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Atrazine adsorption from aqueous solution using powdered activated carbon—Improved mass transfer by air bubbling agitation

Yue Jia[a], Rong Wang[b], Anthony G. Fane[a,*]

[a] *School of Civil and Environmental Engineering, Nanyang Technological University, Block N1,*
Nanyang Avenue, 639798, Singapore
[b] *Institute of Environmental Science & Engineering, Nanyang Technological University, Innovation Center,*
Block 2, 18 Nanyang Drive, 637723, Singapore

Abstract

A set of batch adsorption kinetic tests of atrazine adsorption by powdered activated carbon (PAC) was performed using air bubbling as the mixing method. It was found that air bubbling at appropriate rates could achieve good mixing. Even a slight turbulence generated by a few bubbles could provide a reasonable mixing to facilitate PAC adsorption compared with unstirred processes.

The estimated mass transfer coefficient in the liquid film surrounding the PAC particles increased linearly with the increase in air bubbling rate up to a plateau value. The experimental mass transfer coefficients in the bubbling system compared favorably with values calculated using correlations developed for conventional magnetic stirring systems, with the help of a conversion of the bubbling rates to the equivalent stirring speeds.

The effect of intermittent air bubbling on the adsorption rate was also tested by generating bubbles intermittently at different net air flow rates. It was found that at the same net flow rate, intermittent higher intensity sparging could be more efficient for the PAC adsorption than continuous lower intensity sparging. This suggests that intermittent high intensity bubbling is the preferable operation, with the potential not only to assure good PAC adsorption efficiency but also to reduce the air/energy consumption.

Keywords: Air bubbling; Powdered activated carbon; Mass transfer coefficient; Intermittent bubbling

1. Introduction

For trace organics removal from aqueous solution, the combination of powder activated carbon (PAC) adsorption with microfiltration (MF)/ultrafiltration (UF) membranes is attractive as it takes advantages of both processes to treat the organics [1–3]. The organic solutes can be adsorbed onto the PAC particles with high efficiency, while the fine PAC particles can be separated from the treated water by membrane filtration [4,5]. Submerged membranes can be used for the combined process with PAC in suspension. In this case, the membrane is mounted directly in the adsorption vessel, and air bubbling can be injected into the vessel to provide mixing for adsorption and to improve the filtration efficiency by creating turbulence for control of membrane fouling [6]. Moving bubbles generate shear and liquid movement that can disrupt the concentration polarization layer or cake formation, diminish the boundary layer resistance and accordingly, increase the permeability [7] and prolong the operating lifespan [8].

Although the effect of bubbling on membrane processes has been actively studied and reviewed [6], the role of bubbling on the PAC adsorption process is not yet elucidated. The adsorption process consists of four consecutive steps [9]: (1) external diffusion in the bulk solution; (2) diffusion in the liquid film surrounding the carbon particle; (3) surface diffusion through the pores of the carbon; and (4) the contaminant being adsorbed onto the active sites in the micropores. Of these, bubbling may have an effect on the first two steps.

Mass transfer in gas–liquid–solid systems has been intensively studied in chemical engineering for decades [10–13]. However, different from a typical three-phase system, the bubbling applied to the PAC-submerged membrane system acts only to promote mixing and turbulence without involving mass transfer in the gas phase. The situation is similar to a previous study [14] on the rate of solid–liquid mass transfer with interfacial bubble generation, where mass transfer took place only at the

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* Corresponding author. Tel.: +65 6794 3801; fax: +65 6792 1291.
E-mail address: AGFane@ntu.edu.sg (A.G. Fane).

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Nomenclature

a	specific interfacial area based on the liquid volume (m^{-1})
C_0	initial adsorbate concentration in bulk solution (μg/L)
C_t	adsorbate concentration in bulk solution at any time t (μg/L)
C_{st}	liquid phase adsorbate concentration at solid–liquid interface (μg/L)
d_a	stir bar diamter (m)
d_p	carbon particle diameter (m)
D_v	volumetric diffusivity for dilute liquid solutions (cm^2/s)
K_l	mass transfer coefficient in liquid film (cm/min)
M_B	molecular weight of solvent (g/mol)
n	stirring speed (rmp)
Q_b	bubbling rate (L/min)
Re	Reynolds number
Sc	Schmidt number
Sh	Sherwood number
T	absolute temperature (K)
t	operation time (min)
u_0	velocity of approaching stream (m/s)
u_m	the magnetic stirring speed (rpm)
V_A	molar volume of solute as liquid at its normal boiling point (cm^3/gmol)

