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Effect of superficial gas velocity and ratio of bed volume to reactor volume of inverse fluidized bed biofilm reactor on the removal of ammonia-nitrogen from wastewater

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Hydrodynamic parameters of an inverse fluidized bed biofilm reactor (IFBBR) have been studied using spherical polypropylene (PP) particles having average diameter and density of 5.63 mm and 920 kg/m^3 respectively. Gas-phase holdup (ε_g) was analyzed for various ratios of settled bed volume to reactor volume (V_b/V_r) and superficial gas velocities (U_g) with a liquid recirculation velocity (U_l) of 0.0021 m/s. The ε_g values were found to increase with V_b/V_r ratios up to a certain limit and then decrease with further increase of V_b/V_r ratios. The effect of U_g, V_b/V_r ratios, and initial concentration of ammonia-nitrogen (NH_4^+-N) on the removal of NH_4^+-N from synthetic wastewater were studied. The optimal values of V_b/V_r ratio and U_g were found to be 0.380 and 0.0085 m/s respectively for all initial NH_4^+-N concentrations. Complete removal of NH_4^+-N was achieved in 8 to 44 hours for different initial NH_4^+-N concentrations. It was also observed that with the increase in initial NH_4^+-N concentrations, the nitrification decreases.

Keywords: Ammonia-nitrogen, Biofilm reactor, Inverse fluidization, Nitrification, Wastewater treatment

The detrimental effects of NH_4^+-N present in wastewater are eutrophication; reduction of dissolved oxygen (DO) level; and toxicity to human, animal, and aquatic species[1]. Its removal from wastewater can be achieved by chemical, physical, combined chemical-physical, or biological treatment methods[2]. Among these methods, the biological treatment methods are widely used for the removal of NH_4^+-N from wastewater due to its high efficiency and low operating costs[3]. The biological method of converting NH_4^+-N to unstable nitrite-nitrogen (NO_2^--N) and then to less toxic nitrate-nitrogen (NO_3^--N) is known as nitrification. The conversion of NH_4^+-N to NO_3^--N proceed faster due to which at any instance the level of NO_2^--N in the system is usually low[4]. The basic nitrification reactions[2] can be written as:

$$NH_4^+ + 1.5\ O_2 \xrightarrow{Nitrosomonas} 2\ H^+ + H_2O + NO_2 \quad \ldots (1)$$

$$NO_2^- + 0.5\ O_2 \xrightarrow{Nitrobacter} NO_3^- \quad \ldots (2)$$

The nitrification steps are dependent on *p*H; temperature; alkalinity; DO level; concentrations of NH_4^+-N, NO_2^--N, and NO_3^--N; presence of heavy metals and toxic substances; and concentration of organic substrate[3,5]. The nitrification steps are carried out by two different growth conditions of bacteria: suspended and attached (as biofilm) growth conditions. The reactors based on attached growth condition have several advantages such as minimum wash off of the microorganisms, ease of handling, better stability, and capability to withstand shock loading[6]. These reactors require less floor area for erection and the separation of solids is easier[5]. Furthermore, the growth of the nitrifying bacteria is slow and can easily be swept away from the treatment systems. Therefore, it is essential to use such type of biofilm reactors which can hold the biomass inside the reactor for long duration and can also facilitate their growth[3].

In the fluidized bed biofilm reactors biomass can be hold for long duration as well as their growth can easily be facilitated due to which these reactors were in use for the removal of NH_4^+-N from wastewater[7-11]. Though the use of fluidized bed biofilm reactors is advantageous, the use of IFBBRs has emerged in recent times for the treatment of wastewaters generated from a wide variety of industries[12,13].

In conventional three-phase fluidized bed biofilm reactors (FBBRs), the particle density is higher than the density of continuous liquid phase and the fluidization of solid particles is in the upward direction by the upward flow of both liquid and gas phases. While in the IFBBRs, the particle density is less than the density of continuous liquid phase and the fluidization of solid particles is in the downward direction by either co-current upward flow of both liquid and gas phases or downward flow of liquid with counter current upward flow of gas phase[14]. Some of the critical features of IFBBRs are higher ε_g; higher heat and mass transfer rates; lower bed pressure drop; higher volumetric efficiency; better stability; absence of clogging and channeling of the bed; ease of refluidization in case of power failure; and less power consumption. These reactors also offer uniform biofilm thickness, minimum wash off of biomass, better contact among phases, faster biofilm formation, and greater biodegradation effect. As well as these reactors are easy to operate and have low operating costs[12,13].

