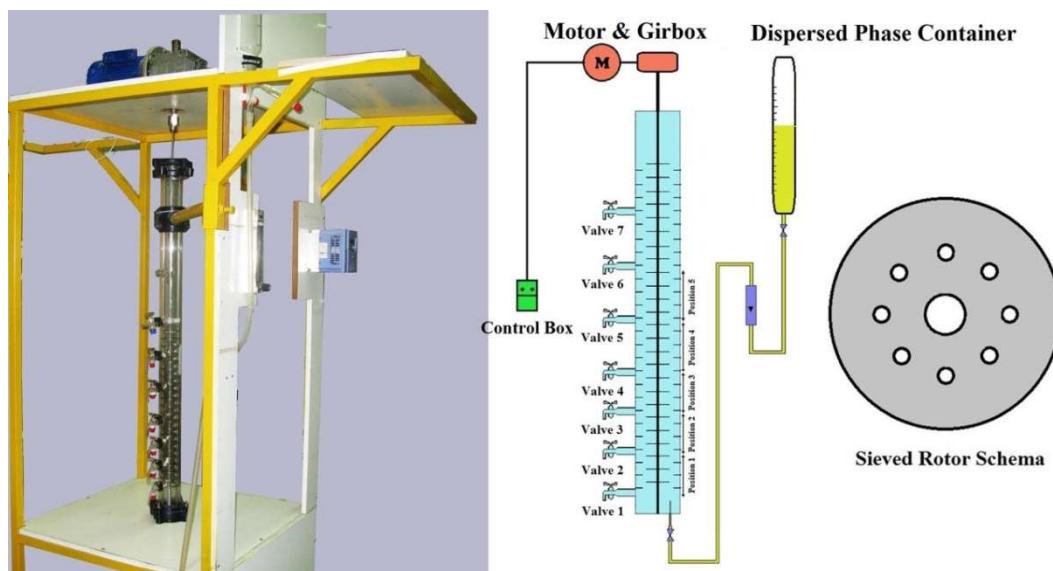
### **2.1. Apparatus**

The used rotary disc column with a glassy body had 21 couples of rotors and stators made up of stainless steel 316. Details of the column geometry have been presented in Tab. 1 in brief. Sampling could be done via seven valves mounted on the body of the column and the numbering method is shown in Fig. 1. Speed of rotors could be increased up to 300 rounds per minute (rpm) by through the electrical motor installed on the column. A glassy nozzle which was 0.4 mm in inner diameter injected dispersed phase in the column in the form of single droplets.

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**Fig.1.** Schema of rotating disc columns.

The only difference between RDC and RSDC columns was their discs. Since the Space under rotors of a rotary disc column is greatly capable of accumulating dispersed phase, creating holes on rotors can reduce the intensity of this accumulation. Rotors of the RDC had no holes but rotors belonged to the RSDC had eight holes with 3 mm diameter which were located in equal distance of each other. Moreover, each one of eight holes was located in the middle of the distance between the center and the edge of the rotor. Holes arrangement for each disc has been shown in Fig. 1.

**Tab.1.** The characteristics of the column

<b>Column Height (cm)</b>	<b>107</b>
Column Inner Diameter (cm)	7
Stator Inner Diameter (cm)	5
Rotor Outer Diameter (cm)	3.2
Rotor Thickness(mm)	1
Compartment Height (cm)	2.5

## 2.2. The chemical system

In all experiments kerosene and distilled water were used as dispersed and continuous phase, respectively. To examine the effects of surface active agent, CTAB as a cationic-hydrophilic surfactant used in five different concentrations in continuous phase. CTAB molecular weight is equal to 364.48 gr/mol in physical form of white powder.

Physical properties of phases for all concentrations of CTAB have been presented in Tab. 2. In addition, all five concentrations are below critical micelle concentration of CTAB. Densities were calculated via measuring mass of certain volume of phases. Visco Clock produced by SCHOTT Corporation, and Digital

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Tensiometer the model of K10ST produced by KRUSS Corporation are devices were used to measure viscosity and interfacial tension, respectively.

