Assessment of spatial representativity of X-ray tomography to study Vertical Flow Treatment Wetlands

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Abstract

French Vertical Flow (VF) treatment wetlands receive raw wastewater and provide simultaneous solids/sludge and wastewater treatment. This feature among others explains why French VF treatment wetlands have become so popular for wastewater treatment of small communities (less 4,000 people equivalent) in France. For proper solids/sludge handling, the treatment wetland must be designed adequately and specific operational and environmental conditions must be maintained. When these conditions are not met, accumulation of biosolids may lead to clogging. Filtration and biological treatment in french VF Treatment Wetlands are governed by mechanisms at the pore-scale. Therefore, they must be better understood to predict reliably biosolid accumulation and clogging. X-ray Computed Tomography (Xray-CT) is a promising technique to characterize in detail the morphology of the filtering media in treatment wetlands. In order to set a solid basis for the use of Xray-CT, the spatial representativity of measurements must be assessed. This issue is addressed in this study by successively analyzing spatial properties at the filter scale using Frequency Domain Electromagnetic Measurements (FDEMs), and at the pore scale using Xray-CT. FDEM was used to obtain a map of the electric conductivity at the surface

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of a French VF treatment wetland. The resulting map shows signs of a high level of homogeneity in the distribution of biosolids to which it is highly correlated. These results support the choice of a random sampling procedure for core samples used for Xray-CT. The following morphological properties were computed from Xray-CT after phase segmentation: phase volume fraction profiles, Specific Surface Area (SSA) profiles and pore size distributions. Samples show several similarities of pore scale properties obtained by Xray-CT independently of the sampling region and especially the same vertical gradients. FDEM measurements and Xray-CT analysis are in agreement to indicate a good influent distribution at the surface of a full-scale mature French VF treatment wetland. A particular focus is put in the definition of the limits of the deposit layer and the dense gravel layer beneath. This division allows to compare layers independently. Finally, a 2D-REV analysis performed at several depths suggests that the selected sample diameter of 5 cm is large enough to be representative of the heterogeneous distribution of phases at the pore-scale as long as no *Phragmites* are present in the 2D-section.

Keywords: Representative Elementary Volume, Pore Size Distribution, Specific Surface Area, Constructed Wetlands, Clogging

1. Introduction

Treatment wetlands are a nature-based technology that present an alternative to conventional wastewater treatment technologies (Sundaravadivel and Vigneswaran, 2001). Among the different possible configurations, French Vertical Flow (VF) treatment wetlands have largely been adopted for treatment of domestic wastewater for communities under 5,000 people equivalent in France (Morvannou et al., 2015). They are often composed of three parallel first-stage filters followed by two parallel second-stage filters filled with granular materials and planted with *Phragmites Australis*.

The first stage typically contains a 30 cm deep layer of coarse gravel (26 mm). The three first-stage cells are fed raw wastewater in an alternating

manner; 3.5 days of feeding followed by 7 days of rest. The rules of design and implementation of French VF treatment wetlands can be found in Molle et al. (2005). As the first-stage filters are fed with raw wastewater, solid particles
15 are retained at the surface by cake filtration and in the filter media by deep bed filtration. In first-stage French VF wetlands, deep bed filtration is the predominant solids retention mechanism in newly commissioned filters, whereas cake filtration is the predominant mechanism once a deposit layer is formed. The deposit layer is the result of the accumulation of solids at the filter surface.

20 The entrapped solids are herein referred to as the biosolids, which are present in the deposit layer as well as the gravel filtration layer. Biosolids are characterized by their high organic matter content which ranges from 50% to nearly 90% (Kania et al., 2018) and form a porous medium whose structure and consistency evolve with time due to water content changes, organic matter mineralization
25 and internal erosion. Kania et al. (2018) characterized the surface deposit of 14 French VF treatment wetlands and conclude that the deposit layer on mature wetlands (> 3 years of operation) has lower OM content, lower biodegradability, and higher humification than newly commissioned wetlands (≤ 3 years of operation). Structure of biosolids impacts water and oxygen transfer as well as
30 biological activity (Molle, 2014). The biosolids in the deposit and gravel layers are home to the microorganisms and micro-fauna responsible for degradation of pollutants. They retain part of the percolating water, and when the wetland is functioning properly, the structure of biosolids maintains a good re-oxygenation capability during rest periods, which is essential to promote mineralization of
35 organic matter (Molle, 2014).

Understanding the structural evolution of biosolids in the deposit and gravel layers is crucial since it constrains maintenance frequency and the lifespan of the treatment wetland. The deposit layer has to be removed every 10-20 years to maintain infiltration capacity, and the fouling material accumulation within
40 the granular material inevitably leads to its clogging and therefore defines the lifespan of the system (Nivala et al., 2012).

Investigations on solid retention in treatment wetlands generally focus on

Horizontal Flow (HF) treatment wetlands, which are more prone to clogging than VF wetlands due to the fact that HF wetlands generally have a much smaller cross-sectional surface area accepting wastewater, thus they experience
 45 a higher areal organic loading rate (Knowles et al., 2011; de Matos et al., 2018). They aim at evaluating the degree of clogging and proper functioning of a treatment wetland, and provide little information regarding the underpinning mechanisms because those depend on pore scale properties. In order to gain insight
 50 into the pore scale structure, a recent study assessed the application of X-ray Computed Tomography (Xray-CT) to obtain high resolution 3D images of the porous media structure (Martinez-Carvajal et al., 2019). Promising results were achieved but the study raised the question of the spatial representativity of the samples used for Xray-CT.

55 Assessing the spatial representativity of Xray-CT images from French VF treatment wetlands is critical to ensure the reliability of pore scale properties that may be derived from these images. French VF treatment wetlands present heterogeneities at both the filter and the pore scales.

