Etude du séchage des boues par conduction

Après avoir proposé le concept du séchage combiné, les transferts de chaleur et de masse présents au cours du séchage conductif sont abordés. L'impédance exhibée par la matrice poreuse à la diffusion de vapeur est évaluée en fonction de la siccité des boues. Des corrélations pour la prévision de ce paramètre sont élaborées à partir de résultats expérimentaux.

L'étude expérimentale met en valeur l'influence de la fréquence de retournement sur les performances du séchage conductif.

1. Résumé de l'article soumis à la revue Journal of Porous Media

1.1. Introduction

Le concept du séchage combiné par énergie solaire et pompes à chaleur consiste à fournir aux boues l'appoint d'énergie nécessaire pour améliorer les conditions d'évaporation au sein et en surface des boues lorsque l'énergie solaire devient insuffisante. Le chauffage par une dalle chauffante a été proposé au chapitre 1 comme solution permettant d'améliorer le transport de vapeur des couches inférieures des boues vers les couches superficielles. Dans ce chapitre présenté sous forme d'article, l'intérêt est porté sur l'étude des mécanismes de transferts qui ont lieu au cours d'un séchage conductif appliqué sur la face inférieure des boues via une dalle chauffante.

Les mécanismes de séchage d'un milieu poreux sont complexes à décrire du fait que les transferts de chaleur et de masse sont étroitement imbriqués et conditionnés par la structure de la matrice poreuse. Les éléments développés dans ce chapitre tentent de constituer une synthèse permettant d'étudier les mécanismes de transferts de chaleur et de masse ayant lieu au sein des boues, dans le but de proposer un modèle prédictif pour le séchage par conduction des boues de stations d'épuration et une meilleure gestion de l'énergie d'appoint.

D'un point de vue bibliographique, l'intérêt porté aux boues résiduaires est assez récent, et les travaux réalisés dans le domaine de la rhéologie portent essentiellement sur les boues liquides, soit dans le cadre de l'optimisation des procédés de traitement, où les paramètres rhéologiques peuvent affecter les opérations de filtration d'épaississement ou de déshydratation, soit dans le calcul des pertes de charge dans le cas de dimensionnement de systèmes de pompage. Depuis quelques années, des laboratoires européens, conscients des enjeux et du manque d'informations scientifiques, ont entrepris des recherches fondamentales dans le domaine des boues. Ces nouveaux développements concernent les procédés de séchage thermique et plus précisément le séchage convectif, ce qui laisse le séchage par conduction une voie non explorée et les propriétés des caractéristiques des boues inconnues.

Le séchage est une opération visant à évaporer l'eau contenue dans les boues. L'évacuation de cette eau engendre généralement des déformations importantes de la matrice poreuse dues au phénomènes de retrait, évoluant en fonction de la teneur en eau des boues. L'aspect rhéologique des boues devient ainsi un paramètre clé pour la modélisation du séchage, et son évolution doit être prise en compte pour la description des phénomènes de transferts mis en jeu.

1.2. Méthodologie

Dans une première partie de l'étude, les notions indispensables à la caractérisation des milieux poreux sont introduites et les équations régissant le séchage conductif établies. L'article présente un recueil de l'étude bibliographique effectuée sur le séchage des matériaux poreux exhibant une porosité ou un comportement à l'évaporation identiques à ceux observés pour les boues. L'étude permet d'introduire un facteur décrivant l'impédance de la matrice poreuse à la diffusion de la vapeur d'eau au cours du séchage. Cependant, l'application régulière du retournement ainsi que la nature et la texture particulières des boues ne permettent pas l'application directe des résultats présents dans la littérature. Il est alors nécessaire de procéder par voie expérimentale dans le but d'élaborer une loi décrivant l'évolution de ce facteur en fonction de la siccité des boues ou de la porosité.

L'approche retenue pour l'évaluation des diffusivités de la vapeur d'eau dans les boues est une approche macroscopique. Elle tient compte uniquement de l'effet de la porosité externe définie par l'agrégation des boues au cours du séchage. Le transfert de vapeur est décrit par une équation de diffusion Fickienne modifiée par l'introduction de l'impédance à la diffusion.

Un dispositif expérimental est réalisé et permet de reproduire les conditions de séchage par conduction d'un échantillon de boues ayant une siccité initiale d'environ 20 %. Le dispositif est instrumenté pour suivre les températures des boues au cours du séchage et évaluer à partir du gradient de température qui s'établit sur l'épaisseur de l'échantillon, la chaleur transmise aux boues par conduction. Un système de pesée en continu permet d'évaluer les capacités évaporatoires. Les expériences sont effectuées avec trois flux thermiques : 300 W/m², 525 W/m² et 700 W/m² afin de détecter l'influence de ce paramètre sur l'évaporation. Dans la plage de flux thermique examinée, aucun effet de la densité de flux imposée sur la diffusivité de la vapeur d'eau dans les boues n'est décelé. Cependant, les résultats expérimentaux permettent d'élaborer des corrélations pour la prévision des résistances à la diffusion de vapeur en fonction de la siccité des boues.

D'autres expériences sont conduites pour étudier l'effet de l'augmentation de la fréquence de retournement sur le comportement des boues. Les résultats montrent que la diffusion de vapeur est d'autant plus accentuée que la fréquence de retournement est élevée.

1.3. Conclusions

Le transport de masse se produisant dans les boues au cours d'un séchage conductif est expérimentalement étudié dans l'objectif de modéliser les transferts de chaleur et de masse qui ont lieu au sein des boues au cours d'un séchage conductif. Les résultats expérimentaux permettent de corréler l'impédance de la matrice poreuse à l'évaporation en fonction de la siccité des boues. L'analyse des résultats montre qu' une augmentation de la fréquence de retournement entraîne une accélération des transferts de vapeur au sein des boues. Toutefois, aucune influence de la puissance de chauffage imposée sur ce paramètre n'a été détectée.

2. Characterization of sewage sludge water vapor diffusivity in low temperature conductive drying (copy of the submitted paper)

Article à paraître dans journal of Porous Media

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2.1. ABSTRACT

In order to study the low temperature conductive drying of urban sewage sludge and evaluate their essential drying characteristics, a laboratory-scale drying device was set up. Sludge was modelled as a coarse aggregated porous medium and experiments were conducted to study its rheological behaviour throughout a drying cycle and the effects of aggregation on diffusion. The investigations are based on a macroscopical model of sludge aggregates where only external porosity is accounted for. The paper presents a method to evaluate water vapor diffusivity within urban sludge, based on the analytical solution of a fickian diffusive model which enables diffusivity determination by simple exponential regression over experimental data. Experiments were carried out with three different levels of heating fluxes: 300W/m², 525W/m² and 700W/m² without any remarkable effect of flux density on water vapor diffusivity over the tested range. Further experiments were conducted to highlight the effect of mixing frequency. Predictive correlations for water vapor diffusivity as a function of sludge dry solid content (DSC) and mixing frequency are reported in this work.