**Tab.2.** Physical properties of used chemical system for different concentrations of CTAB in continuous phase

CTAB Concentration	$\rho_c$ (Kg/m <sup>3</sup> )	$\rho_d$ (Kg/m <sup>3</sup> )	$\mu_c$ (mPa.s)	$\mu_d$ (mPa.s)	$\gamma$ (mN/m)
Zero	998	786	0.87	1.13	48.7
$2.74 \times 10^{-6}$ mol/lit	998	786	0.87	1.13	39.4
$1.37 \times 10^{-5}$ mol/lit	998	786	0.87	1.13	30.0
$6.85 \times 10^{-5}$ mol/lit	998	786	0.87	1.13	23.5
$3.43 \times 10^{-4}$ mol/lit	998	786	0.87	1.13	18.3

### 2.3. Experimental procedure

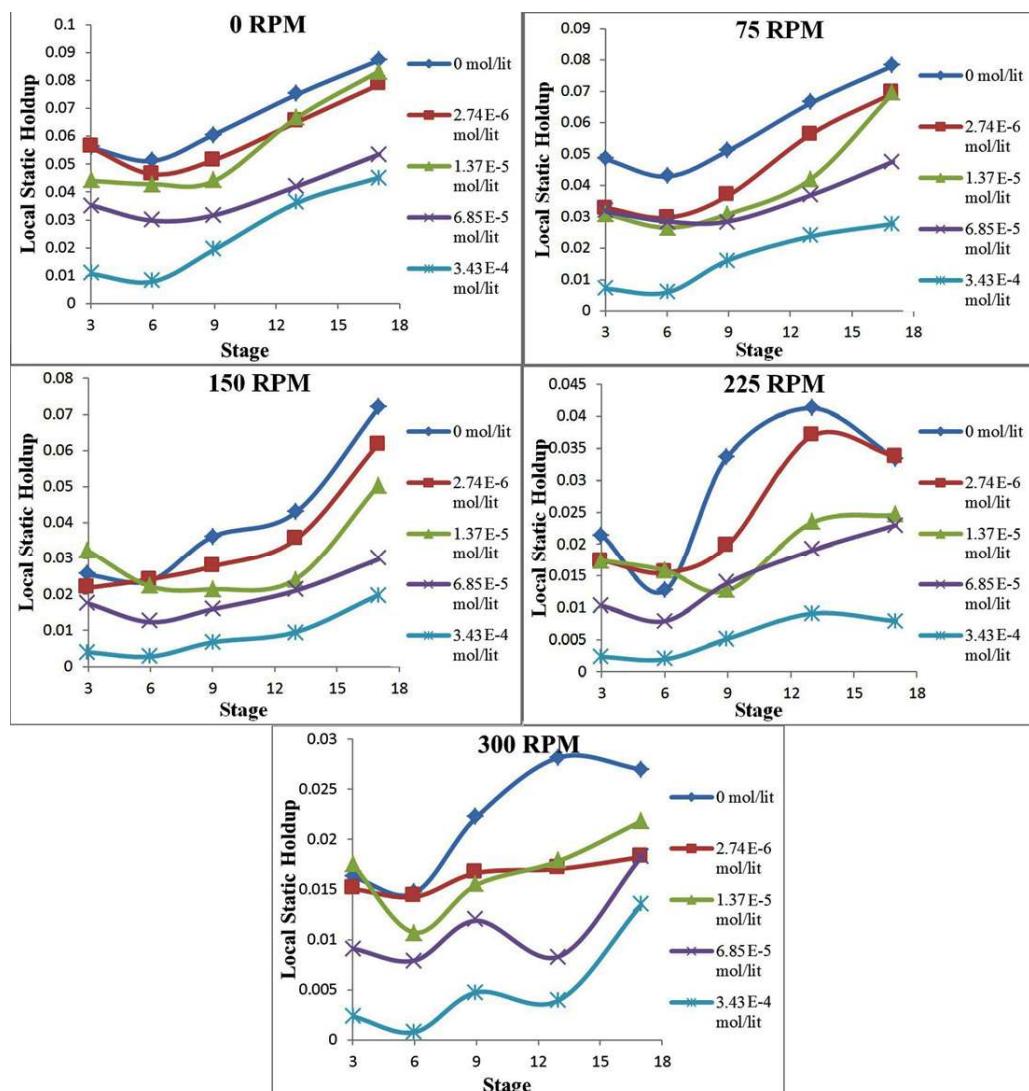
Experiments have been done in five rotor speeds of zero, 75, 150, 225, and 300 rpm. For each rotor speed five concentrations of CTAB in continuous phase have been investigated. Thus, totally 25 experiments have been carried out for each column that the procedure described below was followed for each of them.

Dispersed and continuous phase were mutually saturated in advance to avoid any mass transfer and variation in physical properties of phases during experiments. The column was filled up to the sixth sampling valve with contaminated continuous phase. This filled height of the column contained 17 couples of rotors and stators (five positions). After switching on electrical motor and setting rotor speed at prescribed value, the nozzle valve was opened. Dispersed phase flow rate was maintained in a constant value by a rotameter. As steady state conditions were reached in the column, photographs of the mother drops of the dispersed phase (drops above the nozzle) were captured. Regarding to the reference in all photographs (the diameter of the rotor shaft equal to 10 mm) mother drops' size were extracted.

