

Effect of primary treatment and organic loading on methane emissions from horizontal subsurface flow constructed wetlands treating urban wastewater

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**Abstract**

Methane is emitted in horizontal subsurface flow constructed wetlands (SSF CW) during wastewater treatment. The objective of this work was to determine the influence of primary treatment and organic loading rate on methane emissions from constructed wetlands. To this aim methane emissions from a SSF CW pilot plant were measured using the closed chamber method. The effect of primary treatment was addressed by comparing emissions from wetlands operated either with an anaerobic (HUSB reactor) and with or a conventional settler as primary treatments. Alternatively, the effect of organic loading was addressed by comparing emissions from wetlands operated under high organic loading ( $52 \text{ g COD}\cdot\text{m}^{-2}\cdot\text{day}^{-1}$ ) and low organic loading ( $17 \text{ g COD}\cdot\text{m}^{-2}\cdot\text{day}^{-1}$ ). Results suggest that SSF CW redox status at the middle part of the treatment bed (15 cm depth) is of high reduced nature, regardless the type of primary treatment or organic loading applied. However, redox conditions on the upper part of the wetlands (5 cm depth) are especially affected by the type of primary treatment implemented. Accordingly, significantly lower redox conditions at 5 cm depth in wetlands receiving HUSB effluents are recorded which, in turn, resulted in significant lower organic matter removal efficiencies. Moreover, methane emission rates are affected by the type of primary treatment and, to a lesser extent, by the organic loading applied. Accordingly, a wetland fed with the effluent of a HUSB line were up to 14 times higher than those of the wetland fed with primary settled wastewater. Moreover, systems subjected to three times higher organic loading than that recommended lead to higher methane emission rates, although high data variability resulted in no statistically significant differences.

**Keywords:** Horizontal subsurface constructed wetlands, methane emission rates, HUSB reactor, settler, organic loading, redox potential

## 1. Introduction

Horizontal subsurface flow constructed wetlands (SSF CW) are natural wastewater treatment systems that represent a suitable alternative to conventional technologies. Low energy consumption and operation costs are some of the advantages of this technology that make it a viable option for the sanitation of small communities (PE<2000) (García et al. 2001, Puigagut et al., 2007). In SSF CW organic matter is removed by means of physical, chemical and biological processes that occur naturally and simultaneously within the treatment bed. Although subsurface constructed wetlands are systems subjected to great spatial redox variations (especially in depth) (García et al. 2003) they are considered to be mainly anaerobic (Baptista, 2003) and, therefore, methane emission takes place during the wastewater treatment.

Methane is among the most important gases of greenhouse effect as it has not only increased by ca. three times since pre-industrial times but also its global warming potential is about 25 times higher than CO<sub>2</sub> (IPCC, 2001). Methane in the atmosphere is mainly from biological origin (70-80%) and comes from the activity of methanogenic bacteria in environments where anaerobic pathways predominate. In wetlands methane is produced whenever redox conditions are below -200 mV and only after other electron acceptors such as nitrate or sulphate have been reduced (Mitsch and Gosselink, 2000). Furthermore, besides redox conditions, there are other environmental and operational parameters such as temperature or organic loading that has a great impact on methane emission from wetlands. (García et al., 2010; Sovik et al., 2006; Mander et al., 2014) Moreover, organic loading is of special concern in the context of Spain due to the large number of systems operated under high organic load conditions (Puigagut et al., 2007). Wetlands overloading not only contributes to increase methane emissions during wastewater treatment, but has been also directly linked to one of the main operational problems associated to constructed wetlands: the clogging (Pedescoll et al. 2011b). In order to prevent clogging in wetlands, primary treatments are applied to wastewater. Generally, physical treatments such as settlers or imhoff tanks are used. However,

recently other technologies are being considered as a suitable primary treatment for SSF CW, such as hydrolytic upflow sludge blanket (HUSB) reactors (Pedescoll et al., 2011a). Applying HUSB reactors as primary treatment for wetlands has the advantage of supplying higher biodegradable substrate to the system (Ligero et al. 2001). However, HUSB effluents are also characterized by imposing higher organic loading rates (Barros et al., 2008) and lower redox conditions (Pedescoll et al., 2011a) within the wetland that, in turn, may enhance methane emissions during water treatment.

The main objective of the present study was to determine the influence of both the organic loading conditions and the type of primary treatment (conventional settling vs anaerobic treatment) on methane emissions from horizontal subsurface flow constructed wetlands (HSSF CWs). The effect of redox conditions imposed by either the type of primary treatment or the organic loading applied on plant performance is also discussed.