2.2. Introduction

Knowledge of product moisture diffusivity is essential in simulating and optimizing a drying process. In a phenomenological global approach, the moisture diffusion represents all moisture gradient driven transfer mechanisms such as capillary flow (diffusion in liquid phase), migration in the adsorbed layer, vapo-condensation and true diffusion of vapor in air. Moreover, diffusion in a porous medium is of great importance since it covers a great number of foodstuffs and soils. The study of the diffusion mechanism in a porous medium is more complicated due to additional restrictions to fluid flow generated by the solid matrix of the porous medium.

Many articles were published recently about the determination of the moisture diffusivity for various foodstuffs and soils, using different experimental data processing methods ([MOL0a], [KOH02], [JON03]), especially in soil physics and agro-alimentary studies. The analytical solutions of these models relied on restrictive assumptions such as constant moisture diffusivity and product constant volume. However, no work focused on water vapor diffusivity estimation in sewage sludge throughout a drying cycle. Therefore, this paper aims at studying the evolution of water vapor diffusivity in sewage sludge during a low temperature conductive drying cycle.

Throughout the cycle, the sludge is frequently mixed in order to hygienize and aerate it. This drying process is applied to the sludge, mechanically dehydrated to a dry solid content of 20%, and stopped when a dry solid content of 70% is reached.

The drying mechanisms of a porous medium are complex to describe due to the fact that heat and mass transfers narrowly overlap. Many studies focused on the engineering of a drying process. They either stressed on understanding the physical phenomena occurring in a drying cycle or adopted a system approach disregarding the phenomenological aspects. J.P.Nadeau defines, in his study on drying, three modelling levels evolving successively with increasing geometrical scale [NAD95].

A first microscopic model of knowledge, or “process scale”, analyses the transfer mechanisms within the material and requires the knowledge of many physical properties of the material to be dried. A second scale corresponding to the “product scale” is considered. This macroscopic model offers a complete description of the product behavior in terms of process engineering, usually by means of drying kinetics curves. Finally, a third model is developed and consists of analysing a drier as a system of “megascopic scale” compared to the product dried with no explicit description of the physics of the phenomena.

The interest in a complete model resides in the fine understanding of elementary mechanisms occurring. However, its application is not always conceivable, especially in the case of a work aiming at designing and managing an industrial drier. In fact, the number of independent experiments needed to determine the physical properties of the material as well as the computing time needed to resolve the set of governing equations become very important. Within the framework of this paper, a macroscopic model of urban sludge is developed in order to understand the evolution of water vapor diffusivity in a low temperature conductive drying.

At the beginning of a drying cycle, the urban sludge arises in a pasty form (dry solid content of 20%), and then it acquires a porous structure throughout the drying cycle. Throughout this cycle, the water content evolves causing the modification of the sludge structure: aggregation occurs when primary sludge particles group into aggregates which have a higher density than the bulk density of the sludge as a whole. The random sludge aggregation enables us to refer to them as coarse aggregated porous media with porosity varying relatively to the water content and to the frequency of mixing.

A sludge aggregate consists of an agglomeration of elementary sludge particles showing some cohesion and defining a geometrical form. It is essential to distinguish an aggregate external porosity Φ_e from its internal one Φ_i. Millington and Shearer accounted for internal and external aggregate porosities separately when describing the gas diffusion through an aggregated porous media [MIL71]. Their system was illustrated for idealized spherical aggregates. The internal aggregate porosity contains pores that are much smaller than pores making up the external porosity leading in general to internal pores saturation before external pores wetting. In addition, experiments, led by B. Jones et Al , on the measurement of gas diffusion in soil showed that the internal aggregate porosity contribution to diffusion compared to the external aggregate pore space was minor as described by a dual porosity diffusion model [JON03]. Thus, this work describes the water vapor diffusion through aggregated coarse media where only external porosity is accounted for and internal pores are assumed to be always saturated.

2.3. Literature review of mass transfer in porous aggregated media

Porosity is a key parameter when studying the drying of a porous medium. The pores size influences partly the hygroscopic character of the material. Bulk porosity is defined as the ratio of pore volumes to the apparent volume of the product disregarding the scale of porosity studied.

$$\phi = \frac{V_{bulk}}{V} = 1 - \frac{\rho_{app}}{\rho_{ws}} \qquad [2.1]$$

For instance, at macroscopic scale, one defines external porosity Φ_e as being the ratio of existing porous volume between the aggregates to the total volume of occupied by aggregates, as expressed by equation [2.2]:

$$\phi_e = \frac{V_{e\_bulk}}{V} = 1 - \frac{\rho_{e\_app}}{\rho_{ws}} \qquad [2.2]$$

Sewage sludge structure resembles to great lengths to soil structure where aggregation occurs when primary solid particles group into aggregates themselves denser than the sludge as a whole. When the influence of gravity is negligible and the gaseous phase pressure is uniformly constant (air in void spaces), the major mechanism for gas exchange and transport within a porous medium lacking convective forces is by diffusion through the gaseous and the liquid phases. Along with the set of assumptions stated above, the water vapor migration is best described by a fickian diffusion where coefficients should be identified from a set of laboratory experiments.

The driving force for water vapor diffusion is a gradient of concentrations or partial pressures of water vapor within the sludge air. The dissymmetrical heating of the sample generates a temperature gradient on its thickness. The water vapor transport through the gaseous phase is best described by Fick's first law, expressed in free air as in equation [2.3]:

$$J = -D_0.\nabla C \qquad [2.3]$$

In general, the gas behavior at low density and low pressure is rather accurately described by ideal gases law, since the interaction forces between molecules (Van Der Waals force) become negligible. Therefore, the water vapor transport can be expressed in terms of temperature gradient or vapor partial pressure gradient as in equation [2.4]:

$$J = -D_0 \cdot \rho_0 \cdot \frac{P}{P - P_v} \cdot \nabla \rho_v \qquad [2.4]$$

Macroscopically, one dimensional diffusion of a dilute conservative solute in a saturated porous medium can be described by Fick's second law as stated by [KOH02]:

$$\frac{\partial C}{\partial t} = D_0 \frac{\partial^2 C}{\partial x^2} \qquad [2.5]$$

Yet, the diffusion of gases, vapor and liquids through a solid structure is more complex than in a fluid due to the solid phase heterogeneity and potential interactions with fluids in diffusion. Collis-George et Al introduced a factor of impedance F that represents the ratio of molecular to effective diffusion coefficient and depends mainly on water content and on the diffusion path tortuosity, modifying consequently Fick's second law (Eq. [6]) [COL93]:

$$\frac{\partial C}{\partial t} = D_{eff} \frac{\partial^2 C}{\partial x^2} \qquad [2.6]$$

with $D_{eff} = F.D_0$ [2.7]

In order to improve the predictive capability of transport models, physically based methods including understanding of the control pore continuity and tortuosity on transport are generally required as reported by [COL93].

Conventional gas diffusion measurements in coarse textured and aggregated porous media are severely limited due to hydrostatically induced variations in water content and air filled porosity [JON03]. Consequently, no effective theory describing the diffusion through a porous solid matrix was developed. Researchers established analogies between the heat transfer by conduction and the mass transport by diffusion.

