Iranian Journal of Chemical Engineering

Journal Homepage: www.ijche.com

pISSN: 1735-5397
eISSN: 2008-2355

Regular Article

Investigating the Performance of an Ultrasound-Assisted Rotating Packed Bed Reactor for the Enhancement of the Micromixing Efficiency

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ARTICLE INFO

Article history:
Received: 2022-04-01
Accepted: 2022-05-30
Available online: 2022-07-31

Keywords:
RPB Reactor,
Blade Packing,
Micromixing Enhancement,
Segregation Index,
Ultrasonic Waves

ABSTRACT

The Micromixing plays a key role in the most of industrial processes; enhancing its efficiency is a very important issue. In this study, a typical rotating packed bed (RPB) reactor equipped with the blade packing and high frequency ultrasonic transducers were designed to study the micromixing efficiency using the iodide/iodate reaction. The utilized ultrasonic transducers were ultrasonic atomizer humidifiers with the frequency of 1.7 MHz. Taking advantage of both the controllable high gravitational force and induced effects of the high frequency ultrasound, simultaneously, in a small volume reactor is the novelty of the present work. The effects of different parameters like the rotational speed, volumetric ratio, concentration of acid, ultrasonic power and number of activ transducers were investigated with and without the ultrasonic field. By increasing the rotational speed and volumetric flow, the segregation index decreased and by increasing the concentration of acid and volumetric ratio, the segregation index increased. In all of experiments, the segregation index decreased significantly under the ultrasonic field. Moreover, by increasing the ultrasonic power and number of active transducers the segregation index decreased. The obtained results indicated that the relative segregation index increased up to 41.1 % under the 1.7 MHz ultrasonic field. Therefore, the high frequency ultrasonic waves can intensify micromixing, even in a high efficiency equipment like RPB.

DOI: 10.22034/ijche.2022.335625.1424 URL: http://www.ijche.com/article_154279.html

1. Introduction

Mixing is very important in industrial processes, which has been divided to three stages, including: macromixing, mesomixing and micromixing. The micromixing, as its name implies, investigates the mixing in micro scales. Micromixing is very important in different processes. Many chemical

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systems utilize micromixing techniques (such as; crystallization, polymerization, extraction and so forth) [1]. Micromixing can be helpful from two different viewpoints; design and processing. In the processing phase, it helps to achieve high levels of efficiency, quality and selectivity, while in design phase it indicates the optimum performance of an equipment, like a reactor [2, 3]. Therefore, it is very necessary to find novel techniques that can intensify the micromixing.

Generally, process intensification reduces the energy consumption and operating costs with increasing the mass, heat and momentum transfer in the smaller size of equipment [4]. One of these equipment is a rotating packed bed (RPB). The RPB increases the mass transfer rate by the centrifugal force, which is about 500 times of the gravity force [5]. For the first time, Ramshaw and Malinson have invented RPB (or HIGEE, that is the abbreviation of the high gravity) for being employed in a gas-liquid process. Afterward, it was employed in other processes such as absorption, adsorption, distillation and etc. [6-10]. Nowadays, it has been used in liquid-liquid and liquid-solid-gas processes [11, 12]. The RPBs are made of different parts including a rotor, packing, distributer and reactor casing. The main part of the RPB is the rotor that causes the centrifugal and driving force. By taking advantage of both the high centrifugal force and rotational speed, the RPBs can be employed in systems involved viscous liquids and in reactions with short contact times [13].

Moreover, the RPBs can enhance the micromixing efficiency [11, 12, 14-18, 5]. Some of the researchers investigated the characteristics of an RPB equipped with blade packings [19, 20], and reported that the RPB with blade packings exhibited superior operating characteristics [19, 20]. Nowadays, many innovations have been employed in designing the RPBs in order to increase the micromixing efficiency [17, 11, 12]. In 2009, Yang et al. designed a premixer before entering the liquid stream into the distributer in order to enhance the efficiency of the reactor [11]. In 2014, chu et al. designed a modified nickel foam packing and achieved appropriate results in micromixing performance. Moreover, they reduced the micromixing time with nickel foam packing in comparison with the traditional stainless steel packing from 8.67 ms to 3.28 ms [17]. In 2018, researchers replaced a stainless steel packing with a novel polytetrafluorethylene (PTFE) packing and investigated the micromixing time. They found that, micromixing time can reduce to 2.762×10^{-5} s when they use the PTFE packing [12]. In 2019, Wenzel et al. investigated in detail the influence of the packing depth, liquid distribution, and volumetric ratio on mixing processes for the first time [21]. In 2019, Ouyang et al. developed a Computational Fluid Dynamics (CFD) model to investigate the micromixing efficiency in the premixer and packing zone of an RPB based on the Villermaux-Dushman reaction system and a modified Finite-rate/Eddy dissipation micromixing model [22].

Employing ultrasonic waves is a known technique for enhancing different chemical or physical processes [23]. By taking advantage of physicochemical effects of ultrasonic waves as a proper technique, we can enhance the micromixing efficiency. The propagation of ultrasonic waves in a liquid medium involves different effects, such as: the acoustic cavitation and acoustic streaming, acoustic fountain (the liquid surface deformation), producing OH Radicals and nebulization [24-26].