Greek letters

μ	viscosity of solution (c_p, 10^{-3} kg/ms)
ρ	density of solution (kg/m^3)
ψ_B	association parameter for solvent, for water $\psi_B = 2.6$

solid–liquid interface and no mass was exchanged with the bubbles. This study referred to: (a) the ascending bubble swarm causing an upward flow which reduces the diffusion layer thickness at the solid surface, and (b) the detached bubbles inducing radial momentum transfer which brings a fresh supply of the liquid reactant to the solid surface. Although their discussions were based on a larger flat solid surface, it may still assist on understanding the effect of upward bubble motion on the mass transfer in the suspended PAC particle–liquid system. Other researchers [15] have reported that the liquid–solid mass transfer coefficients increased with gas velocity up to 6 cm/s, but were constant at higher gas throughputs. The particle Reynolds number (*Re*) was correlated to an energy dissipation rate to avoid the difficulty of estimating the relative velocity between the suspended particle and the fluid. This method is more suitable for relatively high Reynolds numbers (>1500), which are not appropriate to our low air flows and small particle sizes. However, we have observed improved adsorption kinetics by bubbling in our previous studies [16], provided no other impurity was introduced during the process of air bubbling.

The enhancement of liquid–solid mass transfer by mechanical stirring has already been well studied [17]. Since the air bubbling in our system plays a similar role to mechanical stirring in enhancing mixing, we aim to link the superficial gas bubbling velocity to a mechanical stirring speed by a mixing experiment. Assuming a relationship between mixing and liquid flow patterns and mass transfer we then use mass transfer correlations developed for conventional mechanically stirred systems to determine the effect of bubbling on mass transfer from liquid to PAC particles. In addition, as intermittent bubbling can be used to improve membrane performance instead of continuous bubbling to reduce energy consumption and cost, the effect of this approach on the PAC adsorption kinetics has been examined. This study should provide insight into the preferred bubbling conditions that could be applied in the hybrid membrane adsorption system process.

2. Background

The dynamics of the adsorption of organic compounds onto activated carbon in water can be described using the well-developed homogeneous surface diffusion model (HSDM) [18]. The model incorporates the liquid film mass transfer coefficient and the surface diffusion coefficient to describe the overall carbon adsorption process. It is commonly recognized [9] that the adsorption rate is controlled by boundary layer film diffusion in the initial stages of adsorption in batch reactors. Eventually, as the carbon becomes loaded with the adsorbates, surface diffusion becomes the controlling factor. Therefore, to observe the bubbling effect on the mass transfer in the liquid film, an alternative to fitting data to the complex HSDM is to use a simple approach based on the general mass balance in a batch kinetic adsorption test to describe only the initial stage of an adsorption process. For the simplified approach we assume that film diffusion is the rate-controlling step in the initial stage of atrazine adsorption [19], and thus, the rate of concentration change is approximately equal to the film diffusion rate. The atrazine concentration change in the bulk liquid with adsorption time can then be described using the following equation:

$$\frac{dC_t}{dt} = -K_l a(C_t - C_{st}) \qquad (1)$$

where C_t is the adsorbate concentration in bulk solution at time t, C_{st} the liquid phase adsorbate concentration at the solid–liquid interface, K_l the mass transfer coefficient in the liquid film and a is the specific interfacial area based on the liquid volume for a completely mixed batch reactor. At the beginning of the kinetic test, C_{st} is near zero and Eq. (1) can be simplified as:

$$\frac{dC_t}{dt} = -K_l a C_t \qquad (2)$$

Solving the first order Eq. (2), the K_l can be written as:

$$K_l = -\frac{1}{at} \ln\left(\frac{C_t}{C_0}\right) \qquad (3)$$

where C_0 is the initial adsorbate concentration in the bulk solution at $t = 0$. Based on the experimental batch data, an estimate of the mass transfer coefficient at different bubbling rates in the initial stage can be obtained.