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**Fig.2.** Variation of local static holdup in the RDC column.

After closing dispersed phase flow, all liquid reached to the top of sixth valve was drained off as waste material. For each position, whole liquid between two respective valves was drained from bottom valve into a marked container based on number of positions separately. For each of marked containers, dispersed and continuous phase were separated and their volume were measured. Then, local static holdup of each position could be calculated (local static holdup of each position is equal to volume of dispersed phase accumulated in that position divided by total volume of that position).

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### 3. RESULTS AND DISCUSSION

#### 3.1. The RDC experiments

##### 3.1.1. Variations of local static holdup in the column

Local static holdup graphs for various concentrations of CTAB and rotor speeds are shown in Fig. 2. Mainly, local static holdup increased from bottom to top of the RDC column. Reduction of drop size due to breakage in the column would lead to decrease in the momentum of each of drops. So, interfacial tension between a drop and dispersed phase located under rotors and stators when contact each others can overcome momentum of drop easier, and then it will be trapped.

##### 3.1.2. Effects of rotor speed on static holdup

As rotor speed goes up, local static holdup shown a decrement in a specific concentration of CTAB. Because by increasing rotor speed, more whirl and turbulence will be created. Furthermore, centrifugal force on surface of rotor increases and as a result, possibility of dispersed phase accumulation under rotors reduces. On the other side, by increasing rotor speed variation process of local static holdup along the RDC column became difference due to change of flow pattern in the column.

Fig. 3 presents average static holdup versus concentration of CTAB in different rotor speeds. The influence of increasing rotor speed on average static holdup is as the same as for local static holdup.

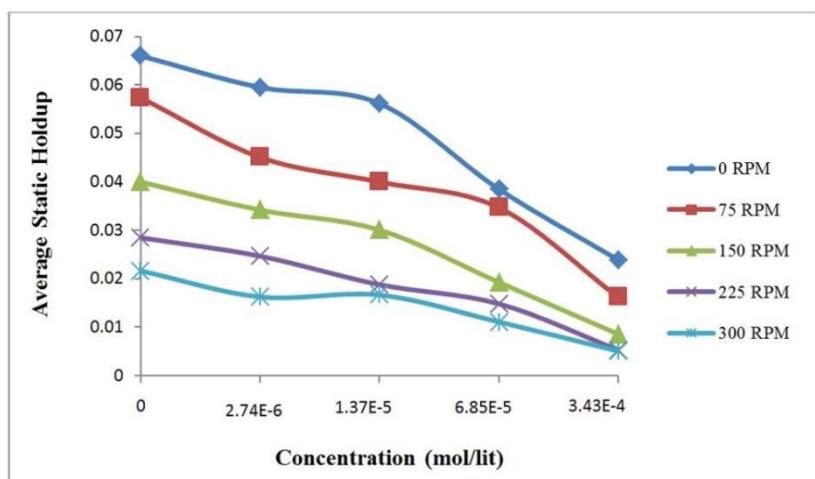


Fig.3. Variation of average static holdup in the RDC column.