Acoustic cavitation is one of the main induced effects of ultrasonic waves. The propagation of these waves in a liquid medium is alternately along with high and low-pressure cycles. These cycles lead to the generation of tiny bubbles where the pressure is less than the liquid vapor pressure. These bubbles grow and then implode during high-pressure cycles [24, 25, 27]. This induces a shock wave and local turbulence through the liquid medium [27-29]. The acoustic cavitation is responsible for the turbulence, mixing and violent collisions between particles. The generation and collapse of millions of these bubbles in a second can affect the liquid properties [24, 25]. The size of cavitation bubbles depends on the wave frequency [30, 27]. There are two types of cavitation: Stable (or the non-inertial cavitation) and transient (or the inertial cavitation) [25, 31]. When the created bubbles exist for less than one cycle, it is called transient cavitation bubbles [25], which are created by low frequency waves. These bubbles are not very small and they can decompose to smaller bubbles. For the frequencies higher than 600 kHz, the created bubbles are smaller and more stable [30, 27, 31]. These stable, tiny bubbles, which are believed to contain mainly gas or vapor, do not collapse during long time and oscillate around their equilibrium size. They are called the stable or non-inertial cavitation [25, 31, 23]. The formation of the stable bubbles produces another important fluid hydrodynamics effect called microstreaming [32, 27]. The induced microstreams lead to the deformation of the free surface of the liquid [32, 27]. In high frequency waves, the liquid is moved along the transducer axis and hits the upper surface and returns to the liquid; this fluid deformation at the liquid-gas interface is known as the acoustic fountain [33, 34, 27, 25]. The acoustic streaming is the other main induced effect of ultrasound, which ensues from the dissipation of the acoustic energy. The creation of the high-speed unidirectional currents of the tiny, stable bubbles causes the momentum gradients and the fluid currents, which are known as the acoustic streaming [35, 25]. The acoustic streaming and microstreaming are the most important induced effects of the stable cavitation [31, 36]. Sometimes, cavitation is known as "cold boiling" [31, 29].

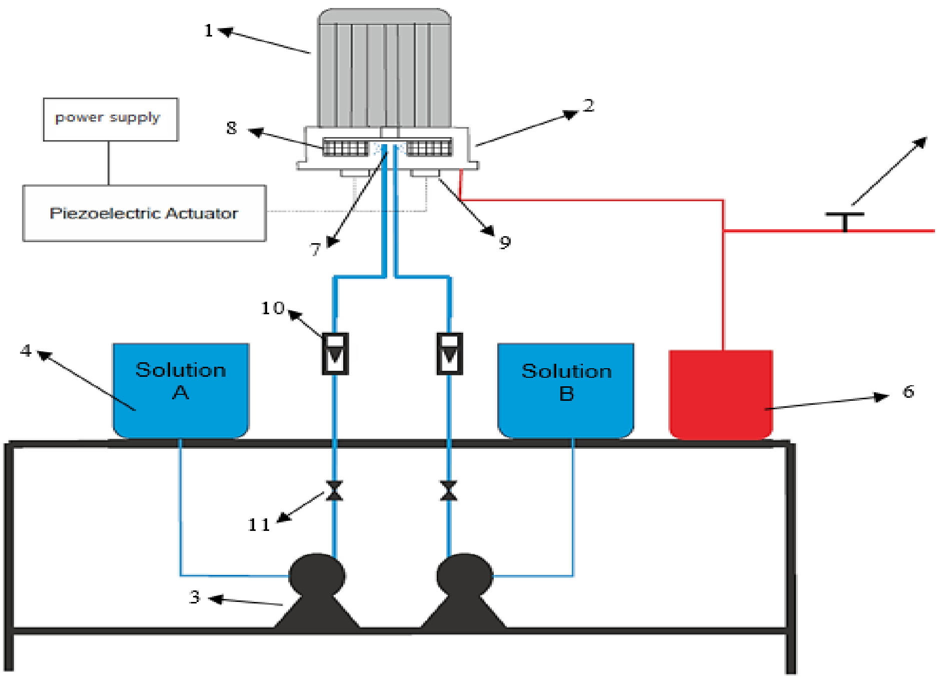
Nowadays, the ultrasonic technique is employed in different processes like the heat transfer enhancement [24, 31], water treatment [37], food processing [38] and etc. [26, 39]. In recent years, using the ultrasonic waves in order to enhance the micromixing efficiency has been reported by some researchers in the literature [24, 39-44]. In some studies, the positive effects of high frequency ultrasonic waves on the micromixing efficiency, in different reactors, were also reported [40, 41, 43, 44]. In 2022, Rabiei Faradonbeh et al. investigated numerically the mixing efficiency of the non-Newtonian fluid flow based on surface acoustic waves [45]. In 2018, Luo et al. [42] presented a feasibility study of using ultrasonic waves with a frequency of 20 kHz for increasing the micromixing efficiency in the RPB. They reported that the segregation index decreased by about 13.8 % in the presence of ultrasonic waves comparing with no ultrasonic condition. In their study, low frequency ultrasonic waves were employed. They showed that ultrasonic waves could intensify micromixing, even in a high efficiency equipment like RPB. In such systems, the micromixing process can take advantage of both ultrasonic effects and centrifugal forces.

