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Influence of ferrous iron on the granularity of a UASB reactor

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ABSTRACT

The effect of ferrous ion addition on the granularity of an upflow anaerobic sludge blanket (UASB) reactor was investigated. Two UASB reactors (R1 and R2) (35 °C; pH 7) were operated for 3 months at a 20-h hydraulic retention time (HRT) at organic loads from 1.4 to 10.0 g COD L^{-1} d^{-1}. Ferrous iron was fed only to reactor R1 in a range of load from 0.014 to 0.100 g Fe^{2+} L^{-1} d^{-1}. The addition of ferrous iron induced a stable and excellent COD conversion rate. The enhancement of the performance of reactor R1 may be accredited to the mean granule diameter increase as well as to the sludge bed porosity decrease. The addition of iron to reactor R1 had a pronounced effect on the quality of the granular sludge, increasing the characteristic settling velocities. The formation of ferrous sulphide was mainly responsible for the promotion of sludge granulation.

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1. Introduction

In the past 20 years, upflow anaerobic sludge blanket (UASB) technology has developed for wastewater treatment [1–6]. More than 900 UASB units are currently operating all over the world [7,8]. The UASB reactor is considered desirable in high-strength organic wastewater treatment because of its high biomass concentration and rich microbial diversity [5,9–12]. The high biomass concentration implies that contaminant transformation is rapid, and highly concentrated or large volumes of organic waste can be treated in compact reactors. As compared to other anaerobic technologies, such as anaerobic filter, anaerobic sequencing batch reactor, anaerobic expanded bed and fluidized bed reactors, the UASB system is highly dependent on its granulation process with a particular organic wastewater. Anaerobic granular sludge is the core component of a UASB reactor [13]. Anaerobic granular sludge consists of dense microbial communities that typically include millions of organisms per gram of biomass [8].

Granulation is the process in which suspended biomass agglutinates to form discrete well-defined granules. Microbial granulation is a complex process, involving different trophic bacterial groups, and their physico-chemical and microbiological interactions [5]. Many factors contribute, in one form or another, to the granulation process [14,15]. Granulation may be initiated by bacterial adsorption and adhesion to inert matters, to inorganic precipitates [10,16], and/or to each other through physico-chemical interactions and syntrophic associations [17]. These substances serve as initial precursors (carriers or nuclei) for further bacterial growth. These initial granules will grow continuously into compact mature granules, if favourable conditions pertaining to bacteria are maintained [18].

It is believed that calcium and iron are necessary for the formation and stabilization of granules [19]. Calcium and iron salts (calcium carbonate and/or calcium phosphate and iron sulphide) were also reported to provide natural inert supports for the bacteria [20]. It was found that extracellular polymers prefer to bind to heavier metals, when they are available, due to the formation of more stable complexes [20,21]. Inorganic precipitates, such as sulphide and carbonate salts, could serve as supports for adhesion of anaerobic bacteria. Ferrous sulphide has been observed to stick firmly to the sheath of *Methanothrix* sp. [22]. By this interaction, ferrous sulphide precipitates might contribute to stabilization of bacterial aggregates within granules [22]. The inorganic precipitates contribute not only to the settling characteristics but also to granule stability [23].

The aim of this work was to investigate the influence of ferrous iron on the granular characteristics of a UASB reactor.

2. Materials and methods

2.1. Composition of feed

Synthetic milk wastewater used in this study was prepared by diluting fat-free fresh milk. This synthetic milk wastewater ensured the presence of various micronutrients in contrast to any chemostatic feed. Furthermore, this substrate could simulate dairy wastewater. The COD of this wastewater ranged from 1500 to

