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BIOFILTER MEDIA CHARACTERIZATION USING WATER SORPTION ISOTHERMS

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ABSTRACT. Compost material has been used extensively as a gas-phase biofilter media for contaminant gas treatment in recent years. One of the biggest challenges in the use of this type of material is adequate control of compost moisture content and understanding its effect on the biofiltration process. The present work provides a methodology for characterization of biofilter media under low moisture conditions. Results indicated that low levels of equilibrium moisture content (EMC) were obtained for high levels of equilibrium relative humidity (ERH), i.e., 99% ERH produced EMC of approximately 20% (dry basis) at 25 °C. Most bacteria struggle to survive in environments with ERH levels lower than 95%. Compost material from the same source was sieved into four compost particle size (PS) ranges to evaluate its water sorption behavior: 4.76 mm > PS₁ > 3.36 mm > PS₂ > 2.38 mm > PS₃ > 2.00 mm > PS₄ > 1.68 mm. Observed data were tested against isotherm models for their goodness-of-fit. Seven isotherm models were compared: (1) Langmuir; (2) Freundlich; (3) Sips; (4) Brunauer, Emmett, and Teller (BET); (5) BET for n-layers; (6) Guggenheim, Anderson, de Boer (GAB); and (7) Henderson. In comparison with the other models, the Henderson model provided the best fit, as determined by the best combination of regression coefficient standard errors (Δ) and coefficients of determination (r^2) for all four particle size ranges tested (95% confidence interval, C.I., and prediction interval, P.I.). The Henderson model was then used to test for significant differences in isotherms by particle size ranges. The four tested particle size ranges were not significantly different from each other ($p < 0.05$), indicating similar water sorption behavior. Data from all four particle size ranges were pooled and regressed, and the minimum required moisture to maintain ERH at or above 95% was 16.41% \pm 2.68% (dry basis).

Keywords. Biofilter, Compost biofilter, Henderson isotherm, Isotherm, Moisture control, Particle size.

Inadequate moisture control is the most common cause of low biofilter performance (Nicolai and Lefers, 2006). Equilibrium relative humidity (ERH) between the compost material and its surroundings is a determinant parameter in the evaluation of media microbial activity. Most bacteria struggle to survive in environments with ERH levels lower than 95% (USDA, 1995). Isotherms are used to study lower levels of moisture in equilibrium with its surroundings. Bound moisture is predominant in these studies and is represented by moisture held loosely in chemical combination, present as liquid solution within the solid, or trapped in the microstructure of the solid owing to capillary forces (Mujumdar and Menon, 1995).

Water sorption isotherms are applied extensively to evaluate the moisture properties of food. The most accepted equation used to relate ERH and equilibrium moisture content (EMC) in food is the Guggenheim, Anderson, de Boer (GAB) isotherm (Rockland and Stewart, 1998; Bell and Labuza, 2000), which is a modification of the original Brunauer, Emmett, and Teller (BET) isotherm. A large number of isotherm models are available, indicating the complexity of the sorption phenomenon. Thousands of data points have been fitted for several food products, and moisture predictions are reliable for those particular materials. Conversely, the final product of stable compost is highly variable from site to site owing to availability of raw materials, blending, and compost processing. The relatively unknown interactions between compost and water in equilibrium should be investigated in a more systematic approach (Maia et al., 2008; Maia, 2010).

The goal of the present work is to introduce a method for characterization of compost-biofilter media using water sorption isotherms. The method can be used as a tool to assess minimum levels of moisture required for microbial activity. To achieve this goal, the following objectives are presented:

- To develop water sorption isotherms for a compost material sieved into four different particle size ranges.
- To test the generated isotherms for the four particle size ranges against seven isotherm prediction models and to select the model with best goodness-of-fit among them.
- To use the selected isotherm prediction model to compare water sorption behavior of the four studied particle size ranges.