## **2. Materials and methods**

### **2.1 Pilot plant**

The constructed wetlands pilot plant was set up in March 2011 and was fed with urban wastewater pumped directly from the municipal sewer. Initially, wastewater was coarsely screened and pumped to a homogenization tank having a hydraulic retention time of five hours. Within the homogenization tank wastewater was kept in constant agitation to avoid solids sedimentation. After the homogenization tank, wastewater was conveyed to the primary treatment that consisted either of one HUSB reactor of 114 L of volume operated at 4 hours of HRT and at 10 g VS.L<sup>-1</sup> or two settlers of 14 L each that were operated in parallel at two hours of sedimentation time. After the primary treatment, wastewater was pumped to the secondary treatment. Secondary treatment consisted of three wetlands of 0,4 m<sup>2</sup> of surface (70 cm length x 55 cm width x 35 cm depth) with a gravel matrix ( $D_{60}=7,3$ ;  $C_u=0,8$ ) having an initial porosity of 40%. Water level inside the wetlands was kept at 30 cm depth (5 cm below the gravel surface). All wetlands were planted from the beginning of its operation with common reed (*Phragmites australis*). For the purposes of this study three experimental lines were considered. The first two lines (named under low organic loading line – LOL and high organic loading line – HOL) consisted of two of the wetlands fed with the HUSB effluents, one at 21 L.day<sup>-1</sup> (2.6 days of hydraulic retention time) and the other at 63

L.day<sup>-1</sup> (0.85 days of hydraulic retention time). The LOL and HOL lines operated at ca. 17 and ca. 50 g COD.m<sup>-2</sup>.day<sup>-1</sup>, respectively, which it was equivalent to approximately 7 and 20 g BOD<sub>5</sub>.m<sup>-2</sup>.day<sup>-1</sup>, respectively. The third wetland (named under settler line – SetLine) was operated at a hydraulic loading rate of 21L.day<sup>-1</sup> but was fed with the conventional settler effluent. The SetLine was operated at ca. 15 g COD.m<sup>-2</sup>.day<sup>-1</sup> which was equivalent to ca. 6 g BOD<sub>5</sub>.m<sup>-2</sup>.day<sup>-1</sup>. The effect of organic loading rate on methane emissions was addressed by comparing the LOL and HOL lines between April and September 2013, whereas the effect of primary treatment on methane emissions was addressed by comparing the LOL line and the SetLine between July 2012 and July 2013. It is important to mention that the HUSB reactor was set in operation in May 2012 and the wetlands fed with HUSB effluents in the present experiment had been previously fed with settled wastewater at a hydraulic loading of 21 L.day<sup>-1</sup>.

Furthermore, each wetland had a PVC cylinder of 20 cm diameter placed at the middle of its surface that was used to implement the closed chamber for methane measurements.

## ***2.2 Methane measurements***

Methane emissions were measured following the closed chamber method (Livingston and Hutchinson, 1995). The closed chamber employed consisted of a PVC cylindrical reservoir of ca. 4 liters of effective volume, having 19 cm and 15 cm to the diameter and height, respectively. The sampling port was located at the top of the chamber and was also equipped with a thermometer (OAKTON) and a rolled vent tube (2 mm of internal diameter and 2 m long). At the end of the sampling port a two-way stopcock was disposed for sample withdrawal. The chamber was implemented with a lap-top power-adjustable 12V fan (0,011 m<sup>3</sup>.s<sup>-1</sup>) attached to the upper part of the chamber with adhering rubber. The fan had a diameter of 120 mm with blades length of 25.4 mm.

Measurements were conducted by placing the closed chamber at the middle zone of the wetlands leaving a headspace where methane accumulated. During experiment deployment the base of the chamber was kept in contact with water to avoid methane leaching. Temperature conditions within the chamber were recorded for each experiment. Once the chamber was placed in the wetland, samples were extracted after 0, 10 and 20 minutes for sampling campaigns carried out in 2012 and after 0, 10, 20, 30

and 40 minutes for sampling campaigns carried out in 2013. Sample withdrawal was conducted with 100 mL syringes and always extracting 60 mL of air from the head space. Methane was analyzed once the experiment had finished (between 2-4 hours after the last sample withdrawal had been carried out) by a gas chromatograph coupled to a FID detector (GC system – Agilent Technologies 7820A). Methane emission rates were then estimated assuming a linear emission pattern.