Due to the wide features of IFBBRs, these reactors find their applications for the removal of pollutants such as phenol[15-17]; ferrous iron[18,19]; sulfate[20]; aniline[21]; and ammonia[2,22]. However, the use of IFBBRs for the removal of NH_4^+-N from wastewater is not widely reported.

Bougard *et al.*[22] were probably the first group of investigators to report the nitrification of a high-strength ammonia wastewater (250 - 2000 mg NH_4^+-N L^{-1}) using an inverse turbulent bed reactor with V_b/V_r ratio of 0.2 and with light mineral granular solids as support particles. The study was carried out at temperatures of 30 and 35°C and *p*H of 7.2 at U_g of 6 mm/s for a period of 350 days. Complete removal of NH_4^+-N was reported with nitrification of 68-100%. Devi and Setty[2] achieved 92.8% removal of NH_4^+-N within 22 h using a three-phase bioreactor with lighter density PP beads at an air flow rate of 2 lpm, *p*H of 7, temperature of 30°C, and V_b/V_r ratio of 0.00334 for an initial NH_4^+ concentration of 100 ppm. The nitrification was reported to be 81.04%.

Literature review reveals that the phase holdup, particularly ε_g, is a significant hydrodynamic parameter. Sufficient literatures were not found for the study of ε_g with respect to the variations of V_b/V_r ratios and U_g. Also, the studies of the effect of V_b/V_r ratios and U_g on the removal of NH_4^+-N from wastewater was found to be missing. As well as these studies are limited to a very narrow range of operating parameters. Keeping this in mind, an IFBBR was used in the present study to find the effect of hydrodynamic parameters (V_b/V_r ratios and U_g) on the removal of NH_4^+-N from wastewater and find their optimal values.

Experimental Section

IFBBR setup

The IFBBR used in this study (Fig. 1) is a batch type of reactor consisting of an acrylic column having overall height of 100 cm and internal diameter of 10 cm (having internal volume of 7.8 L). A tank was used along with the IFBBR to provide a total working volume of 10 L. Air inlet was provided at the bottom of the column through an air-sparger and air-distributor plate. Water was fed from the top of the IFBBR through a water-distributor plate. Temperature control system was provided to the outer tank to which a centrifugal pump was connected to recirculate the water in to the column. Distributor plates distribute gas and liquid phases uniformly as well as prevent the escape of the solid particles from the column. Rotameters were used to control the air and water flow rates. A height-adjustable discharge pipe was attached at the bottom of the column to maintain the desired water level inside the column. An air-vent was provided at the top of the IFBBR to allow the waste gases to be vented out and to avoid pressure buildup in the column. Spherical PP particles (density of 920 kg/m^3 and average diameter of 5.63 mm) were used as biomass support particles. PP particles were treated with solutions of ferric chloride and chlorosulfonic acid to make the surfaces hydrophilic[23].

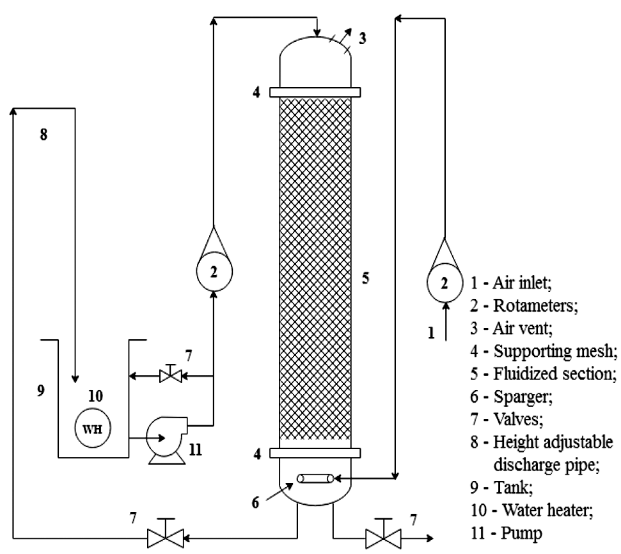

Fig. 1 — Schematic representation of the IFBBR.