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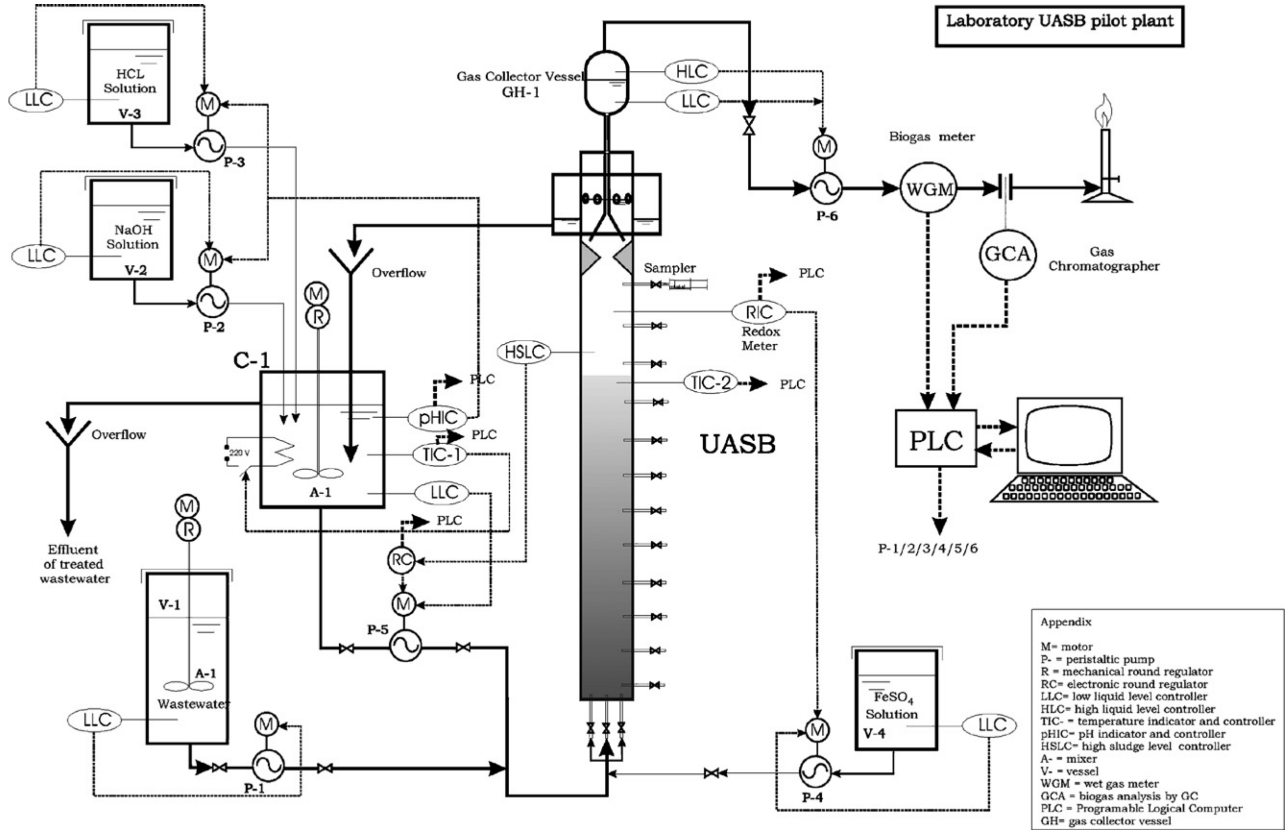

Fig. 1. Laboratory-scale UASB reactor.

11,000 mg L^{-1}. The total nitrogen and total phosphorous of the wastewater ranged from 75 to 550 mg L^{-1} and 12 to 88 mg L^{-1}, respectively. pH varied between 6.5 and 7.0.

2.2. Reactor operating conditions

Two Plexiglas laboratory-scale UASB reactors (Fig. 1) built in house with a volume of 20 L each, were operated at 35 °C. Hydraulic retention time (HRT) was 20 h. The two UASB reactors were operated under identical operational conditions, except for the influent iron concentration. One reactor (R1) was supplemented with iron solution, whereas no iron was supplied to the second reactor (R2) which served as the control reactor. Ferrous iron was supplemented to R1 in the form of a ferrous chloride solution (2%, w/v). The experimental schedule for COD load and ferrous iron load is shown in Fig. 2. The ratio of Fe^{2+}/influent COD was maintained stable and equal to 0.01 g g^{-1}, which was found to be the optimum ratio for the complete retention of iron in the sludge bed, as ferrous sulphide given the sulphur content of the wastewater [24].

During the reactor operation, COD, gas production, total soluble iron and total sulphur concentrations and pH in the influent and effluent were measured daily. For each loading rate, the performance data mentioned above were collected until steady-state conditions were obtained. The steady-state condition in the biological system implied that the daily changes in the biogas production and effluent COD were not more than ±15 and ±16%, respectively [25]. After steady-state conditions were achieved, sludge samples from all the sampling ports of the sludge bed of the UASB reactor were collected for the determination of total suspended and volatile solids content, iron content and settling characteristics. All analysis for pollution parameters were conducted in accordance with the standard methods [26]. Each analysis was repeated three times and the results presented are the mean values. All chemicals were of analytical or biological grade and purchased from E. Merck AG. The produced gas was measured with a wet gas meter (Ritter Gas meter Drum type TG01).