At the filter scale, two main types of macro-scale heterogeneities (Costanza-
 60 Robinson et al., 2011)) could be considered: uneven distribution of biosolids and uneven reed density. These heterogeneities are most likely controlled by the general design of the filter including its shape and the location of feeding points. Forquet et al. (2011) computed the relative water content changes in the deposit layer over a vertical cross sectional area of a mature French VF treatment
 65 wetland (20 cm thick deposit layer) during a feeding period using time-lapse Electrical Resistivity Tomography (ERT). Water content in the deposit layer increased by 14% in a region of 50 cm around feeding points and by less than 4% for the rest of the analyzed vertical cross-section. Heterogeneities in the deposit layer are likely to evolve in French VF treatment wetlands due to the reed growth
 70 and colonization, the progressive accumulation of biosolids in the pore space of the gravel layer, and the mineralization of biosolids in both the deposit layer and gravel layer. The older a French VF treatment wetland is, the smoother these heterogeneities become. This is especially the case for plants because

it may take several years before the plant cover is fully established. Factors
75 affecting the speed of plant establishment include plant species, age, availability
of adequate micronutrients, and adaptation to the environmental conditions
found in in treatment wetlands (Lombard-Latune et al., 2018; Chazarenc et al.,
2009).

At the pore scale, the French VF treatment wetlands are comprised of gravel,
80 biosolid, water and void space. The representativity of samples at this scale de-
pends on the distribution of these phases in terms of volumetric contents and
characteristic sizes. Many studies rely on the computation of the Representa-
tive Elementary Volume (REV) to assess the spatial representativity of samples
(Costanza-Robinson et al., 2011; Al-Raoush and Papadopoulos, 2010; Li et al.,
85 2009). The 'p'-REV is the minimal volume of a sample from which the volume-
weighted average value of the property 'p' is no longer dependent on the volume
of the sample and then becomes representative. Several methods have been pro-
posed to assess the REV size. They are all based on iterative processes where
the volume integral of the property 'p' is computed over volume increasing in
90 size but they differ on the origin of this sampling volume: a single point taken
in the middle of the sample (Costanza-Robinson et al., 2011; Al-Raoush and
Papadopoulos, 2010) or multiple points randomly distributed over the sample
(Zhang et al., 2000; Ramírez et al., 2019). In porous media, the most common
property analyzed is the void volume fraction (e.g., porosity), but REV of other
95 properties, such as specific surface area (SSA), moisture saturation, coordina-
tion number, or local void ratios are also studied (Al-Raoush and Papadopou-
los, 2010). A subject of great interest is to assess whether or not achieving
the porosity-REV implies that REV for other properties is achieved too. Some
authors have investigated this question and their conclusions vary. Al-Raoush
100 and Papadopoulos (2010) found that the porosity-REV is smaller than the REV
for particle size distribution, local void ratio and coordination number for sand
samples with different uniformity/gradation degrees. Costanza-Robinson et al.
(2011) found larger porosity-REVs than REVs for interfacial area (A_i) and mois-
ture saturation (S_w) in wet samples of glass beads and silica sands. However,

105 these authors stated that REV deterministic computation is difficult because
of the lack of a robust criteria to determine whether the averaged value of the
property 'p' is constant or not.

This work demonstrates the spatial representativity of samples extracted
from a first-stage French VF treatment wetland by examining the heterogeneities
110 present at both the pore scale and the filter scale. This is achieved by (i)
determining whether the size of the samples is large enough to ensure spatial
representativity at the pore scale, and (ii) quantifying the similarity of pore
scale structure of samples from the same filter extracted at different locations.

2. Materials and Methods

115 2.1. Study site

The study was carried out on a full scale French VF treatment wetland in
Montromant (Rhône, France). It was designed according to French standards
defined in Molle et al. (2005) for 200 people equivalent. It has two treatment
stages: a first stage consisting of four parallel filters, and a second stage con-
120 taining two parallel filters. Each filter in the first stage is approximately 64
 m^2 . Since its commission in 1994, three of the four filters in the first stage
were in operation. In 2009, the deposit layer of these filters was removed and
the fourth filter in the first stage was put into operation. The duration of the
feeding period remained 3.5 days and the rest period was increased from 7 days
125 to 10.5 days. The filtration layer is 30 cm thick and is made of 1 to 6 mm gravels
($d_{10} = 2$ mm and uniformity coefficient = 1.5). Based on regulatory monitoring
measurements performed in 2013, the suspended solids (SS) and the Chemical
Oxygen Demand (COD) loads applied to the first stage filter in operation were
 $158 g \cdot m^{-2}d^{-1}$ and $262 g \cdot m^{-2}d^{-1}$, respectively. More details on the site de-
130 scription can be found in Martinez-Carvajal et al. (2019). All measurements
were conducted on a single first stage wetland cell.

2.2. Electrical conductivity mapping

Gravel is a geomaterial having low Electrical Conductivity (EC). On the other hand, the water and organic matter constituting the biosolids present a high EC. Geophysical methods, such as Frequency Domain Electromagnetic Method (FDEM) and Electrical Resistivity Tomography (ERT), thus present interesting capabilities to estimate the spatial distribution of biosolids in the top layer of a French VF treatment wetland. In particular, FDEM presents a suitable sensitivity for highly conductive media (McNeill, 1980). Its application on French VF treatment wetlands is promising considering that EC (the inverse of electrical resistivity) measurements using ERT have shown values between 2.5 and 50.0 $mS \cdot m^{-1}$ in French VF treatment wetlands (Morvannou, 2011). FDEM has been widely used by soil scientists to study the spatial variability of soil properties in the field (Doolittle and Brevik, 2014; Lardo et al., 2016). It provides a 2D map of the EC. Soil EC measured by FDEM does not allow a direct characterization of soil characteristics like water content or hydraulic conductivity, but their correlation is used to compute estimates of these properties using petro-geophysical models (e.g. Archie's Law).

The FDEM sensors generate a primary oscillating electromagnetic (EM) field, which propagates through the soil. It induces alternating currents that in turn produce a secondary EM field measured by the sensor. Under conditions known as 'low induction number', a linear relationship exists between the quadrature component of the secondary EM field and the electrical conductivity of the soil penetrated by the EM field (Calamita et al., 2015; Callegary et al., 2007). This relationship allows the measurement of EC.

