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Moving bed bio trickling filters: an innovative solution for hydrogen sulphide removal from gas streams

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ABSTRACT

This work reports the results of the first worldwide full scale application of the concept of MBBTF (moving bed bio trickling filter) applied to biological gas treatment. A full scale prototype of an innovative MBBTF for for gaseous effluents treatment was designed and installed in a tannery wastewater treatment plant (Cuoiodepur, Pisa, Italy). The prototype was operated for 9 months with empty bed retention time of 3.5 s and treated 8000 $m^3 h^{-1}$ of a gaseous stream with a concentration of hydrogen sulphide up to 300 mg S m^{-3} (in average 170 mg S m^{-3}). The removal efficiency was on average 80% and the highest elimination capacity (EC) was 18 kg S-H_2S d^{-1} (corresponding to a specific EC of 90 g H_2S m^{-3} bed h^{-1}). The rotation of the bed was demonstrated to be a suitable solution to promote the detachment of the biomass and, thus, to control the pressure headlosses caused by the biofilm growth. The start up phase lasted 2 weeks and, the role of several process parameters (including pH, recirculated water flow rate and rotation frequency) was evaluated.

Keywords: Biological reactor; Gas treatment; Hydrogen sulphide removal; Moving bed bio trickling filter

1. Introduction

Biotrickling filters (BTFs) technology is a suitable solution for the removal of several contaminants from gaseous effluents, including: chlorinated and non-chlorinated volatile organic compounds (VOCs) [1], hydrogen sulphide (H_2S), organic reduced sulphur compounds [2,3] and ammonia [4–6]. Hydrogen sulphide can be found in wastewater (mainly as sulphide) and biogas, causing corrosion, toxicity and odours problems. BTFs are prone to clogging when the load of H_2S or other compounds is high due to biofilm growth, with the consequence of increasing energy consumption for gas pumping [7,8]. Especially in the case of biogas treatment, the clogging of the support media is challenging, mainly due to both the high concentration of hydrogen sulphide and the production of elemental sulphur in electron acceptor limiting conditions [9]. Recent tests on BTFs, applied to biological sweetening of energy gases [10] and biogas [11–13], highlighted how long-term performance struggles with reactor clogging with sulphur. The clogging is particularly problematic and occurs when the specific load is higher than 45 mg S m^{-3} [14]; moreover, above this value the proneness to irreversible clogging is accelerated. Conventional BTFs are usually designed to achieve an empty bed retention time (EBRT) of 10–40 s [15,16]; a few investigations refer to a gas contact time of less than 10 s [7,17]; however, for increased volumetric loads of H_2S, pressure head loss increases over the long term due to biomass accumulation inside the reactor [18]. This is also the case of gaseous effluents deriving from tannery wastewater treatment [7], where sulphide concentration

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is in the range of 50–300 mgS L^{-1} and sulphate concentration can reach up to 2 gS L^{-1}; given the fact that sulphate reduction easily occurs in equalisation tanks and thickeners, the potential H_2S desorption may significantly increase the cost of gaseous effluents treatment. Chemical scrubbers are the most common technology applied to H_2S removal from gaseous effluents; however, this technology presents high costs, both from environmental and economic point of views, due to the consumption of reagents and energy. Biological systems, able to reduce the operational cost and, potentially, to eliminate the need for chemicals, represent a possible alternative [19]. The possibility of removing the biofilm and, consequently, to optimize the solids retention time (SRT) and biofilm surface in the trickling filter, would allow to limit clogging and maintain a high volumetric elimination capacity (EC) in the long term. The most investigated process parameters and solutions include: optimising the packing material [15,20] and the recirculation flow rate of the liquid [21], the occasional mixing of the bed [22] and the recovery of elemental sulphur [23]; nevertheless, only a few authors suggested to apply mechanical forces to remove the excess biomass as a solution to allow the BTFs exploitation in high load context [24].

In the context of the UE LIFE+ Project BIOSUR (rotating bioreactors for sustainable hydrogen sulphide removal), a full scale prototype of a moving bed biological reactor (MBBTF-mobile bed biotrickling filter) was designed and constructed. The aim of this research was to verify the applicability of these novel biotrickling filter for odour treatment in highly loaded streams with an EBRT of less than 10 s and, at the same time, to demonstrate the efficiency of bed rotation to stably remove excess biomass. The prototype was the first full scale moving bed BTF applied to the treatment of a real effluent.