In this study, the effect of high frequency ultrasonic waves on the micromixing process was investigated in a typical RPB, which was equipped with the blade packing. Taking advantage of both the controllable high gravitational force and induced effects of the high frequency ultrasound, simultaneously, in a small volume reactor is the novelty of the present work. For this reason, high frequency transducers with the frequency of 1.7 MHz were embedded in the designed RPB in order to investigate the effects of high frequency waves on the segregation index and micromixing characteristics under different experimental conditions.

2. Materials and experimental methods

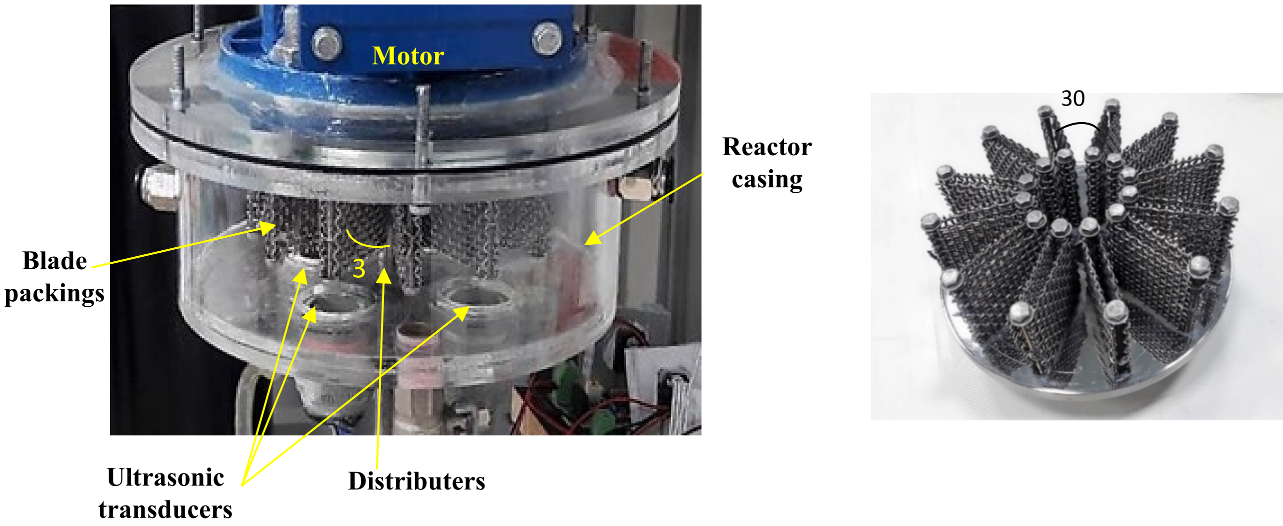
2.1. Experimental setup

In this study, a typical RPB reactor equipped with the blade packing was designed. The schematic representation of the experimental setup is depicted in Figure 1. The twelve-blade packing which was made of the stainless steel grille, was placed on the rotating bed, upside down. The angle between blades was 30º. In order to take advantage of the high frequency ultrasonic field, four transducers with the frequency of 1.7 MHz and diameter of 2 cm were placed on the bottom of the reactor. This design allows the tiny droplets of the liquid to be thrown up by the vibration of the surface of the transducer and easily pass through the packing blades. Four distributers, with a holes diameter of 1 mm, were used to create the symmetric distribution of the liquid entering the reactor. Two of them were used for the acid line (solution B) and the other two for the buffer line (Solution A, containing $H_2BO_3^-$, I^-, and IO_3^-). Figure 2 shows the different parts of the reactor and blade packings. Table 1 shows the dimensions of the different parts of the designed reactor.



1: motor, 2: RPB reactor, 3: pump, 4: stock tank, 5: sampling valve, 6: waste tank, 7: distributer, 8: blade packing, 9: ultrasonic transducer, 10: rotameter and 11: valve.

Figure 1. Schematic representation of the experimental setup.



(a) (b)

Figure 2. (a) Different parts of the reactor, (b) blade packings.

Table 1
Dimensions of different parts of the designed reactor.

Item	Value
Reactor casing:	
Axial height	73 mm
inner diameter	190 mm
Packing:	
Axial height	37mm
Outer diameter	138 mm
Inner diameter	39 mm
Outer diameter of rotating bed	150 mm
Mesh dimensions	1×1 mm^2

2.2. Parallel competitive reaction

The most common reaction in parallel competitive reactions, which is used for investigating micromixing, is the iodide/iodate reaction. This reaction is known as the Dushman reaction and consists of following three stages [42]:

$$H_2BO_3^- + H^+ \leftrightarrow H_3BO_3 \quad \text{(instantaneous)} \tag{1}$$

$$5I^- + IO_3^- + 6H^+ \leftrightarrow 3I_2 + 3H_2O^- \quad \text{(fast)} \tag{2}$$

$$I^- + I_2 \leftrightarrow I_3^- \tag{3}$$

The stages that were mentioned above can be simply defined under the ideal condition where all protons (H^+) are consumed and the next two stages do not happen. But, under the non-ideal conditions, an excessive amount of protons remain in the liquid medium and causes the reaction (2) to progress and create iodine. The created iodine causes triiodide ions to be generated during reaction (3). The concentration of triiodide ions describe the efficiency of micromixing. The efficiency of micromixing is characterized by the Segregation index (X_S), which can be calculated from the following equations:

$$X_S = \frac{Y}{Y_{ST}} \quad (4)$$

$$Y = \frac{2\times(n_{I_2}+n_{I_3^-})}{n_{H_0^+}} \quad (5)$$

$$Y_{ST} = \frac{6(IO_3^-)_0}{6(IO_3^-)_0+(H_2BO_3^-)_0} \quad (6)$$

where Y is the selectivity of iodide (the ratio of the acid mole number that is consumed in reaction (2) to the initial acid mol number) and Y_{ST} is the value of Y in the case of the total segregation [5]. The value of X_S is between 0 and 1 that will be 0 under an ideal mixing condition.

