

**CHARACTERIZATION OF CLOGGING MATERIAL FROM HORIZONTAL
SUBSURFACE FLOW CONSTRUCTED WETLAND SYSTEMS**Doi: <http://dx.doi.org/10.1590/1809-4430-Eng.Agric.v37n3p463-470/2017>**SUYMARA T. MIRANDA^{1*}, ANTONIO T. MATOS², MATEUS P. MATOS³,
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ABSTRACT: This study aimed to characterize the material composition responsible for clogging the porous medium of horizontal subsurface flow constructed wetland (HSSF–CW) systems, which is detrimental to a proper system operation. Six completely clogged HSSF–CWs were used after treatment of swine wastewater. Operating conditions of these systems were named CW–C (HSSF–CW 1 and HSSF–CW 4, non-cultivated, i.e. controls), CW–T (HSSF–CW 2 and HSSF–CW 5, cultivated with Tifton 85 (*Cynodon* spp.)), and CW–A (HSSF–CW 3 and HSSF–CW 6, cultivated with alligator weed (*Alternanthera philoxeroides*)). The results showed that most of the clogging material was composed of total fixed solids (95, 84, and 82% in CW–C, CW–T, and CW–A, respectively). However, total volatile solids (TVS) mostly affected pore clogging. The larger accumulations and productions of TVS in CWs might have originated from dead plants.

KEYWORDS: constructed wetlands, clogging, porous medium.

INTRODUCTION

Constructed wetlands (CWs) are artificial systems used for wastewater treatment and consist of ponds or porous, media in which macrophytes are cultivated (plants adapted to flooded environments). The main components of these systems are a support medium or substrate (gravel, sand, soil, among others), plant species, and microorganisms associated with these constituents (MATOS et al., 2011).

Horizontal subsurface flow (HSSF) configuration has been studied with great intensity because generates less odor and attracts fewer vectors, in addition to being operationally simple (PRATA et al., 2013; NIVALA & ROUSSEAU, 2009). However, operational problems can be found in all treatment systems, mainly obstruction of the porous medium the so-called clogging phenomenon. Clogging can provide a decreased hydraulic conductivity in a porous medium, appearance of dead zones or short-circuit and, in a more advanced degree, water runoff (PEDESCOLL et al., 2009). These effects provide a reduction in hydraulic detention time (HDT), leading to a lower treatment efficiency and impairing treatment system overall performance, as well as its operational life cycle.

Accumulation of different solids in interstitial space associated with the formation of a biofilm adhered to the support medium decreases the useful system volume, leading to a reduction in bed infiltration/percolation capacity (CASELLES-OSORIO et al., 2007; HUA et al., 2014; KNOWLES et al., 2010). According to PEDESCOLL et al. (2009), clogging occurs due to material accumulation associated with treatment and operational factors. Material amount and composition vary with the specificities of each system (configurations), management, wastewater characteristics, and environmental conditions to which CWs undergo. According to KADLEC & WALLACE (2008), COOPER (2010), KNOWLES et al. (2011), and PAOLI & VON SPERLING (2013), the main contributing mechanisms to filter medium clogging are organic and inorganic solid accumulations on CW surface, deposition of chemical precipitates, biofilm growth, besides plant root and rhizome development on the porous material bed.

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Nonetheless, many authors disagree on the main factors contributing to porous medium clogging. For WINTER & GOETZ (2003), affluent characteristics are preponderant whereas, for TANNER et al. (1998), plant solids would be more important for decreasing the useful volume. CASELLES-OSORIO et al. (2007) attributed clogging to recalcitrant organic material accumulated in porous medium whereas WANG et al. (2010) credited this phenomenon to microbial growth and biofilm-associated substances (exopolymers). PEDESCOLL et al. (2009), differently from the aforementioned authors, observed that most of clogging solids are inorganic.

Thus, this study aimed to characterize the material composition responsible for clogging the porous medium of horizontal subsurface flow constructed wetland (HSSF–CW) systems at rest (without receiving wastewater) and cultivated with two plant species, as well as to assess the importance of solids present in the porous medium under system operating conditions.