2. Materials and methods

2.1. The prototype

The prototype was a cylinder with horizontal axis, 2.5 m of diameter and 6 m of length divided in to four zones (sectors). Each sector consisted of two rotating discs (biodiscs). The gas flowed along the horizontal axis while liquid phase was recirculated from the bottom to the top and discharged independently in each zone. (Fig. 1). Before the first sector, and after the last one, two empty sectors were designed to uniform the gas distribution on the biodisc surface.

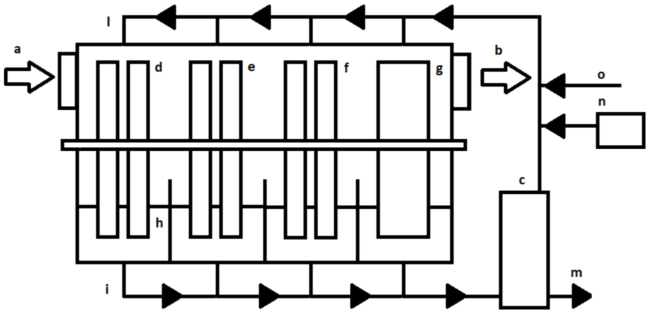

Fig. 1. Schematic of the prototype: a) inlet gas, b) outlet gas, c) recirculation water tank, d) first sector, e) second sector, f) third sector, g) fourth sector, h) water level, i) recirculation water flow from the bottom l) recirculation water flow from the top, m) discharge water, n) nutrients tank, o) make up water.

The design allowed to use specific reagents for eventual recovery cleaning operation as well as to maintain different process conditions in each sector. The biodiscs had a diameter of 2.38 m; each sector (1–3) was composed by two discs (32 cm of thickness each), while in the last sector (4) there was only a single biodisc with a thickness of 64 cm (the total length of the bed was 2.5 m). The depth of the liquid phase at the bottom was 40 cm and sectors were hydraulically separated up to a height of 50 cm by vertical barriers. The water from each sector was pumped to an external tank and recirculated at the top of the biodiscs to keep them wet and to mix the substrates in the liquid phase. A nutrient solution (120 mg L^{-1} di P-H_3PO_4 and 264 mg L^{-1} di N–NH_4Cl) was dosed in the recirculation flow; the concentration of nutrients was monitored and controlled in order to avoid limitations for biomass growth. Since biological oxidation of H_2S produces acidity, make up water was added for the maintenance of the pH set point; the water level in the MBBTF was kept constant and the excess water discharged. The biomass grew on the rotating discs filled with polyurethane foam partially submerged in water; the total volume of the bed was 8 m^3. The polyurethane foam had a specific surface area 600 m^2 m^{-3}, a density of 35 kg m^{-3} and a porosity of 390 pores m^{-1}.The last biodisc had a different composition and was filled with polypropylene pall ring with a diameter of 10 mm. The rotation of the discs, allowed to apply shear stress on the biofilm in order to remove the excess biomass. The MBBTF has been implemented in the treatment train of the consortial tannery wastewater treatment plant (WWTP) operated by Cuoiodepur (Pisa, Italy). The prototype was designed to treat up to 12000 m^3 h^{-1} of gas and it was equipped with a flow meter and an air pump. The MBBTF was connected to the piping system that collected contaminated air from the wastewater treatment covered tanks of Cuoiodepur WWTP to the actual chemical scrubbers. The actual chemical scrubbers (six vertical in parallel and two horizontal for refining) remove H_2S by washing the gas phase in counter current with a NaOH solution. The prototype intercepted the gas stream originated from the thickener and from an equalization tank of the WWTP (the most loaded gas stream) and was subsequently collected to the chemical scrubber where it was originally treated. This strategy was aimed at testing the MBBTF as pretreatment before a further refining step, with the goal of maximizing H_2S mass flow removal and, thus, of reducing NaOH consumption. The EC was considered as the key parameter, while achieving a low concentration in the effluent was not a primary objective.