3. Settleability test

The settling properties of granules were evaluated by the fractions of granules exited under certain upflow velocities in a fractionating device [27]. About 8 mL of granules from the reactor were separated into 10 fractions under 10 upflow velocities and at each velocity for 5 min. Each fraction from the fractionating device

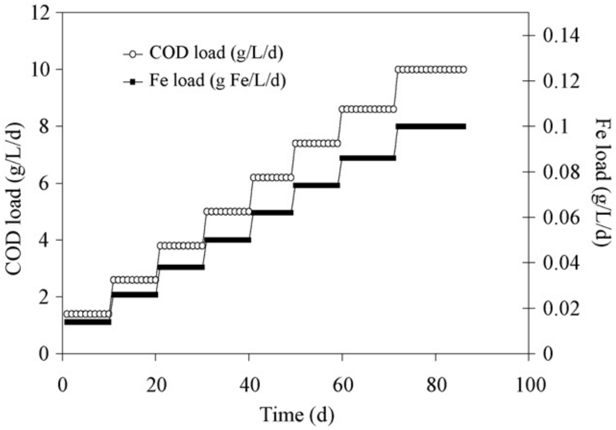

Fig. 2. COD and iron experimental schedule.

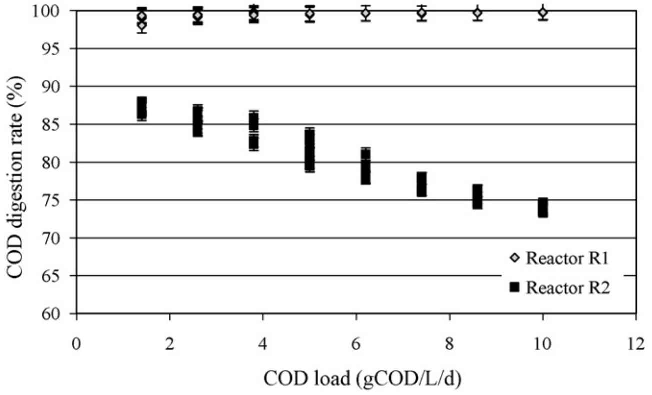

Fig. 3. Digestion rate of reactors R1 and R2.

was collected in a Whatman (1PS) filter paper and the TSS in each fraction was determined.

4. Iron content in sludge

Iron in the granules was determined by atomic absorption spectroscopy. Freeze-dried granules were ashed in a muffle furnace at 550 °C for 4 h. The ash was dissolved in 5 mL of hot concentrated HCl and filtered (Whatman No. 2) into a 50-mL volumetric flask. In all cases, the glassware was thoroughly washed with nitric and hydrochloric acid. A PerkinElmer Atomic Absorption Spectrometer (2380) was used with hollow cathode lamps and acetylene–air flame. Each analysis was repeated three times and the results presented are the mean values.

5. Results and discussion

5.1. Performance of reactors

The term “performance of reactors” is defined in this case as

$$\text{COD digestion rate } (\%) = \frac{\text{COD}_{\text{in}} - \text{COD}_{\text{out}}}{\text{COD}_{\text{in}}} \times 100 \qquad (1)$$

For low loading rates (<3 g COD L^{-1} d^{-1}), the COD digestion rate in each reactor was excellent (higher than 85%). However, when the COD loading rate was increased, the COD digestion rate in reactor R2 in which iron was not supplied decreased. The digestion rate in each reactor is shown in Fig. 3. The data of Fig. 3 indicate that COD digestion rate was significantly affected by the supplement of iron.

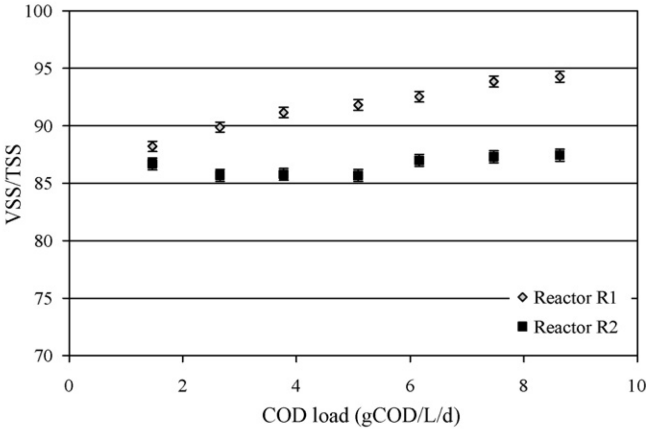

Fig. 4. Evolution of VSS/TSS ratio of reactors R1 and R2.