MATERIAL AND METHODS

The experiment consisted of six horizontal subsurface flow constructed wetland (HSSF–CW) systems of fiberglass boxes with dimensions of 0.6 m in height \times 0.5 m in width \times 2.0 m in length installed in a greenhouse. These boxes were filled with gneissic gravel #0 ($D_{60} = 9.1$ mm, coefficient of uniformity $C_u D_{60}/D_{10} = 3.1$, and initial void volume of $0.398 \text{ m}^3 \text{ m}^{-3}$) up to a height of 0.55 m with a free edge of 0.05 m (water level was maintained at 0.05 m below the support material surface). The units assessed have no bottom slope and were in operation from July 2011 to December 2013 for treating swine wastewater (FERRES, 2012).

These six HSSF–CWs were distributed in three different treatment groups: two controls or non-cultivated (HSSF–CW 1 and HSSF–CW 4) named CW–C; two cultivated with Tifton 85 (*Cynodon* spp.) (HSSF–CW 2 and HSSF–CW 5) named CW–T; and two cultivated with alligator weed (*Alternanthera philoxeroides*) (HSSF–CW 3 and HSSF–CW 6) named CW–A. Because all HSSF–CWs were completely clogged at the end of the experiment conducted by FERRES (2012), a characterization of clogging material was carried out at each experimental unit, proceeding to the following steps to collect the clogging material:

- Previous drainage of all wastewater present in HSSF–CW beds.
- Three samples of support material together with solids retained in their interstices at each HSSF–CW, one sample at each third of the bed. For this, a PVC tube of 100 mm in diameter and 60 cm in height was inserted into the bed, being removed manually all material contained in its interior.
- In the cultivated HSSF–CWs, the first 20 cm of material was first collected, separating them from the remaining present in the tube (20 cm posterior) for a separate analysis, totaling six different samples at each HSSF–CW. This analysis was not performed in the non-cultivated CWs because when the PVC was inserted into these systems, a lowering of support material was observed, which led to a mixture of upper and lower layers. This problem was also observed during the manual collection of support material. In addition, in the cultivated CWs, the exact compact layer was known, contrary to that observed for non-cultivated CWs, making complex the task of separating the samples by depth.
- Collected material at CWs was spread out on a bench for 48 h in order to obtain their air-drying.
- After drying, the material was sent to the Laboratory of Civil Construction Materials (LMC) at the Department of Civil Engineering of the Federal University of Viçosa (UFV), Viçosa - MG, Brazil. These materials were passed through sieves with different mesh sizes (19.1, 12.7, 9.25, 6.35, 4.76, and 2.38 mm) aiming at separating them by particle size, allowing the removal of most of the interstitial and adhered solids to the support medium (gneissic gravel). Solids adhered to support medium, being released during stirring and sieving, were collected in a bottom catch tray positioned at the base of the set of sieves.

– All material from the bottom catch tray was passed through other two smaller-opening sieves (1.0 and 0.212 mm) at the Laboratory of Soils and Solid Residues of the Department of Agricultural Engineering of UFV. All material retained and that passed through the 0.212-mm opening sieve were packed in different containers.

– The remaining support material was washed using a maximum volume of 1.5 L of distilled water in order to provide the largest possible removal of adhered organic material. For this, a bowl and a 1.3-mm mesh sieve were used in order to facilitate the support medium washing procedure.

– The suspensions generated after washing each support material sample were placed in 2-L beakers and dried on a hot plate at a temperature of 60 °C. Subsequently, a complementary drying of the beaker residue was performed in a forced air oven at a temperature of 65 °C for 24 h. After dried, all samples were declodded by using a mortar and passed through a 0.212-mm mesh sieve.

– Finally, the material that passed through the 0.212-mm mesh sieve, named as fine material, and the material that passed through the 1.0-mm mesh sieve but retained on the 0.212-mm mesh sieve, named as coarse material, were analyzed. This separation was performed since the organic material continued adhered to smaller particles of the support material after washing.

After these procedures, the following analyses were performed on the dry organic material: total solids content, obtained by the gravimetric method; total volatile and fixed solids contents, obtained after material calcination in a muffle at a temperature of 550 °C for 2 h; total and soluble phosphorus content, obtained by spectrophotometry; nitrate content, obtained by spectrophotometry as described by MATOS (2012), and Standard Methods for the Examination of Water and Wastewater (APHA et al., 2012). Total nitrogen content (TN) was quantified using the semi-micro Kjeldahl process with the addition of salicylic acid and following the method presented by MATOS (2012).

RESULTS AND DISCUSSION

The analysis of variance of data, presented in Table 1, showed no significant interaction between vegetation and position factors. This means that these factors will interfere independently in the clogging process of the CWs under study. Thus, the effects of vegetation and position factors will be discussed separately.