The experimental liquid film mass transfer coefficients surrounding a particle in the completely mixed batch reactor can be compared with available correlations [17] via the Sherwood number:

$$K_1 = \frac{Sh\,D_v}{d_p} \tag{4}$$

where Sh is the Sherwood number, D_v the volumetric diffusivity for dilute liquid solutions and d_p is the carbon particle diameter. For mass transfer to an isolated sphere, the Sherwood number can be estimated from [17]:

$$Sh = 2.0 + 0.6\,Re^{1/2}\,Sc^{1/3} \tag{5}$$

where Re is the Reynolds number and Sc is the Schmidt number. Eq. (5) is claimed to be accurate for Reynolds number up to 1000. The Reynolds number for a particle in a fluid is defined as:

$$Re = \frac{u_0 \rho d_p}{\mu} \tag{6}$$

in which u_0 is the relative velocity of the particles to the fluid, ρ the density of the solution and μ is the viscosity of the solution. For a mechanically stirred vessel the upper limit of u_0 would be the tip speed of the impeller, nd_a. In our situation d_p is of the order 10 μm (10^{-5} m) which requires $u_0 > 100$ m/s to give $Re = 1000$. In the bubbled vessel, the used u_0 will be <1 m/s, so Eq. (5) should apply.

The Schmidt number is defined as:

$$Sc = \frac{\mu}{\rho D_v} \tag{7}$$

Volumetric diffusivity for dilute liquid solutions can be calculated approximately from the following equation [17], which is valid only at low solute concentrations:

$$D_v = 7.4 \times 10^{-8} \times \frac{(\psi_B M_B)^{1/2} T}{\mu V_A^{0.6}} \tag{8}$$

in which ψ_B is association parameter for solvent (for water $\psi_B = 2.6$), M_B the molecular weight of solvent, T the absolute temperature and V_A is the molar volume of solute as liquid at its normal boiling point.

3. Experimental

3.1. Materials

Powdered activated carbon (Norit SA-2) with mass median diameter (d_{50}) of 6.3 μm (milled) was used as a representative adsorbent in this study. Atrazine, a common herbicide was chosen as the targeted compound to represent trace organics typically found in contaminated surface water. The preparation procedure has been described elsewhere [16].

3.2. Methods

The bubbling mixed batch kinetic tests were conducted using the same set-up as in our previous study [16]. Air was transported by stainless steel tubing in all the tests to avoid contaminant uptake. The air bubbles, which were generated through a ceramic bubble diffuser, had a median diameter of 3 mm, measured photographically. Intermittent bubbling was conducted using the same apparatus and a solenoid valve, which was installed between the gas cylinder and the reactor. Two intermittent regimes were compared with on/off periods of 0.5 s-on-1.0 s-off and 0.5 s-on-2.0 s-off, respectively. Samples were taken at predetermined time intervals and atrazine concentration was analyzed using a high-performance liquid chromatography (WATERS 2695, Separations module, XTerra C_{18} column, 50% acetonitrile + 50% 10 mM ammonium bicarbonate buffer).

To correlate air bubbling with magnetic stirring, a series of mixing tests were conducted. The vessel used for mixing was the same as that used for batch adsorption, which was 0.06 m in diameter, 0.44 m in height and with an operating volume of 1 L. Acridine Orange Base (AOB dye) was used as the target compound and a spectrophotometer (Jasco) was used to detect the target concentration at UV491 nm. At the start of mixing, 5 mL of the AOB dye at a concentration of 1 g/L was injected quickly into the bottom of the vessel in which mixing was provided by either magnetic stirring or air bubbling. Immediately, a series of samples from the top of the liquid were taken by a peristaltic pump sipper (Jasco NPF-509) installed inside the spectrophotometer. Sample measurement was performed automatically after sample suction and the AOB concentrations were recorded after measurements by a connected computer.

4. Results and discussion

4.1. Continuous bubbling

The effect of air bubbling rate on the adsorption rate at a carbon dosage of 5 mg/L in the batch kinetic tests is compared with magnetic stirring results in Fig. 1. Air bubbling was beneficial to adsorption provided air bubbles generate a little turbulence. For the 0.5 L/min bubbling rate, atrazine adsorption was slightly slower than that of magnetic stirring. However, when compared with the unstirred test, the adsorption process performed much better if a few bubbles were sparged into the system. The low bubble rate could still provide adequate mixing to facilitate PAC adsorption. For 1.5, 2.7 and 5.0 L/min bubbling tests, the initial slopes of the kinetic curves dropped more rapidly than that of magnetic stirring, indicating a faster adsorption rate. Therefore, bubbling is an effective mixing method for adsorption by suspended PAC and the bubbling rate is an important parameter to be considered for maximizing the adsorption rate economically.