Moreover, semi-empirical correlations were developed describing diffusion in porous media. Extensive efforts have been undertaken to measure the soil gaseous diffusivity in laboratory and correlations were introduced to predict gaseous effective diffusivity, defined as the ratio between the diffusion coefficient in soil, D_{eff} and in free air, D_0. These equations include relatively easy measurable properties such as total porosity, air filled porosity or volumetric air content.

A first generation of models consisted of models based only on ε representing the air filled porosity (volumetric soil air content). The first ε-based model was introduced by Edgar Buckingham, as part of his research on water and gas transport in soils during the period of 1902 and 1906. He suggested that the relative oxygen diffusion coefficient in soil is best described by equation [2.8]. At air saturation, ε is replaced by Φ representing soil total porosity.

$$\frac{D_{eff}}{D_0} = \varepsilon^2 \qquad [2.8]$$

Later on, other classical linear models were proposed by Penmann in 1940 in his work on gas and water movement in soils, Marshall and Millington in 1959 [MOL00a]. The latter two can be considered mechanistically based (cutting and randomly rejoining pores) models.

A second group of models consisted of simple empirical nonlinear models taking into account both air-filled porosity ε and soil total porosity Φ. These predictive models introduce a minor soil type effect through Φ dependant on soil type and management. Among these models are the Millington and Quirk model proposed in 1961 for the evaluation of the porous media permeability, which is almost universally accepted and used in vadoze zone transport and fate models to describe both gas and solute diffusivity.

$$\frac{D_{eff}}{D_0} = \frac{\varepsilon^{10/3}}{\phi^2} \qquad [2.9]$$

Moldrup et Al. combined the Penman and Millington-Quirk approaches into the general PMQ model described by equation [2.10] [MOL97]:

$$\frac{D_{eff}}{D_0} = 0.66\varepsilon\left(\frac{\varepsilon}{\phi}\right)^{\frac{12-m}{3}} \qquad [2.10]$$

They showed that m=3 best describes gas diffusivity in undisturbed soils while m=6 corresponds to gas diffusivity in sieved and repacked soils.

The latest works on gas diffusivity in soils use the soil water retention curve as an additional input to take into account the effect of soil type on gas diffusivities. In the case of undisturbed soils, a soil water characteristic – and thus pore size distribution – dependent model for predicting gas diffusivity was presented by Moldrup et Al. (Eq. [2.11]) by modifying the Burdine-Campbell relative, unsaturated hydraulic conductivity model to describe gas diffusivity in unsaturated soil [MOL00a]. The model requires measurement of the soil water retention curve at a minimum of two different soil water potentials, using a reference measurement at a water potential of –100 cm² H_2O corresponding to a water pressure of 10kPa).

$$\frac{D_{eff}}{D_0} = \left(2\varepsilon_{100}^3 + 0.04\varepsilon_{100}\right)\left(\frac{\varepsilon}{\varepsilon_{100}}\right)^{2+\frac{3}{b}} \qquad [2.11]$$

In equation [2.11], *b* is the Campbell soil water retention parameter in the Burdine-Campbell model [MOL04]. This model is highly recommended by Moldrup et Al for it gives accurate predictions even when extrapolated to nearly dry soil (ε/Φ > 0.9) where the classical soil type independent gas diffusivity models failed to adequately describe gas diffusivity [MOL00b].

On the other hand, far from soil sciences, a linear model predicting gaseous diffusivity in porous media was introduced by Pruess combining the effect of the porous medium tortuosity ζ, total porosity Φ, and in the case of unsaturated media, the gaseous saturation S_g [PRU85].

$$D_{eff} = D_0 \cdot \frac{\phi}{\tau} \cdot S_g \qquad [2.12]$$

Pruess also stated that their effects can be treated as a single combined fitting parameter for gaseous phase since the parameters representing fluid tortuosity and the filled pore space connectivity are generally not derived from theoretical considerations.

These results can not be applied directly to predict the water vapor diffusivity for sludge; indeed, Moldrup et Al. recommended more investigations before applying these formulations to soils having high C concentration which is the case of sludge [MOL01]. In addition, in the case of sludge drying, the frequent mixing leads to porosity change throughout a drying cycle, which is not included in the above. Therefore, an experimental mock up was realized in order to investigate the evolution of Pruess fitting parameter or Collis impedance factor with respect to water content or dry solid content and to external porosity.

2.4. Preliminary Assumptions

In addition to the complexity generated by the coupling of heat and mass transfer in a porous medium, the frequent mixing of the sludge layer throughout the drying process adds difficulty to the problem studied. Therefore, a number of simplifying assumptions is introduced beforehand:

- The sludge is dried over large surfaces which permits to consider a 1 dimensional model and neglect the edge effects;
- A thermodynamic equilibrium exists between present phases at a given point of the porous medium. Thus, one temperature is enough to describe the existing phases.
- The various chemical reactions occurring between phases are not investigated.
- The heat transfer by radiation is not taken into account.
- As previously stated, the paper describes the water vapor diffusion through aggregated coarse media where only external porosity is accounted for. Internal pores are assumed to be always saturated whereas no unbound water exists between aggregates. Hence, our work considers a two-phase porous medium: the gaseous phase constituted by air filling the pores between the aggregates, saturated with water vapor and the porous matrix or the solid phase.

2.5. Laboratory Experimental set-up and Procedure

In order to determine the water vapor diffusivity in sludge, some properties should be known such as sludge apparent and bulk densities, dry solid content, thermal conductivity as well as porosity accordingly with sludge water content. Therefore, an experimental device reproducing sludge conductive heating by the lower face was set up in order to describe properly the various transfers occurring during the drying cycle.

Thermal conductivity measurements prove to be difficult and thermal conductivity models are often used. The most widespread model, when investigating foodstuff and agricultural products dehydration and drying, is based on calculating a weighted average of various phases thermal conductivities as stated in equation [2.13].

$$k_{eff} = k_s(1-\phi) + k_w \phi S_g + k_g \phi \left(1 - S_g\right) \qquad [2.13]$$

In this work, the sludge thermal conductivity is evaluated experimentally: 4 samples were taken at several dry solid contents during a drying cycle and their thermal conductivities were measured at the laboratory L'Ecole des Mines of ALBI using the hot disc method. This method is regarded as being the most reliable apparatus and most widespread for measurement of thermal conductivity [MUJ95]. The sludge thermal conductivity (k_{eff}) showed a decreasing linear trend when plotted against sludge dry solid content (figure 2.1) and can be described by equation [2.14]:

$$k_{eff} = -0.5592.DSC + 0.7116 \quad [2.14]$$

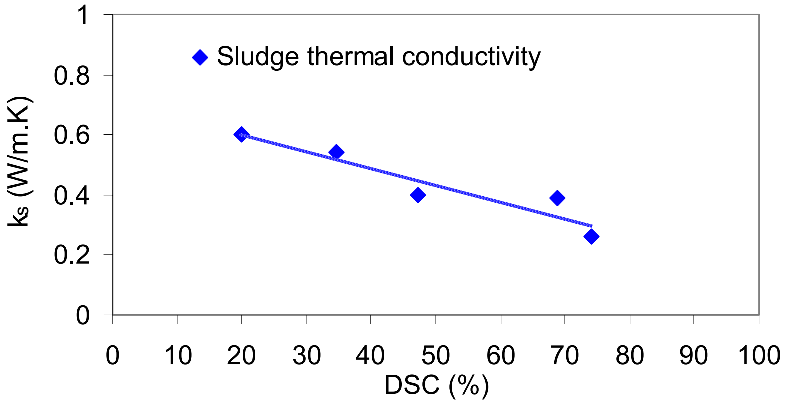

Figure 2. 1Sludge apparent thermal conductivity vs.DSC

Figure 2.2 shows a picture of the test device used to reproduce the drying cycle. The device consists of a highly insulated box containing a heating resistance connected to a power regulator and a wattmeter to measure and control the heating flux applied. The box is placed on a balance to evaluate sludge mass losses and therefore calculate the dry solid content evolution throughout a drying cycle.