The effect of the type of primary treatment on methane emission was experimentally addressed by conducting 4 sampling campaigns in July, September and October 2012 and in July 2013 (n=4). The effect of organic loading on methane emissions was experimentally addressed by conducting 18 sampling campaigns from April to September 2013 (n=18).

### ***2.3 Water quality parameters and redox potential***

Water quality parameters surveyed during the experiment were COD and ammonia. Sampling was conducted at the inlet and outlet of the wetlands around the time were methane sampling campaigns were conducted. Analyses were performed according to Standard Methods (APHA-AWWA-WEF, 2005).

Redox potential was measured before, during and just after methane analysis were conducted at each sampling campaign during periods ranging between 1 and 9 days. Wetland redox status was monitored at 5 and 15 cm depth by means of (Digimed TH-404) equipped with a platinum electrode (Ag/AgCl reference system - accuracy:  $\pm 10$  mV). Sensors were connected to a datalogger (DATATAKER DT50 series 3) that recorded one redox value every 15 minutes. Data obtained was transformed to express results in terms of the standard hydrogen electrode ( $E_H$ ).

### ***2.4 Statistical analyses***

Differences among experimental conditions for any of the considered parameters (organic and ammonia loading, redox conditions and methane emissions) were determined by carrying out an ANOVA test of variance. Data normality and homogeneity of variances were determined by performing the Kolmogorov-Smirnoff and Levenne test, respectively. Differences among experimental conditions were

considered significant at p values below 0.05. All statistical analyses were performed using the software package SPSS v. 16.0.

### **3. Results and discussion**

#### ***3.1 Effect of primary treatment on redox status and plant performance***

Removal of contaminants such as organic matter and ammonia is related to the redox conditions within the wetland (Pedescoll et al (2011a), Dusek et al (2008), Faulwetter et al. (2009)) HSSF CWs have both aerobic and anaerobic zones (García et al. 2003). Plants may impose micro-aerobic zones along the depth of the wetland due to the oxygen released by roots (Stottmeister et al, 2003; García et al. 2010). However, it is generally accepted that redox potential decreases along with wetlands depth being especially high in water zones in close contact with the atmosphere (García et al. (2003), Dusek et al (2008)). Redox potentials measured in this study at 15 cm depth showed that both wetlands were under severe anaerobic conditions, regardless the type of primary treatment considered. More precisely, redox potentials recorded were, in average, that of  $-219,82 \pm 32,82$  mV and  $-208,61 \pm 66,41$  to the HUSB and the settler line, respectively, which are in the range of those previously reported in current literature (García et al., 2003, Dusek et al. 2008, Corbella et al. 2014, Pedescoll et al., 2013). Moreover, although the HUSB line tended to show slightly lower redox conditions than the settler line at 15 cm depth, no statistical significances were recorded.

Regarding redox potentials measured at 5 cm depth, a higher variability was detected in both lines when compared to redox at 15 cm. Figure 1 shows an example of the redox variation encountered at 5 and 15 cm depth. Such variability could be attributed to the fact that planted systems are subjected to high evapotranspiration rates that lead to important water level variations within the wetland and increases the redox state within the upper wetland zones (Pedescoll et al. 2013). The redox variability at 5 cm depth was especially pronounced for the HUSB line (Figure. 2) since it remained under lower redox conditions for longer time periods when compared to the settler line. More precisely, average redox values recorded were significantly lower ( $-89,73 \pm 172,51$  mV) for the HUSB line when compared to the settler line ( $136,72 \pm 124,00$  mV) (p value < 0.001), which is in accordance to that previously described by Pedescoll et al. (2011a).