Fig. 2 shows the results of similar experiments as in Fig. 1 but for double the PAC dosage. Comparing these two figures, the adsorption rate increased with the increase of PAC dosage at all conditions. In the 10 mg/L PAC tests, it was found that the adsorption rate could be improved significantly (compared with no stirring) by introducing only a low air flow rate (0.2 L/min).

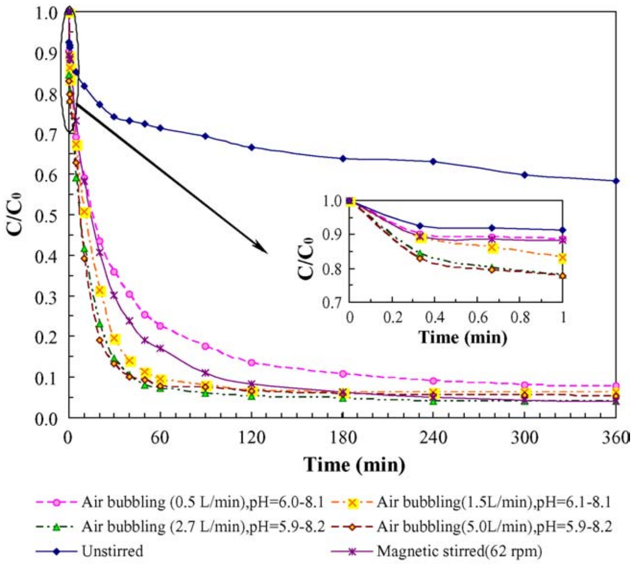

Fig. 1. Effect of air bubbling on atrazine adsorption by PAC, stainless steel tubing ($C_c = 5$ mg/L, $C_0 = 200$ μg/L, $d_{50} = 6.3$ μm).

Almost complete atrazine removal could be rapidly achieved by introducing bubbles at the rate of 3.0 L/min. For the two carbon dosages, further increase in bubbling to 5.0 L/min (data not shown in Fig. 2) did not noticeably enhance removal rate indicating that there is a limit to bubble-induced mass transfer. From economic considerations, the optimum bubbling rate should be sought first through batch tests before being applied to the hybrid membrane system. However, the scale-up of vessel size and sparging needs to be considered.

4.2. Correlation between the bubbling rate and the magnetic stirring speed

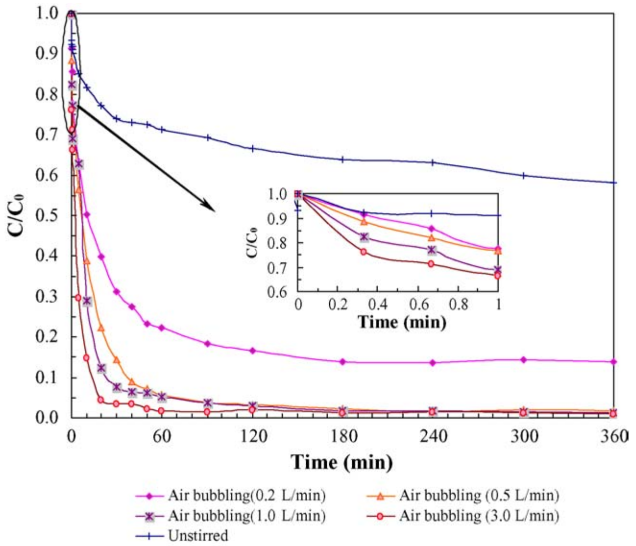

Fig. 2. Effect of air bubbling on atrazine adsorption by PAC, stainless steel tubing ($C_c = 10$ mg/L, $C_0 = 200$ μg/L, $d_{50} = 6.3$ μm).

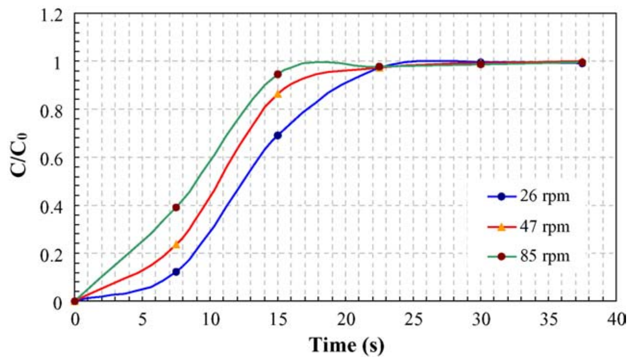

Fig. 3. Response of AOB dye concentration at the top of liquid (magnetic stirring).