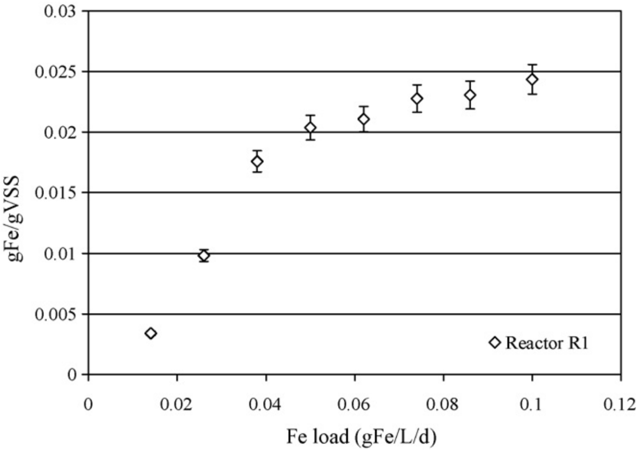

Fig. 5. Iron concentration in the sludge of reactors R1.

In spite of the high loading rates, the COD digestion rate in R1 was maintained very high, over 98%.

In the effluent of R1, the concentrations of total iron and sulphur were very low. From the mass balances, it derived that both elements were retained in the sludge in molecular proportion. Their retention was over 97%. This fact indicates that both elements are retained in the form of the inorganic precipitate of ferrous sulphide (FeS).

5.2. Composition and appearance of granules

The VSS/TSS ratio is important because it indicates the amount of biomass in total sludge measured as suspended solids. The greater VSS/TSS ratio indicates larger percentage of biomass. When the loading rates were increased, higher VSS/TSS ratios were obtained, in accordance with Ghangrekar et al. [11]. During experimental runs, greater change in TSS and VSS concentrations in the sludge bed was observed for R1 as compared to the inoculum sludge of R2 (Fig. 4). When no iron was supplied, a lower concentration of biomass (or VSS) was detected in the reactor R2. In contrast, the accumulation rate of biomass in R1 was higher.

Increasing iron load resulted in an increase of iron concentration in the sludge (Fig. 5). The concentration of iron in granules corresponded directly to their concentration in the feed. The increasing iron content in granules is not in accordance with literature [11,23,28]. According to Zandvoort et al. [28], where the anaerobic degradation of methanol was studied in a UASB and iron was added in the form of $FeCl_2$ too, the addition of higher

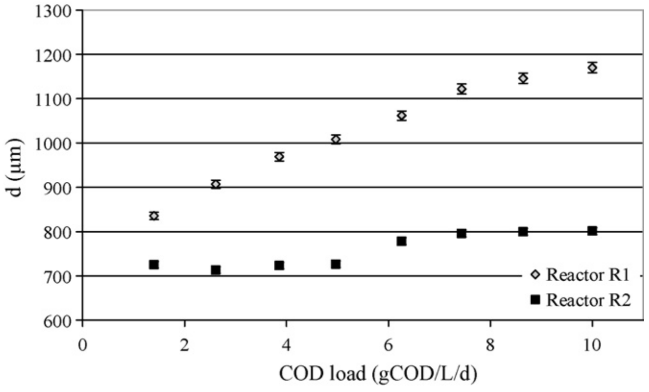

Fig. 6. Evolution of the mean granule diameter for reactors R1 and R2.