2.2. Reactor start up

For the inoculum preparation, a completely mixed reactor (1 m^3) was, in turn, inoculated with primary sludge and mixed liquor collected from Cuoiodepur WWTP and fed with a solution of tap water, sodium sulphide (NaS) (1 g S L^{-1}) and nutrients [25]; the reactor was maintained in aerobic conditions through a membrane diffuser and pH was set at 3. After 60 days of selection in aerobic conditions, the inoculum was dosed inside the liquid recirculation of the prototype. The average temperature in the first week of start up was 16°C, the stream flow was 5000 $m^3 h^{-1}$ and was mixed with clean air (H_2S loads <50 g H_2S $m^{-3} h^{-1}$).

2.3. Process operation and monitoring

The test phase lasted 9 months and it is still ongoing. The aim of this preliminary test was to evaluate the suitability of biodisc rotation to control biomass removal. The goal was to obtain a better understanding of the behaviour of the system as a function of process conditions. The planned time for the start up was 1 month, for testing each operating condition 2 months were planned (pH, recirculation flow rate, rotation duration and speed). The temperature during the test was between 7 and 26°C.

2.4. Operating conditions

The experimental plan is reported in Table 1: there were two start up (the second one after August, when the reactor was stopped due to the lack of H_2S load). In February, the cover of the prototype was removed and the internal structure was inspected. In the first experimental period the pH set point was gradually varied from 3 to 6.5 and an average EBRT of 3.5 s was maintained. The second one was designed to test different rotation speeds and to study the effect of the intermittent rotation. The following intervals between two subsequent rotation phases were tested: 1, 5, 15, 60, 90, 180, 240, 720 and 1440 min. The duration of rotation tested were 1, 5 and 15 min. The velocity range tested varied in the range 0.1–3 rpm. The aim was to find an operational strategy able to optimise biomass removal through rotation as a trade-off between the need of maximizing biological activity and biofilm surface and the goal of minimizing bed clogging. During the last phase the recirculation flow rate was varied (3–10 $m^3 h^{-1}$ with a consequential hydraulic residential time in the tanks at the bottom of the reactor were of 20–60 min^{-1}) and also intermittent recirculation was tested.

2.5.Monitoring

The monitoring of the prototype included the analysis of biological, chemical and physical parameters. Recirculation liquid was sampled three times a week (composite samples) and in the collected samples the following parameters were measured: chemical oxygen demand (COD) and soluble (filtered at 0.45 µm) COD with Hach Lange kits; dissolved organic carbon (DOC, in triplicate with a Shimazdu analyser); Sulfate (in triplicate with an ion exchange chromatography Dionex); ammonium and phosphate with Hach Lange kits; total suspend solids (TSS); volatile suspended solids (VSS) (APAT CNR IRSA 2003 [26]). The removal of H_2S was evaluated through sulphur mass balance on the liquid phase and during specific periods with a gas chromatograph (Agilent 7890B) equipped with flame photometric detector (GC-FID) to measure the concentrations of H_2S in the influent and in the effluent. Probes were installed to measure pH, turbidity, suspended solids, gas flow rate and pressure head loss. The parameters measured by the probes were recorded every 5 min and stored in a memory card. The biofilm on polyurethane foam and the recirculation water were analysed (mean composite samples) for the characterization of biomass twice a week. Culture isolation attempts were performed for the samples (liquid and biofilm) of the prototype in order to obtain cultures of sulphur-oxidizing bacteria. The mineral medium, modified from Vishniac & Santer [27] was composed of solution A in 200 mL ($Na_2S_2O_35H_2O$ 10 g and NH_4Cl 1 g), solution B in 100 mL (KH_2PO_4 0.6 g), solution C in 100 mL ($CaCl_2$ $2H_2O$ 2 g), solution D in 100 mL ($FeCl_3$ 0.02 g) and solution E in 500 mL ($ZnSO_4$ 0.008 g $CaCl_2$ 0, 1 g $MnCl_2$ 0.06 g $CuSO_4$ $5H_2O$ 0.040 g $Na_2Ba_4O_7$ 0.020 g). It was rendered highly selective with the addition of an antifungal drug (Amphotericin B 3 mg L^{-1}) and it was characterized by low pH values (4–5). Medium was replaced with fresh one every 15 d, and every time a subculture on solid medium was performed. Isolated strains were characterized by mean of 16S rRNA gene sequencing. The bacterial DNA was stored as described elsewhere [25]. Total genomic DNA was extracted and the 16S rRNA gene was amplified with C1000 TouchTM Thermal Cycler (Bio-Rad) by PCR. Primers used in the PCR amplification were F7 bac_2 deg (5′-GAGTTTGAT(CT)(AC)TGGCTCAG-3′, modified from Lane [28]) and BAC R1492 (5′-GG(CGAT)(AT)ACCTTGTTACGACTT-3′, modified from Lane). Amplified and purified fragments were sequenced in both directions with proper internal primers for the bacterial 16S rRNA gene sequence: 16SF343ND 5′-TACGGGAGGCAGCAG-3′; 16SF785ND 5′-GGATTAGATACCCTGGTA-3′; 16SR515ND 5′-ACCGCGGCTGCTGGCAC-3′. During the opening phase the TSS and VSS were measured in water samples after detachment of the biomass from the open-pore polyurethane foam of the first tree discs. Metals were measured with an inductively coupled plasma spectrometer (Perkin Elmer).