TABLE 1. Summary of analysis of variance of the variables total solids (TS), total fixed solids (TFS), total volatile solids (TVS), and easily oxidizable organic carbon (EOC) submitted to the following HSSF–CW operating conditions: control (CW–C), cultivated with Tifton 85 (CW–T), and cultivated with alligator weed (CW–A).

SV	DF	Fine material				Coarse material			
		TS	TFS	TVS	EOC	TS	TFS	TVS	EOC
Vegetation (V)	2	5.0**	257.8**	257.8**	27.5**	12.1**	404.2**	404.2**	47.2**
Residual (a)	3	0.2	1.1	1.1	0.9	1.0	9.5	9.5	3.0
Position (P)	2	1.1 ^{ns}	75.2*	75.2*	11.1*	2.9*	113.9*	113.9*	11.2*
V × P	4	0.1 ^{ns}	9.9 ^{ns}	9.9 ^{ns}	1.2 ^{ns}	0.6 ^{ns}	13.9 ^{ns}	13.9 ^{ns}	1.3 ^{ns}
Residual (b)	6	0.3	12.8	12.8	1.0	0.4	17.8	17.8	1.4
CV (%) plot		0.4	1.2	7.6	19.4	1.0	3.5	24.2	39.3
CV (%) subplot		0.6	4.1	26.6	20.4	0.7	4.8	33.1	26.9

ns, **, and *: non-significant, significant at 1 e 5% probability by the F-test, respectively.

SV – source of variation; DF – degrees of freedom; CV – coefficient of variation

According to the results presented in Table 2, a significant difference was observed between all analyzed variables when comparing cultivated and non-cultivated systems for both fine and coarse material.

TABLE 2. Average values, in dag kg⁻¹, of total solids (TS), total fixed solids (TFS), total volatile solids (TVS), and easily oxidizable organic carbon (EOC).

Treatment	Fine material				Coarse material			
	TS	TFS	TVS	EOC	TS	TFS	TVS	EOC
CW-C	98.76 A	93.95 A	6.04 C	2.40 B	99.32 A	96.78 A	3.28 B	1.21 B
CW-T	97.18 B	84.37 B	15.63 B	5.70 A	96.97 B	83.07 B	16.93 A	5.86 A
CW-A	97.20 B	81.42 C	18.58A	6.41 A	96.77 B	81.99 B	18.00 A	6.25 A

Means followed by the same letter on columns do not differ significantly from each other by the Tukey's test at 5% probability.

CW-C, CW-T, and CW-A – HSSF-CWs non-cultivated, cultivated with Tifton 85, and cultivated with alligator weed, respectively.

Higher TS contents are expected in cultivated HSSF-CWs due to a higher organic material production in these systems. However, this effect was not observed since this variable is related to the fresh matter, in which is included the water content effect. Thus, a greater amount of water can be found proportionally in the solid material of cultivated CWs when compared to those non-cultivated, which may have led to these results.

As in the studies conducted by TANNER & SUKIAS (1995), CASELLES-OSORIO et al. (2007), and PEDESCOLL et al. (2009), most of the solids accumulated in CW beds were of non-degradable material, characterized by the fixed solids fraction. For these authors, the fixed solids fraction was 90, 85, and 75% respectively. In this study, the fixed solids fraction (average of fine and coarse material) was higher, presenting values of 95, 84, and 82%, for CW-C, CW-T, and CW-A, respectively.

Although CW clogging was more evident in cultivated systems due to a greater surface flow, TFS content was higher in non-cultivated systems. These results indicate that TFS would be the least associated with porous medium clogging. In this case, the best association was obtained for TVS content, which presented values 2.6 and 3.1 times higher for CW-T and CW-A, respectively, when compared to CW-C regarding fine material, and 5.2 and 5.5 times higher regarding the coarse material. The contribution of organic material deposits produced by plants was more important than the external contribution of wastewater solids. Therefore, this could have been the main clogging source in the studied HSSF-CWs. Similarly, TANNER & SUKIAS (1995) found higher rates of organic matter accumulation in cultivated HSSF-CWs when compared to those non-cultivated.