The device is equipped with measuring instruments and temperature sensors to calculate the energy balances. Knowing the thermal conductivity for a given dry solid content and imposed flux, one can deduce the diffusive part of the heating flux and thereby the water vapor effective diffusivity.

The box is made of five laminated wooden panels maintained together by wooden cleats. The box 6^{th} face is left open and will be covered with the material which conductivity is to be measured.

Figure 2.2 shows the box 6^{th} face covered with a polystyrene panel to evaluate the share of heat crossing it. In order to minimize the box thermal losses at the time of measurements, the box walls are isolated with extruded polystyrene panels (XPS). The XPS panels are 5 cm thick. Dimensions of limps are presented in table 2.1.

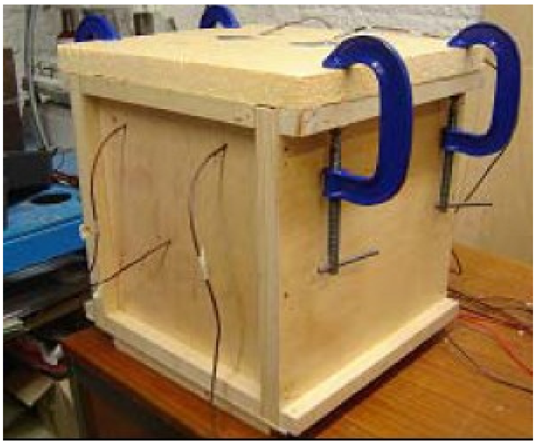

Figure 2. 2 Photo of the experimental set up

Table 2. 1Mock up internal dimensions

Dimensions in mm (length .width .depth)		
	Internal dimensions without polystyrene	Internal dimensions with polystyrene
Box	35 x 39 x 41	25 x 29 x 36

Figure 2.3 shows the electric installation: the power regulator also plays the role of power generator. The wattmeter inlets are the current and the voltage measured at the resistor borders and the reporting is in Watt.

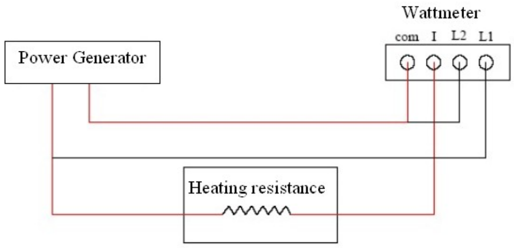

Figure 2. 3 experimental device electrical installation

2.6. Characterization of the test bench losses

Prior to any material characterization, one should identify the box thermal losses with respect to ambient conditions. Once the thermal losses evaluated, it is possible to define the share of flux crossing the material to be characterized. Therefore, a reference case is necessary and the open face is covered with a material of known thermal conductivity such as a XPS panel.

Figure 2.4 illustrates the test assembly scheme. T type thermocouples are fixed on the box faces, the temperature of the room is also reported. The measuring devices are connected to an acquisition module which transmits data to a computer and stores it in an Excel ® sheet format. The used sensors uncertainties are indexed in table 2.2.

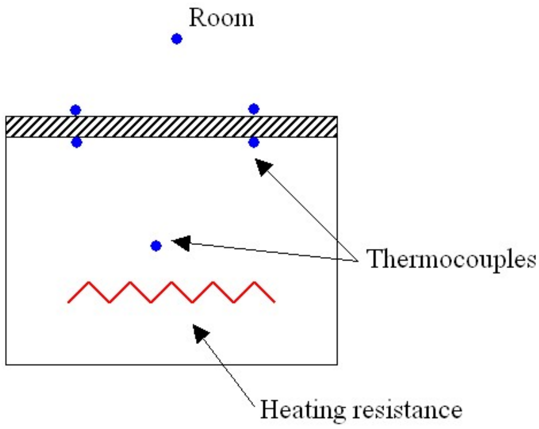

Figure 2. 4 Scheme of the test bench

Table 2. 2 Sensors sensitivity level

Measurement	Sensor Type	Uncertainty
Temperature	T type thermocouple	± 0,2 K
Power	wattmeter	± 0,05 W
Dimensions (length, width)	ruler	± 0,001 m
thickness	ruler	± 0,05 mm

The thermal conductivity of polystyrene being known, the temperature measurement on the XPS panel both sides makes it possible to evaluate the flow crossing it (Eq.[2.15]). The heating power being measured by the wattmeter, thermal losses are deduced by difference. The power regulator allows the testing of various power levels and consequently various temperature gradients between room air and the interior of the box.

$$Q_{poly} = \frac{k_{poly} \cdot S_{poly}}{e_{poly}} \cdot \Delta T_{poly} \qquad [2.15]$$

A series of tests is carried out in order to evaluate the box thermal losses according to the temperature gradient between the box interior and the ambient air. For each test, in steady state conditions, the heating power and the temperatures are recorded. Several power levels are tested. Table 2.3 depicts the values recorded for different parameters.

Table 2. 3 test bench characterization results

test	T room (°C)	T box in (°C)	ΔT box - room	U (V)	I (A)	Power (W)	ΔT polystyrene	Q poly (W)	Box losses (W)
1	17.56	35.25	17.69	19.85	0.29	5.70	13.78	1.26	4.43
2	17.45	49.63	32.18	28.60	0.42	12.01	26.19	2.40	9.61
3	17.42	49.44	32.02	27.96	0.41	11.46	26.57	2.43	9.03
4	17.98	60.04	42.06	33.20	0.49	16.15	35.35	3.24	12.91
5	17.42	54.22	36.80	30.40	0.45	13.53	30.11	2.76	10.77
6	17.24	66.06	48.82	35.30	0.53	18.71	40.55	3.71	15.00

From table 2.3, one can deduce the power lost by the box walls to the environment (column 7) for each imposed power (column 10), as well as the temperature gradient between the box interior and the room air(column 4). Thus, thermal losses can be plotted temperature gradient using a linear function: L_{box}= 0.298 $\Delta T_{box\text{-}room}$ as shown on figure 2.5.