Procedure for study of hydrodynamic parameters

Filtered tap water and oil and moisture free compressed air were used for studying the hydrodynamic parameters. For a particular volume of solid particles (bed volume, V_b), calculated weight of PP particles was fed to the column. Air and water were introduced counter-currently to the column. The water level in the column was maintained by adjusting the height of the discharge pipe. Air and water flow rates and V_b/V_r ratios were varied to estimate the ε_g. Nearly 10 min time gap was given between two successive set of readings for achieving the steady state. All the experiments were carried out in duplicate. The hydrodynamic parameters with their operating range are mentioned in Table 1.

Microorganisms and culture medium

Sludge was collected from the wastewater treatment plant of a local steel industry. Acclimatization of this sludge (1% v/v) with the growth medium was done in an incubator shaker at 30^0C and 100 rpm[2]. At every 48 h time interval, 1% v/v of the sludge was treated with the growth medium afresh. This procedure was repeated for a period of two weeks to allow the adaption of the microorganisms with the growth medium. The culture thus obtained was used as inoculum for the IFBBR for the development of biofilm over the PP particles. Table 2 shows the composition of the growth medium[8]. The synthetic wastewater was also prepared with the same composition corresponding to an initial NH_4^+-N concentration of 40 mg/L. The synthetic wastewater corresponding to other higher NH_4^+-N concentrations (100 and 200 mg/L) were also prepared according to the recommended composition ratio. All the solutions were prepared using the water obtained from Milli-Q RiOs™ 3 Water Purification Unit (Millipore, ZR0S0P3WW, Germany) which were further sterilized at 121°C and 15 psi for 20 min in an autoclave to prevent contamination[24].

Table 1 — Operating range of the hydrodynamic parameters.

Parameter	Range
V_b/V_r ratio	0.152 - 0.445
U_g	0.0064 - 0.0127 m/s
U_l	0.0021 m/s

Table 2 — Composition of the growth medium.

Compounds	Composition (mM/L)
NH_4Cl	2.861
Na_2HPO_4	0.328
$NaHCO_3$	5.572
NaCl	0.352
KCl	0.130
$CaCl_2$	0.087
$MgSO_4.7H_2O$	0.137

Reactor inoculation and operation

The IFBBR was acid washed (0.1 N HCl solution) followed by water washed several times and then drying at 60°C using hot air blower. The IFBBR was filled with the calculated volume of PP particles corresponding to a particular V_b. Calculated volume of filtered tap water with the growth medium corresponding to low strength of NH_4^+-N (40 mg/L) was pumped to the reactor to which oil and moisture free air was fed at 0.0064 m/s. The U_l was maintained at 0.0021 m/s. The temperature and *p*H were kept at 30±0.3°C and 8.3±0.1 respectively as it was observed from the literature[25] that the optimum temperature and *p*H for nitrification are 30°C and 8.1 to 8.5 respectively. The 1% (v/v) culture prepared in incubator shaker was added to the reactor for the inoculation of the IFBBR and for the development of biofilm. The IFBBR was then allowed to run for a period of two weeks' time with NH_4^+-N feeding rate of 40 mg/L/d under liquid recirculation mode for faster formation of biofilm over the solid particles[26]. Liquid recirculation ensures the solution to be homogeneous and removes the stationary regions from the reactor[15]. Suspended biomass was continuously filtered out during liquid recirculation to prevent them from entering the IFBBR[27].

After the formation of biofilm and the reactor was filled with calculated volume of synthetic wastewater corresponding to NH_4^+-N concentration of 40 mg/L. The reactor was operated under non-sterile conditions. Loss of liquid was observed due to aeration. Therefore, double-distilled water was added (0.2 to 0.5% v/v) at 4 h interval to maintain a constant liquid volume of 10 L. As the volume of double-distilled water added was very less the change in concentrations of NH_4^+-N was assumed not to vary much. The effects of U_g (0.0043 to 0.0127 m/s), V_b/V_r ratios (0.228 to 0.445), and initial concentrations of NH_4^+-N (40 to 200 mg/L) on the removal of NH_4^+-N were studied by using this IFBBR. The *p*H and temperature of the contents were maintained at 8.3±0.1 and 30±0.3°C respectively. The U_l was maintained at 0.0021 m/s for all experiments. Duration of 48 to 96 h was given between two consecutive set of operating conditions. This was done to allow the microorganisms to get adapted with the new operating conditions. All the experiments were carried out in triplicate.