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ISOTHERM MODELS

The relation between EMC (dry basis) and ERH is usually described in terms of water sorption isotherms. Moisture content (dry basis) is defined as follows:

$$MC_{dry-basis} (\%) = \left(\frac{m_w - m_d}{m_d} \right) \times 100 \quad (1)$$

where m_w is the mass of wet material (kg), and m_d is the mass of dry material (kg) determined by placement of the sample in a convective oven at 100 °C for 24 h. Moisture content on a dry basis ($MC_{dry-basis}$) is expressed as a percentage (%).

Several isotherm models have been used to determine water sorption properties of materials. Rockland and Stewart (1981) presented more than 70 different isotherm models formulated under several different assumptions. One of the earliest theoretical models based on classic kinetic theory is the Langmuir isotherm (Langmuir, 1918):

$$EMC = \frac{L_1 \times L_2 \times ERH}{1 + (L_2 \times ERH)} \quad (2)$$

where EMC is the equilibrium moisture content (%), (dry basis), and ERH is the corresponding equilibrium relative humidity (%), which is function of EMC . L_1 is the moisture content (%), (dry basis) to achieve saturation (monolayer coverage), and L_2 is the Langmuir affinity constant, which is temperature dependent. The assumptions for the model include: (1) a uniform and energetically homogeneous surface, (2) weak capillary forces, (3) adsorption energy is constant through all surface sites, and (4) surface sites accommodate one molecule of thickness (monolayer model).

The Freundlich isotherm (Freundlich, 1932) is a fully empirical model applicable for a small spectrum of partial pressures (Do, 1998). This model assumes a continuous increase in water adsorbed as water vapor partial pressure increases:

$$EMC = F_1 \times ERH^{F_2} \quad (3)$$

where F_1 and F_2 are temperature-dependent dimensionless empirical parameters.

The Sips isotherm (Sips, 1948) combines the Langmuir and Freundlich models in an attempt to improve the Freundlich model by incorporating key Langmuir properties. The Langmuir factor in this hybrid equation forces a decrease in the rate of adsorption as the partial pressure increases. The Sips equation is described as follows:

$$EMC = \frac{S_1 \times S_2 \times ERH^{S_3}}{1 + (S_2 \times ERH^{S_3})} \quad (4)$$

where S_1 is the moisture content to achieve saturation (monolayer coverage) (%), (dry basis); S_2 is the Langmuir affinity constant, which is temperature dependent; and S_3 is the parameter that describes the heterogeneous nature of the media (Do, 1998).

The BET equation, formulated by Brunauer et al. (1938), is a semi-theoretical model in which the assumptions include (1) infinite layers of adsorbate molecules are accommodated in the media surface (absorbent); (2) the media surface is energetically homogeneous, as in the Langmuir model; and (3) there is no interaction among adsorbed molecules. The BET equation is described as follows:

$$EMC = \frac{B_1 \times B_2 \times ERH}{(1 - ERH) \times [1 + (B_2 - 1) \times ERH]} \quad (5)$$

where B_1 and B_2 correspond to the monolayer moisture content and BET constant, respectively.

Brunauer et al. (1940) modified their original model (eq. 5) to describe isotherms with different shapes. The BET n -layers equation is described as follows:

$$EMC = \left(\frac{Bnl_1 \cdot Bnl_2 \cdot ERH}{1 - ERH} \right) \cdot \left\{ \frac{1 - [(Bnl_3 - 1) \cdot ERH^{Bnl_3}] + (Bnl_3 \cdot ERH^{Bnl_3+1})}{1 + [(Bnl_2 - 1) \cdot ERH] - (Bnl_2 \cdot ERH^{Bnl_3+1})} \right\} \quad (6)$$

where Bnl_1 and Bnl_2 correspond to the B_1 and B_2 parameters in the original BET equation (eq. 5), respectively. They assume that the number of layers covering the surface is finite and defined by the parameter Bnl_3 , which is the number of finite layers on the media surface. Assumptions for the original BET (infinite layers, eq. 5) apply to the BET with a finite number of layers (eq. 6).