2.3. Experimental method

A buffer solution, solution A containing $H_2BO_3^-$, I^-, and IO_3^- iones, with a constant concentration was prepared according to Table 2. The concentration of solution B (acid) was 0.05-0.09 mol/L. The volume flow rates of both solutions were measured by two rotameters. The concentration of triiodide ions in final samples was measured by a UV-spectrophotometer (PG instruments T80$^+$) and calibration curve. The effect of different experimental conditions on the segregation index was investigated under the ultrasonic field and without the ultrasonic field at room temperature. The effects of the buffer to acid volumetric ratio (R), concentration of H^+ ($[H^+]$), rotational speed of the rotating packd bed (N), ultrasonic power and number of active transducers were investigated. The ultrasonic power, or dissipated power, was measured by a calorimetric experiment in a batch flow mode [23, 35]. All experiments were repeated at least three times to insure the repeatability of experiments.

Table 2

Concentration of reactants (solution A).

Reactant	Concentration (mol/L)
H_3BO_3	0.1818
NaOH	0.0909
KIO_3	0.00233
KI	0.0117

3. Results and discussion

3.1. The effect of the rotational speed on X_S

The effects of the rotational speed (N) on X_S under two different conditions, the ultrasonic assisted RPB reactor (UAR) and non-ultrasonic assisted RPB reactor (NUAR) are shown in Figure 3. R, $[H^+]$, V_A and P_{us} were adjusted to 10, 0.1 mol/L, 100 L/h and 22.5 W respectively. As it is observed from this figure, the ultrasonic waves have greatly reduced the segregation index. Moreover, X_S decreases by increasing the rotational speed. Therefore, the micromixing efficiency increases by increasing the rotational speed. By increasing the rotational speed, the relative velocity between the liquid droplets and packing increases which causes a reduction in X_S. In addition, the residence time distribution (RTD) reduces by increasing the rotational speed. Most of the collisions between the two liquid elements happen when they are captured by the rotating packing. When RTD decreases, the two liquid elements are captured by packing in a shorter time. Besides, both the micromixing rate and coalescence-dispersion frequency of the liquid elements will be accelerated [46].

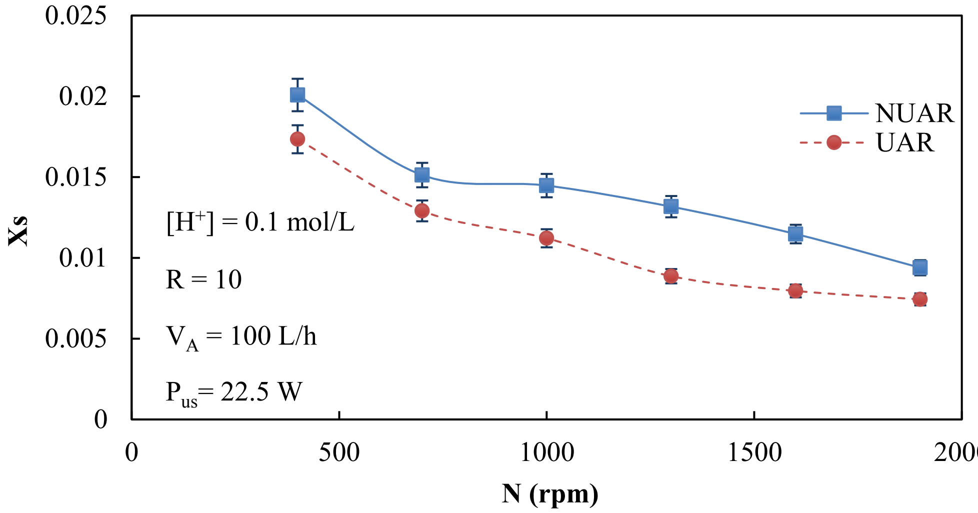


Figure 3. Effect of the rotational speed on the segregation index.

For a better evaluation of the effects of the high frequency ultrasound on micromixing, the relative segregation index, σ, is defined as follows [42]:

$$\sigma = \frac{Xs_0 - Xs_{us}}{Xs_0} \times 100 \qquad (8)$$

where, Xs_0 and Xs_{us} are the segregation indexes without and with the activation of ultrasonic transducers. Since, in the designed reactor, micromixing is intensified by both the ultrasonic waves and centrifugal forces of RPB, this dimensionless parameter represents the contribution of ultrasound on the segregation index [42]. The value of this parameter varies from zero to one. The larger values of σ indicate the greater contribution of ultrasound to the progression of micromixing.

Figure 4 illustrates the values of the relative segregation index in different rotational speeds. According to this figure, at a rotational speed higher than 1300 rpm, the contribution of ultrasound for enhancing the micromixing efficiency has decreased and the effect of the rotational speed was dominant. Because, at very high rotational speeds due to very high collision speeds, liquid droplets are numerous and tiny enough that even the presence of waves does not affect their number and size. In these experiments, the value of σ varies from 13.6 to 32.7 %.