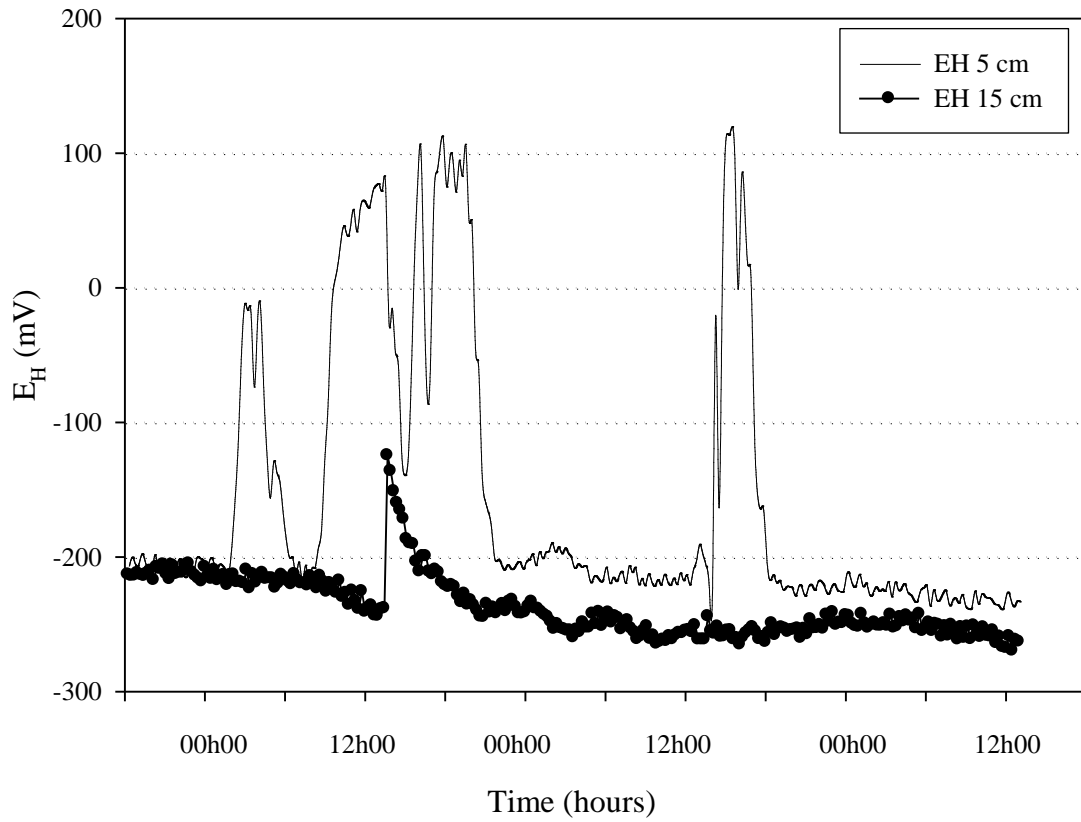


Figure. 1 Example of the redox variations recorded from the wetland of the LOL line.

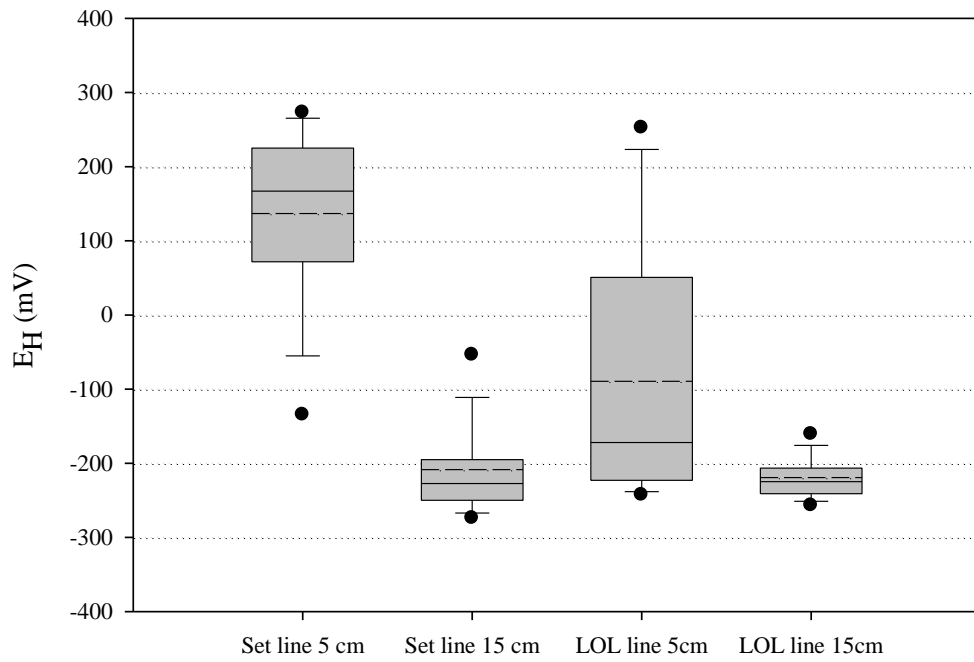


Figure 2. Redox potentials at 5 and 15 cm depth from the SetLine and the LOL Line during all the experimental period. Lower and upper bars represent the 10<sup>th</sup> and 90<sup>th</sup> percentiles, respectively. The beginning and the end of the boxes are the 25<sup>th</sup> and 75<sup>th</sup> percentiles, respectively. Upper and bottom dots represent the 95<sup>th</sup> and 5<sup>th</sup> percentile, respectively. The solid lines and dashed lines represent the median and the mean values, respectively. *Note:* SetLine boxplot is based on n=1297 and LOL Line boxplot on n=1479