The GAB isotherm model (Anderson, 1946) assumes: (1) a finite number of layers on the media surface, (2) layers above the monolayer contain less adsorption energy than layers closer to surface, and (3) the surface available for adsorption in each subsequent layer is smaller. The GAB equation is described as follows:

$$EMC = \frac{G_1 \cdot G_2 \cdot G_3 \cdot ERH}{(1 - G_3 \cdot ERH) \cdot [1 + G_3 \cdot (G_2 - 1) \cdot ERH]} \quad (7)$$

The GAB equation is similar to the BET equation with the introduction of the parameter G_3 , which is intended to quantify the difference in free enthalpy between molecules in the monolayer and layers above the monolayer (Timmermann, 2003). When comparing equation 7 with equation 5, G_1 is analogous to B_1 , and G_2 is analogous to B_2 .

The Henderson isotherm has been extensively used in agricultural systems to describe the water sorption behavior of biological materials. The advantage of the original Henderson formulation is the small number of model parameters and frequent high correlation with experimental data. The Henderson equation (Henderson, 1952) is described as follows:

$$EMC = \left(\frac{-\ln(1 - ERH)}{H_1 \cdot T} \right)^{(1/H_2)} \quad (8)$$

where T is the temperature (K), H_1 and H_2 are empirical constants of the material, and ERH is given as a decimal and EMC as a percent.

MATERIALS AND METHODS

A methodology was developed to determine compost media water sorption isotherms together with detailed data analysis comparing seven different isotherm models: Langmuir (1918), Freundlich (1932), Sips (1948), BET (Brunauer et al., 1938), BET with limited number of layers (Brunauer et al., 1940), GAB (Anderson, 1946), and Henderson (1952), repre-

sented by equations 2 through 8, respectively. This systematic approach, including the classical models, is expected to provide future researchers with useful information regarding proper selection of isotherms for compost media moisture control.

Isotherms for the four different compost particle size ranges were fitted to acquired moisture data using the seven selected models using nonlinear regression (SigmaPlot, version 10.0; SPSS, 2006). The best goodness-of-fit was used as the criterion to evaluate differences among particle size ranges. The best model was used for predictions based on confidence intervals of the regression and new observations (prediction intervals).

EXPERIMENTAL PROTOCOL

Material Characteristics

Compost material used as biofilter media was collected from a modern compost facility at the University of Kentucky C. Oran Little Research Center (UK LRC) located in Versailles, Kentucky. Active ingredients used as compost material included horse manure, cattle manure, and chicken waste. Bulking agents were added to these organic ingredients to improve aeration. Bulking agents and carbon sources

primarily included woodchips (usually available throughout the year), sawdust, leaves, ground hay, tobacco stalks (seasonal), grass hay, and others. Compost ingredient ratios at the LRC farm were not constant owing to several factors, primarily the seasonal availability of composting materials. Ideally, constant ratios of ingredients would be recommended for consistency of compost characteristics. Nevertheless, sustainable waste management requires use of those biodegradable materials that are available.

Selection of Particle Sizes

Selection of compost particle size was based on use of available compost material at the University of Kentucky Research Farm as part of a sustainable recycling program. The most homogeneous and available particle size range (PS) after removing finer particles (<1.68 mm) was 4.76 mm $>$ PS $>$ 1.68 mm. Compost was sieved (shaker model B, W.S. Tyler, Inc.) and separated into four compost particle size ranges (PS₁, PS₂, PS₃, and PS₄) to evaluate water sorption properties: 4.76 mm $>$ PS₁ $>$ 3.36 mm $>$ PS₂ $>$ 2.38 mm $>$ PS₃ $>$ 2.00 mm $>$ PS₄ $>$ 1.68 mm. The visual observation of the material showed that particle size ranges PS₁ and PS₂ were composed of larger woodchips, whereas PS₃ and PS₄ were composed of smaller woodchips. It was also observed that, even after siev-



Figure 1. Environmental chamber controlled for temperature and relative humidity (Parameter Generation and Control, Black Mountain, N.C.): (a) rear view, (b) front view with door open, (c) input parameter control display, and (d) interior with 16 sieved compost samples.

ing, smaller woodchips (PS₃ and PS₄) presented a greater number of fines attached to them in comparison with PS₁ and PS₂, owing to the fact that smaller particles of the same material have higher surface area per mass unit.