##### 3.1.3. Effects of CTAB concentration on static holdup

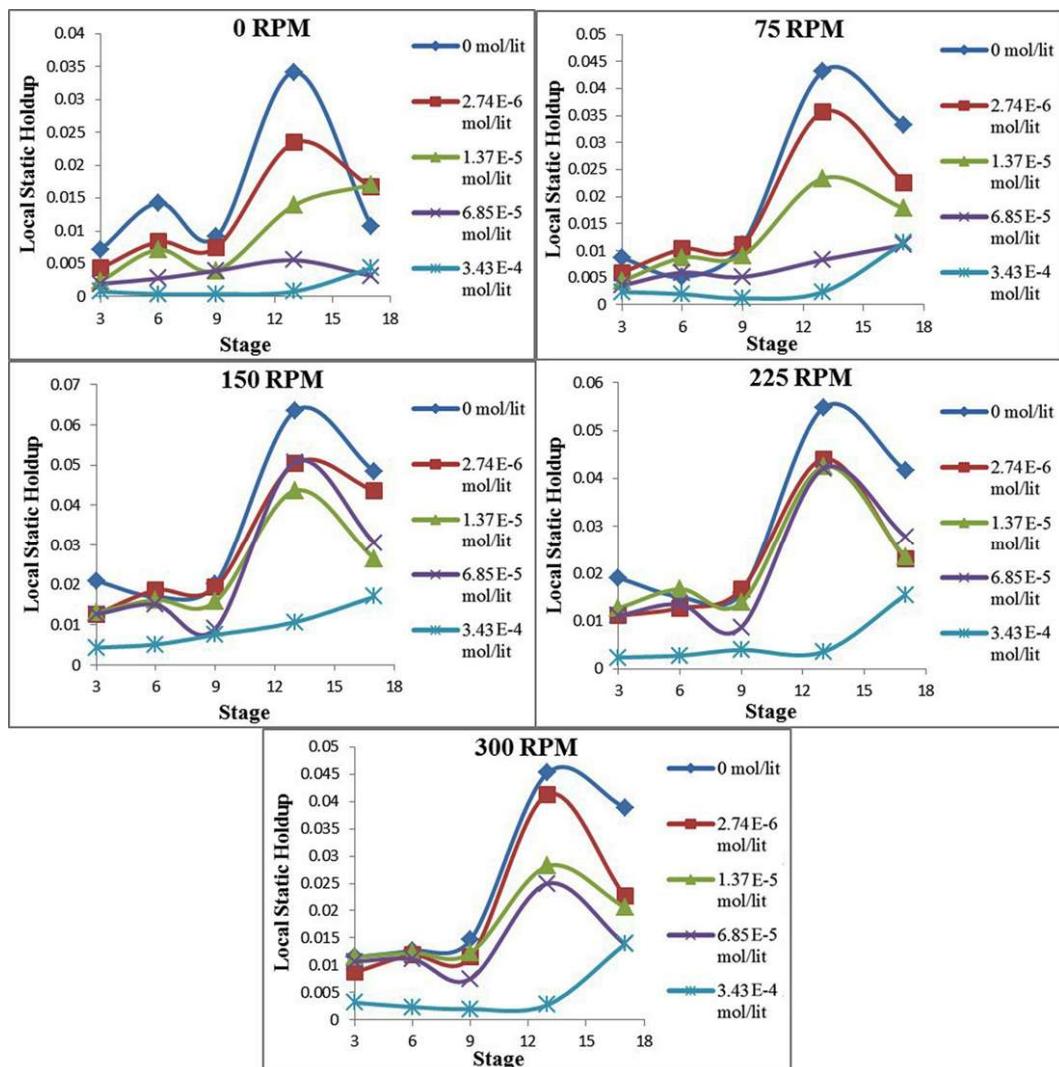
Regarding to Fig. 2, increase in concentration of CTAB would lead to a decrement in the amount of local static holdup because of decrement of interfacial tension between dispersed and continuous phases. On the other hand, the presence of the surface active agent had effects on the form of the graphs, too. Indeed, by increasing CTAB concentration, the variation process of local static holdup along

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the column has changed. It has intensified for rotor speeds of 225 rpm and 300 rpm. Respecting to Fig. 3, as concentration of CTAB goes up in a constant rotor speed, reduction rate of the amount of average static holdup increased.



**Fig.4.** Variation of local static holdup in different concentrations of CTAB for a certain rotor speed in the RSDC.

### 3.2. The RSDC experiments

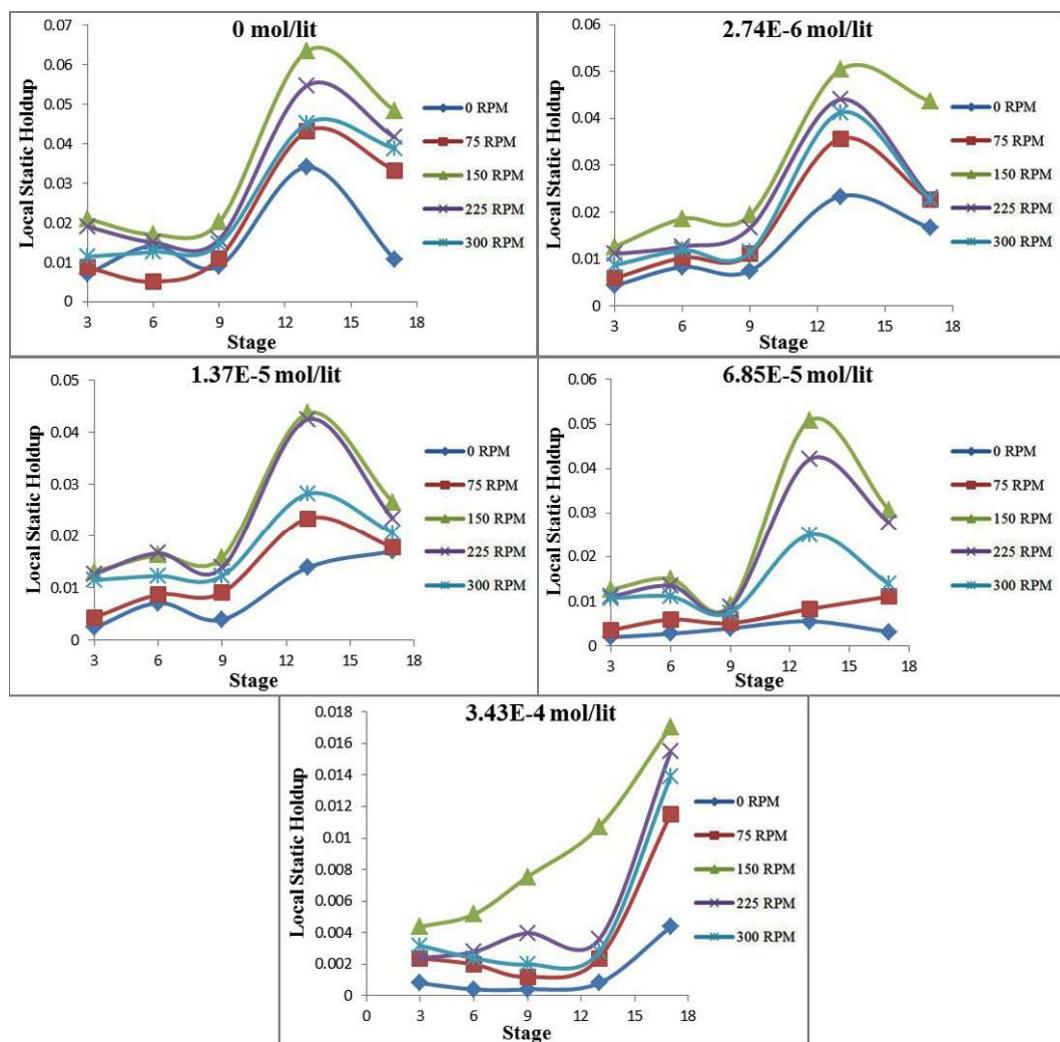
#### 3.2.1. Variations of local static holdup in the column