Water quality parameters analyzed during the study period are summarized in Table 1. It is well documented that anaerobic reactors carry out a hydrolysis of organic matter that make the wetlands work under higher organic loading rates than those fed with the effluent of conventional settlers (Barros et al., 2008). Therefore, as expected, the wetland of the HUSB line was always subjected to a higher organic matter concentration when compared to the settler line (Table 1). Moreover, the fact that the HUSB line was operated at higher organic loading and lower redox conditions resulted in a significantly lower plant performance for the whole study period when compared to the SetLine (p value<0.01). Ammonia removal efficiency was higher for the settler line than for the HUSB line, though without significant differences. Moreover, ammonia removal efficiency was generally above 90%, regardless the experimental line considered. To this regard, wetlands here employed are shallower than those generally described in literature. Accordingly, shallow wetlands have been related to higher ammonia removal rates due to a higher oxygen transfer to the bulk liquid (Garcia et al 2005). It is worth mentioning that even though the average redox conditions recorded either at 5 or 15 cm were low enough to avoid nitrification to take place (this is especially evident for the LOL line – Figure 2), at 5 cm the redox variation caused by water level fluctuation resulted in daily periods were redox reached values above 262 mV, regardless the experimental line considered.

Overall, the wetland fed with the HUSB effluent was operated under higher organic loading and lower redox conditions than the settler line that resulted in significantly higher effluent organic loading and, not significant, but still higher ammonia effluent loadings.

Table 1. Average and standard deviation (in brackets) of both concentration and loading for the surveyed water quality parameters.



	LOL Line (n=15)			Set Line (n=15)		
	IN	OUT	%	IN	OUT	%
<i>COD</i> ( <i>mg O<sub>2</sub>.L<sup>-1</sup></i> )	327,24 (126,08)	148,48 (65,86)	55%	271,78 (105,39)	98,67 (63,59)	64%
<i>COD</i> ( <i>gO<sub>2</sub>.m<sup>2</sup>.day<sup>-1</sup></i> )	17,64 (6,80)	8,01 (3,55)		14,65 (5,68)	5,32 (3,43)	
<i>AMMONIUM</i> ( <i>mg NH<sub>4</sub>-N.L<sup>-1</sup></i> )	38,98 (19,6)	3,02 (3,36)	92%	31,80 (20,72)	1,12 (1,30)	96%
<i>AMMONIUM</i> ( <i>g NH<sub>4</sub>-N.m<sup>-2</sup>.day<sup>-1</sup></i> )	2,10 (1,06)	0,16 (0,18)		1,71 (1,12)	0,06 (0,07)	

### 3.2 Effect of organic loading on redox status and plant performance

Wetlands overloading has several consequences on both plant performance and redox status. Accordingly, Dusek et al. (2008) and Pedescoll et al (2013) reported lower redox potentials in wetlands subjected to higher flow rates and Faulwetter et al. (2009) and Headley et al. (2005) reported less reducing conditions as function of longer HRT. Moreover, higher oxidizing conditions caused by evapotranspiration are enhanced in wetlands working under lower flow rates (Pedescoll et al. (2013)).

Our results suggest that even though redox condition at the upper part of the wetlands are higher than those recorded at deeper depths, overall redox potential is significantly lower for highly loaded wetlands, regardless the depth considered. (p value<0.001) (Figure 3). More precisely, average values of redox recorded were that of -148.39±183.44 and -250.32±34.90 at 5 and 15 cm depths, respectively for the HOL line. Whereas it was that of -98.69±167.10 and -219.11±29.05 at 5 and 15 cm depths, respectively, to the LOL line.