The samples were placed in a controlled environmental chamber (Parameter Generation and Control, Black Mountain, N.C.; fig. 1) with fixed temperature (25 °C) while varying values of relative humidity from 45%, 55%, 65%, 75%, 85%, 95%, and 99%. Initial relative humidity was set at 45% in the environmental chamber, and one day was allowed for the chamber to reach equilibrium. Relative humidity was increased every five days. Compost sample moisture contents were measured inside the environmental chamber by weighing the samples with a digital scale.

STATISTICAL ANALYSIS

Seven sorption isotherm models were fitted to the observations: Langmuir, Freundlich, Sips, BET, BET with limited layers, GAB, and Henderson. Nonlinear regression was performed using the Dynamic Wizard Fit tool in SigmaPlot 10.0 (SPSS, 2006). Constrained optimization of parameters for Langmuir and Sips isotherms was performed assuming that the moisture content to achieve a monolayer could not exceed 100%.

Each isotherm model was evaluated using the coefficient of determination (r^2), regression coefficient standard error (SE), and coefficient of variation (CV) for each regression coefficient. The best model among the seven isotherms was used to compare differences among the four selected particle sizes by using a t-test with the regression standard errors at the 95% level of confidence.

RESULTS AND DISCUSSION

EVALUATION OF THE BEST ISOTHERM MODEL

The BET isotherm did not fit the data and is not presented here. The Henderson and GAB isotherm models yielded higher r^2 values (>0.9) compared with the other five models (Langmuir, Freundlich, Sips, BET, BET n -layers). The GAB model had a slightly higher r^2 value than the Henderson model; however, the standard error of the parameter G_2 was much larger than the parameter mean value (CV >> 1). This suggests an inability of the GAB model to be extrapolated or to predict accurate values of the water monolayer, demonstrating a substantial limitation of the applicability of equation 7 for this type of material. Four of the remaining six isotherms (Langmuir, Sips, BET n -layers, and GAB) had one or more of the regression parameters with large CV (>>1), in which case the parameter is of limited utility in fitting the data. Thus, these four models were rejected.

The remaining two isotherms (Henderson and Freundlich) were compared based on their r^2 values and model regression SE. The Henderson model presented the higher r^2 and lower SE (both model and individual coefficients) for each particle size range and was thus selected as the best model for the isotherm data. Values for model-specific regression parameters along with their associated standard errors are presented in table 1.

Regression plots for the Henderson model were constructed for the selected particle size ranges, as shown in figure 2. The r^2 values were 0.9655 (PS₁), 0.9683 (PS₂), 0.9687 (PS₃), and 0.9171 (PS₄). Particle size range PS₄ showed the poorest goodness-of-fit (larger SE and poorest r^2) compared to the other three particle size ranges.

The observed water sorption differences have important implications with regard to biofilter media moisture

Table 1. Parameter values with standard errors and model coefficient of determination.