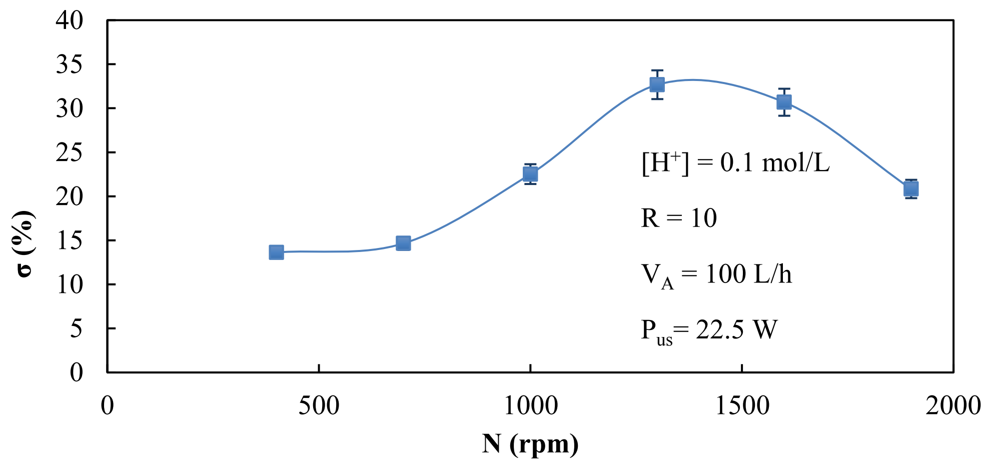


Figure 4. Effect of the rotational speed on the segregation index.

A real view of the different situations of RPB is shown in Figure 5. In Figure 5 (a), the cone-shape acoustic fountain, as soon as the transducers were activated without the packing rotation (with N=0 rpm), is illustrated. Figure 5 (b) shows how a large amount of liquid is atomized and the generated fog fills the space inside the reactor about 15 s after actuating the transducers with N=0 rpm. Figure 5 (c) shows the created liquid droplets with N=1000 rpm without ultrasonic waves and Figure 5 (d) shows the tiny droplets and large amount of fog created with N=1000 rpm and at 15 s after actuating the transducers.

The main mechanism for improving micromixing in the presence of ultrasonic waves is related to the high cavitation activity, fog formation and acoustic streams in RPB. In the case of the RPB that is assisted by high-frequency waves, the acoustic cavitation induces the shock waves and local turbulence through the liquids [27-29]. The generation and collapse of some bubbles cause turbulence, mixing and collisions between particles [24, 25], which are intensified by the collision of the packing blades. Moreover, the fog or tiny droplets will also be generated by the transducers. In this case, the liquid atomization can be made easier by creating weak spots (bubbles) by cavitation [47]. Therefore, using RPB and the high frequency ultrasound at the same time, intensifies the liquid atomization, fog generation, more collisions, and more local turbulence, which lead to more efficient micromixing.

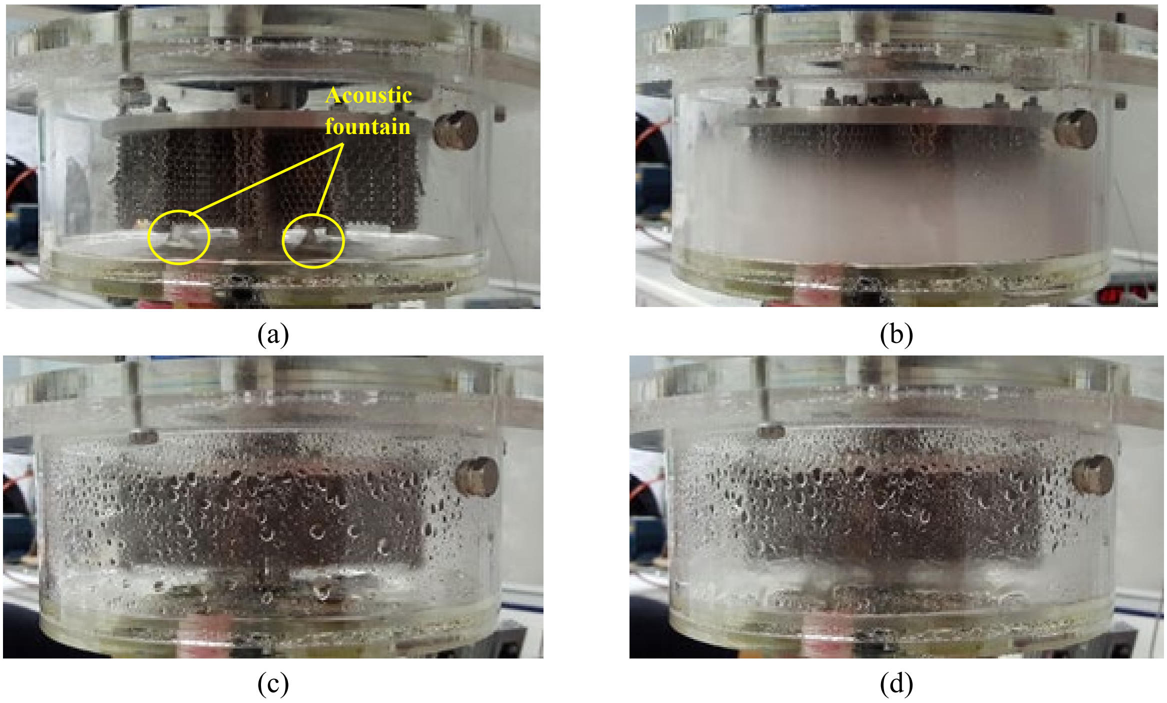


Figure 5. Real view of the different situations of RPB, (a) N=0 rpm and at the beginning of actuating the transducers (b) N=0 rpm and at 15 s after actuating transducers (c) N=1000 rpm without ultrasonic waves (d) N=1000 rpm and at 15 s after actuating the transducers.