In this study, a CMD-TINY electromagnetic induction tool (GF Instruments) was used. It simultaneously collects both in-phase and quadrature component data. The device is a frequency electromagnetic induction sensor with a frequency of 9 KHz composed of two coils, an emitter and a receiver, with an inter-coil spacing of 0.45 m. The depth of the magnetic field penetration was estimated to about 35 cm for the vertical dipole mode. To obtain measurements, the device was swept over the investigated area of the filter. Therefore

measurements were carried out at the end of winter 2018, when *Phragmites* were cut off. The pipes that dispose wastewater at the treatment wetland surface are made of plastic and thus do not interfere with the measurements. Data were collected on a regular grid ($\Delta x = 0.5$ m, $\Delta y = 0.5$ m). The map of the EC was computed using a linear interpolation between the points of the grid.

EC data were filtered to eliminate defective points by applying two thresholds:

- Electrical conductivity values below $0.1 \text{ mS} \cdot \text{m}^{-1}$, the limit of detection of CMD-Tiny, were not taken into account
- Phase changes exceeding 2.5 ppm were eliminated. Measurement of the in-phase component of the signal indicates magnetic disturbance in the ground. If the phase change is above this threshold, the linear relationship between the electrical conductivity and the quadrature component is not verified and the McNeil relationship is thus not applicable (McNeill, 1980).

2.3. Sampling

FDEM measurements were also used to drive the sampling procedure for Xray-CT. Based on the map of electrical conductivity, three regions, approximately 1 m in diameter, were selected. At the beginning of a rest period, two samples per region, named A and B, were extracted. The samples consisted of cylinders of 5 cm in diameter and 16 cm in height. The extraction method is described in detail in Martinez-Carvajal et al. (2019). Samples were identified with a number corresponding to the sampling region and a letter (A or B). Cores A and B were sampled as close as possible to each other. The height of the samples was large enough to contain all the deposit layer and at least 5 cm of the gravel layer. The cross-sectional area of samples was approximately 19 cm^2 . After the extraction, all the samples were sealed with a plastic film and stored at 5 °C to avoid water evaporation. They were scanned by X-ray tomography within the following 72 hours.

2.4. X-ray tomography and segmentation

Tomographies were performed with a tomograph EasyTom XL (RX Solutions) at University Grenoble Alpes. The scanner was equipped with a 150 kV tungsten X-ray tube. It was operated with an accelerating voltage of 140 kV and a current of 200 μA . A 0.5 mm copper foil filter was put in front of the X-ray source to reduce beam-hardening artifacts. 4480 projections distributed among 360 °were recorded. The sensor area was 250×200 mm with 2084×2084 pixels. Scans were obtained using a helicoid acquisition. The data were reconstructed using a cone-beam algorithm (X-Act 2.0, RX Solutions). 3D images consist of approximately $1500 \times 1500 \times 4600$ voxels ($l \times w \times h$) with a voxel size of 35 μm . No contrast enhancing was performed during the reconstruction. The reconstructed data were then processed and visualized with the public domain ImageJ/Fiji (Schindelin et al., 2012) and Python (Van Rossum and Drake Jr, 1995) sharewares. A three-phase segmentation of the scans was performed based on a region growing algorithm developed by Hashemi et al. (2014) and adapted in a previous study (Martinez-Carvajal et al., 2019). Segmentation consists in the attribution of the voxels of a digital image to a single phase based on their gray values and those of their neighbors. This algorithm allows to successfully segment three phases: voids, biosolids, and gravels.

In images containing multiple phases, the ranges of gray values corresponding to each phase overlap due to small differences in absorption coefficients, thus generating noise. Segmentation is often preceded by pretreatment routines to facilitate the voxels attribution such as smoothing filters to reduce noise. In this work, the median filter used in Martinez-Carvajal et al. (2019) has been replaced by an anisotropic filter (Bhattad et al., 2013; Pilny and Janacek, 2006; Tschumperl and Deriche, 2003). A simple diffusion filter reduces noise by smoothing values proportionally to the gradient of gray value. However, it tends to blur the edges between different phases (i.e. interfaces between gravel, voids and biosolids). In anisotropic diffusion, noise is reduced by applying smoothing in the edge tangential direction but not in the normal direction which preserves the location of interfaces better.

The application of the anisotropic filter leads to a better recognition of voxels presenting a Partial Volume Effect (PVE). PVE appears when two phases are taken into account into a single voxel, thus making it more difficult to assign
225 the voxel to a single phase. These voxels are preferentially located at edges of phases, where the gray level gradient is maximum. PVE voxels were defined by thresholding the image of the gradient of gray values using the FIJI's routine 'Find Edges' (Martinez-Carvajal et al., 2019).

2.5. Post-treatment

230 2.5.1. Morphological properties

Three pore scale measurements were obtained by image post-processing (after segmentation): volume fraction profiles for each phase (voids, gravel, and biosolids), SSA profiles for the interface between voids and biosolids and pore size distributions of the void phase.

235 Volume fraction profiles were obtained by computing the ratio between the number of voxels belonging to each phase and the total number of voxels (the sum of the three phases) for each horizontal slice. Profiles were smoothed using a moving average window of 57 voxels in depth with a displacement of one voxel at a time. 57 voxels (2 mm) corresponds to the d_{10} value of the gravel size
240 distribution.

The SSA is obtained by dividing the surface of the interface between voids and biosolids by the bulk volume. The surface of the interface between voids and biosolids was computed using the Cauchy-Crofton cutting line method (Legland et al., 2011; Lehmann and Legland, 2012; Santaló, 2004). This method allows to
245 numerically estimate the interface areas with a better accuracy than the direct counting of interface voxels, which presents a "staircase effect". In addition, it has a much lower computational cost than methods based on surface meshes. For each voxel belonging to the void phase located at the interface with the biosolid phase, the algorithm consists in counting the number of neighboring
250 voxels in the biosolid phase with respect to three families of connection: 1-connected (face-connection), $\sqrt{2}$ -connected (2D diagonal/edge connection) or

$\sqrt{3}$ -connected (3D diagonal/corner connection). The number of connections of each type is then multiplied by a characteristic coefficient obtained from the Voronoi tessellation of the unit sphere. The sum of these contributions results
 255 in the value of the surface. The specific surface area profile was obtained by computing the specific surface areas of subsamples 57 voxels in height without overlay.