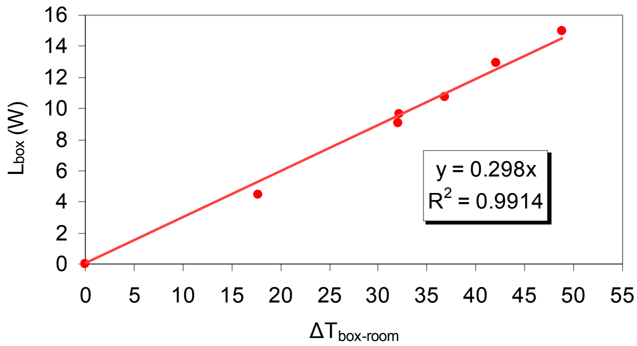

Figure 2. 5 Box thermal losses vs. temperature gradient

Having characterized the thermal losses across the five isolated box faces, the share of flow crossing the 6th face can be deduced using equation [2.16]. The sludge sample is placed on the 6th face in order to study the sludge conductive drying. A plastic container is embedded in the polystyrene panel to contain the pasty sludge sample. The container has the following dimensions: a height of 14 cm, a length of 19 cm and a width of 15.1 cm. Since the thickness of the container material is less than a millimetre, its conductivity can be neglected when calculating the heat flux crossing the sludge sample.

$$Q_s = Q_{cond} - Q_{poly} \quad [2.16]$$

Figure 2.6 shows the experimental device with sludge container embedded. T-type thermocouples are placed at two different points on both sides of the polystyrene panel. Other thermocouples measure the box interior temperature as well as room temperature. In order to quantify the mass losses during a drying cycle, the box is placed on a balance having an error of ± 2%. The data are received by Fieldpoint modules connected to a PC and recorded each 300s.

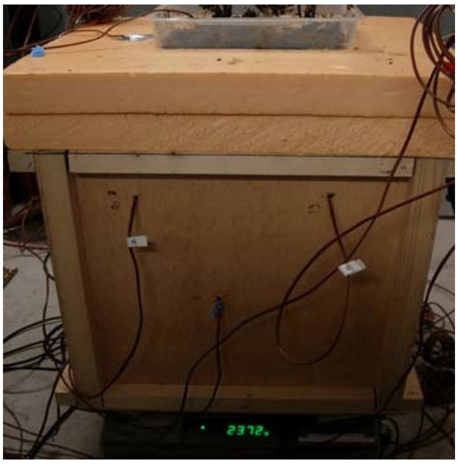

Figure 2. 6 Sludge container embedded in experimental device

2.7. Mathematical model used to interpret the results

Knowing the temperatures of the sample bottom and surface, one can calculate the heat transferred to the sample by conduction. Since the sample is being mixed throughout the test, no thermocouple was placed in the sludge and no intermediate temperature was recorded. The temperature profile remains unknown but is certainly not linear due to coupled heat and mass transfer. The vapor flux is determined by the vapor-pressure gradient across the air filled pores. Thus, for a more accurate evaluation of the impedance factor F, a multilayer four node model is applied to predict the temperature profile within the sludge sample as shown in figure 2.7.

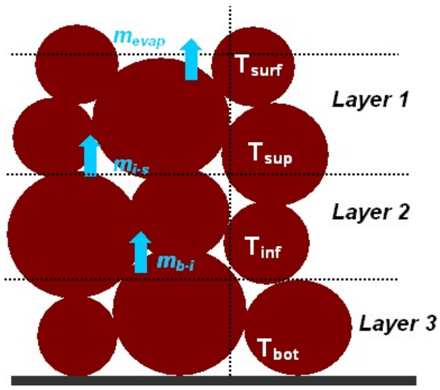

Figure 2. 7 Multilayer four-node-model of sludge

Thus, four sludge temperatures are taken into account when writing the energy balances on the sludge layers. This approach provides additional accuracy in the F factor evaluation and allows to predict a non linear temperature profile across the sludge sample.

The time–independent energy balances are derived for the surface and bottom nodes and also for the two internal nodes as described by equations stated below. Equation [2.17] states the energy balance of the lowest layer (Layer 3).

$$Q_s = \frac{k_s}{e_s} \cdot \left(T_{bot} - T_{inf} \right) + \Delta \dot{m}_{f-i} L_v \qquad [2.17]$$

The vapor diffusion is expressed, according to the Fick's law modified for a porous media in equation [2.18]:

$$\Delta\dot{m}_{f-i} = -D_0\rho_0 F \frac{P_{vsat}\left(T_{bot}\right) - P_{vsat}\left(T_{inf}\right)}{P_{atm}} \tag{2.18}$$

Replacing equation.[2.18] in.[2.17], the energy balance on the lowest layer becomes (equation [2.19])

$$Q_s = -\frac{k_s}{e_s}\cdot\left(T_{bot} - T_{inf}\right) - D_0\rho_0 F \frac{P_{vsat}\left(T_{bot}\right) - P_{vsat}\left(T_{inf}\right)}{P_{atm}}\cdot L_v \tag{2.19}$$

The energy balance of intermediate layer is written in terms of 3 unknowns: T_{inf}, T_{sup} and *F* (equation.[2.20]):

$$\begin{aligned}&\frac{k_s}{e_s}\cdot\left(T_{bot} - T_{inf}\right) + D_0\rho_0 F \frac{P_{vsat}\left(T_{bot}\right) - P_{vsat}\left(T_{inf}\right)}{P_{atm}\cdot e_s}\cdot L_v = \frac{k_s}{e_s}\cdot\left(T_{inf} - T_{sup}\right)\\ &+ D_0\rho_0 F \frac{P_{vsat}\left(T_{inf}\right) - P_{vsat}\left(T_{sup}\right)}{P_{atm}\cdot e_s}\cdot L_v\end{aligned} \tag{2.20}$$

Equation [2.21] states the energy balance for sludge layer 1 :

$$\begin{aligned}&\frac{k_s}{e_s}\cdot\left(T_{sup} - T_{surf}\right) + D_0\rho_0 F \frac{P_{vsat}\left(T_{sup}\right) - P_{vsat}\left(T_{surf}\right)}{P_{atm}\cdot e_s}\cdot L_v = \frac{k_s}{e_s}\cdot\left(T_{inf} - T_{sup}\right)\\ &+ D_0\rho_0 F \frac{P_{vsat}\left(T_{inf}\right) - P_{vsat}\left(T_{sup}\right)}{P_{atm}\cdot e_s}\cdot L_v\end{aligned} \tag{2.21}$$

Since air filled pores are assumed to be vapor saturated, the vapor partial pressure P_v is equal to the saturation water-vapor pressure P_{vsat}, and is calculated using the psychrometric formulas of Hyland and Wexler to predict the saturation water-vapor pressure P_{vsat} at a given air temperature [HYL83].

Dry solid content (DSC) is defined as being the mass ratio of dry matter over wet sludge. The initial mass and DSC being measured, the dry matter mass is calculated. The dry matter mass quantity is assumed constant throughout a drying cycle. Hence the mass loss recorded on a time interval refers to the water loss and allows the calculation of the DSC evolution. The thickness is measured with a ruler at each mixing and linear interpolation is applied to evaluate it between two mixing times. Once solved, the temperature profile can be plotted as a function of the sample thickness and the evolution of the impedance factor F can be determined as a function of DSC.