In Fig. 4, a decrement in static holdup from position 2 to position 3 and also from position 4 to position 5 can be seen almost in all graphs. It should be pointed out that the variation process of this hydrodynamic parameter in the RSDC has become different from the RDC. Moreover, due to holes on rotors of RSDC column values of static holdup are less than RDC column.

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**Fig.5.** local static holdup in different rotor speeds for a certain concentration of CTAB in the RSDC.

### 3.2.2. Effects of rotor speed on static holdup

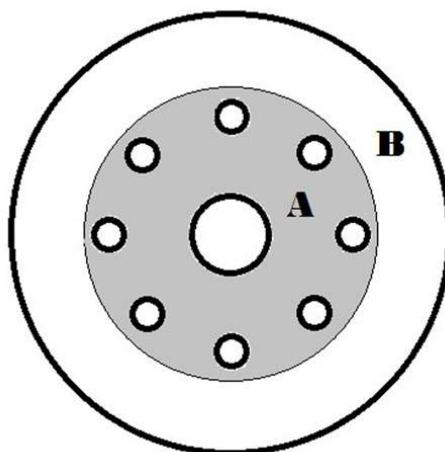
Fig. 5 demonstrates the influence of rotor speed on local static holdup. Respecting to the graphs for any concentration, the ascending order of amount of static holdup based on rotor speed is zero, 75, 300, 225, and 150 rpm which is different from the RDC column.

The schema of RSDC rotors is shown in Fig. 6. In low rotor speeds, dispersed phase which was trapped under rotors, was located mainly in zone A. By increasing rotor speed and consequently increase of centrifugal force on the surface of the rotor, the tendency of dispersed phase to be accumulated in zone A was reduced and was tended to zone B. Indeed, as rotor speed went up the role of holes was getting less importance and in the following, static holdup increased in rotor speed of zero to 150 rpm. Since dispersed phase under each rotor could cross its holes in general. Therefore, holes played key role in reducing static holdup.

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**Fig.6.** Sieved rotor and respective zones

As the speed of rotating discs raised more than 150 rpm in a constant concentration, static holdup decreased. This fact can be realized by considering graphs in Fig. 5 for rotor speeds of 150, 225, and 300 rpm in all CTAB concentration. This arrangement of graphs based on rotor speed was observed for RDC column in all speed of discs. The effect of holes on static holdup was eliminated gradually as rotor speed increased. Therefore, variation in static holdup by enhancement of rotor speed became like that of RDC column.

### 3.2.3. Effects of CTAB concentration on Static holdup

Regarding to graphs in Fig. 4, increasing concentration of the surfactant would lead to decrease of local static holdup. This is due to decrement in interfacial tension of two phases. But the point is that the presence of CTAB not only has shown itself by decreasing local static holdup, but it has also influenced the variation process of this hydrodynamic parameter along the column especially in high concentrations. As for concentration of  $3.43 \times 10^{-4}$  mol/lit the variation process of static holdup has changed considerably.