In order to compare the mass transfer coefficients in the bubble mixed batch kinetic tests with predictions for mechanical stirring, the air bubbling rate was correlated to the magnetic stirring speed through a set of mixing experiments. Fig. 3 shows the results of the AOB dye tracer tests with concentration increase with time at different magnetic stirring speeds. It can be seen that the higher the stirring speed, the faster the concentration reaches a maximum value which represents a fully mixed solution. The times for achieving 60% of maximum concentration are 13.6, 11.8, 10.0 s at magnetic stirring speeds of 26, 47 and 85 rpm, respectively. Sixty percent was chosen as the point of comparison because the curves of mixing extent over time were smooth in this middle region so that the sharp changes in the slopes of the curves around lower and higher percentages can be avoided for more reliable comparison. Plotting these times and stirring speed values gives a linear relationship (Fig. 4):

$$u_m = -16.39t + 246.1 \tag{9}$$

where u_m is the magnetic stirring speed (rpm). Eq. (9) provides a means for estimating what magnetic stirring speed has a similar mixing effect as a certain air bubbling rate.

As shown in Fig. 5, the response to the air bubble mixing has a similar trend to the magnetic stirring. The higher the bubbling rate, the faster the liquid was fully mixed.

The times to reach 60% of the maximum concentration are 12, 10.7, 4.6, 4.0 s at 0.5, 1.5, 2.7, 5.0 L/min bubbling rates, respectively. Using Eq. (9), the equivalent stirring speeds can be estimated, as listed in Table 1. It can be seen that 0.5, 1.5, 2.7,

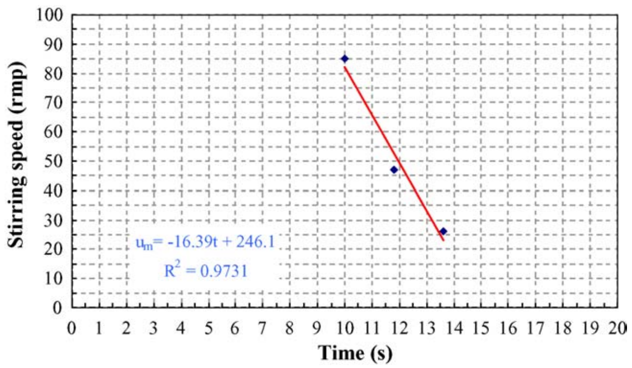

Fig. 4. Linear regression of the stirring speeds vs. time required to achieve 60% of complete mixing.

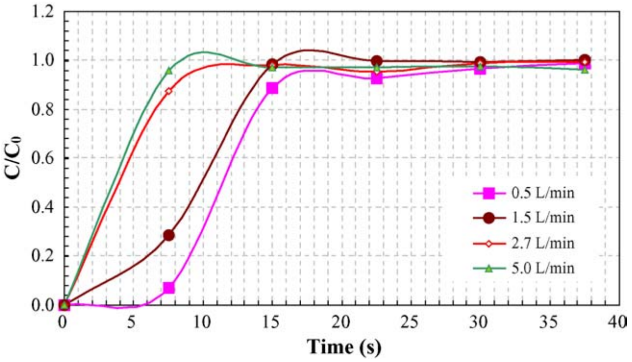

Fig. 5. Response of AOB dye concentration at the top of liquid (air bubbling).

Table 1
Conversion of the bubbling rates to equivalent stirring speeds

Bubbling rate (L/min)	Time (s)	Converted to stirring speed (rpm)
0.5	12.0	49
1.5	10.7	71
2.7	4.6	172
5.0	4.0	181

5.0 L/min bubbling can be converted to 49, 71, 172, 181 rpm of magnetic stirring, respectively, with an equivalent mixing ability using the same stirrer bar (16 mm in diameter). This is consistent with the observation in Fig. 1 that 1.5 L/min bubbling results in an improved adsorption rate over that of the 62 rpm magnetic stirred test, and that high bubbling rates do not continue to improve adsorption significantly.