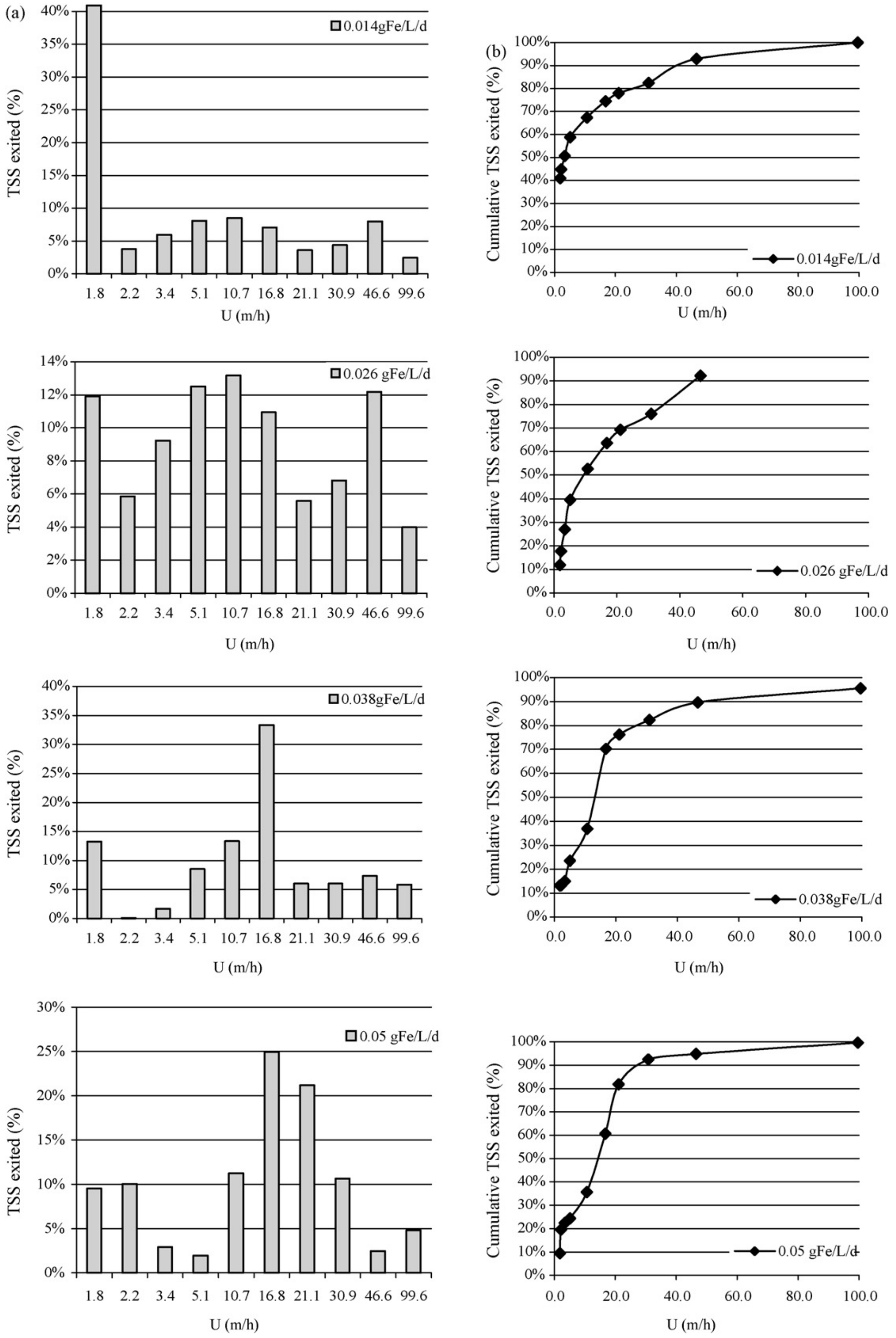

Fig. 7. Upflow velocities profiles (a) and cumulative solids loss plots (b) for 0.014, 0.026, 0.038, and 0.05 g Fe L^{-1} d^{-1} iron loads for reactor R1.