Variations of average static holdup versus concentration of CTAB for different rotor speeds have been presented in Fig. 7. This parameter, as local static holdup, is reduced by increasing concentration. Moreover, the graph belongs to average static holdup in 150 rpm is located above other ones absolutely.

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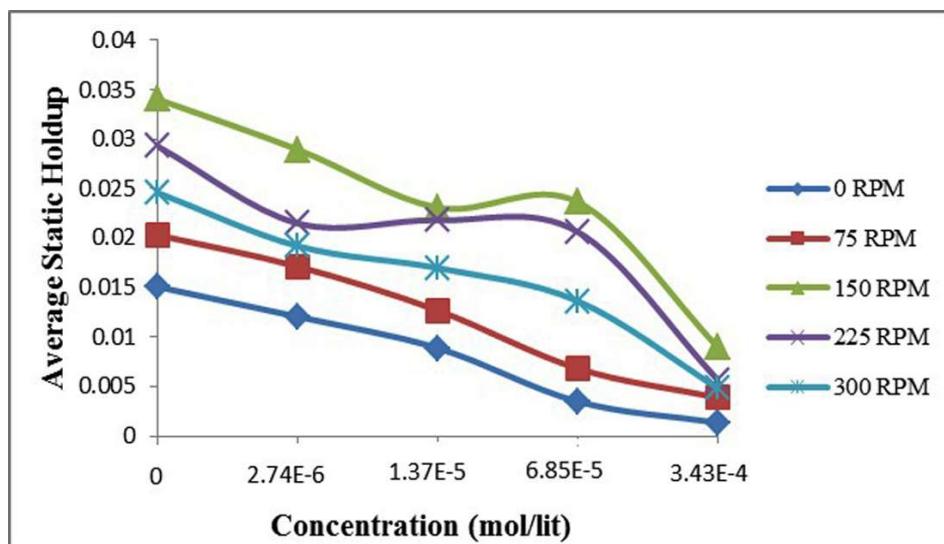


Fig.7. Average static holdup in the RSDC column

### 3.3. Effects of CTAB concentration on mother drops' size

Fig. 8 shows mother drops' size versus concentration of the used surfactant for different rotor speeds. For a constant rotor speed, decreasing in mother drops' size can be observed by enhancement of CTAB concentration.

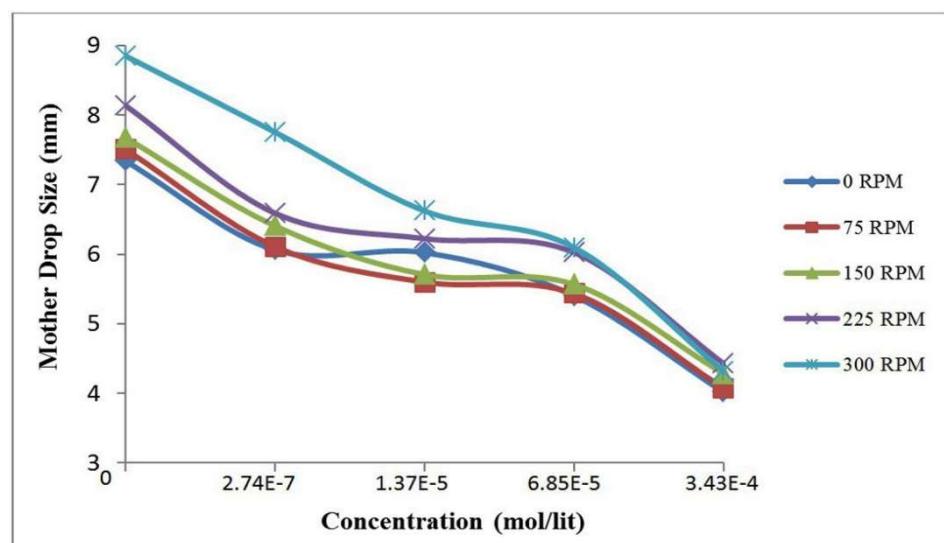


Fig.8. Mother drops' size versus concentration of CTAB for different rotor speeds

Another point worth mentioning is that by increasing the concentration, variation span of mother drops' size from rotor speeds of zero to 300 rpm decreases. Variation of mother drops' size due to change in rotor speed from zero to 300 rpm belongs to concentration of zero is 25.5% whereas, for concentration of  $3.43 \times 10^{-4}$  mol/lit is only 4.46%.

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## 4. CORRELATIONS

For deriving the following correlations, “DataFit” software version 9.0.59 was used which applies the least square technique method to calculate constants.