Measurement and analysis

The V_b was calculated for a known weight of particles by dividing the weight with the particle density. The liquid height was measured from the air distributor plate for all readings from which the V_r was calculated. The ε_g was measured by volumetric method using the following equation[28,29].

$$\varepsilon_g = \frac{H_a - H_d}{H_a} \quad \ldots (3)$$

where, H_a = height of aerated liquid, m and H_d = height of de-aerated liquid, m.

Biofilms formed over solid particles were observed through environmental scanning electron microscopy (ESEM) images (FEI, Quanta FEG 250, US) for which the solid particles were treated by glutaraldehyde fixation method[30]. Samples from the reactor were collected at 4 h intervals to estimate the concentrations of NH_4^+-N using the spectrophotometer (JASCO, V530, US) for which initially the standard calibration curve for NH_4^+-N was prepared using the standard solutions. All measurements were carried out according to the APHA standard methods[31].

Results and Discussion

Gas-phase holdup

The study of ε_g is important for the aerobic wastewater treatment. This was carried out for V_b/V_r ratios of 0.152 to 0.445 each at U_g of 0.0043 to 0.0127 m/s. For low V_b/V_r ratios of 0.152 and 0.228, fluidization of the bed of particles was observed at the lowest U_g of 0.0043 m/s. While for other higher V_b/V_r ratios of 0.304 to 0.445, the fluidization was observed to occur at U_g of 0.0064 m/s. This fact that, bed with higher V_b/V_r ratios has higher may be due to the weight which needs higher U_g for fluidization[32].

The effect of V_b/V_r ratios on ε_g for different U_g values at U_l of 0.0021 m/s is shown in Fig. 2. It is seen that, the ε_g increases with the increase in U_g values for all V_b/V_r ratios. The ε_g was also found to increase with the increase in V_b/V_r ratios and decrease after reaching the limiting V_b/V_r ratio. These observations are found to be identical with the literature findings[33]. It was also observed that, the limiting V_b/V_r ratio decreases from 0.380 to 0.228 with the increase in U_g from 0.0064 to 0.0127 m/s. This may be due to the breakage of bubbles at higher V_b/V_r ratio and higher U_g. The liquid recirculation in the IFBBR causes higher ε_g at lower U_g with higher V_b/V_r ratios and at higher U_g with lower V_b/V_r ratios. This may be due to the fact that the liquid recirculation in the inverse fluidized column causes more gases to be trapped at these conditions and the minimum fluidization velocities to be decreasing at increasing U_g with decreasing V_b/V_r ratios. At the lowest U_g of 0.0043 m/s, fluidization is not occurring for bed materials having V_b/V_r ratios higher than 0.304. The reason may be the lowest U_g of 0.0043 m/s is not sufficient enough to fluidize higher volumes of bed materials when the inverse fluidized column is operated with liquid recirculation condition. Because with the increase of U_g, the maximum ε_g was found at lower V_b/V_r ratios, the study for the removal of NH_4^+-N was carried out for a wide range of V_b/V_r ratios and U_g to know the optimal operating conditions.

Scanning electron micrographs

Figure 3 shows the ESEM images of the solid support particles before and after the formation of biofilm. Figure 3b suggests the formation of the biofilm over the chemically treated PP particles.

Removal of NH_4^+-N

Effect of time

The study on the removal of NH_4^+-N was carried out for initial NH_4^+-N concentrations of 40 to 200

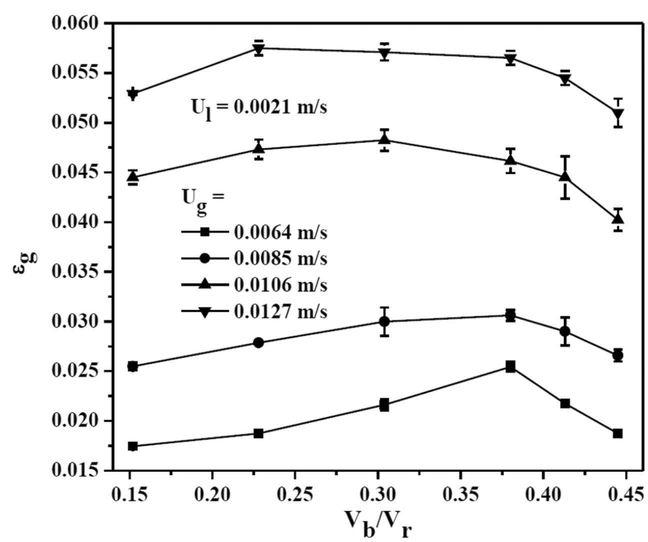

Fig. 2 — Effect of V_b/V_r ratios on ε_g for various U_g at U_l of 0.0021 m/s.