Isotherm Models	Parameter ^[a]	Particle Size Range (mm)			
		4.76 < PS ₁ < 3.36	3.36 < PS ₂ < 2.28	2.28 < PS ₃ < 2.00	2.00 < PS ₄ < 1.68
Langmuir	$L_1 \pm \Delta L_1$	100.0 ± 193.8	100.0 ± 193.7	100.0 ± 188	100.0 ± 334.8
	$L_2 \pm \Delta L_2$	0.0019 ± 0.0042	0.0019 ± 0.0041	0.0019 ± 0.0042	0.0016 ± 0.0062
	r^2	0.628	0.620	0.633	0.544
Freundlich	$F_1 \pm \Delta F_1$	0.0014 ± 0.0012	0.0015 ± 0.0012	0.0015 ± 0.0012	0.0001 ± 0.0002
	$F_1 \pm \Delta F_2$	2.072 ± 0.1880	2.066 ± 0.1866	2.075 ± 0.1871	2.603 ± 0.3278
	r^2	0.869	0.870	0.870	0.844
	SE (%)	2.1	2.0	2.1	2.4
Sips	$S_1 \pm \Delta S_1$	100.0 ± 313.4	100.0 ± 314.7	100.0 ± 300.3	100.0 ± 416.9
	$S_2 \pm \Delta S_2$	5.820E-6 ± 6.977E-6	6.009E-6 ± 7.0542E-6	5.753E-6 ± 7.320E-6	4.043E-7 ± 9.822E-7
	$S_3 \pm \Delta S_3$	2.310 ± 1.005	2.302 ± 1.000	2.321 ± 1.003	2.880 ± 1.523
	r^2	0.854	0.855	0.855	0.831
BET n -layers	$Bnl_1 \pm \Delta Bnl_1$	394.4 ± 12191	394.2 ± 12178	411.8 ± 12811	359.4 ± 16755
	$Bnl_2 \pm \Delta Bnl_2$	1.549E-6 ± 4.168E-5	1.590E-6 ± 4.2786E-5	1.513E-6 ± 4.104E-5	1.043E-7 ± 4.228E-6
	$Bnl_3 \pm \Delta Bnl_3$	2.099 ± 0.953	2.093 ± 0.948	2.101 ± 0.948	2.646 ± 1.451
	r^2	0.868	0.868	0.869	0.843
GAB	$G_1 \pm \Delta G_1$	3.479 ± 0.3579	3.465 ± 0.3758	3.583 ± 0.3331	2.688 ± 0.5524
	$G_2 \pm \Delta G_2$	1973212 ± 3.5980E+11	3257686 ± 1.031E+12	3315781 ± 9.7548E+11	1109430 ± 2.6182E+11
	$G_3 \pm \Delta G_3$	0.008 ± 0.0002	0.008 ± 0.0002	0.008 ± 0.0001	0.008 ± 0.0003
	r^2	0.970	0.972	0.974	0.926
Henderson ^[b]	$H_1 \pm \Delta H_1$	1.514E-4 ^a ± 2.47E-5	1.520E-4 ^a ± 2.368E-5	1.455E-4 ^a ± 2.275E-5	3.056E-4 ^a ± 8.151E-5
	$H_2 \pm \Delta H_2$	1.494 ^a ± 0.059	1.495 ^a ± 0.056	1.492 ^a ± 0.06	1.285 ^a ± 0.098
	r^2	0.966	0.968	0.969	0.917
	SE (%)	1.1	1.0	1.0	1.8

^[a] Δ = standard error of the parameter; SE (%) = standard error of the nonlinear regression.

^[b] Different superscripts within a row denote significant difference of the Henderson parameter between particle sizes.