3.2. The effect of the concentration of acid on X_S

The effect of the concentration of acid on the micromixing efficiency in different rotational speeds in the presence and absence of ultrasonic waves is shown in Figure 6. In these experiments, R, V_A and P_{us} were adjusted at 10, 100 L/h and 22.5 W respectively. As it can be seen, by increasing the concentration of acid, the segregation index increases. The main reason is remaining more excessive protons in medium, by increasing the concentration of acid. In accordance with Le Chatelier's principle, increasing the concentration of H^+ ions will shift the equilibrium to the right side of the reaction (2) that would reduce the change in concentration. The higher amounts of the produced iodine creates the higher concentration of triiodide ions, which means the poorer quality of the micromixing. Therefore, excessive protons produce more triiodide ions and increase the segregation index. This effect enhances in the presence of the ultrasonic field. In these experiments, the value of σ varies from 4.1 to 32.7 %.

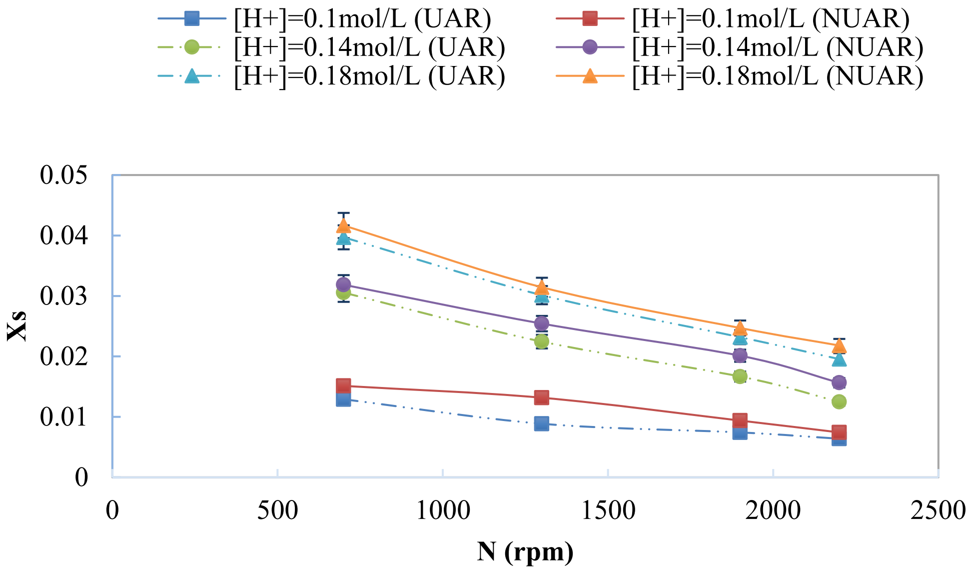


Figure 6. Effect of the concentration of acid on the segregation index.

3.3. The effect of the volumetric flow rate on X_S

The investigation of the effect of the buffer volumetric flow rate on X_S under the ultrasonic filed and without ultrasound is shown in Figure 7. In these experiments, R, $[H^+]$, N and P_{us} were set at 10, 0.1 mol/L, 2000 rpm and 22.5 W respectively. It can be seen that, by increasing the volumetric flow rate, the segregation index reduces. In this case, the higher volumetric flow rate leads to the higher injection velocity of the liquid exiting from the distributor's holes. So, the higher injection velocity causes higher collision speed between liquid elements that results in a better micromixing [46]. Furthermore, RTD decreases by increasing the volumetric flow rate. A shorter RTD leads to more collision-collapse and hence the higher micromixing efficiency, like the effect of the rotational speed [46]. In these experiments, the value of σ varies from 14.4 to 29.7 %.

3.4. The effect of the volumetric ratio on X_S

In this section, the effect of the volumetric ratio (R) on X_S in the presence and absence of ultrasonic waves has been investigated. The obtained results are illustrated in Figure 8. In these experiments, N and P_{us} were set at 2000 rpm and 22.5 W respectively. This figure shows that, X_S raises by increasing the volumetric ratio, because by increasing the volumetric ratio, the total flow rate decreases. This reduction declines the impact strength of the liquid elements in the packing [17]. It should be noted that for increasing the volumetric ratio, the acid flow rate must be reduced. Therefore, the amount of protons will be variable in each experiment. In order to fix the amount of protons in each experiment, the concentration of H^+ has been raised by increasing the volumetric ratio [5]. In these experiments, the value of σ varies from 14.4 to 41.1 %.