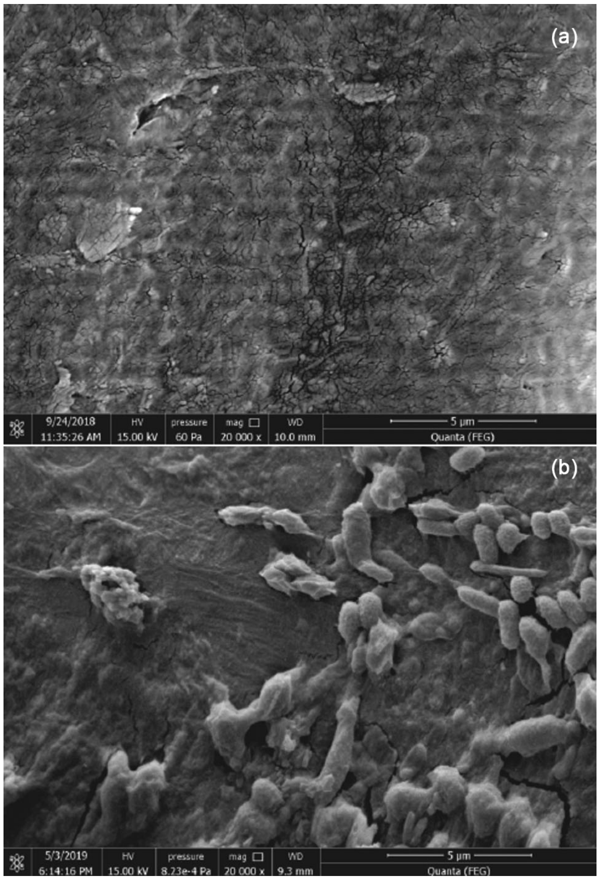

Fig. 3 — ESEM images of the support particles: (a) before the formation of biofilm and (b) after the formation of biofilm.

mg/L, V_b/V_r ratios of 0.228 to 0.445, and U_g values of 0.0043 to 0.0127 m/s. Reductions in concentration of NH_4^+-N with time for initial NH_4^+-N concentrations of 40, 100, and 200 mg/L for different values of U_g and V_b/V_r ratios are shown in Fig. 4A, 4B, and 4C respectively. It is observed that, the maximum reduction was occurring at 4 h time interval for all values of U_g and V_b/V_r ratios for all initial NH_4^+-N concentrations. For this reason, the time duration of 4 h is considered for determining the optimum NH_4^+-N removal conditions. Complete (100%) removal of NH_4^+-N is observed for an initial NH_4^+-N concentration of 40 mg/L within 8 to 16 h (Fig. 4A). Similar reduction trends are also observed for initial NH_4^+-N concentrations of 100 and 200 mg/L. Complete NH_4^+-N removal are observed to occur within 16 to 32 h (Fig. 4B) and 28 to 44 h (Fig. 4C) for initial NH_4^+-N concentrations of 100 and 200 mg/L respectively. With the progress of treatment time, the NH_4^+-N concentration decreases because the nitrifying bacteria consume the NH_4^+-N content for their growth.

Effect of U_g

From Fig. 5a it is observed that for low initial NH_4^+-N concentration of 40 mg/L treated with bed having V_b/V_r ratio of 0.228, maximum reduction of NH_4^+-N occurred at a U_g of 0.0064 m/s. This may be due to the reason that at this combination, the oxygen requirement was sufficient for the growth of microorganisms. With further increase in U_g, the reduction of NH_4^+-N is found to decrease. This may be due to the detachment of biofilm from the surfaces of solid support particles at higher U_g because of higher particle-particle collision and attrition at higher air velocity. For higher initial NH_4^+-N concentrations of 100 and 200 mg/L treated with bed having V_b/V_r ratio of 0.228, maximum reduction of NH_4^+-N occurred at a U_g of 0.0085 m/s. This may be due to the reason that, higher initial NH_4^+-N concentrations require more oxygen for metabolic reactions which was made available at higher U_g[15]. For other higher V_b/V_r ratios of 0.304 to 0.445 the maximum reduction occurred at a U_g of 0.0085 m/s. This may be due to the reason that, with the increase in V_b/V_r ratios the large mass of biofilm formed over solid particles might be requiring more oxygen for metabolic reactions.