Table 1
The experimentation plan during 2014 and 2015

Months	May	June/ July	August	September	October/ November	December/ January	February
Phases	Start up	pH tests	Stop	Start up	Rotation tests	Recirculation tests	Opening
pH	3	3–6.5		3	3	3	
Rotation velocity	off	0.1 rpm		off	0.1–3 rpm	0.1 rpm	
Rotation frequency	off	5 min on 720 min off		off	1–15 min on 1–1440 min off	5 min on 720 min off	
Recirculation	5 $m^3 h^{-1}$	5 $m^3 h^{-1}$		5 $m^3 h^{-1}$	5 $m^3 h^{-1}$	3–10 $m^3 h^{-1}$	

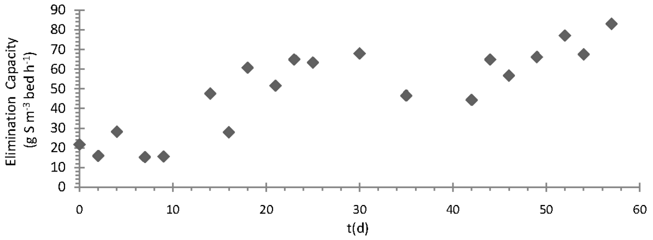

Fig. 2. The increase of the EC during the start up. Average RE 80%.

3. Results and discussion

3.1. Start up

After the inoculum, as shown in Fig. 2, the EC increased to 50 g S m^{-3} within 2 weeks. The results showed that the duration of immobilization and acclimation stages were comparable to those reported for static bed BTFs

The H_2S loads in the influent were very variables depending on the seasons and during the day (0–300 mg S m^{-1} an example is shown in Fig. 4); this is an important factor since inhibition by sulfide itself (Ki, S^{2-} = 42.4 mg S L^{-1}) [30] S^{2-} = 42.4 mg S L^{-1} may affect process performance.The MBBTF received no H_2S loads throughout August. The MBBTF was started up again in September, but as shown in Fig. 3, the EC growth quickly and there was no need for inoculating again. The recovery capacity of the biofilm after starvation was comparable as found also in the literature (36 h) [31]. In this and in the following test phase, after the start up, the average RE calculated was 80%.

3.2. Biodiscs rotation effects

The intermittent rotation was able, at tested operating conditions, of removing the excess biomass; either continuous or very frequent rotation cleaned thoroughly the biodiscs and 0.3 rpm resulted as sufficient speed to achieve this goal (as shown in Figs. 5 and 6). During the biodiscs rotation there was a sharp increase of suspended solids and a decrease of pH in the recirculation water due the removal of biomass from biodiscs. The continuous oxidation of H_2S resulted in an accumulation of hydrogen and sulphate ions in the liquid phase close or inside the biofilm; this liquid was, then, released due to polyurethane squeezing during the rotation leading to a decline in pH. It was observed that changing the timespan of the rotation phase and the time between rotations, it was possible to fine tune the removal of biomass. The biofilm was stable

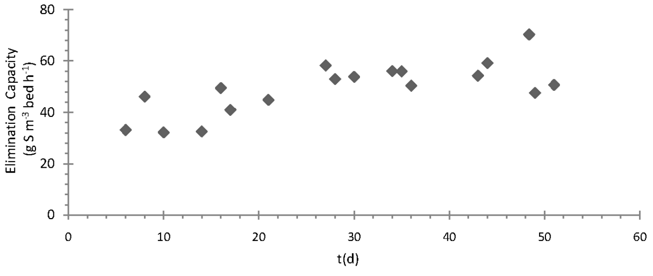

Fig. 3. The growth of the EC at September and October. Average RE 80%.