The pore size distribution was obtained by computing the local thickness map of the void phase of the segmented images. The local thickness map of
 260 a 3D object is another 3D image where each voxel stores the diameter of the largest sphere that fits inside the object and contains the voxel (without being necessary centered at the voxel). The computation of the map was conducted using the FIJI plugin called Local Thickness Map (Hildebrand and Rüeggsegger, 1997; Saito and Toriwaki, 1994). This method has previously been used to
 265 compute pore size distributions in sandstone (Chang et al., 2016).

2.5.2. Representative Elementary Volume (REV)

The REV for the volume fraction of voids, biosolids and gravels were computed for one of the samples (MON1A) at several 2D slices (1-voxel thick) corresponding to four different depths in the sample. They are herein referred
 270 to as void-REV, biosolid-REV and gravel-REV. The average volume fraction of each of the three phases is computed for concentric circular windows of increasing radius ranging from 1 voxel to the sample radius. The volume fractions of each phase are then plotted as a function of the radius of the circular windows. The analysis of several 2D sections using concentric circular elements was preferred
 275 to a 3D analysis (using concentric cylinders) because (i) the influence of *Phragmites* on the sample structure is easier to analyse and (ii) French VF treatment wetlands present a natural vertical gradient of phase distribution in the direction of the water flow due to cake and bed filtration of solid particles (Martinez-Carvajal et al., 2019). Therefore, 3D REV would integrate vertical
 280 variations of phase volume fractions that are already analyzed in volume fraction profiles and introduce a bias regarding the minimum representative sample

Figure 1: Electrical conductivity map of the filter. FP stands for feeding points. Samples regions are indicated on the map. $\bar{\sigma}$ is the electrical conductivity mean value and $\Delta\sigma$ is the chosen threshold value for the measurement sensitivity

diameter.

3. Results and Discussion

3.1. FDEM and choice of sampling points

285 A map of the electrical conductivity of the topmost layer of the filter is
 drawn according to the methodology presented in SECTION 2.1 (Figure 1).
 This method captures the spatial variation of biosolid content over the entire
 filter surface. In highly conductive materials, Clement et al. (2018) and McNeill
 (1980) advised to only consider variation ($\Delta\sigma$) larger than 6% of the mean
 290 value of EC ($\Delta\sigma = 1 \text{ mS} \cdot \text{m}^{-1}$, in our case) as significant compared to the
 manufacturer specification which considers variations larger than $\Delta\sigma = 0.1 \text{ mS} \cdot$
 m^{-1} as significant. EC values within the ranges ($\pm\Delta\sigma$) and ($\pm 2\Delta\sigma$) correspond

to 75% and 95% of the filter surface, respectively. The high values of EC ($13-17 \text{ mS} \cdot \text{m}^{-1}$) observed for almost the entire surface of the filter indicate a homogeneous distribution of organic matter and water embedded in the biosolids because biosolids present a high moisture content due to capillarity effects and the presence of biomass.

The remaining surface corresponds to circular areas of less than 1 m in diameter located around the feeding points. A significant increase of the EC in these regions is observed, which is most likely a result of the higher organic matter content and water content in the biosolids and/or a larger content of biosolids in depth. These results are complementary to those presented in Forquet et al. (2011) where electrical resistivity measurements showed that, during a feeding period, the water content in a first stage French VF treatment wetland increased by approximately 7-14% at distances less than 0.5 m from the feeding points and by less than 4% for the rest of the filter. When water exits the feeding pipes, it infiltrates predominantly in the vicinity of a feeding point, leading to a preferential accumulation of biosolids and water retained by capillarity. Except for these localized effects, variations of EC in Figure 1 do not depend on the distance to the feeding points. This homogeneity may be explained by the age of the French VF treatment wetland (25 years) as it is observed that the homogeneity of the biosolid distribution depends on the age of the facility and the applied load (Molle, 2014).

Values in the white regions on Figure 1 are not plotted as they correspond to parts of the filter with specific features that induce artefacts (Clement et al., 2018): (i) the freeboard produces an artifact that extends over 1m from the filter edges. (ii) at the bottom-right part, a metal lid covers the water collector at the outlet of the the French VF treatment wetland which also induces an artifact.

Xray-CT complements the data acquired by electromagnetic mapping. Xray-CT gives insight on the vertical structure and phase distribution of the filter as its resolution (about 100 times better than FDEM data) gives more detail. FDEM can drive the choice of the Xray-CT sample location by identifying areas

with homogeneous water and biosolid content. Consequently, Xray-CT sample
 325 location may be chosen wisely to achieve good spatial representativity with a
 limited number of samples. As the surface of the filter is considered to be
 homogeneous away from the immediate surrounding of the feeding points and
 in order to compare samples to each other, the sampling points were chosen
 randomly over this area. The 3 sampling regions are shown in Figure 1.