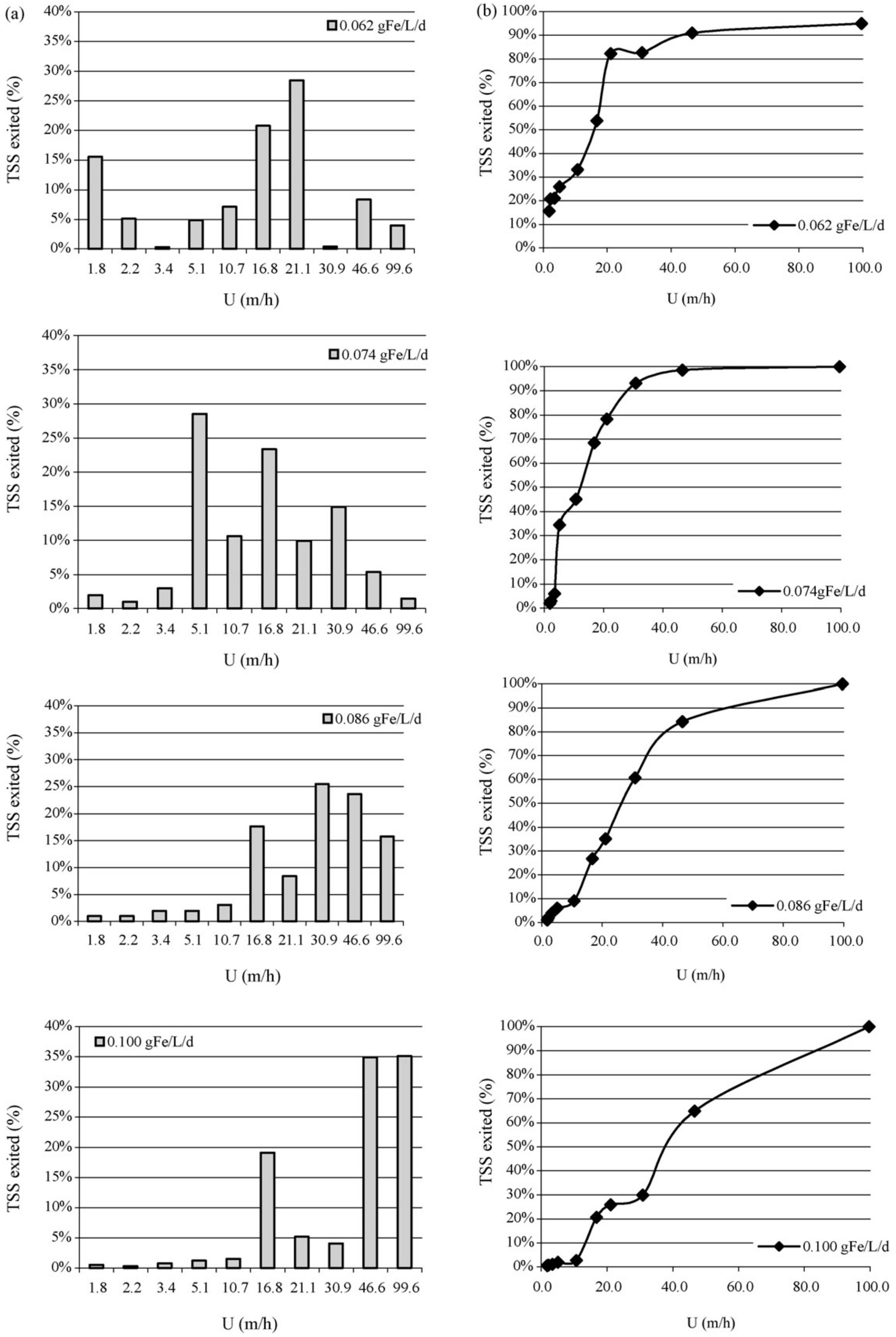

Fig. 8. Upflow velocities profiles (a) and cumulative solids loss plots (b) for 0.062, 0.074, 0.086, and 0.100 g Fe L^{-1} d^{-1} iron loads for reactor R1.

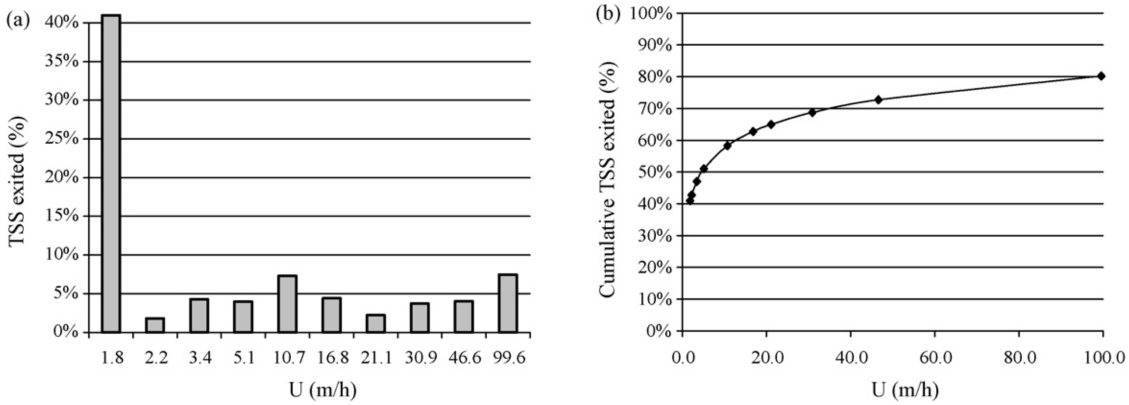

Fig. 9. Upflow velocities profile (a) and cumulative solids loss plot (b) for reactor R2 for all COD loading rates.

concentrations of iron (up to 16.846 nM) resulted in decrease of iron concentration in the sludge. According to Ghangrekar et al. [11] who studied the influence of different loading conditions on the anaerobic sludge characteristics, added iron in the form of ferrous sulphate as a trace metal. It was proved that the uptake of iron in the sludge was as high as 16.18–27.80 mg Fe g^{-1} dry sludge, which could not be linearly correlated with organic and ferrous loads. Shen et al. [23] studied the properties of anaerobic granular sludge as affected by yeast extract, cobalt and iron supplements. The concentration of supplied iron (Fe) was 1.0 ppm in chloride form. It is shown that the concentration of Fe in the granules decreased throughout the whole study. The inconsistency with the literature can be attributed to quality and quantity of iron added and to the presence of sulphur in the bioreactor. It is more likely that iron was retained in the sludge as iron sulphide (FeS) precipitates. Furthermore, in our experiments iron is not added as a micronutrient but in higher concentrations.