2.8. Results and discussions

The studied sample is provided by the Saint Arnoult wastewater treatment plant in France after having undergone a preliminary dehydration treatment by centrifugation. A sample was sent to the industrial partner laboratory in order to measure an initial DSC of 15%. Within the scope of this work, a 70% dry solid content is to be obtained at the drying cycle end. Therefore, a cycle is defined as weel as the time needed to reach this final DSC.

The sample initial dimensions are: e_s = 0.11 m (corresponding to the container insulated height), S_c= 0.151x0.19 m², M_{ws}= 3.85 kg. The sample volume is considered representative since it's larger than the porous media aggregates, and smaller than the distance between dissimilar regions. In these series of tests, the mixing is done manually by means of a stem having a width of 0.03 m and a thickness of 0.007 m identical to industrial mixing tooth. A mixing frequency of 1 mixing per 48 hours is first tested.

The whole experimental procedure was carried out for three different fluxes using identical sludge samples having an initial DSC of 15%. The fluxes selected in this work correspond to the typical range that can be reached for low temperature conductive drying (with applied temperatures lower than 60°C). In each series, a different density flux is imposed at the sample bottom in order to analyse the heating flux effect on the sludge rheological behavior throughout the drying process. The first sample was tested with a heating flux of 350 W/m², the second one with 525 W/m² and the third one with 700 W/m².

2.8.1. Sample behavior

Organic sludge has a specific behavior which influences considerably the kinetics of a drying process. This behavior depends on the reached DSC. According to the "Memento Technique de l'eau" [DEG05], the dry solid content evolution can be divided into three zones:

- The first zone includes DSC between 20 and 35% where sludge presents a low viscosity and a pasty coherence.
- The second zone is spread out over dry solid contents from 35 to 60%: sludge presents a strong viscosity and a binding coherence.
- For dry solid contents higher than 60%, a last zone is reached where sludge aggregates of different dimensions and shapes are observed.

In fact, as long as its solid composition does not change, the water content of the sewage sludge influences its rheological behavior [BAUD04]. The water content evolution generates deformation and shrinkage, causing the modification of the porous structure which flashes back on the drying characteristics by decelerating and even by blocking the transfers. This evolution is clearly observed during the tests and photos of the sample behavior throughout a drying cycle are illustrated in figure 2.8:

DSC= 18% DSC = 24% DSC=33% DSC=75%

Figure 2. 8 Deformation and shrinkage due to water content evolution

2.8.2. Estimation of water vapor diffusivity during a bottom conductive heating

The environmental conditions had slight fluctuation during the drying cycles, with the dry bulb temperature oscillating within ±1.5°C around the value of 19.5°C. The evolution of the impedance factor F with respect to DSC for the three fluxes is plotted in figure 2.9. For each applied heating flux, the F factor increases with the DSC and reaches an upper limit for a DSC of 70%. The reached upper value is 1 where the effective diffusivity in porous media coincides with the diffusivity in free air. The increased porosity of sludge does not constitute any additional resistance to water vapor diffusion.

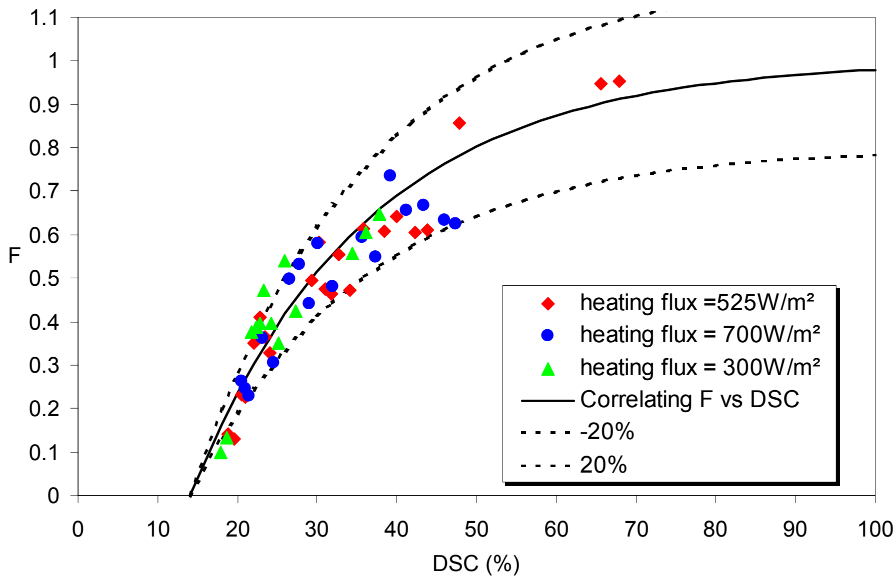

Figure 2. 9 Impedance factor F for different conductive heating fluxes :
a) 350W/m², (b) 525 W/m², (c) 700W/m²

Furthermore, the comparison of the series illustrated in figure 2.9 did not show any remarkable slope variation relative to the applied heating flux. The analytical fitting of F yielded a close approximation of data for the three heating fluxes. Therefore, for a constant mixing frequency and in the range of fluxes investigated in this work, F depends only on the sample dry solid content since the bottom temperature did not exhibit any effect on the rheological behavior of the sewage sludge.

The exponential correlation stated in equation [2.22] was determined from the experimental plot and can be used subsequently in F determination for different DSC throughout the drying cycle.

$$F = \left(1 - exp^{\left(-\left(DSC - DSC_0\right)\cdot k\right)}\right) \qquad [2.22]$$

where DSC_0 stands for the DSC where mass transfer start to be detected and k a correlation factor.

For this series, the initial DSC was 14%, and a good agreement with the measured points was found for a k value of 4.5. Equation [2.23] states the corresponding expression of the impedance factor. The experimental curves agree well with the correlated evolution curve as shown in figure 2.9.

$$F = \left(1 - exp^{\left(-\left(DSC - 0.14\right)\cdot 4.5\right)}\right) \qquad [2.23]$$

The experimental error, estimated from exponential fitting error is around 20% and corresponding error margins are plotted in figure 10. According to this correlation, and for this mixing frequency, no water vapor diffusion occurs before 14%, but as DSC increases, the diffusion phenomenon is enhanced and reaches an upper limit for 70% of DSC corresponding to a F of 1.

2.8.3. Estimation of water vapor diffusivity as a function of mixing frequency

A second set of experiments was carried out with a different mixing frequency to highlight the mixing frequency effect on F evolution and thereby on porosity: one mixing per 24 h is applied in the second set. A heating flux of 525 W/m² is measured and results are compared to those found previously for an identical heating flux but with lower mixing frequency (one mixing per 48 h). The comparison is illustrated in figure 2.10.