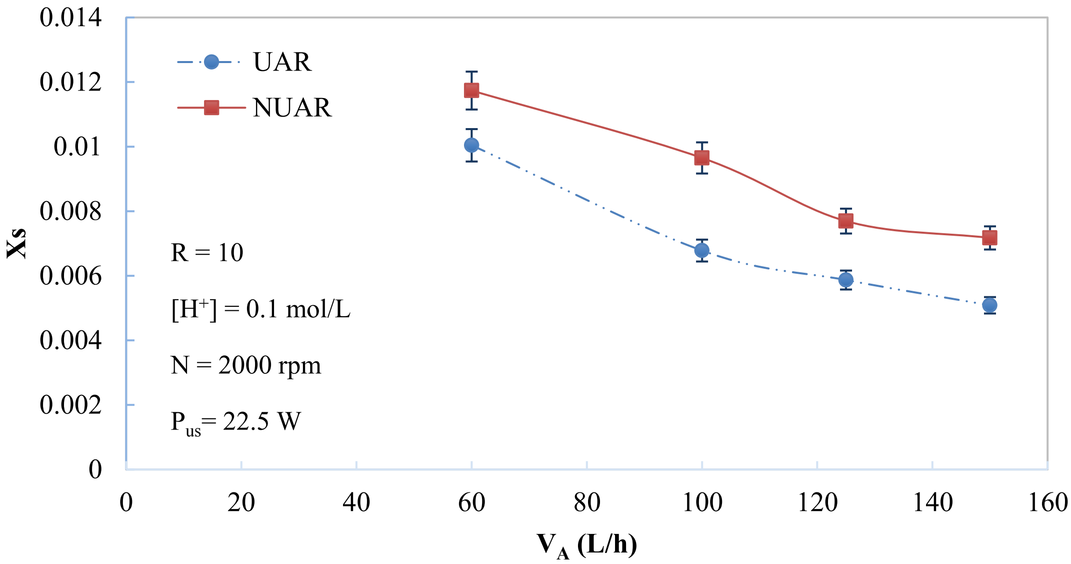


Figure 7. Effect of the volumetric flow rate on the segregation index.

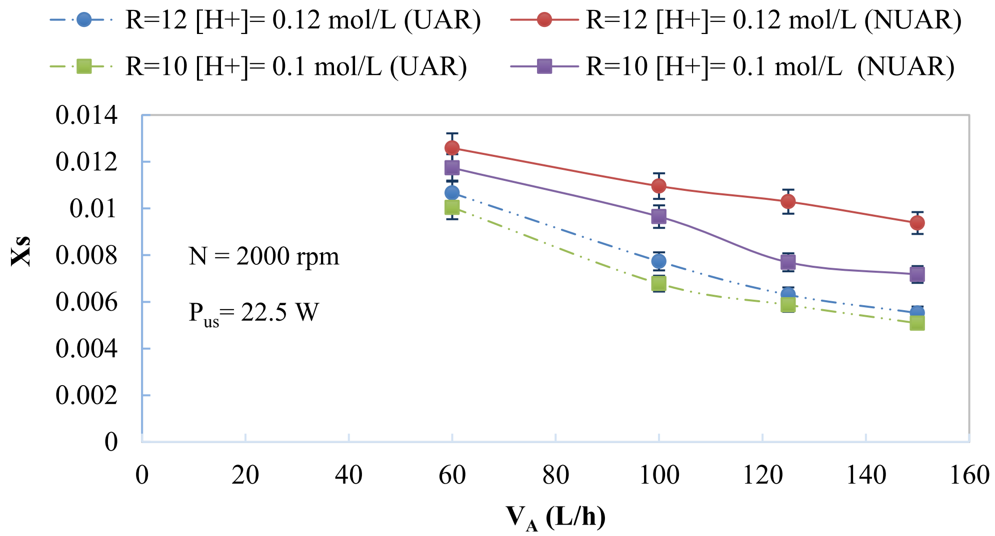
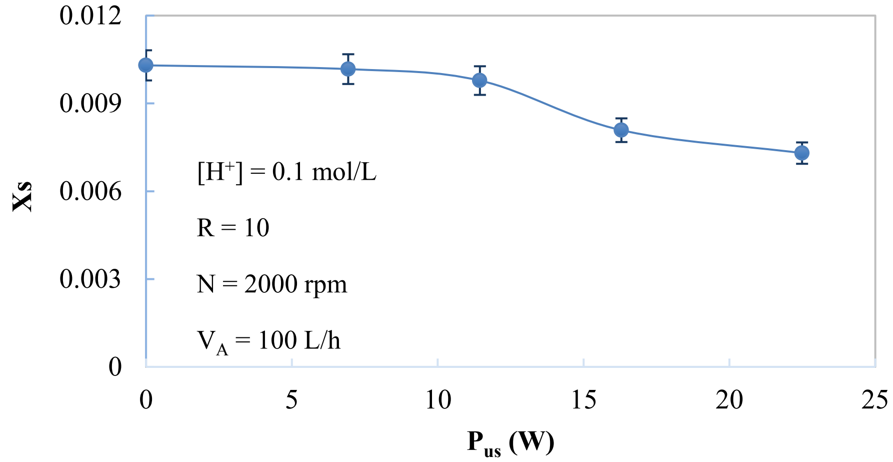