### 4.1. The RDC experiments

In order to predict local static holdup in the RDC column in the condition of immovable rotor discs, the following equation has been proposed:

$$\varphi_{L_n} = 0.0544 + \frac{3.54 \times 10^{-12}}{Mo} - \frac{7.08 \times 10^{-23}}{Mo^2} + \frac{4.18 \times 10^{-34}}{Mo^3} - 0.0762 \ln(n) + 0.0237 \ln(n^2) \quad (1)$$

where  $\varphi_{L_n}$ ,  $Mo$ , and  $n$  are local static holdup for the  $n$ th stage, Morton number, and number of stages, respectively. The average absolute relative deviation (AARD) was used to investigate the accuracy of the correlations. AARD can be described as follows:

$$\%AARD = \frac{1}{NE} \sum_{i=1}^M \left| \frac{Y_{i,exp} - Y_{i,cal}}{Y_{i,exp}} \right| \times 100 \quad (2)$$

where  $NE$ ,  $Y_{i,exp}$ , and  $Y_{i,cal}$  are the number of experiments, experimental value of  $Y$ , and calculated value of  $Y$ , respectively. The %AARD and  $R^2$  values for Eq. 1 are 8.54% and 0.97, respectively which show an acceptable agreement with experimental data.

In the case of movable rotors, the correlation below is derived to calculate local static holdup in the RDC:

$$\varphi_{L_n} = 0.251(Fr^{0.679})(We^{-1.010})(n^{0.522}) \quad (3)$$

where  $Fr$  is Froude number and  $We$  is Weber number. The  $R^2$  and %AARD for this correlation are 0.91 and 15.67%, respectively.

Average static holdup in the RDC column can be predicted by using the following equations.

In the condition of immovable rotors:

$$\varphi_{ave} = 0.0077 + \frac{3.54 \times 10^{-12}}{Mo} - \frac{7.08 \times 10^{-23}}{Mo^2} + \frac{4.18 \times 10^{-34}}{Mo^3} \quad (4)$$

In the case of rotating rotors:

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$$\varphi_{ave} = 0.827(Fr^{0.691})(We^{-1.019}) \quad (5)$$

The AARD values for Eq. 4 and 5 are 0.26% and 7.91%, and the  $R^2$  are 0.99 and 0.93, respectively.

#### 4.2. The RSDC experiments

As it was observed in section 3, variation process of local and average static holdup in the RSDC column was different from the RDC. The correlations for predicting local static holdup when rotors were immovable and were rotating can be written as Eq. 6 and 7, respectively:

$$\begin{aligned} \varphi_{L_n} = 0.0166 + \frac{7.94 \times 10^{-14}}{Mo} - 7.45 \times 10^{-3}(n) - \frac{8.06 \times 10^{-24}}{Mo^2} + 7.95 \\ \times 10^{-4}(n^2) + 9.88 \times 10^{-14} \left( \frac{n}{Mo} \right) + \frac{6.43 \times 10^{-35}}{Mo^3} - 2.49 \\ \times 10^{-5}(n^3) - 2.66 \times 10^{-15} \left( \frac{n^2}{Mo} \right) - 3.85 \times 10^{-25} \left( \frac{n}{Mo^2} \right) \end{aligned} \quad (6)$$

$$\begin{aligned} \varphi_{L_n} = 0.0232 - \frac{2.65 \times 10^{-3}}{Fr} + \frac{2.78 \times 10^{-5}}{Fr^2} - 1.90 \times 10^{-2} \ln(We) \\ + 1.47 \times 10^{-5} \ln(We)^2 + 3.46 \times 10^{-2}(n) - 7.37 \\ \times 10^{-3}(n^2) + 6.23 \times 10^{-4}(n^3) - 1.73 \times 10^{-5}(n^4) \end{aligned} \quad (7)$$

The AARD values for Eq. 6 and 7 are 17.18% and 25.43%, and the  $R^2$  are 0.94 and 0.91, respectively.

The equations below describe average static holdup for the RSDC column in the condition of immovable rotor discs (Eq. 8) and rotating rotor discs (Eq. 9):

$$\varphi_{ave} = -0.0024 + \frac{7.17 \times 10^{-13}}{Mo} - \frac{1.18 \times 10^{-23}}{Mo^2} + \frac{6.46 \times 10^{-35}}{Mo^3} \quad (8)$$

$$\begin{aligned} \varphi_{ave} = 0.0917 - \frac{5.70 \times 10^{-3}}{Fr} + \frac{3.01 \times 10^{-4}}{Fr^2} - \frac{4.90 \times 10^{-6}}{Fr^3} - 1.80 \\ \times 10^{-2} \ln(We) - 3.10 \times 10^{-4} \ln(We)^2 \end{aligned} \quad (9)$$