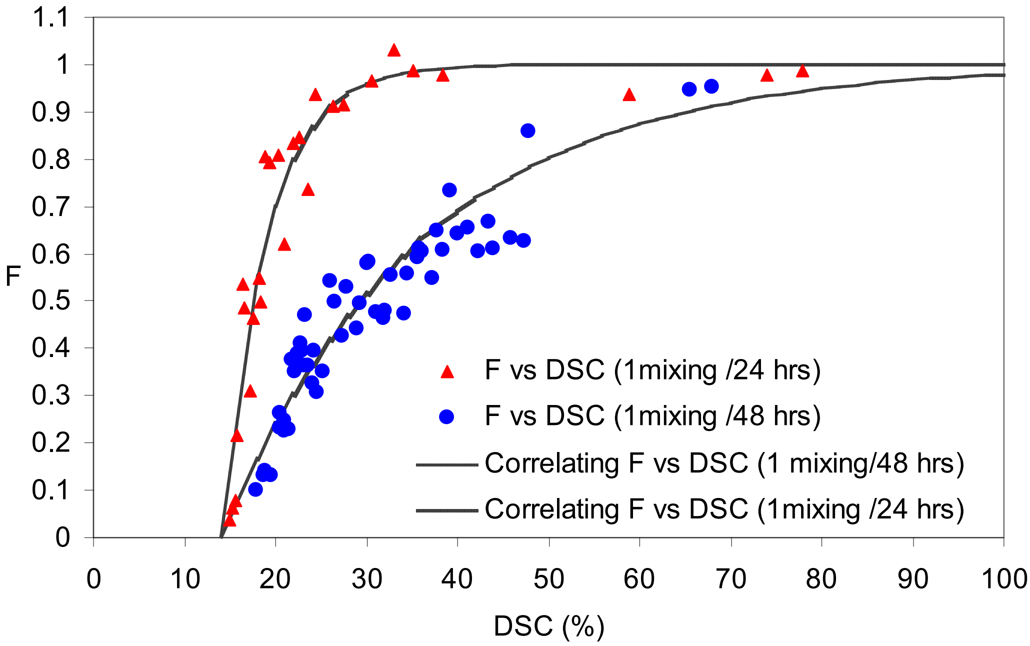

Figure 2. 10 Mixing frequency effect on sludge rheological behaviour for 2 different frequencies

It is quite obvious that increasing the mixing frequency enhances greatly the diffusion within the sludge. A higher slope is observed when plotting the impedance factor evolution according to the DSC. When the mixing frequency is increased, the sludge is textured faster and is more aerated . The expression of F factor becomes as stated in equation [2.24]:

$$F = \left(1 - exp^{\left(-\left(DSC - 0.14\right)\cdot 20\right)}\right) \quad [2.24]$$

For the same starting DSC, the k factor is increased approximately by a factor of 4. Only two frequencies were tested and results demonstrated that the higher frequency yielded to better performances, however this value of mixing frequency is not optimised and it is an interesting perspective to determine an optimal value and find frequencies limitations.

2.8.4. Correlating with porosity (Bulk density Vs apparent density)

The objective of this paragraph is to correlate the diffusivity or the impedance factor with external porosity of aggregates in order to compare the results to previous expressions developed for soils or coarse aggregated porous media. The external porosity can be determined using equation [2.2]. Hence, both bulk and apparent densities must be calculated. Since the sludge container section is known, and the sample weight and thickness are reported at every mixing during the drying cycle, it is possible to evaluate its apparent density with respect to DSC using equation [2.25]:

$$\rho_{e\_app} = \frac{M_{ws}}{e_s S_c} \quad [25]$$

Figure 2.11 shows ρ_{e_app} evolution for different heating fluxes and mixing frequencies. For the same mixing frequency, this evolution was found identical when different heating fluxes were tested and thus can be correlated by a unique linear function as stated in equation [2.26].

$$\rho_{e\_app} = -1000.3DSC + 1242 \quad [2.26]$$

The dashed lines show the error margins corresponding to a maximum deviation of 15% from correlated values. Since all the points fall within this range of error, it is adequate to use a unique apparent density expression for the series tested with different heating fluxes but one mixing frequency.

For a higher mixing frequency, the sludge apparent density followed a decreasing linear trend for an increasing DSC, however a higher slope was found. For this mixing frequency the expression of apparent density is stated in equation [2.27]

$$\rho_{e\_app} = -1167.8DSC + 1209.7 \quad [2.27]$$

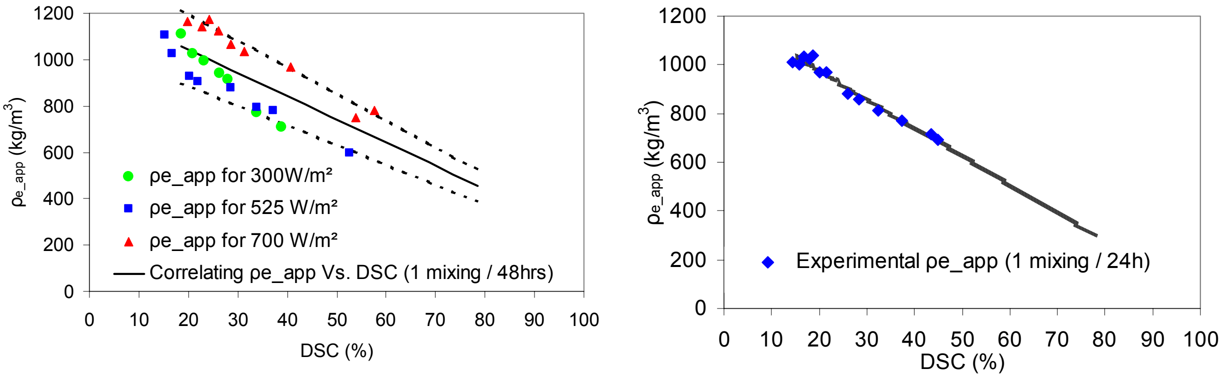

Figure 2. 11 Apparent density evolution for two mixing frequencies:
To the left, one mixing per 48 hours
To the right, one mixing per 24 hours

In order to evaluate the sludge external porosity, the sludge bulk density ρ_s must be known. Since the sludge is made of dry matter and water, the resulting bulk density can be calculated using a binary mixture law stated in equation [28]. However, the dry matter density must be known. A series of tests were carried out in the Ecole Des Mines of Albi using a Helium pycnometer in order to evaluate the missing property. Its was found equal to 1645 kg/m^3.

$$\rho_{ws} = \rho_w \cdot (1 - DSC) + \rho_{DM} \cdot DSC \quad [2.28]$$

Φ_e can be determined for each DSC by replacing apparent and bulk densities with their values in equation [2.2]. Hence, F can be plotted as a function of Φ_e for previously measured points. The evolution of F is showed on figure 2.12 for the tested heat fluxes. This evolution can be correlated with the sludge external porosity using an exponential function similar to the expression developed with respect to DSC previously (equation[2.29]).

$$F = \left(1 - exp^{-k\left(\phi_e - \phi_{e0}\right)}\right) \quad [2.29]$$

Applying equation [2.2] for an initial DSC of 18%, Φ_{e0} is equal to 0.024. For one mixing per 48 hrs, the fitting parameter k is equal to 3.92. Equation [2.28] establishes the correlation relating F to *Φe.* The external continuous lines of figure 2.12 show the margins of error corresponding to 20% maximum deviation.