KNOWLES et al. (2011) stated that the biodegradability of organic matter accumulated in CWs is responsible for predicting its impact on clogging. Initially, organic matter was thought to decompose sufficiently rapidly so that only inorganic solids would contribute to system clogging. However, according to the obtained data, clogging of CW porous medium occurred mainly by organic matter accumulation, evidenced by TVS contents.

According to the data showed in Table 3, a significant difference was observed in TS contents from the first to the second and third thirds of the HSSF-CW beds for coarse material. However, a higher concentration of these solids is expected in the first third of the beds since it is the affluent inlet zone of HSSF-CWs, acting as a filter and retaining a large part of solids present in the wastewater used.

TABLE 3. Average values, in dag kg⁻¹, of total solids (TS), total fixed solids (TFS), total volatile solids (TVS) and easily oxidizable organic carbon (EOC) in the solid material present in the interstices of porous medium considering their position in the HSSF-CW bed.

Position	Fine material			Coarse material			
	TFS	TVS	EOC	TS	TFS	TVS	EOC
1st third	82.65 B	17.35 A	6.24 A	96.88 B	82.36 B	17.63 A	5.91 A
2nd third	87.56 AB	12.44 AB	4.75 AB	98.01 A	88.74 AB	11.26 AB	4.19 AB
3rd third	89.53 A	10.47 B	3.53 B	98.16 A	90.69 A	9.31B	3.22 B

Means followed by the same letter on columns do not differ significantly from each other by the Tukey's test at 5% probability.

A decreased TVS content was observed along the HSSF–CW length, with higher values at the first and second thirds, which correspond to the thirds closer to the point where organic matter entered into the systems. In addition, the highest material levels of easier degradation were found in these positions, which can be evidenced by EOC analysis.

The presence of a compact layer was observed at the sampling phase of material from cultivated CWs. In these systems, a vertical gradient of solid distribution could be observed, with a greater accumulation in the layer close the surface, as can be observed in Figures 1 and 2 for HSSF–CWs cultivated with Tifton 85 and alligator weed, respectively. This profile is possibly due to the plant root growth and solid deposition on the support medium from the wastewater and plant residues.

Samples collected in cultivated CWs were manually extracted from inside the PVC tubes from the bed surface layer and had from 5 to 19 cm of thickness. The highest thicknesses were observed in CWs cultivated with alligator weed. Some authors have shown the presence of this layer with thicknesses ranging from 10 to 30 cm (TANNER & SUKIAS, 1995; ALCABAR, 2010; KNOWLES et al., 2011).



FIGURE 1. Samples extracted from the surface layer of HSSF–CW bed cultivated with Tifton 85.



FIGURE 2. Samples extracted from the surface layer of HSSF–CW bed cultivated with alligator weed.

Much has been studied regarding the functions and benefits of plants in wastewater treatment: removal of nutrients, oxygen transfer to the substrate, support for the growth of biofilm of bacteria (rhizomes and roots), as well as improvement in substrate permeability (KADLEC & WALLACE, 2008). In some studies, differences in growth and quantification of root biomass in CWs were focused.

CHEN et al. (2007) studied two root type growth (rhizomatic and fasciculated) of eight plant species in CWs aiming at finding out which root type would better adapt for wastewater treatment. The authors observed that fasciculate root growth was faster and provided a greater mass and surface area when compared to rhizomatic roots. In addition, they concluded that the cultivation of plant species with fasciculated root system in CWs favored wastewater decontamination. However, few studies consider root system propagation and its influence on clogging process in CWs. In this

study, a greater accumulation of organic and inorganic material in the porous medium was observed in CWs cultivated with alligator weed when compared to those cultivated with Tifton 85. This greater accumulation of dead material on the surface and surface layer of CWs cultivated with alligator weed is due to the pest attack observed in this plant species. Thus, as previously mentioned in the Material and Methods section, studying separately the clogging material collected in the first 20 cm and that collected in the deepest layer, below the first 20 cm in cultivated CWs was considered as relevant.

Table 4 shows the data related to the descriptive statistics of variables corresponding to the solid series performed at depths from 0 to 20 cm and from 20 cm to CW bottom for systems cultivated with Tifton 85 (CW-T) and alligator weed (CW-A) along the CWs (first, second, and third third).

TABLE 4. Descriptive statistics of average values, in dag kg⁻¹, of variables related to solid series (TS, TVS, and TFS) in samples collected at two depths in the studied HSSF-CWs.