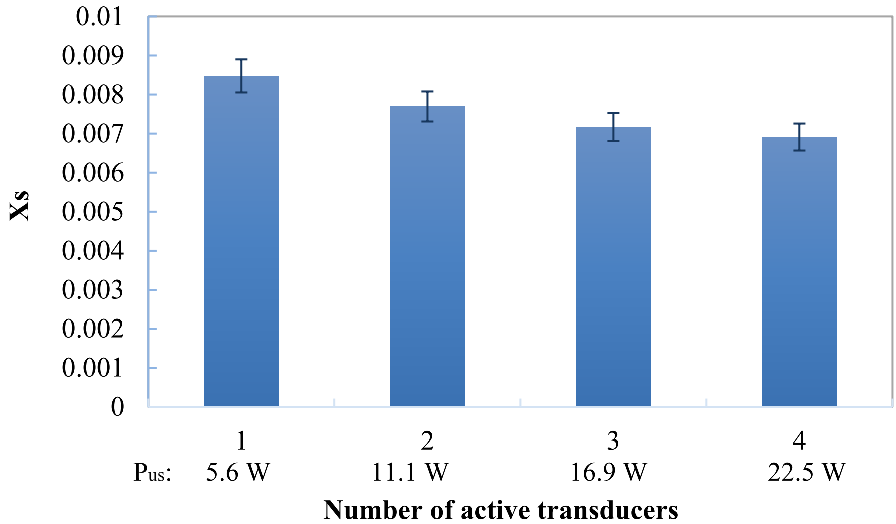
Figure 8. Effect of the volumetric ratio (R) on the segregation index.

3.5. The effect of the power and number of the active transducers on X_S

In this section, the effect of the ultrasonic power for four active transducers and number of the active transducers on Xs has been investigated. The obtained results are illustrated in Figure 9 (a) and (b). It is obvious that, under the non-ultrasound condition, the ultrasonic power (P_{us}) is equal to 0 W. As it is obtained from Figure 9 (a), by increasing the ultrasonic power, all induced effects of ultrasonic waves will be intensified. More ultrasonic power leads to a lower segregation index and hence the higher micromixing. This result is obtained because of more cavitation activities, more atomization, more turbulence, and more collisions between the two liquid droplets. By increasing the ultrasonic power of four active transducers from 6.9 W to 22.5 W, Xs decreases by almost 28.2 %. By employing two, three, and four active transducers, Xs decrease by about 9.2 %, 15.4 %, and 18.5 % respectively (See Figure 9 (b)). As the number of active transducers increases, the ultrasonic power increases and the volume of the reactor exposed to the ultrasonic radiation, and their induced effects are increased. Therefore, increasing the number of active transducers increases the micromixing efficiency.



(a)



(b)

Figure 9. Effects of (a) ultrasonic power of four active transducers, and (b) the number of active transducers on the segregation index.

4. Conclusions

In this study, the micromixing efficiency in a typical RPB reactor has been intensified by high frequency ultrasonic waves. Taking advantage of both the controllable high gravitational force and induced effects of the high frequency ultrasound, simultaneously, in a small volume reactor is the novelty of the present work. For this reason, four ultrasonic transducers (ultrasonic atomizer humidifiers) with the frequency of 1.7 MHz were employed in a typical RPB with the 12-blade packing. These waves take advantage of the stable acoustic cavitation and acoustic streams to atomize liquids and generate large amounts of tiny droplets (fog or cold vapors) in the reactor. The rotational speed, buffer volume flow rate, volumetric ratio and concentration of acid were different experimental parameters for investigating the segregation index. All experiments in the RPB were repeated under the ultrasonic field. With increasing the rotational speed and volumetric flow, the segregation index decreased and by increasing the concentration of acid and volumetric ratio, the segregation index increased. In all of experiments, the segregation index decreased significantly under the ultrasound field. Moreover, by increasing the ultrasonic power and number of active transducers the segregation index decreased. Totally, the relative segregation index, σ, was increased by up to 41.1 % under the ultrasound field. In these experiments, the highest enhancement in the micromixing efficiency was related to N = 2000 rpm, P_{us}= 22.5 W, V_A=150 L/h, R=12 and $[H^+]$=0.12 mol/L. In the RPB reactor, centrifugal forces lead to violent collisions between the two liquid elements while they are captured by the rotating packing. In addition, RTD decreases as the two liquid elements are captured by packing in a shorter time. Therefore, RPB can accelerate both the micromixing rate and coalescence-dispersion frequency of the liquid elements [46]. High frequency ultrasonic waves intensify the collision between liquid elements by increasing the fog formation and atomizing more liquids. Therefore, high frequency ultrasound waves can effectively enhance the micromixing efficiency in the RPB reactor.

Nomenclature

A	buffer solution containing $H_2BO_3^-$, I^-, and IO_3^-.
B	solution of sulfuric acid.
$[H^+]$	initial concentration of H^+ ion [mol/L].
$[H_2BO_3^-]_0$	initial concentration of $H_2BO_3^-$ [mol/L].
$[IO_3^-]_0$	initial concentration of IO_3^- [mol/L].
$n_{H_0^+}$	mole number of initial H^+ [mol].
n_{I_2}	mole number of produced I_2 [mol].
$n_{I_3^-}$	mole number of I_3^- [mol].
P	power [W].
R	volumetric ratio, V_A/V_B.
V_A	liquid flow rate of solution A [L/h].
V_B	liquid flow rate of solution B [L/h].
X_S	segregation index.
Y	selectivity of iodide.
Y_{ST}	selectivity of iodide in the case of total segregation.
Greek symbols	
σ	relative segregation index.

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