3.2. Comparison of pore scale properties between the samples

3.2.1. Phase Volume Fraction Profiles

In figure 2, the volume fraction profiles of each phase (voids, biosolids, and
 gravels) are drawn for each sample and presented along with an example of 2D
 vertical slice. Similar trends, observed in all the samples, can be highlighted:

- 335 (i) The void volume fraction is the highest near the surface (50-85 %) and
 decreases with depth to levels lower than the two other phases,
- (ii) In the first centimeters, the biosolid and the void volume fraction are
 always predominant
- (iii) At greater depths, the biosolid and gravel volume fractions are the pre-
 340 dominant phases

These observations match the current understanding of filtration mechanisms in
 French VF treatment wetlands. The deposit layer is built up by cake filtration.
 This leads to a higher content of biosolids in the deposit layer than in the
 gravel layer. The high resolution of Xray-CT provides interesting details on
 345 the structure of the deposit and biosolids within the filter. Figures 2a to 2f
 illustrate that there is no clear delimitation of the end of the deposit layer and
 the beginning of the gravel layer at the micro-scale. In particular, the end of the
 deposit layer cannot be defined as the depth at which the first gravel appears,
 as some gravel can be observed near the surface at depths where the biosolid
 350 fraction is predominant. This may be explained by segregation mechanism
 (Rosato et al., 1987): fine particles of the biosolids move beneath some pieces
 of gravel forcing them to migrate upwards. However, a systematic transition

Figure 2: Volume fraction versus depth of each phase (voids, biosolids, and gravels) (on the left), 2D vertical slice of the segmented image (on the right)

Table 1: Thickness and average volume fractions of the constitutive phases for the different layers

	Sample	Volume fraction (-)			Thickness (mm)
		Voids	Biosolids	Gravels	
Deposit layer	MON1A	0.18	0.65	0.16	69
	MON1B	0.13	0.71	0.16	81
	MON2A	0.19	0.71	0.10	100
	MON2B	0.21	0.72	0.06	89
	MON3A	0.14	0.77	0.09	99
	MON3B	0.11	0.80	0.09	97
Gravel layer	MON1A	0.21	0.21	0.58	73
	MON1B	0.08	0.53	0.40	42
	MON2A	0.10	0.34	0.56	41
	MON2B	0.06	0.31	0.63	33
	MON3A	0.03	0.32	0.65	38
	MON3B	0.03	0.34	0.63	44
Deposit layer + Gravel layer	MON1A	0.21	0.42	0.36	142
	MON1B	0.23	0.57	0.20	123
	MON2A	0.19	0.59	0.22	141
	MON2B	0.28	0.54	0.18	122
	MON3A	0.16	0.61	0.23	137
	MON3B	0.10	0.65	0.25	141

is observed between the deposit layer (where the void and biosolid phases are predominant) and the gravel layer (where the biosolid and gravel phases are predominant). The inversion of phase predominance is readily identified at the intersection of the biosolid and gravel curves which occurs around 40-50% of volume fraction. This systematical behavior is used to define the lower limit of the deposit layer. Similarly, the upper limit of the deposit layer is defined at the intersection of the void and biosolid curves at the top of the samples. The thickness of the deposit layer is represented by the red-shadowed regions in Figures 2a to 2f. Furthermore, by setting this criterion, it is possible to compare specific features of the deposit layer and gravel layer (Table 1).

Deposit layer. Most of the volume in the samples corresponds to the deposit layer. The total thickness of the deposit layer ranges between 7 and 10 cm. Samples MON2A, MON3A and MON3B have a deposit layer thicknesses of ap-

proximately 10 cm. Samples MON1A, MON1B and MON2B have a slightly thinner deposit layer: 7 cm, 8 cm and 9 cm, respectively. Samples from sampling region 1 show the lowest biosolid volume fraction (0.65-0.70) followed by sampling region 2 (0.71) and 3 (0.77-0.80). Xray-CT can capture small variations of the biosolid volume fraction and of the layer thickness. Nevertheless, the differences obtained among the samples (from 0.1 to 3.0 cm in the deposit layer, and from 1% to 15% in the biosolid volume fraction) may be explained by local compaction associated to maintenance operations (i.e. person walking on the filter) or local accumulation of stems and leaves. It creates a micro-topography that differs from the heterogeneities usually induced by uneven effluent distribution. This micro-topography is not captured by spatial averaging technique like FDEM but is readily observable by Xray-tomography.

To better estimate how the void volume fraction evolves in depth, the contribution of *Phragmites* has to be removed. For over half of the sample, the void volume fraction reaches values ranging from 0% to 15%. High values near the surface may visually be explained by the presence of a large number of macropores which is probably the sign of an ongoing consolidation of fresh biosolids or caused by movement of *Phragmites* stems. Finally, it must be pointed out that despite the low void volume fraction observed at the bottom of the deposit layer, water infiltrates normally on the surface of the filter.

Gravel layer. The phase distribution in the gravel layer (the region below the deposit layer in Figures 2a to 2f) tends to be more homogeneous than in the deposit layer with the exception of samples MON1B and MON2B. In other samples where *Phragmites* are not present, gravel volume fraction ranges from 0.55 to 0.65 which is in agreement with the value of 0.65 reported by Kim and Forquet (2016). Depending on the sample, the average value of biosolid volume fraction ranges from 0.21 to 0.53 with a decreasing gradient with increasing depth. The average void volume fraction are less than 0.1 with the exception of sample MON1A. Similar to what has been mentioned earlier for the deposit layer, the high biosolid content and the low porosity appears to not be detrimental

to filter operation. It seems that the volume of void space is still large enough to provide gravity-driven infiltration. The void phase identified by tomography corresponds to pores larger than the tomography’s resolution ($35 \mu m \cdot voxel^{-1}$). These large pores are often referred-to as the ‘open porosity’. To access the micro-porosity inside the biosolid phase it would be necessary to enhance the resolution, which would require smaller sample size. The choice of sample size is discussed in SECTION 3.3. In a gravel area with no biosolids, the gravel fraction goes up to 80%, whereas samples show that its fraction could be 5% to 10% lower (sample MON2B and MON2A, respectively) where fouling material is present. This result contradicts the usual understanding of clogging that considers a constant gravel volume fraction. Such a decrease in gravel density is likely due to the mechanical action of the biosolids during its invasion of the gravel pore space as mentioned in previous section.

Singularities. The more accurate the measurement is, the more likely heterogeneities are likely to be seen. These singularities may be explained for most of the cases by specific local features like the presence of reeds.