$$F = \left(1 - exp^{-3.92(\phi_e - 0.024)}\right) \quad [2.29]$$

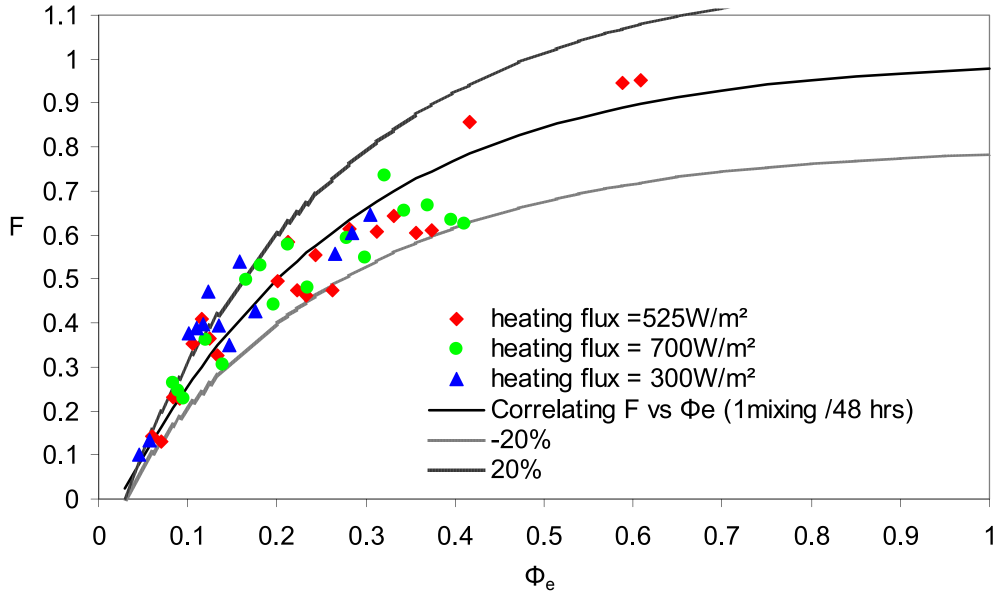

Figure 2. 12 Impedance F factor vs. external porosity for different flux densities

For a higher mixing rate, the correlation of F as a function of porosity is illustrated in equation [2.29] and figure 2.13. Here also, the correlation coefficient k is nearly four times higher than in the case of 1 mixing per 48 hrs. It seems that the mixing rate does not effect only the porosity evolution as a function of the DSC but also the whole rheology of the sludge leading to a higher impedance.

$$F = \left(1 - exp^{-10.2(\phi_e - 0.024)}\right) \quad [2.30]$$

For the values of initial porosity and correlation coefficient k, 76% of the experimental points fall within the range of 20% error from the correlated values for both mixing frequencies. The maximum error was found for low porosity where the impedance factor is low and more accuracy in measurement is needed.

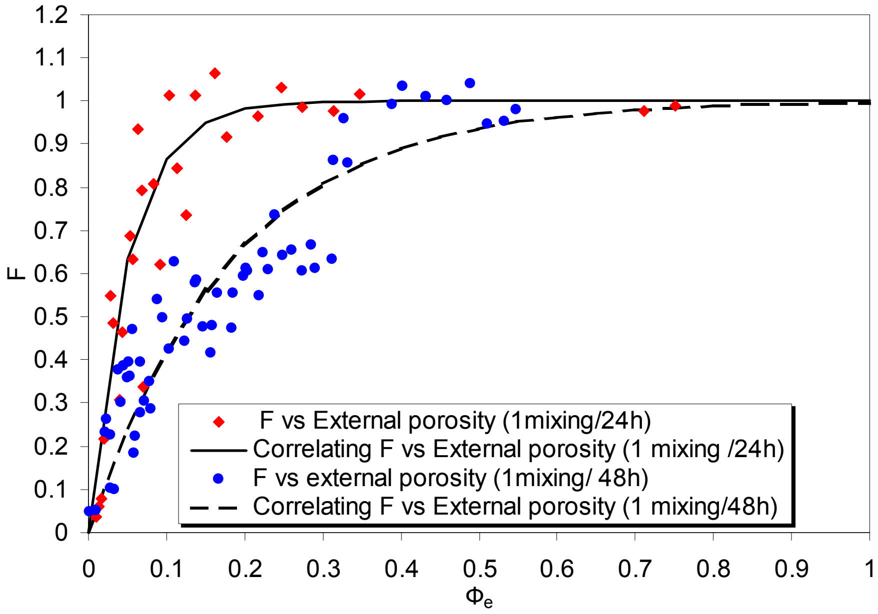

Figure 2. 13 Correlating F against external porosity for 2 mixing frequencies

The exponential behaviour of the measured impedance is compared to the correlations found in literature and stated previously. Figure 2.14 shows a clear difference between the literature correlations and the one found in this work. The difference is mainly related to the mixing applied frequently during the drying cycle even if differences in sludge composition may have an impact.

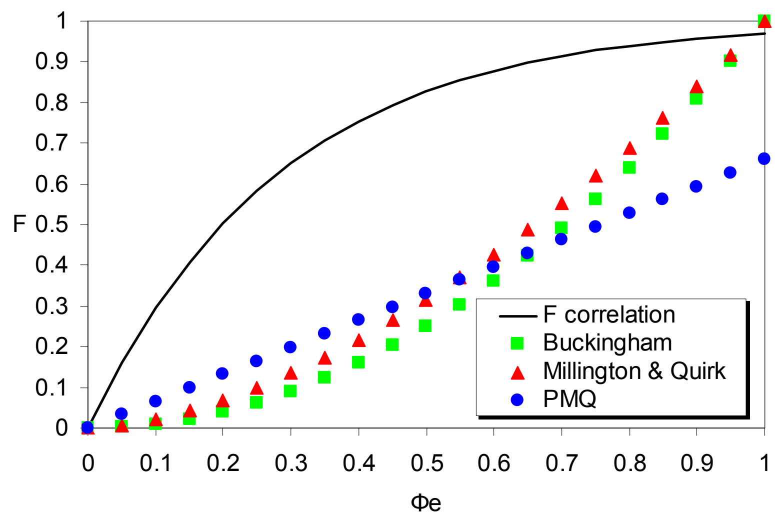

Figure 2. 14 Comparison with soil proposed models

2.8.5. Conclusions

The diffusive mass transfer occurring within the sludge throughout low temperature conductive drying is experimentally investigated and theoretically modelled to predict water vapor effective diffusivity. The investigations are based on a macroscopical one-dimensional diffusive model where only external porosity is accounted for.

An experimental diffusion device was developed for the determination of the diffusion impedance factor F or the effective diffusion coefficient of a group of sewage sludge aggregates during a conductive drying process. The heating fluxes applied showed no remarkable effect of flux density on water vapor diffusivity. On the other hand, experiments highlighted the effect of increased mixing frequency on the enhancement of the water vapor diffusion.

Predictive correlations for impedance factor F as a function of sludge dry solid content (DSC) or porosity, and mixing frequency were established and a good agreement was found between predicted and measured parameters.