From Fig. 5a it is also observed that, the least NH_4^+-N reduction occurred at V_b/V_r ratio of 0.445 for all initial NH_4^+-N concentrations even if higher ε_g was measured at this V_b/V_r ratio as observed in Fig. 2. This may be due to the poor phase mixing caused by higher particle loading inside the IFBBR. Similar phenomenon was observed by Begum and Radha[15] for the treatment of phenol in an IFBBR. Greater NH_4^+-N reduction was observed for V_b/V_r ratio of 0.380 for all initial NH_4^+-N concentrations at U_g of 0.0085 m/s. This may be due to fact that, at this combination the oxygen requirement is adequate for the metabolic reactions and higher rate of nitrification.

Effect of V_b/V_r ratios

From Fig. 5b it is observed that the reduction of NH_4^+-N increases with V_b/V_r ratios upto 0.380. This may be due to the increase in volume of biomass attached with the solid support particles. Further increase in V_b/V_r ratios beyond 0.380 might have led to the decay in biomass because of death phase that in turn gets added to the wastewater within the IFBBR thereby increasing the NH_4^+-N concentrations. This may be due to vigorous fluidization (higher U_g/U_{lmf}). Thus the result is as observed decrease in reduction of NH_4^+-N concentration. This may also be due to the

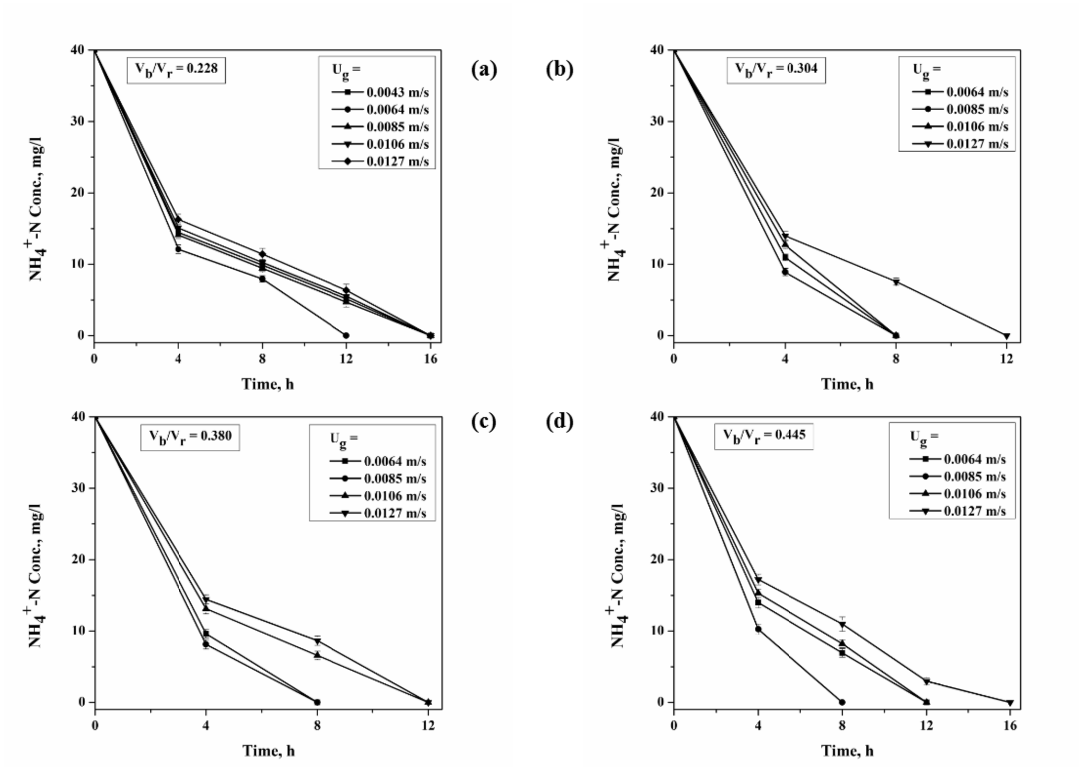

Fig. 4 (A) — Reduction of NH_4^+-N concentration with time for initial NH_4^+-N concentration of 40 mg/L for various U_g values at V_b/V_r ratios of: (a) 0.228, (b) 0.304, (c) 0.380, and (d) 0.445.