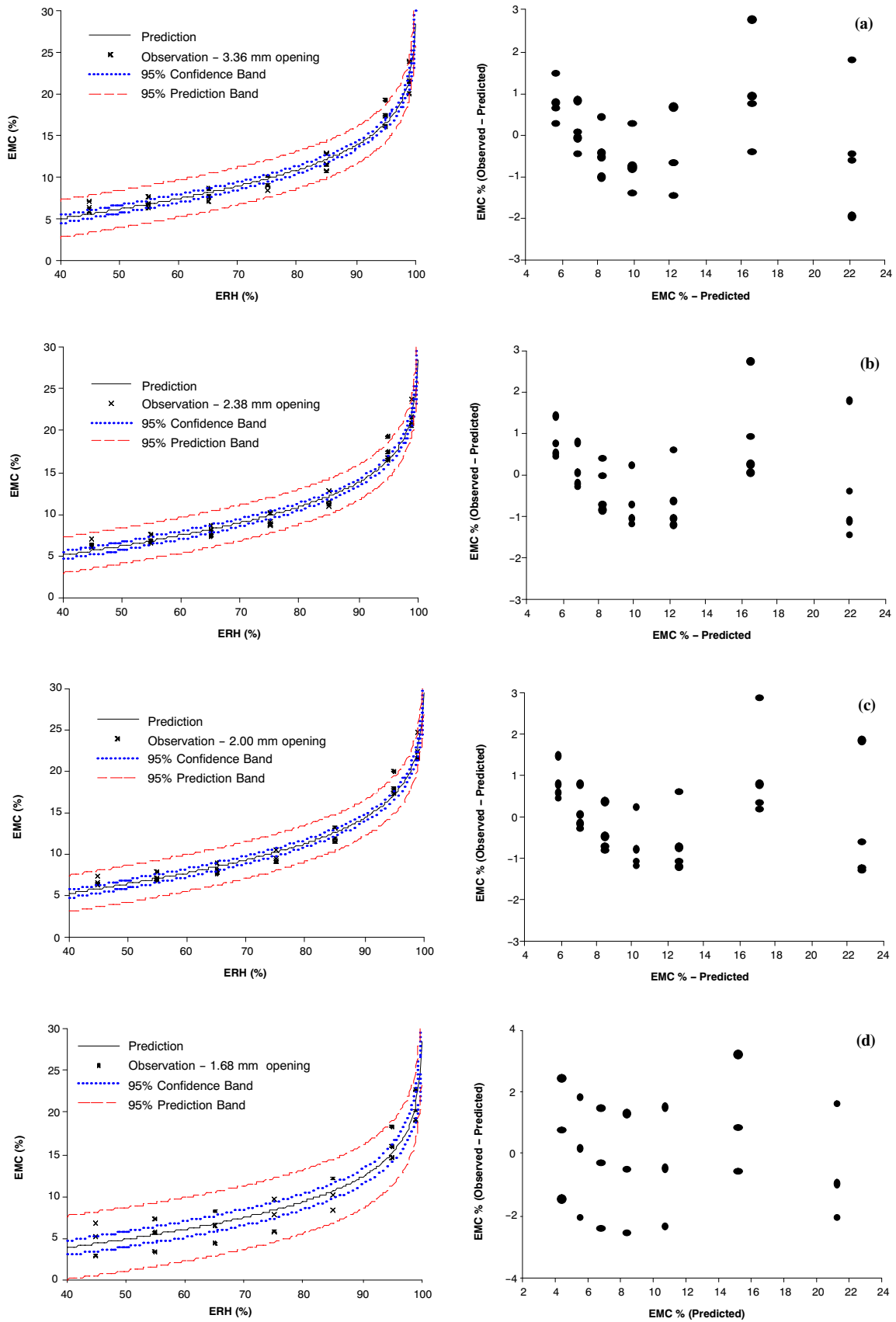


Figure 2. Henderson isotherm regression plots with 95% C.I. for regression and 95% P.I. with residuals: (a) $4.76 \text{ mm} > \text{PS}_1 > 3.36 \text{ mm}$, (b) $3.36 \text{ mm} < \text{PS}_2 < 2.38 \text{ mm}$, (c) $2.38 \text{ mm} < \text{PS}_3 < 2.00 \text{ mm}$, and (d) $2.00 \text{ mm} < \text{PS}_4 < 1.68 \text{ mm}$.

management. To allow $ERH > 95\%$ (friendly growth environment for most microbial organisms), PS_1 , PS_2 , PS_3 , and PS_4 should be kept above the following minimum moisture contents (dry basis): 19.9%, 19.8%, 20.6%, and 17.9%, respectively (dry basis, 25°C). For all particle size ranges, moisture content at or above 20.6% (dry basis) would be required to ensure $ERH > 95\%$.

The simplicity of the two-parameter Henderson model, along with its goodness-of-fit, showed its promise for use as a management tool in the determination of minimum levels of biofilter media moisture required for microbial activity. Moisture levels found in the literature (Nicolai and Lefers, 2006; Nicolai and Janni, 2001) are substantially higher (54% to 186%, dry basis) than the moisture levels found here (approx. 21%, dry basis) to allow bioconversion of ammonia into nitrate and nitrite. More investigation is needed to identify if biofilters in current use are actually operating in the gaseous phase or if the excess of moisture is making them bio-trickling filters or scrubbers (Devanny et al., 1998).

PARTICLE SIZE COMPARISON USING THE HENDERSON MODEL

The practical significance of different model parameters for the particle size ranges is that differing equilibrium moisture contents are required to maintain an equilibrium relative humidity at or above 95% (a reasonable threshold for microbiological activity). Given a regression standard error of about 1.3% of EMC, these four particle size ranges are practically similar at 95% ERH, although the smaller range PS_4 tends to be driest and PS_3 tends to be wettest.