4.3. *Determination of mass transfer coefficient K_l*

To estimate K_l, the averaged C/C_0 values in the first 1 min were used. These values were thought to be sufficiently early in the kinetic test (duration 6 h) to be controlled by film diffusion but not be subject to 'start-up' errors. The correlation between the experimental K_l and the bubbling rates is plotted in Fig. 6. It can be seen that the mass transfer rate K_l increased linearly with increase of the air bubbling rate ($R^2 = 0.9809$) over a certain range of the air bubbling rate, but further increase in the air bubbling rate (e.g. to 5.0 L/min) could not result in a more enhanced mass transfer. A similar trend was also seen in 10 mg/L PAC dosage tests.

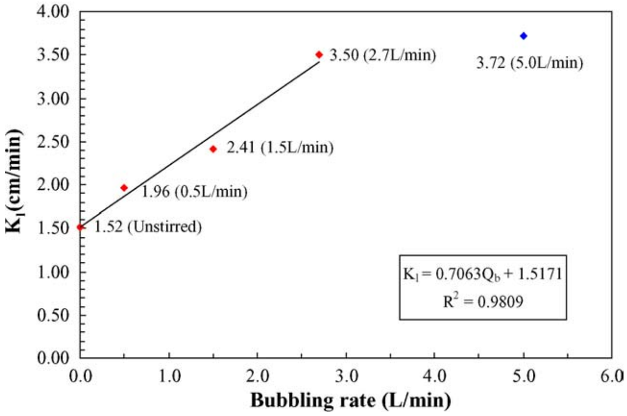

Fig. 6. Effect of bubbling rate on mass transfer rate in the initial stage of adsorption ($C_c = 5$ mg/L).

Other researchers [20] have also found that the solid–liquid mass transfer at a stationary single sphere placed in a bubbled shallow column was improved by increase of the superficial gas velocity up to a limit value. The mass transfer enhancement was attributed to the microscale high-intensity eddies induced by the rising bubbles. At very high gas velocity, the formed slug flow was not able to completely transfer energy into microscale eddies but only induce an overall circulation of liquid in the column. Similarly, in this study, increasing the bubbling rate to a certain value may induce more microscale eddies which associated with more generated shear. Accordingly, the thickness of the liquid film layer surrounding the particles is attenuated by the fluid shear. Therefore, the mass transfer through the liquid film surrounding the carbon particles can be improved by increasing the bubbling. However, above the limiting bubbling, the thickness of the film layer cannot be easily reduced and thus the mass transfer rate does not increase further. Therefore, optimization of the bubbling rate for the enhancement of adsorption efficiency is needed from the energy consumption point of view.

In our experiments, the PAC particles were well suspended in the liquid and moved with the eddies generated by the bubbles. However, direct contact of the particles and the bubbles were not obvious because of the low carbon dosage; there was no evidence of carbon segregation to the surface by flotation processes. Bubble coalescence during the upward motion in the vessel was also considered negligible because the operating conditions in this study were in the homogeneous regime of a multiphase system. According to Kluytmans et al. [21], the transition point from homogeneous to heterogeneous regimes for distilled water was at a superficial gas velocity of 0.03 ± 0.005 m/s and was delayed to 0.035 ± 0.005 m/s by the addition of PAC particles ($d_p = 30$ μm) at 0.1–1.0 g/L carbon concentrations. Compared to their tests, the carbon concentration in this study was much lower (5, 10 mg/L) and the bubbling flow rates used were in the range 0.003–0.03 m/s. Therefore, the liquid conditions in this study were closer to the homogeneous regime and the bubble coalescence should not be significant. There is clearly more that could be done in terms of bubble characterization and measurement of induced shear. This study will be reported in future publications.

The mass transfer coefficient K_l relevant to the bubbling condition can also be estimated from available mass transfer correlations using Eqs. (4)–(8). The Reynolds number of a carbon particle in the bubbling system was estimated using the corresponding stirring speeds via the conversion data in Table 1 by assuming that the fluid velocity adjacent to the carbon particles is the same as the imposed fluid velocity in the vessel caused by the stirrer bar. The results are listed in Table 2 and compared with the experimental K_l obtained from the mass balance of Eq. (3). The difference between the two calculations is below 27%. This result shows us how the mass transfer in the bubbling system can be characterized quantitatively based on the correlations used for conventional mechanically stirred systems.