The presence of *Phragmites* alters considerably the void distribution and biosolid accumulation in the samples. This is particularly the case for sample MON1B (Figure 2b), where the criterion to define the limits of the deposit layer proposed in this work fails to describe accurately the sample. The green regions in Figures 2a to 2f show clearly how the major fluctuations in phase distribution are caused by *Phragmites*. A more homogeneous description could be obtained if *Phragmites* and their *Aerenchyma* were segmented separately. To assess this hypothesis, the image treatment methodology should evolve to a 4-phase segmentation procedure. This is particularly challenging since the contrast between the plant tissues and the biosolids in the gray-scale image is weak. A methodology based on shape recognition might enable such a segmentation.

There are also some fluctuations in the profiles that are not caused by the presence of a *Phragmites* (blue-shadowed regions in Figures 2a to 2f). For instance, in samples MON2A and MON2B, the gravel layer contains a zone of

Figure 3: Specific Surface Area (SSA) versus depth. Wide lines correspond to the deposit layer

relatively low biosolid volume fraction. A simulation of solids transport at the pore scale may help to understand why there is no biosolids in some part of the gravel.

3.2.2. Specific surface area profiles

430 The Specific Surface Area (SSA) is an important parameter for biochemical reactions and for multiphase solute transport modeling. In French VF treatment wetlands, the SSA of the interface between voids and biosolids is of particular interest. Oxygen transport is a limiting factor for nitrification and mineraliza-
 435 tion (Dotro et al., 2017). Oxygen diffuses quickly in the air-filled porosity and most of the biological degradation occurs within biosolids (Molle, 2014), therefore the oxygen transfer from the air-filled porosity to the biosolids depends on the SSA of the interface between biosolids and voids.

The SSA profiles of the interface between biosolids and voids in Figure 3 show

Table 2: Mean Specific Surface area (SSA) of the sample constitutive layers

	Specific Surface Area (SSA) ($m^2 \cdot m^{-3}$)					
	MON1A	MON1B	MON2A	MON2B	MON3A	MON3B
Deposit layer	539	362	507	468	367	384
Gravel layer	437	222	275	184	112	126

similar trends independently of the sample location. The sections where lines
440 are wider correspond to the limits of the deposit layer following the criterion
defined in SECTION 3.2.1. The fluctuations in the SSA profiles are due to local
variations of void volume fraction and pore size, but the global trends are similar
: (i) The highest values are located in the deposit layer ranging from 200 to 1000
 $m^2 \cdot m^{-3}$ for all the samples, while (ii) in the gravel layer, the range is lower
445 and narrower (100 to 400 $m^2 \cdot m^{-3}$), except for sample MON1A. This may be
explained in part by the larger volume of voids in the deposit layer compared
to the gravel layer (see Table 1). The mean SSA for each layer is presented in
table 2. The measurement of the SSA following the methodology detailed in
SECTION 2.5.1 depends strongly on the resolution of the tomography (35 μm
450 in this study). Increasing the resolution would allow the visualization of smaller
pores, which if filled with air would increase the SSA of the interface between
biosolids and voids.

Table 3 compares results of this work with predicted and measured values
of SSA for different treatment wetland media reported in the literature. Values
455 vary mainly depending on the experimental methodology, and on the 'type' of
surface. The comparison must be conducted carefully as each measurement
is representative of different characteristics of the media (gravel surface, root
surface, biofilm surface, micro-porosity surface) that are relevant at different
space scales.

460 Results agree with theoretical approximations of SSA in other subsurface
flow wetland designs (Kadlec and Knight, 1996; Khatiwada and Polprasert,
1999; Austin et al., 2007). In these approaches, the SSA is computed or mea-

sured considering macroscopic features of the media, such as the surface of granular media or the surface of the biofilm developing around the granular material and plant roots. The predicted values in Polprasert and Khatiwada (1998) and Khatiwada and Polprasert (1999) are considerably lower than the values obtained in this study as they computed the 'effective specific surface area' based on bio-kinetic models. The effective SSA is the active surface where biological reactions can take place. In fact, bacteria would only develop in a small fraction of all the available surface and thus the effective SSA should be lower than the value estimated by this method.

Measurements relying on adsorption/desorption methods yield values 10000 times larger than those of this study. These are molecular scale measurements, which depend on the molecule that is being adsorbed/desorbed (typically $H_2O(g)$ or $N_2(g)$). In the work of Kania et al. (2018), the studied samples were taken from the deposit layer of French VF treatment wetlands. Values with a similar order of magnitude of SS were found by Poots and McKay (1979) and Szafraniek-Nakonieczna and Bennicelli (2010) when measuring the specific surface of peat using N_2 adsorption, mercury porosimetry and solution isotherms. However the measurements vary depending on the chemical and physical interactions between the adsorbate and the absorbent. Most of these surfaces are not accessible to X-ray-CT methodology due to their location within the biosolids and because characteristic pore sizes are smaller than the scan resolution.

The use of SSA values for the interface between voids and biosolids as parameters in models for transport and reaction in French VF treatment wetlands must be carried out carefully. The measurements from this work are relevant for macroscopic transport of solutes within the open porosity, such as oxygen transport from the atmosphere to the biosolids, and are not relevant for microscopic transport phenomena inside the biosolids, such as adsorption and desorption or biochemical kinetic models.

Compared to other methods, the advantages of using X-ray tomography images to measure the SSA are: (i) a high spatial resolution compared to experimental methods that usually provide a global estimate only, (ii) measurements

rely on the actual geometry extracted from the segmented images and there
 495 is no assumption of specific pore shape, or particle shape (iii) there is no bias
 induced by chemical or physical interactions between the adsorbate and the ad-
 sorbent. However, the computed values do not represent SSA of pores smaller
 than the resolution of the tomography, and thus, this approach does not take
 into account the small capillary pores within the biosolid phase. Nonetheless,
 500 small pores are often saturated with water thus limiting oxygen transfer during
 the feeding period and a part of the rest period.

3.2.3. Pore Size Distribution (PSD)

The description of the pore structure of treatment wetlands is critical for the
 understanding of pore scale phenomena like fluids flow or solute and suspended
 505 solid transport. In Figure 4, the PSD of all the samples is plotted for the deposit
 and gravel layers separately. There is a remarkable similarity among samples
 regarding the mode, and the dispersion of pore sizes around the median values
 (Figure 5).