The colour of granules from reactor R1 was black, while some white conglomerates were present in the granules from reactor R2, whose feed was not supplemented with iron. The colour of granules and the colour of ash obtained after combustion of the granules remained the same when granules were taken from the same reactor at all operating conditions. This suggests that the presence or lack of iron determine the colour of natural and ashed granules. The black colour of granules is due to the formation of large amounts of iron sulphide precipitate [17,21,29].

5.3. *Size of granules*

A significant correlation between the mean granule diameter in the sludge bed and the ratio VSS/TSS was reported by Vlyssides et al. [30]. Eq. (2) depicts this correlation:

$$d = -8 \times 10^{-4} \log \left(1 - \frac{\mathrm{VSS/TSS}}{97.27} \right) \qquad (2)$$

where d is the granule diameter (m) and VSS/TSS is the percentage of volatile suspended solids.

Fig. 6 demonstrates the evolution of the mean granule size with COD loading rate for both reactors using Eq. (2). In reactor R1 where iron was added, a considerable increase of 40% in the mean granule diameter was observed. This was not the case for reactor R2, where the mean diameter just slightly increased.

5.4. *Settling properties of granular sludge*

Plotting upflow velocity versus percent of TSS lost from the glass test device depicts a settling velocity profile of a particular sludge (Figs. 7a, 8a and 9a). In general, this type of plot is rather descriptive, however it does give a basic understanding of the quality and nature of a particular sludge.

As one might expect, a poor settling sludge would have a large proportion of biomass exiting from the system at low upflow velocities, while sludge with good settling properties would remain within the test system at much higher velocities. From Figs. 7a and 8a, it is evident that, as ferrous ion was added, less TSS exited from the system at lower upflow velocities. If the majority of sludge exited within a small range of upflow velocities, it is characterized as a homogeneous sludge.

A plot of cumulative percentage of TSS lost from the glass test device versus upflow velocity proved to be more informative and an easier way to interpret the results. Figs. 7b and 8b are the mean cumulative profiles for the five runs for each iron load applied on reactor R1. The sludge characteristics of reactor R2 remained almost intact without being affected by the COD loading rate. The mean cumulative profile of the R1 sludge is presented in Fig. 9b.

From the cumulative plots, the upflow velocities corresponding to wash out of 10%, 30% and 60% of the sludge (U10%, U30%, and U60%) were determined. The lower the upflow velocities U10%, U30% and U60%, the worse the settling characteristics of the sludge. According to Andras et al. [27], who introduced this settleability test, a sludge sample with U60% lower than 17 m h^{-1} is considered to have poor settling properties, while the corresponding values for moderate and good settling characteristics are 30 and 40 m h^{-1}, respectively. Fig. 10 represents the variations of U10%, U30% and U60% for each iron load for reactor R1. U10% increased from 0.23 to 13.21 m h^{-1}, U30% from 0.87 to 29.11 m h^{-1} and U60% from 6.63 to 49.82 m h^{-1}. On the contrary, for reactor R2, the upflow velocities U10%, U30% and U60% were almost independent of the COD loading rate and equal to 0.05, 1.60 and 12.71 m h^{-1}, respectively. It is obvious that the addition of iron enhanced the settling properties of the sludge, since samples with poor settling properties (U60% = 12.71 < 17 m h^{-1}) after the addition of iron obtained good settleability (U60% >40 m h^{-1}).

5.5. *Sludge bed porosity*

For the estimation of sludge bed porosity, ε, the algorithm suggested by Elmaleh and Grasmick [31] for biological sludge granules with inert nuclei was used. According to them, the porosity of the sludge bed can be estimated by the following equation:

$$\varepsilon = \left[\frac{18Re + 2.7Re^{1.69}}{\mathrm{Ar}} \right]^{1/4.7} \qquad (3)$$