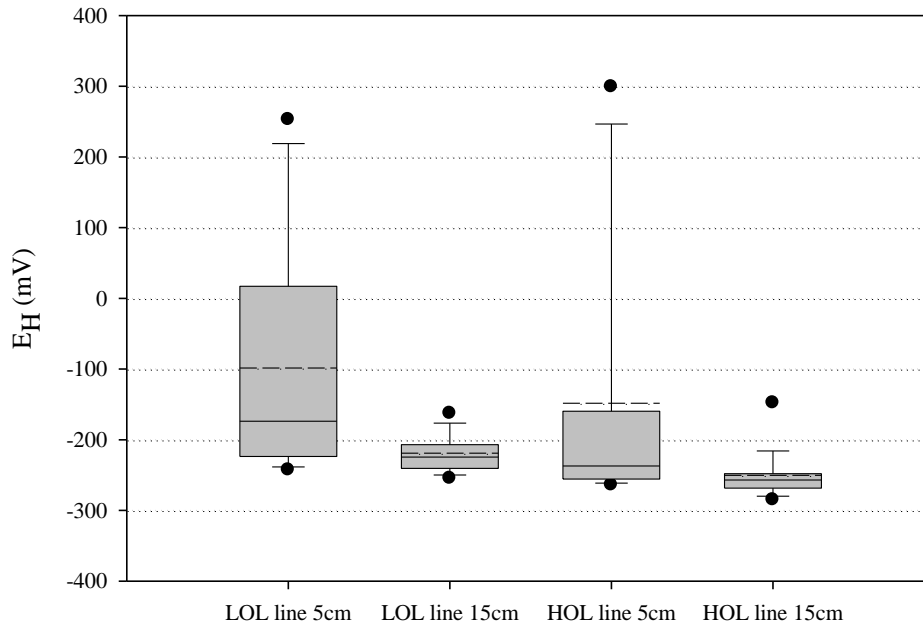


Figure 3. Average redox potentials recorded for the LOL and the HOL lines during all the experimental period. Lower and upper bars represent the 10<sup>th</sup> and 90<sup>th</sup> percentiles, respectively. The beginning and the end of the boxes are the 25<sup>th</sup> and 75<sup>th</sup> percentiles, respectively. Upper and bottom dots represent the 95<sup>th</sup> and 5<sup>th</sup> percentile, respectively. The solid lines and dashed lines represent the median and the mean values, respectively. *Note:* LOL Line boxplot is based on n=1430 and HOL Line boxplot on n=597

Ammonia loading for the HOL line was significantly higher than that of the LOL line (Table 2); this fact altogether with that of the wetland of the HOL line showing a more reduced status than the LOL line (Figure 3) lead to lower ammonia removal efficiencies (ca. four times lower) (Table 2). To this regard, Akratos et al. (2007) described an increase of ammonia effluent concentration in wetlands subjected to higher loading rates. Furthermore, Vymazal (2007) also reported a relationship between removal of total nitrogen and inflow loading. Despite the wetland of the HOL line was operated at three times higher organic loading than the LOL line, no significant differences among treatment lines were recorded for organic matter removal efficiencies (Table 2). This result is in accordance to previous findings in literature where it has been described that overloaded systems can perform similarly to those operated under lower organic loading conditions (Puigagut et al., 2007). Overall, our results suggest that organic loading has a significant effect on the status redox of the wetland which translates in significant differences in terms of ammonia removal rather than on organic matter removal.

Table 2. Average and standard deviation (in brackets) of water quality parameters for the Set Line and of the LOL line.

	HOL Line (n=6)			LOL Line (n=6)		
	IN	OUT	Removal (%)	IN	OUT	Removal (%)
<i>COD</i> ( <i>mg O<sub>2</sub>.L<sup>-1</sup></i> )	322,31 (97,63)	127,26 (30,69)	61	322,31 (97,63)	148,49 (47,24)	54
<i>COD</i> ( <i>g O<sub>2</sub>.m<sup>2</sup>.day<sup>-1</sup></i> )	52,13 (14,48)	20,58 (4,60)		17,38 (4,83)	8,01 (2,52)	
<i>AMMONIUM</i> ( <i>mg NH<sub>4</sub>-N.L<sup>-1</sup></i> )	26,11 (3,98)	21,30 (2,31)	18	26,11 (3,98)	5,35 (4,52)	80
<i>AMMONIUM</i> ( <i>g NH<sub>4</sub>-N.m<sup>-2</sup>.day<sup>-1</sup></i> )	4,22 (0,64)	3,44 (0,37)		1,41 (0,21)	0,29 (0,24)	

### 3.3 Methane emissions as function of primary treatment and organic loading

Methane is emitted in constructed wetlands as a consequence of the degradation of organic matter under anaerobic conditions. As previously discussed, anaerobic conditions in wetlands are enhanced whenever anaerobic primary treatment is applied instead of conventional settling or whenever organic loading exceeds the design recommendations. Therefore, we expected that wetlands fed with the effluent of an anaerobic digester and/or operated under higher organic loading conditions may show higher methane emission rates.