Table 2
Comparison of K_l from different methods

Bubbling rate (L/min L)	K_l from data and Eq. (3)	Calculation from Eq. (4)–(8)			
	Average in 1 min (cm/min)	K_l (cm/min)	u_0 (m/s)	*Re*	*Sh*
0.5	1.96	2.66	0.01	0.09	3.97
1.5	2.41	2.93	0.02	0.13	4.37
2.7	3.50	3.81	0.05	0.32	5.69
5.0	3.72	3.88	0.05	0.34	5.79
Magnetic stirred 62 rpm	2.06	2.82	0.02	0.12	4.22

Note: $D_v = 7.0 \times 10^{-6}$ (cm^2/s), $Sc = 1274.54$.

4.4. Intermittent bubbling

Fig. 7 shows the trends of the adsorption rate at different bubbling rates which were introduced to the reactor intermittently at 0.5 s-on-1.0 s-off and 0.5 s-on-2.0 s-off, respectively. It is clear from Fig. 7(a–c) that a shorter bubbling pause (1.0 s off) is more effective than a longer bubbling pause (2.0 s off), but less efficient than the continuous bubbling. This indicates that more bubbles per unit time injected facilitate the PAC adsorption. By comparing Fig. 7(a–c), it is also easily seen that the difference in the adsorption performance between the intermittent and continuous bubbling becomes less with increase in bubbling rates. This implies that intermittent bubbling may be more suitable for higher air flow rate operations in terms of both the energy consumption and adsorption performance.

Fig. 8 shows the required time to achieve 20% of the initial atrazine concentration for two intermittent operations which have approximately the same net air consumption of 0.5 L/min but different intensities of air injection compared with 0.5 L/min continuous bubbling. The intermittent conditions were 1.5 L/min bubbling with 0.5 s-on-1.0 s-off intermittence and 2.7 L/min bubbling with 0.5 s-on-2.0 s-off intermittence, respectively. It can be seen that 2.7 L/min intermittent bubbling gave the most benefit ($t_{0.2} = 27$ min) to the adsorption rate despite the 2.0 s pause interval, followed by the 1.5 L/min bubbling ($t_{0.2} = 36$ min) with shorter pause interval of 1.0 s. 0.5 L/min continuous bubbling brings the least benefit ($t_{0.2} = 71$ min) to the PAC adsorption. This example shows that for the same amount of air injection, intermittent higher intensity sparging is significantly more efficient than continuous lower intensity sparging