Omitting small peaks at the largest sizes, (> 3 mm) which correspond to reed
 510 cavities, the distributions in Figures 4a and 4b can be described as unimodal.
 The mode ranges from 0.40 to 0.75 mm, (~ 10 to 20 voxels) independently of
 the layer (deposit or gravel) and the sample location.

In the deposit layer (Figure 6), pores smaller than 4mm represent more
 than 90% of the void volume for all samples, except for sample MON2B which
 515 presents a *Phragmites* occupying a large volume within the deposit layer. In
 the gravel layer (Figure 6), the range of pore size is narrower. Pores smaller
 than 3 mm correspond to more than 95% of the void volume, except for samples
 MON1A and MON1B which have a *Phragmites* at the bottom of the sample.
 Some pores in the deposit layer may have been enlarged by movement of *Phrag-*
 520 *mites* as it can be seen in sample MON2A (Figure 2c), but this behavior has
 been observed clearly only once. In contrast, samples MON1B and MON2B
 contain large *Phragmites* deeper into the sample and no pore enlargement is
 observed around them. The only relevant difference among the samples is the

(a) Deposit layer

(b) Gravel layer

Figure 4: Cumulative (at the top) and frequency (at the bottom) PSD for the deposit layer and the gravel layer

(a) Deposit layer

(b) Gravel layer

Figure 5: Pore Size Distribution (PSD) boxplots for the deposit layer and the gravel layer. Dash line indicates the presence of *Phragmites*.

Figure 6: 3D rendering of pore local thickness map in the deposit layer and the gravel layer.

diameter of the largest pores, which correspond to *Phragmites* (Figure 5).

525 Kim and Forquet (2016) describe a bi-modal distribution of pore size. The mode of larger pores they observed matches the one obtained in this study (0.5 mm). The mode of the smaller pores in their work (near 50 μm) is almost at the same order of magnitude than the resolution of the scans in this study, explaining why those pores cannot be observed. This comparison must be interpreted very
530 carefully as (i) the method used in Kim and Forquet (2016) to compute the PSD relies on a different algorithm (Dougherty et al., 2017), (ii) the pore-size distribution was calculated on 2D thin section images and (iii) the procedure to produce the thin sections may affect pore shape.

Compared to the thin section method, X-ray tomography provides three
535 main advantages: (i) it provides 3D informations with an acceptable resolution for larger volumes, (four times the thickness of samples used for thin sections), (ii) 3D tortuosity and 3D pore connectivity can be studied (Rezanezhad et al., 2009), which gives a better description of the sample's morphology, and (iii) no sample pretreatment is needed.

540 The representativity of tomography has been discussed by comparing pore properties of different samples extracted at different locations in the filter. The heterogeneity within the sample and the smallest representative sample's diameter are discussed in the next section.

3.3. Representative Elementary Volume (REV)

545 The void-REV, biosolid-REV and gravel-REV are deduced by analyzing the convergence of the volume fractions to a constant value in Figure 7. When convergence is reached, at a certain diameter of circular window, the volume fractions reach a plateau (i.e. plateau of region II in the work of Costanza-Robinson et al. (2011)) with a value close to the global volume fraction of the
550 slice. This implies that the sample diameter is large enough to be spatially representative of the heterogeneities of phases distribution that can be observed at the pore scale. Results of this study indicate that a 2D-REV is reached in the deposit and gravel layers, as long as no reed is present in the 2D-slice and

(a) Depth = 0 mm

(b) Depth = -45 mm

(c) Depth = -85 mm

(d) Depth = -124 mm

(e) Depth = -124 mm, four phase segmentation

Figure 7: Phase volume fraction versus relative diameter at four different depths

the slice is not located near the surface. It seems that for phases with more than
555 50% of volume fraction, REV is reached when variations are less than $\pm 5\%$. For
phases with less than 50% of volume fraction, REV is reached when variations
are less $\pm 2.5\%$. To our knowledge, this is the first time an analysis of the REV
in Xray-CT on first-stage French VF wetlands has been conducted. These
560 results could be complemented by the analysis of the REV for other pore scale
properties different than phase volume fraction (Al-Raoush and Papadopoulos,
2010).

Usually, REV assessments in pore scale studies focus on the porosity-REV
(i.e. voids-REV). In this study, a particular attention is given to biosolids
because most of the microbial activity and the treatment of water-soluble pol-
565 lutants takes place in this phase (Molle, 2014). The gravel phase is studied as
well as it has been observed that the compaction of the gravel layer decreases
as biosolids are retained within the filter.

Figures 7a to 7e correspond to four different horizontal (perpendicular to
the main water flow direction) 2D-slices taken in sample MON1A: two slices
570 from the deposit layer (one at the top of the deposit and other at a location of
maximal biosolid accumulation) and two slices from the gravel layer (with and
without *Phragmites* identified inside the slice).

In Figures 7b and 7c, no *Phragmites* is present. The volume fractions for all
the phases converge to a global value when the sub-sampling diameter reaches
575 30% of the total diameter of the sample (1.5 cm). There are no large oscillations
of the volume fractions beyond this point. The reader must be aware that this
assessment remains mainly qualitative.

At the top of the deposit layer, the void and biosolid distributions are very
heterogeneous. This reflects the fact that the surface of the deposit layer is
580 naturally very irregular. As a consequence the curves for biosolid and void
phases in Figure 7a show no satisfactory convergence.

When a *Phragmites* is present, the biosolid and void distributions exhibit
a high degree of heterogeneity, and thus no convergence is reached (figure 7e).
For this slice, a manual segmentation of the reed was performed and is shown in

585 green. Then the REV is deduced for the volume fraction of four phases (void, biosolid, gravel and *Phragmites*) in Figure 7e. The convergence of the void and biosolid volume fractions occurs for smaller radius and a representative volume is reached at 70% of the total diameter of the sample. This does not affect the convergence for the gravel phase. This figure shows that *Phragmites* have
 590 an effect on the distribution of biosolids and gravels in its surroundings. It is then relevant to optimize the methodology presented here to obtain four phase segmentations of the samples in order to describe better the morphology of the samples.