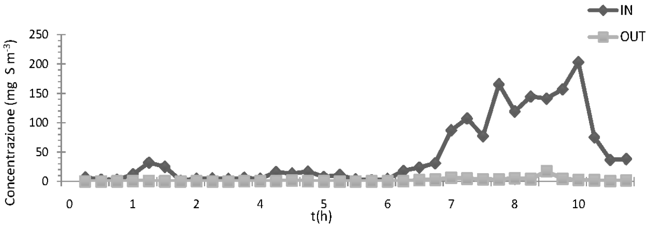

Fig. 4. Performance at the end of the start up measured with the GC-FID.

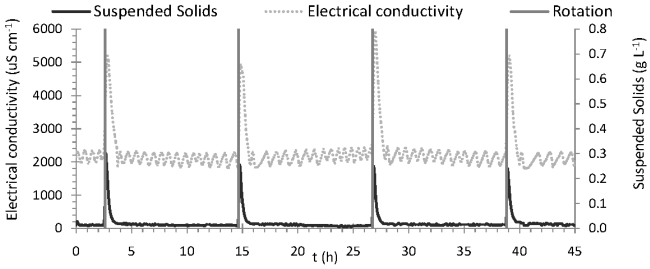

Fig .5. The rotation effect (5 min at 0.3 RPM) in recirculation water.

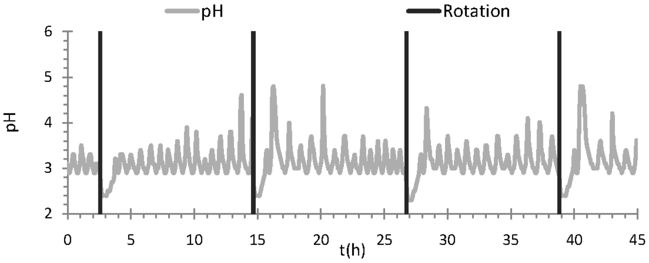

Fig. 6. The rotation effect on pH in recirculation water (in the same period of the previous Fig. 6).

Table 2
The average value of COD, sCOD and DOC in recirculation water of each sector during a period with the same process parameters

	COD mg O_2 L^{-1}	sCOD mg O_2 L^{-1}	DOC mg C L^{-1}
First sector	133.3 ± 28	63.0 ± 44	47.2 ± 33
Second sector	118.4 ± 56	58.3 ± 36	45.6 ± 30
Third sector	94.0 ± 50	58.7 ± 58	47.3 ± 35
Fourth sector	73.6 ± 33	42.3 ± 30	41.6 ± 27

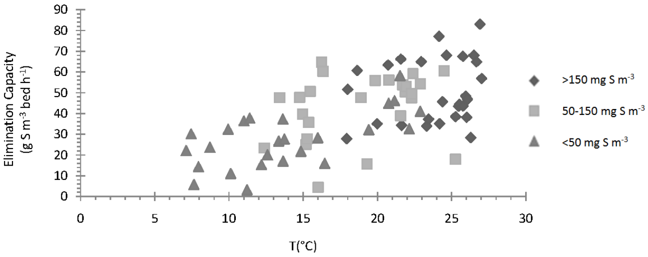

Fig. 7. The prototype temperature and the EC with different concentration of H_2S in the polluted gas stream. The volumetric mass load are reported with different shapes as described in the legend.

with 5 min rotations and 720 min of interval between two consecutive rotations. There are only a few examples in the literature of MBBTFs: Cox and Deshusses [32] tested a rotating filter at bench scale; however, they mainly focused on the removal performance; moreover, the filter was different since the entire filter was connected to an axis and rotated of 180°; Chumpling [33] tested a rotating drum biofilter at bench scale, however, in this case the aim of the study was to avoid the uneven distribution of pollutant in the filter and the axis of rotation was perpendicular to the gas flow; Vinage [24] successfully tested in lab scale, for one year, a rotating contactor to remove toluene from a synthetic gas stream; Ryu [34] realized a vertical rotating filter at pilot scale to remove ammonia and remove clogging with rotation, an intermittently automatic agitation device was used to automatically remove the excess biomass. Due to the different design of the prototype the forces applied were different.