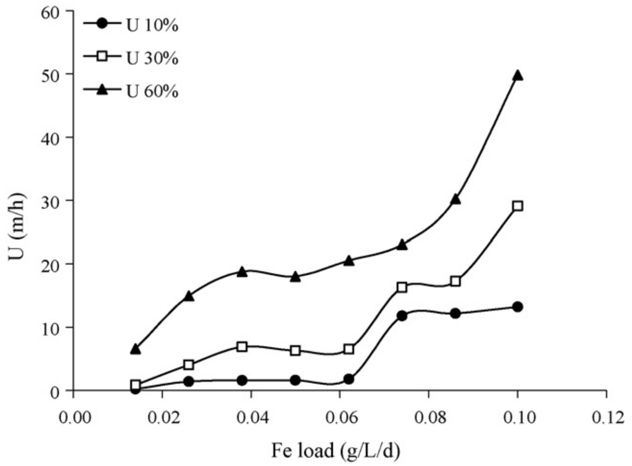

Fig. 10. Variations of U10%, U30% and U60% for each COD and iron load.

where *Re* is the Reynolds number for a particle and Ar is the Archimedes number.

$$Re = \frac{\rho u_{\mathrm{up}} d}{\mu} \qquad (4)$$

$$\mathrm{Ar} = \frac{\rho g(\rho_{\mathrm{p}} - \rho) d^3}{\mu^2} \qquad (5)$$

where ρ is the liquid's density (kg m^{-3}), ρ_{p} the density of a spherical particle (kg m^{-3}), d the diameter of a spherical particle (m), u_{up} the upflow velocity of the liquid (m s^{-1}), μ the dynamic viscosity of the flowing liquid (kg m^{-1} s^{-1}), and g is the acceleration constant (=9.81 m s^{-2}).

For the calculation of the sludge bed porosity (ε) of the UASB reactor the equations above were used where it was assumed that the liquid of the bioreactor behaved as water. Thus, it is $\rho = 995$ kg m^{-3}, $\mu = 8777 \times 10^{-4}$ kg m^{-1} s^{-1} for 35 °C. The upflow velocity of the liquid in all experiments was kept constant and equal to 1 m h^{-1}.

The sludge bed porosity was estimated taking into consideration the granule diameter and the VSS/TSS ratio. In Fig. 11, the sludge bed porosity of both reactors is presented. It is evident that the larger the granule, the less the porosity of the sludge bed. The higher COD digestion rates of reactor R1 can be attributed to the porosity of its sludge bed, since the substrate–granule contact time was increased.

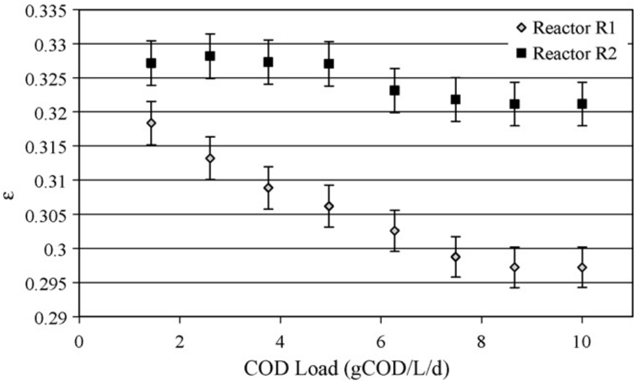

Fig. 11. Sludge bed porosity versus mean granule diameter for reactors R1 and R2.

6. Conclusions

The addition of ferrous iron induced a stable and excellent COD conversion rate. Moreover, the addition of iron to the reactor R1 influent resulted in its steady accumulation in the granules. The enhancement of the performance of reactor R1 may be accredited to the mean granule diameter increase as well as to the sludge bed porosity decrease.

According to Ghangrekar et al. [11], the settling characteristics of UASB anaerobic sludge deteriorate as the COD load increase. On the contrary, from the experiments described above, it is obvious that this is not the case. As the COD load increased from 2.0 to 10.0 g COD L^{-1} d^{-1}, the settling characteristics of sludge improved in reactor R1, while for reactor R2, they did not alter. This conclusion can be drawn from the values of the upflow velocities U10%, U30% and U60%. Specifically, U10% increased from 0.23 to 13.21 m h^{-1}, U30% from 0.87 to 29.11 m h^{-1} and U60% from 6.63 to 49.82 m h^{-1} for reactor R1 while for reactor R2 the upflow velocities U10%, U30% and U60% were independent of the COD loading rate and equal to 0.05, 1.60 and 12.71 m h^{-1}, respectively. The increase of the upflow velocities of reactor R1 corresponds to a sludge bed with well-defined, larger granules which may resist to high hydraulic pressures and proves that the addition of iron to the UASB reactor had a pronounced effect on the quality of the granular sludge. The formation of ferrous sulphide is mainly responsible for the promotion of sludge granulation.

In other words, the addition of ferrous iron significantly contributes to the formation of granular sludge which is a key function for the good operation of a UASB reactor.

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