For the study of pore scale phenomena using Xray-CT, results demonstrate
 595 that the representativity of the sample regarding phase volume fractions is achieved at sample diameters larger than 1.5 cm (7.5 times the size of d_{10} of the gravels in this study). However, considering the sensibility of the samples to mechanical deformations during sampling, it is still recommended to extract samples of minimum 5cm in diameter, which will give a security factor of more
 600 than three times the REV size observed in this study.

4. Conclusion

This study assesses the spatial representativity of pore-scale measurements obtained by Xray-CT of the filtering media in first-stage French VF treatment wetlands. Both the heterogeneity of phase distribution inside a single sample
 605 and the differences of pore scale properties between several samples of a treatment wetland have been considered. The results suggest that a sample diameter of 5cm is large enough to (i) be representative of the heterogeneous phase distribution (voids, fouling material and gravel) at the pore scale, independently of the location of the sample and (ii) preserve the structure of the sample during
 610 the sampling by limiting shear stress impact at the core of the sample.

Prior to Xray-CT, the distribution of biosolids in a mature French VF treatment wetland was assessed based on electromagnetic measurements to select sampling points representative of its heterogeneity. FDEM allows a 2D-

estimation of the biosolid distribution as electrical conductivity is highly correlated to the thickness of the deposit layer due to its high organic matter and water contents. The electrical conductivity measurements were found to be homogeneous over nearly 95 % of the treatment wetland surface. This is explained probably by the age of the wetland and its good maintenance. The remaining 5 % of the surface showed high values of electrical conductivity in regions of 1 m in diameter around the feeding points which are explained by preferential water retention and biosolid accumulation at those locations. This information leads to the conclusion that large scale heterogeneities in biosolid distribution cannot be considered as a cause of non-representative Xray-CT for the treatment wetland in this study.

After scanning and segmenting the tomographies, the volume fraction and specific surface area vertical profiles are computed for all the samples. They show several similarities independently of the sampling point. All samples show a vertical stratification of these properties which results from the vertical filtration of wastewater in the treatment wetland. A special focus is put on the transition from the deposit layer to the gravel layer. In order to make better comparisons of pore scale properties, a criterion to delimit the deposit layer is introduced. The upper and lower limits of the deposit layer are located at the intersection of the void and biosolid volume fraction profiles and at the intersection of the biosolid and gravel volume fraction profiles, respectively. Though Xray-CT capture very well variation of these properties, samples are not found to be significantly different from each other in the context of first stage French VF treatment wetlands.

The global biosolid volume fraction for the deposit layer and the gravel layer, and the thickness of the deposit layer measured by Xray-CT show an interesting correlation with small variations of EC ($> 1\mu S \cdot m^{-1}$) measured by FDEM. This could be further exploited to characterize biosolid distribution in French VF treatment wetland being aware that the contributions to the EC measurement of organic matter, moisture, biosolid and gravel volume fractions and the thickness of the deposit layer cannot be distinguished solely by FDEM.

645 Pore size distributions are similar for all samples; the mode and the range
of pores size distribution depend only on the vertical location in the sample
(deposit layer or gravel layer). The presence of *Phragmites* is the main cause of
non-systematic differences between pore-scale properties of the samples.

A study of the 2D-REV at several depths was conducted for one of the sam-
650 ples. It consists in the analysis of the dependency of the average value of phase
volume fractions (voids, biosolids and gravels) on the size of a sub-sampling
circular window. Based on a qualitative assessment of this relationship, REV is
estimated to be achieved at one third of the sample diameter (1.6 cm) when the
analyzed depth does not include *Phragmites* or strongly heterogeneous features
655 as observed near the top of the sample.

This study sets a solid base for the spatial representativity of further investi-
gations on mechanisms that govern pore-scale filtration and biological treatment
in French VF treatment wetlands, including water flow, as well as transport of
particulate matter and dissolved oxygen. This is valid for mechanisms depending
660 predominantly on macropore structure, such as gravity driven flow, and advective/
diffusive oxygen transport from the atmosphere to the biosolids during the
feeding and rest period of the first-stage filters.

Table 3: Comparison of different studies on treatment wetland Specific Surface Areas (SSA)

Mass SSA (m^2/g)	Volumetric SSA (m^2/m^3)	Type of media	Type of surface	Method	Pretreatments	Comments	Reference
	10000	Free Water Surface (FWS) treatment wetland	gravels/voids + root Surface/voids	Theoretical approximation			Polprasert and Khatiwada (1998)
	4.4	FWS treatment wetland	gravels/voids + root Surface/voids	Predicted value			Polprasert and Khatiwada (1998)
	2.94-4.20	FWS treatment wetland	biofilm/voids	Predicted from kinetic model for COD degradation		Slab geometry assumption	Khatiwada and Polprasert (1999)
	360 - 24000	SubSurface Flow (SSF) treatment wetland	gravels/voids + root surface/voids	Theoretical approximation			Kadlec and Knight (1996) and Khatiwada and Polprasert (1999)
	3.6- 47.37	SSF treatment wetland	biofilm surface surrounding roots/voids	Predicted from kinetic model for COD degradation		Cylindrical geometry assumption. Values depend on the biofilm thickness and the root diameter	Khatiwada and Polprasert (1999)

8.9×10^{-4}	810	SSF treatment wetland	granules/voids	water gravimetric	None	surface estimated from water film thickness ($10\mu m - 60\mu m$), spherical granules	Austin et al. (2007)
13.5-218	$1.35 \times 10^7 - 21.8 \times 10^7$ ⁶	Deposit of French VF treatment wetlands	deposit/voids	Dynamic vapor sorption	Drying (1 day, 35 °C) + sieving (1 mm)	Mixing samples	Kania et al. (2018)
	100 - 1000	deposit + gravel French VF Treatment Wetland	Biosolids/voids	Tomography	None	Depending on the depth of the sample	This study

⁶Assuming bulk density of 1000 kg/m^3

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