During the whole period a decreasing concentrations of particulate COD detaching from the first to the last one biodisc were observed; however, no significant differences for sCOD and DOC were observed, as shown in Table 2 (the numbering of sector follows the direction of the gas influent). The biomass (particulate COD) seems to growth more in the first biodisc, due to the higher concentration of hydrogen sulphide in the gas stream.

3.3. Temperature

Temperature was shown to be an important parameter influencing the EC and efficiency, as shown in Fig. 4. In the prototype, there was no control system for the temperature, that was dependent on the environmental conditions. The high variability of load and EC (Fig. 7) strongly depended on the operating conditions of the WWTP. Generally, the H_2S load coming from the wastewater tank increase with the temperature in spring and summer. The best performance for the EC was at high temperature (25–27°C) in correspondence with the highest influent load. In the literature, the effect of temperature on H_2S removal efficiency was studied in the temperature range (in the inlet gas) from –1.5 to 103°C [35]; the removal efficiencies are high and stable in the range from 25 to 50°C; however, the H_2S removal efficiency, was shown to drop rapidly with decreasing temperature in the lower temperature range [35].

3.4. pH

The pH control system plays an important role since the biomass works better at constant pH. The best EC was observed at acid pH as shown in Fig. 8. During a period of 60 d the pH set point was gradually changed (0.1 each day) from 3 to 6.5 to favour H_2S transfer from gas to liquid phase. Sulphur oxidizing bacteria can grow in environments having a wide range of pH depending on the strain. Isolation attempts showed the abundance of bacteria belonging to the genus *Acidithiobacillus* (99% similarity with the sequence of *Acidithiobacillus sp.* KJ473429). It is therefore probable that this was the dominant active microbial component. Bacteria of the genus *Acidithiobacillus* are sulphur-oxidizing acidophiles whose growth is favoured by a pH value close to 3. H_2S oxidation kinetics have not been studied extensively for the SOB strain at low pH conditions [36]; however, Takano et al. [37] isolated and identified a bacterial strain from volcanic soil, which was capable of removing the sulphur compounds at a pH 1.5. In addition, an SOB strain, *Acidithiobacillus thiooxidans*, showed high activity for the H_2S oxidation at a pH of 1.5 [38]. Ramirez et al. [39] also studied H_2S removal rate at pH 2.5 using the same SOB strain, and found EC higher than 100 g S m^{-3} h^{-1}.In this study the higher value of the EC 90 g S-H_2S m^{-3} h^{-1} was measured at pH 3.5; EC is however, dependent not only on biological process, since gas liquid transfer plays also an important role.

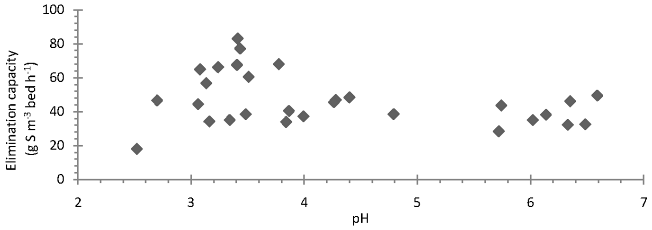

Fig. 8. EC as a function of pH (at 20–27°C).