An analysis was conducted to evaluate whether the isotherms differed by particle size range (table 1, superscripts on parameters). The numerical values for nonlinear regression coefficient H_1 was smallest for PS_3 , similar for PS_1 and PS_2 , and largest for PS_4 . For parameter H_2 , PS_4 had the smallest numerical value. No significant differences were found between the four particle size ranges for either of these parameters; thus, the data for all of ranges were pooled, and the nonlinear regression was repeated (fig. 3). The following equation was obtained:

$$EMC = (-19.35 \times \ln(1 - ERH))^{0.6892} \quad (9)$$

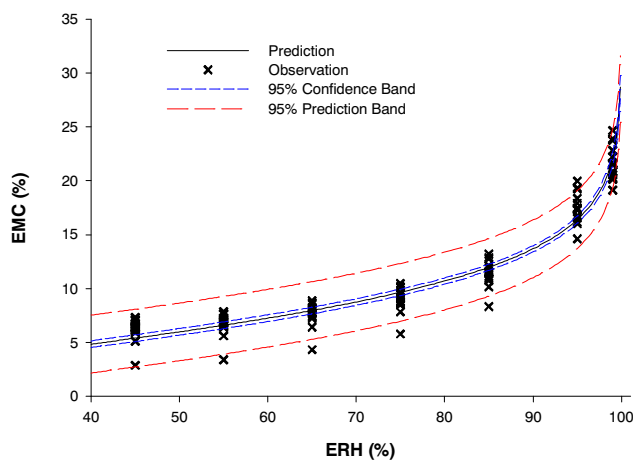


Figure 3. Henderson isotherm regression plots with 95% C.I. for regression and 95% P.I. for particle size ranges PS_1 , PS_2 , PS_3 , and PS_4 combined. Parameter values: $H_1 = 1.733E-4$ ($\pm 1.774E-5$) and $H_2 = 1.451$ (± 0.037), regression SE = 1.34%, $r^2 = 0.945$.

The model applies for $T = 298.15$ K, where ERH is expressed as decimal, $r^2 = 0.945$, model SE = 1.3%, and the SE of H_1 and H_2 are $1.774E-5$ and 0.037, respectively. Equation 9 may be used as a reasonable estimate for composted media with this size of particles.

Using equation 9, for $ERH = 95\%$, the required minimum moisture to allow microbial activity is 16.41% with absolute uncertainties of $\pm 0.34\%$ (95% C.I.) and $\pm 2.68\%$ (95% P.I.). Moisture content above 19.1% can be considered excessive for this media, and anything below about 13.7% is too dry to support microbial activity.

SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

Seven isotherm prediction models were applied to compost material from the same source, separated into four particle size ranges (PS_1 , PS_2 , PS_3 , and PS_4), and compared based on the combination of model r^2 and SE of the regression parameters. The Henderson equation was chosen and used as a tool to compare water sorption differences among particle sizes. The following conclusions were drawn from this work:

- The Henderson equation presented the best combination of SE and r^2 in comparison with the other tested models and with regard to the statistical analysis performed on the collected experimental data.
- There were no significant differences among particle size ranges based on a statistical comparison of the Henderson equation regression coefficients.
- From the combined pooled parameters for all particle size ranges, to provide $ERH > 95\%$ (friendly growth environment for most microbial organisms), EMC of a mixture of this media must be at or above 16.41% $\pm 2.68\%$ (at 25°C).
- The optimum media moisture content to support microbial activity in gas-phase biofilters is strongly dependent upon the type of the media used; thus, recommendations for optimum media moisture content should be linked to the sorption isotherm behavior of each individual material used as biofilter media.
- Different candidate materials for gas-phase biofilters may exhibit differing water sorption isotherms, and thus provide opportunities for testing the applicability of the presented method as a biofilter media management tool. The use of the method with different materials used in gas-phase biofilters is recommended as future work.

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