Our results confirmed this hypothesis since methane emissions from wetlands receiving the effluent of an anaerobic digester were up to 12 higher than those receiving primary settled effluents (Figure 4). Alternatively, although wetlands receiving three times the recommended organic loading showed higher maximum methane emission rates (up to two times higher) than those operated at design organic loading rates, high data variability resulted in no significant differences among experimental conditions (Figure 5).

Differences on methane emission as function of the type of primary treatment were especially evident for September and October 2012 and, to a lesser extent, for July 2013. More precisely, flux densities ranged from 235.8 to 571.6 mg CH<sub>4</sub>.m<sup>2</sup>.day<sup>-1</sup> and from 20.2 to 161.2 mg CH<sub>4</sub>.m<sup>2</sup>.day<sup>-1</sup> to the HUSB and settler line, respectively (Figure 4). It is worth mentioning that no significant differences were detected among treatment lines for the first sampling campaign (July 2012). Authors believe that this result was due to the fact that methane was measured just few weeks after the on-set of

experiments and, therefore, environmental conditions that may favor methane emission differences were of similar extent among treatment lines.

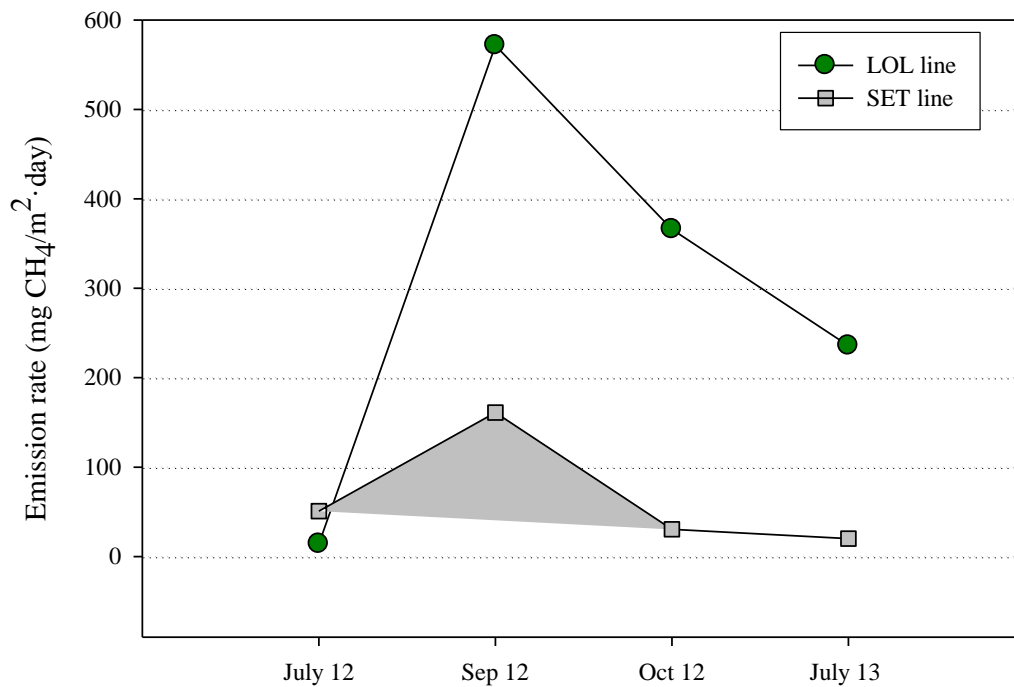


Figure 4. Emission rates estimated as function of the primary treatment and sampling campaign

In spite of the lack of significant differences for methane emission rates as function of the organic loading here considered, average methane emission for the wetlands working at higher organic loading conditions was, in average, ca. 1.5 times higher. This trend observed in our results agrees with that previously described in literature (Sovik et al., 2006; García et al., 2007; García et al., 2010; Mander et al., 2014). Furthermore, in a very extensive review paper Mander et al. (2014), describes a positive correlation between methane emissions and influent TOC.