The intermittent flow of recirculated wastewater was demonstrated to negatively affect H_2S removal due to a sharp (within minutes) reduction of bed humidification (this aspect is also dependent on the rotation frequency

Table 3
The total sulphur, total nitrogen and DOC concentrations measured in the recirculation water measured after the opening of the prototype

	Total sulphur mg S L^{-1}	Total nitrogen mg N L^{-1}	DOC mg C L^{-1}
First sector	1445	243	388
Second sector	963	96	73
Third sector	845	92	75
Foursector	661	63	60

Table 4
Main metals concentrations measured in the recirculation water measured after the opening of the prototype

	mg Cu L^{-1}	mg Cr L^{-1}	mg Ni L^{-1}	mg Pb L^{-1}	mg B L^{-1}	mg Cd L^{-1}	mg Al L^{-1}	mg Zn L^{-1}	mg Fe L^{-1}	mgP L^{-1}
First sector	–	0.16	0.05	<0.01	–	<0.01	0.25	0.61	1.53	29.44
Second sector	–	0.03	0.03	–	–	<0.01	0.13	0.24	0.28	4.49
Third sector	–	0.03	0.03	–	–	<0.01	0.11	0.45	0.25	5.74
Four sector	–	0.01	0.02	<0.01	–	<0.01	0.08	1.88	0.06	3.70

Table 5
Comparison between recent studies with biotrickling filter

Duration d	Bed length m	Specific surface m^2 m^3	Max H_2S load ppmv	Flow velocity m h^{-1}	EBRTs	Pressure drop Pa m^{-1} bed	Max EC S-H_2S m^{-3} bed h^{-1}	Reference
267	1	500	360	565	63	105	24.7	[41]
365	0.48	480	140	62	40	400	20	[8]
110	1.1	600	389	1132	3.7-6.2	1091	245	[14]
270	2.56	600	215	1799	3.5	117	90	This study

and air flow rate); moreover, the impact of recirculation on biomass removal was clearly lower than that of rotation. After 9 months the prototype was stopped for 1 month and the upper cover of the prototype was removed and a detailed evaluation of biological and chemical parameters was carried out. A decreasing trend in the biofilm thickness was observed from the first biodisc to the last one; also the DOC value was higher than in the first one than in the other biodiscs (more than five times), the majority of metals measured shown a similar trend (Table 3). The analysis of the water samples taken from the sector, after the opening of the prototype, revealed that the VSS were around 90% of TSS and the majority of sulphur in the water was sulphate. The measurement reported in Tables 3 and 4 were done in triplicate and standard deviation was less than 10%.

When the packing material specific surface is high, such as in polyurethane foam [15,16,18,40], the excess biomass is difficult to remove, and its accumulation becomes the main limitation for a high rate treatment. Pressure drop influences the operational costs due to energy consumption in bio-trickling filters where gas is forced to pass through. From this point of view, the prototype of the MBBTF showed excellent performance: at operational flow rate, the pressure drop of the clean bio filter was 1–2 mbar, when operated at high H_2S load the head loss remain less than 3 mbar for all the test period.The pressure drop in this study was significantly lower when compared with previous studies on static bed BTF, as shown in Table 5. In recent studies, with similar conditions (such as media specific surface, duration and load) other authors obtained similar EC [8] and pressure drop [41]; however, these results were obtained with higher EBRT and slower gas flow velocity; in others cases, with similar EBRT and flow velocity, higher pressure drop were observed [14]. Furthermore, in the others studies the authors were not able to remove the excess biomass in comparison with the MBBTF. This highlight the potential of the moving bed concept applied to contaminated gas treatment.

The prototype was tested in aerobic conditions; however, based on the effectiveness of the technology in controlling biomass detachment and, thus, pressure headloss, the application will be potentially suitable also for the treatment of more loaded streams: in particular, the simultaneous removal of H_2S from the biogas and nitrogen from the supernatant recirculated from the anaerobic digestion (after the oxidation of ammonia to either nitrite or nitrate) may result in an optimal solution to apply the MBBTF technology, even though this type of application requires a deeper study of the process to evaluate the applicability.

4. Conclusions

A MBBTF for the removal of H_2S from gas streams was tested for the first time at full scale. The removal efficiency was higher than 80% on average; the highest measured value of EC was 90 g S-H_2S m^{-3} h^{-1}. The accumulation of biomass was controlled through by rotation of the discs at low speed (0.1 rpm) and, consequently, the pressure drop between inlet and outlet were found to be very low (less than 3 mbar after 9 months of operation). These results suggest that the limits of the technology have not yet been achieved and so further studies need to be done with higher H_2S load.

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