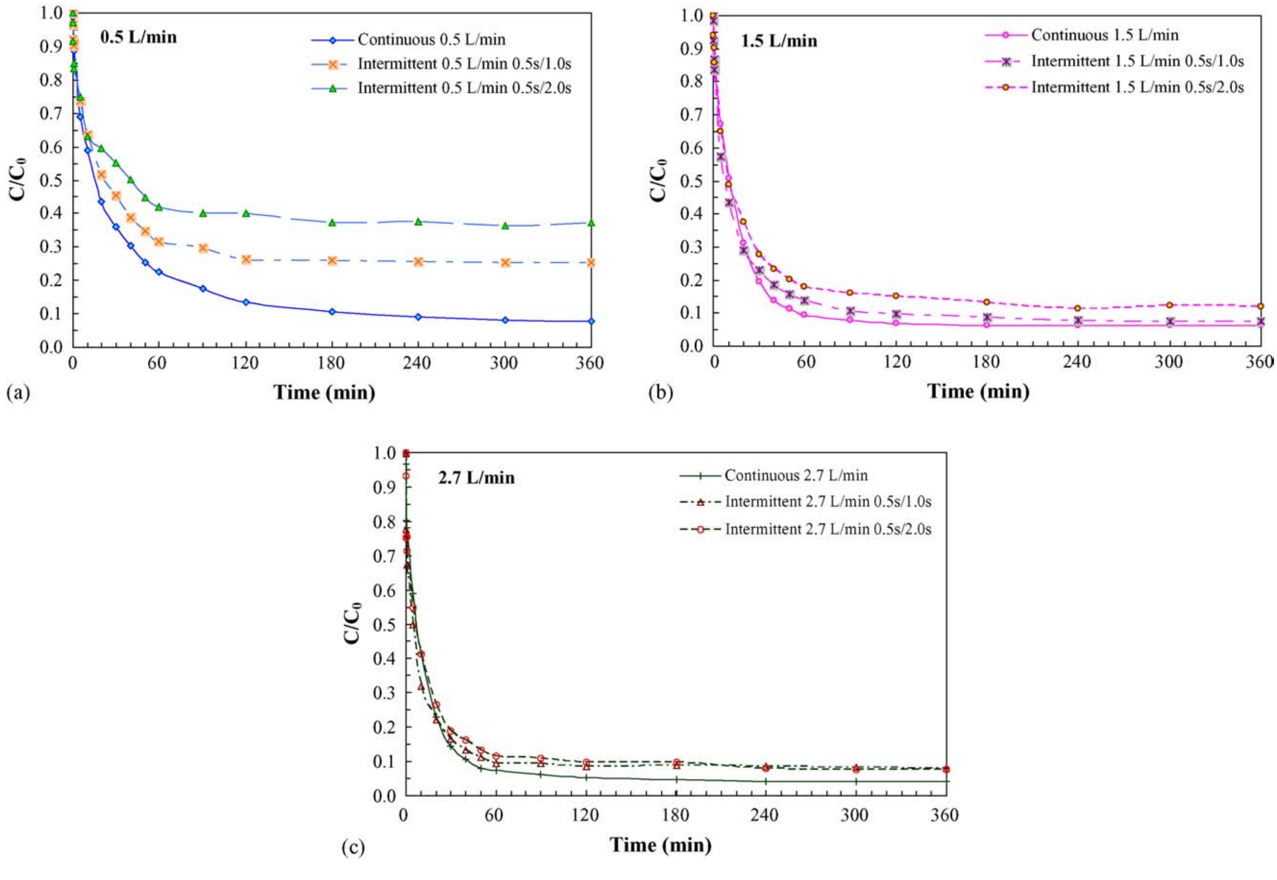

Fig. 7. (a–c) Effect of intermittent bubbling adsorption kinetics ($C_c = 5$ mg/L, $C_0 = 200$ μg/L, $d_{50} = 6.3$ μm).

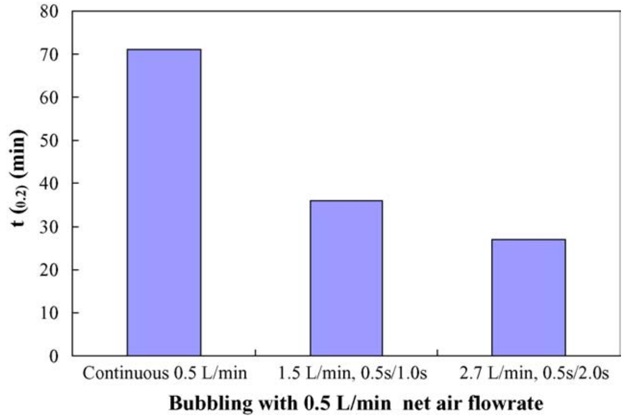

Fig. 8. Comparison between intermittent and continuous bubbling with equivalent flow rate ($C_c = 5$ mg/L, $C_0 = 200$ μg/L, $d_{50} = 6.3$ μm).

in terms of PAC adsorption rate. The adsorption approaches the equilibrium faster in the former mode of bubbling. This result points to an important approach for improving the system performance economically. Obviously, the intermittency and net bubbling rate can be optimized to minimize air and power usage.

5. Conclusions

Air bubbling was found to be a favorable approach to enhance PAC adsorption of a dilute organic solute as the mass transfer coefficient in the liquid film surrounding the PAC increased linearly with increase of air bubbling rate over a certain range. Good mixing could be achieved by air bubbling at moderate flow rates. Even the slight turbulence generated by a few bubbles could provide a reasonable mixing to facilitate the PAC adsorption compared with the unstirred process.

The mass transfer coefficient through the liquid film in the bubbling system was calculated from the initial experimental data and the first order mass balance equations. These values were compared with mass transfer correlations, which were developed for conventional magnetic stirring systems, with the help of the conversion of the bubbling rates to the equivalent stirring speeds. The differences between experimental and predicted values were within 27%. In general, the results show that the mass transfer coefficient K_l is improved by the increase in air bubbling rate to a plateau value.

In addition, the use of intermittent air bubbling was found to have potential for higher air flow rate operations in terms of both the energy consumption and adsorption performance. For the same amount of net air injection applied to the process, intermittent higher intensity sparging is more efficient for PAC adsorption than continuous lower intensity sparging. It is recommended to use intermittent bubbling with slightly higher air flow rates during injection, as this would not only assure the PAC adsorption efficiency but also potentially reduce the air/energy consumption.

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