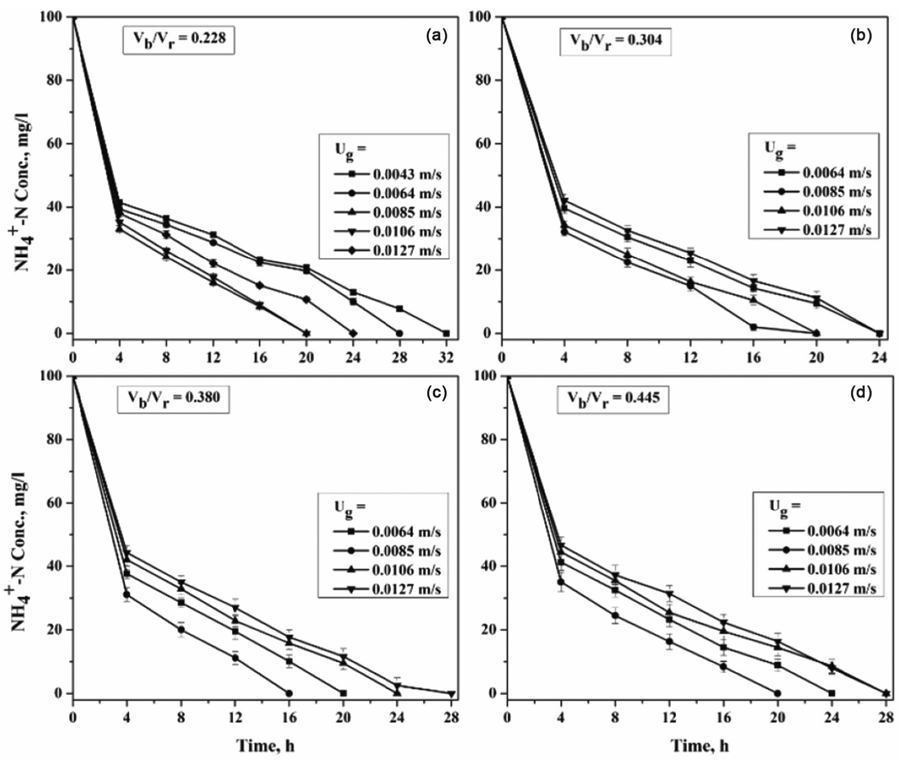

Fig. 4 (B) — Reduction of NH_4^+-N concentration with time for initial NH_4^+-N concentration of 100 mg/L for various U_g values at V_b/V_r ratios of: (a) 0.228, (b) 0.304, (c) 0.380, and (d) 0.445.