Depth (cm)	Solid	Treat.	Fine material			Coarse material			
			1st third	2nd third	3rd third	1st third	2nd third	3rd third	
0 to 20	TS	CW-T	Mean	94.1	96.5	95.5	93.0	95.5	95.5
			CV	0.93	0.24	1.60	0.64	0.27	2.45
		CW-A	Mean	95.0	95.7	95.8	91.7	95.3	95.7
			CV	0.12	0.36	1.49	1.85	0.71	2.19
	TVS	CW-T	Mean	32.0	19.0	21.8	37.9	25.1	25.3
			CV	2.93	4.63	4.44	1.25	4.29	5.73
		CW-A	Mean	39.6	31.2	20.4	45.9	28.6	21.2
			CV	1.70	1.67	1.34	5.13	8.71	1.72
	TFS	CW-T	Mean	68.0	81.0	78.2	62.1	74.9	74.7
			CV	12.19	1.09	12.36	7.63	1.43	19.44
		CW-A	Mean	60.4	68.8	79.6	54.1	71.4	78.8
			CV	1.11	0.76	3.44	4.35	3.49	4.65
Below 20	TS	CW-T	Mean	98.9	99.0	99.1	99.0	99.3	99.5
			CV	0.33	0.07	0.41	0.17	0.16	0.26
		CW-A	Mean	98.6	99.0	99.1	99.2	99.3	99.5
			CV	0.03	0.31	0.06	0.39	0.15	0.08
	TVS	CW-T	Mean	7.7	7.6	5.7	5.4	4.9	3.0
			CV	0.65	0.21	0.32	0.36	0.46	0.55
		CW-A	Mean	8.8	5.9	5.7	5.7	3.9	2.6
			CV	0.69	0.24	0.45	0.47	0.41	0.34
	TFS	CW-T	Mean	92.3	92.4	94.3	94.6	95.1	97.0
			CV	0.54	1.73	1.91	0.21	2.36	1.71
		CW-A	Mean	91.2	94.1	94.3	94.3	96.1	97.4
			CV	0.66	1.53	0.27	2.85	1.69	0.09

CW-T and CW-A – HSSF-CWs cultivated with Tifton 85 and alligator weed, respectively.
 CV – coefficient of variation.

The greatest difference in solid series values between depths can be observed in TVS. This variable presented contents in systems cultivated with Tifton 85, at a depth of 0 to 20 cm, 4.2, 2.5, and 3.8 times higher when compared to those obtained at a depth below 20 cm, respectively for the first, second, and third thirds of beds for fine material, and 7.0, 7.5, and 8.4 times higher for coarse material, respectively. For systems cultivated with alligator weed (CW-A), the relation of depth from 0 to 20 cm in relation to the material collected at higher depths (below 20 cm) was also higher, i.e. 4.5, 5.3, and 3.6, respectively for the first, second, and third thirds of HSSF-CWs for fine material, and 8.7, 3.0 and 7.1, respectively, for coarse material. The greater presence of this organic material at the upper layer of HSSF-CWs is apparently due to the contribution of dead organic

matter deposits and deposition of solids on the support medium. TANNER & SUKIAS (1995) also observed a greater accumulation of organic material close to CW surface. However, according to these authors, at lower layers of cultivated systems, concentrations were, in some cases, lower compared to those obtained at the same positions in beds without vegetation, pointing out the importance of vegetal material contribution in TVS concentration of clogging material up to the depth of 20.0 cm of the porous medium. In addition, the importance of having been performed the analyses considering two particle sizes (fine and coarser) of the material needs to be emphasized since a greater presence of coarse organic material was observed at a depth from 0 to 20 cm in the profile of cultivated HSSF-CWs when compared to fine material.

CONCLUSIONS

Most of clogging material was composed of total fixed solids, with values of 95, 84, and 82% for non-cultivated (controls) HSSF-CWs (CW-C) and those cultivated with Tifton 85 (CW-T) and alligator weed (CW-A), respectively. However, total volatile solids appear to be more important for pore clogging phenomenon. In addition, the higher TVS accumulation/production in cultivated CWs is possibly due to dead plant tissue contribution.

A greater accumulation of organic material was observed in cultivated CWs when compared to those non-cultivated. This difference is due to foliar renewal, forming thin but dense layers of dead biomass on CW surface, as well as the presence of a compact layer in the first 20 cm of CWs because of the presence and accumulation of plant roots and rhizomes.

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ERRATUM

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It should read:

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