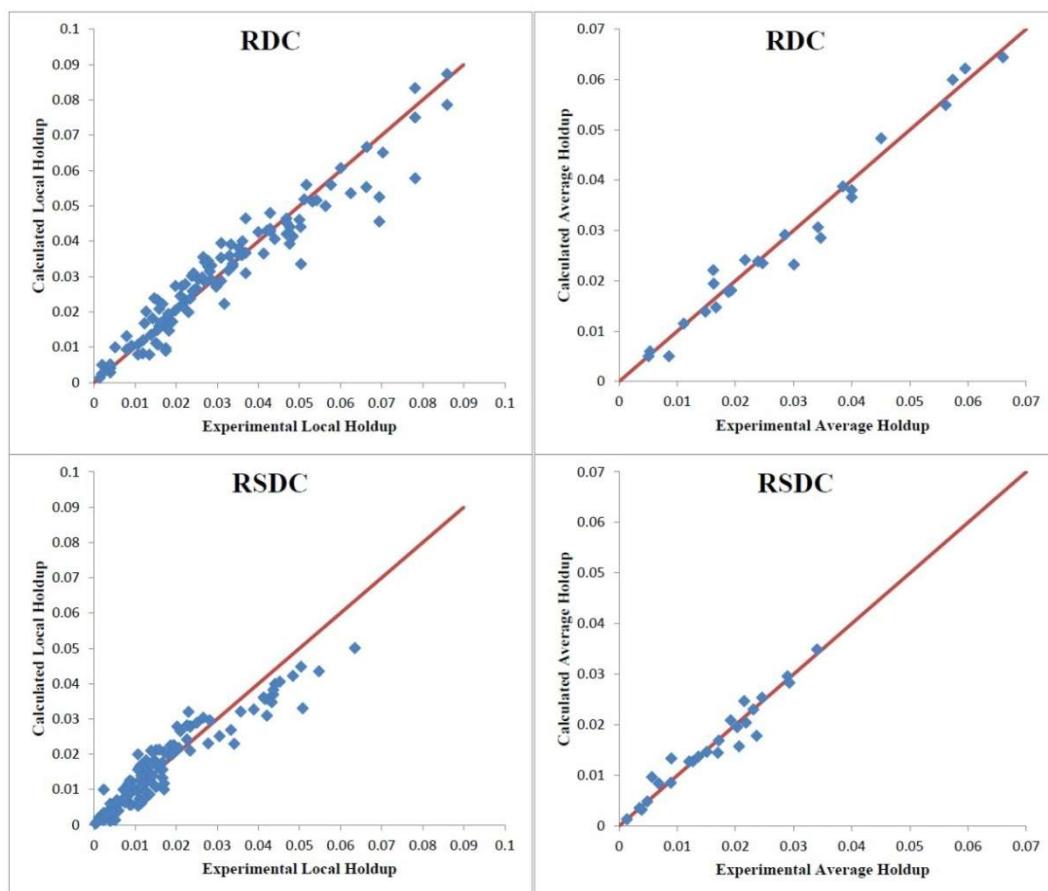
The  $R^2$  values of Eq. 8 and 9 were calculated 0.99 and 0.91, and the AARD values were 9.44% and 15.88%, respectively.

For comparing experimental data with calculated values of holdup from proposed correlations, they are demonstrated in Fig. 9, simultaneously. As it can be observed in Fig. 9, most of the points are located close enough to bisector line that confirms preciseness of the correlations.

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**Fig.9.** comparison between experimental and calculated static holdup.

## 5. CONCLUSION

The effects of the presence of CTAB surfactant, rotor speed, and length of column on local and average static holdup and also mother drops' size were studied in RDC and RSDC columns. Experiments were done in five concentrations of CTAB and also five different rotor speeds for each concentration. Results were summarized as following:

Respecting to findings, the presence of the used surface active agent caused a decrement in local and average static holdup and this matter was intensified by increasing concentration. In addition, CTAB changed variation process of local static holdup along the column particularly in the concentration of  $3.43 \times 10^{-4}$  mol/lit. The presence of CTAB surfactant reduced mother drops' size in both columns.

Local static holdup almost increased along the RDC but on the contrast to that, this hydrodynamic parameter was reduced from position 2 to 3 and position 4 to 5 in the RSDC column. Generally, static holdup in the RSDC column was less than the RDC.

Enhancement of rotor speed resulted in reduction of static holdup in the RDC. Whereas, by increasing speed of discs in the RSDC column from zero to 150 rpm

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and from 150 rpm to 300 rpm, static holdup showed increment and decrement, respectively.

To predict local and average static holdup, correlations were proposed for RDC and RSDC columns separately which had good agreement with experimental data. The maximum and minimum values of AARD were calculated 25.43% and 0.26%, and  $R^2$  values varied from 0.91 to 0.99.

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