As stated before, emission rates observed as function of the organic loading applied were very variable. Variability associated to methane emissions has been widely described in current literature (i.e. García et al. (2010) and Teiter and Mander (2005), Sovik et al. (2006)). Emissions here reported are within the range of those described in for HSSF CWs. Accordingly, Sovik et al. (2006) measured average methane emission rates ranging from 149 mg CH<sub>4</sub>·m<sup>-2</sup>·day<sup>-1</sup> to 766 mg CH<sub>4</sub>·m<sup>-2</sup>·day<sup>-1</sup> during summer time and Mander et al. (2014) reported average emission rates ranging from -1 to 480

mgCH<sub>4</sub>.m<sup>-2</sup>.day<sup>-1</sup>. Other studies have also mentioned a wide range of emission (negative values included) ranging from -0.25 to 10,199 mgCH<sub>4</sub>.m<sup>-2</sup>.day<sup>-1</sup> (Mander et al. 2008). Overall, emission rates here reported and those cited from current literature are very variable. However, it is clear that both the type of primary treatment and, to a lesser extent, the organic loading in the range here considered, has a great effect on methane emissions in wetlands.

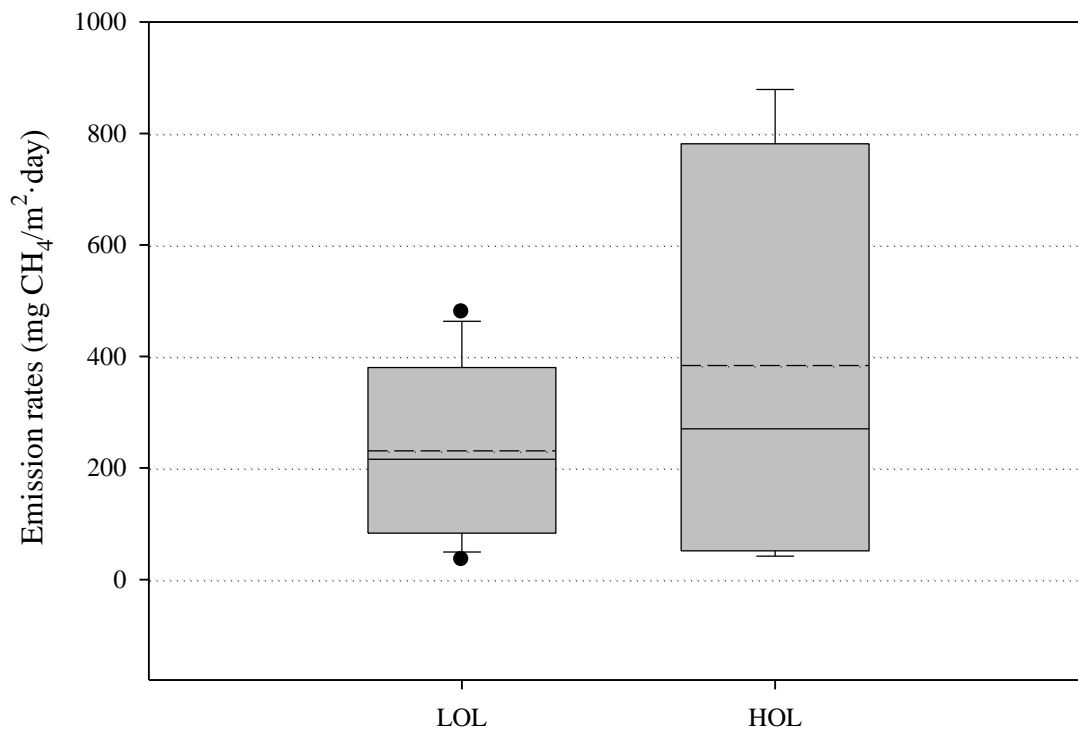


Figure 5. Emission rates recorded for the LOL and the HOL Lines. Lower and upper bars represent the 10<sup>th</sup> and 90<sup>th</sup> percentiles, respectively. The beginning and the end of the boxes are the 25<sup>th</sup> and 75<sup>th</sup> percentiles, respectively. Upper and bottom dots represent the 95<sup>th</sup> and 5<sup>th</sup> percentile, respectively. The solid lines and dashed lines represent the median and the mean values, respectively. *Note*: LOL Line boxplot is based on n=12 and HOL Line boxplot on n=6.

#### 4. Conclusions

Organic matter removal efficiencies are significantly higher for a wetland receiving primary settled wastewater than a wetland receiving the effluent of a HUSB reactor.

Organic loading has a significant impact on ammonia removal efficiencies in constructed wetlands. More precisely, a wetland operated at three times the recommended organic loading shows lower percentage of ammonia removal when compared to a wetland operated under the recommended organic loading.

The type of primary treatment and, to a lesser extent the organic loading, influences the methane emission rates during wastewater treatment. More precisely, methane emission from a wetland receiving the effluent of a HUSB reactor is up to twelve times higher than that of a wetland receiving primary settled wastewater.

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