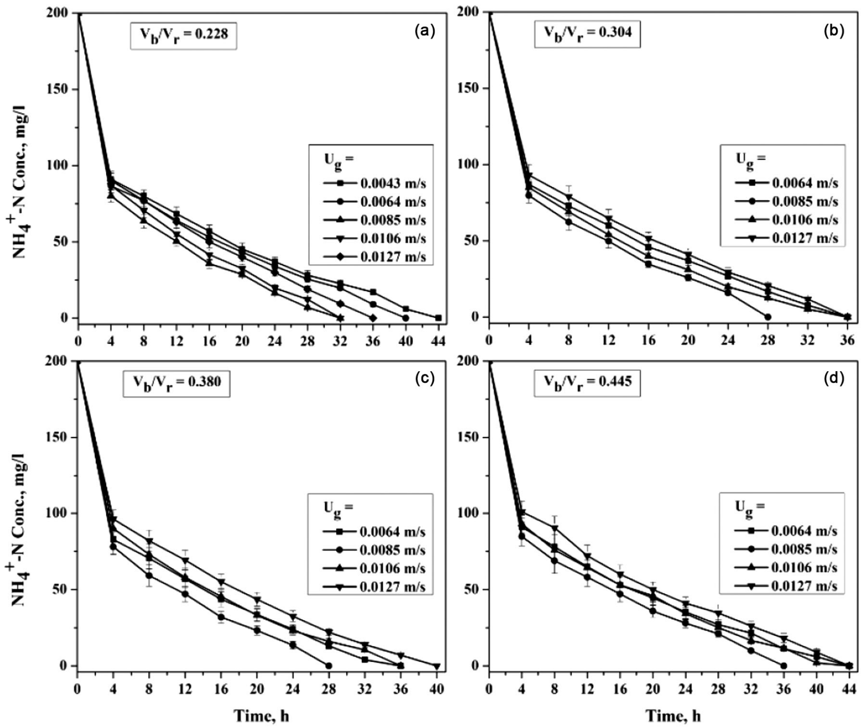

Fig. 4 (C) — Reduction of NH_4^+-N concentration with time for initial NH_4^+-N concentration of 200 mg/L for various U_g values at V_b/V_r ratios of: (a) 0.228, (b) 0.304, (c) 0.380, and (d) 0.445.

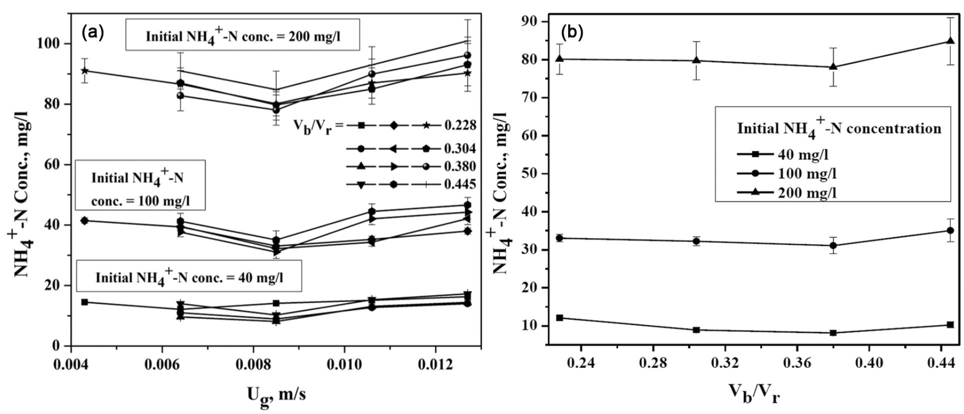

Fig. 5 — (a) Effect of U_g on the removal of NH_4^+-N for various initial NH_4^+-N concentrations and (b) Effect of V_b/V_r ratios on the removal of NH_4^+-N for various initial NH_4^+-N concentrations.

reduction of ε_g, poor phase mixing, and poor mass transfer characteristics at higher V_b/V_r ratio[33,34].

Conclusion

Study of hydrodynamic parameters in the present IFBBR indicate that for higher V_b/V_r ratios (higher volume of solid particles, constant reactor size) higher U_g is required to fluidize the bed of solid particles. The ε_g was found to increase with the increase in U_g and V_b/V_r ratios till a limiting value of V_b/V_r ratio was reached. For U_g values upto 0.0085 m/s the critical V_b/V_r ratio was observed to be 0.380. Further increase in the V_b/V_r ratios resulted in the decrease of ε_g. It is also observed that, with the further increase in U_g value to 0.0127 m/s the limiting V_b/V_r ratio decreased to 0.228. The ESEM images suggested the formation of biofilm over the solid support particles. The results obtained from the reduction of NH_4^+-N revealed that the maximum reduction occurred at 4 h of treatment. Reduction of NH_4^+-N increased with the increase in U_g and V_b/V_r ratios till the limiting values of U_g and V_b/V_r ratios. Thereafter, the reduction decreased for all initial NH_4^+-N concentrations (40, 100, and 200 mg/L). For all values of initial NH_4^+-N concentrations, the maximum reduction occurred at U_g of 0.0085 m/s and V_b/V_r ratio of 0.380. Complete removal of NH_4^+-N was observed between 8 to 44 h depending on the initial NH_4^+-N concentrations. Thus, the study of hydrodynamic parameters can be considered as the base for proper design of the IFBBR which can be used in the industrial scale for the removal of toxic NH_4^+-N from the wastewater resulting in clean environment.

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