Modeling biosolids drying through a laminated hydrophobic membrane

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Abstract

The adaptation of the membrane distillation process as a low-cost and sustainable approach to biosolids drying and stabilization is investigated, which may have application in container-based sanitation systems proposed in low-income urban regions. Three-layer laminated, breathable, hydrophobic membranes enclose the biosolids, facilitating drying but preventing transport of contaminants. The membranes used in this process are non-wetting with pore spaces that only allow vapor transport. Water vapor can be expelled due to a moderate vapor pressure gradient. Other constituents, including both particulate and dissolved matter are retained. The permeate, therefore, is expected to be of high purity and pathogen-free. This study presents experimental results showing usable rates of moisture transfer through the laminated hydrophobic membranes with temperature gradients, $\Delta T = -2^{\circ}\text{C}$, corresponding to the condition that biosolids do not receive external heating in which laminate-enclosed biosolids are two degrees Celsius cooler than outside, as well as conditions that samples are 2°C and 10°C warmer than the ambient temperature ($\Delta T = 2$ and 10°C , respectively). The conditions result in reduction in the moisture content of the laminate-enclosed biosolids from about 97% to 12-30% and the permeate is observed to be free of fecal coliforms, indicator organisms for pathogens. The initial constant-rate drying period is described well with a stagnant film model that accounts for different temperature gradients, laminate surface area, and ambient relative humidity. The proposed model may be used to assess the feasibility of incorporating laminated hydrophobic membranes to enhance biosolids drying in container-based sanitation systems as well as other applications.

Keywords

Biosolids drying; Hydrophobic membrane; Mass transfer; Modeling; Pathogen transport

1. Introduction

The quantity of municipal biosolids produced annually in the United States had increased dramatically, from roughly 4.6 million dry tons in 1972 (Bastian (1997)) to 8 million dry tons in 2010 (Arlabosse et al. (2012)). In China over 9.2 million tons of dry biosolids were produced from wastewater treatment plants in 2009 (Deng et al. (2009)). Since most biosolids are originally about 3% solids or 97% water (although this number varies widely), the actual mass of biosolids generated in the United States is over 250 million tons per year. A major challenge of biowaste management is efficient removal of water from biosolids before disposal. Its moisture should be reduced to decrease transportation and management costs for land application, composting, landfilling, or incineration (Tao, Peng and Lee (2005)).

Handling wastewater biosolids accounts for up to 50% of total operating costs for wastewater treatment plants (Spinosa et al. (2011)). In managing wastewater biosolids, the water content may be reduced first mechanically, e.g., centrifugation or filtration, and then by drying to below 5% moisture content (Bennamoun (2012)). At this low water content, the mass and volume of biosolids is reduced considerably, thus decreasing costs for handling, transport, and storage of the final product (Bennamoun, Arlabosse and Léonard (2013); Bennamoun (2012)). Thermal drying is frequently used to remove the moisture from biosolids and is utilized as a final pretreatment step before land application, composting or incineration (Tao, Peng and Lee (2005); Bennamoun (2012)).

One limitation of centrifugation or filtration is that water separated from biosolids during dewatering requires costly conditioning agents and energy intensive filtration or centrifugation processes. In addition, there is the additional cost and effort in re-treating highly concentrated

supernatants, filtrates, and centrates generated in the sludge separation processes (Ahuja (2014); Marzooghi (2013)).

In some applications, biosolids dewatering/drying may be needed where there are heightened concerns for environmental contamination and human exposure. For example, in dense urban areas in developing countries, traditional waterborne sewerage does not exist and is too costly for construction (Russel et al. (2015); Tilmans et al. (2015)). Instead, on-site sanitation practices such as toilets with septic tanks or pit latrines are frequently used in low-income households in urban areas (Tilmans et al. (2015)). Because of narrow, irregular streets, use of suction trucks to empty and remove septic or latrine pit sewerage may not be possible, and instead emptying takes place manually with the concomitant exposure of fecal contaminants to both laborers and community members (Russel et al. (2015)). Any biosolids dewatering/drying takes place away from initial waste storage in septic tanks or latrine pits.

Recently, container-based sanitation (CBS) systems have been proposed as a way to mitigate many of the challenges for biosolids disposal in low-income urban regions (Tilmans et al. (2015)). CBS systems include a toilet that collects fecal matter and urine, sometimes separately, which are then stored in removable containers that minimize human exposure, odor, and insects. At weekly or twice weekly intervals, the containers are sealed and transported safely for disposal or composting. CBS systems utilize household toilets that permit users convenient 24-h access not easily achieved in community-based public toilets. In a recent pilot-scale study in Haiti, CBS systems resulted in a 3.5-fold decrease in the proportion of feces that were unmanaged, nearly eliminating the reported use of open defecation (Tilmans et al. (2015)). CBS systems were also popular, with 71% of users electing to continue the CBS system as paying subscribers at the completion of the free testing period (Russel et al. (2015)). However, the pilot-scale service was

approximately two to three times more costly than large-scale conventional waterborne sewerage, with most of the CBS system cost associated with labor (Russel et al. (2015)). Since water is the largest component of fecal biosolids, technologies that allow biosolids to dry during toilet usage might reduce the mass of biosolids for transport, thus decreasing the frequency and cost of toilet emptying. Or, biosolids drying in a temporary storage/treatment facility after removal from the CBS system might reduce biosolids disposal costs. In both cases, biosolids drying must limit human exposure of pathogens while ensuring sufficiently rapid drying.

In this work, we evaluate the potential application of a breathable, hydrophobic membrane to allow rapid biosolids drying while limiting transport of pathogens, where fecal coliforms were used as indicator organisms. This hydrophobic membrane may be an integral component of a container-based toilet, allowing for fecal biosolids drying at the point of collection, or an important component of a biosolids storage/treatment facility in a CBS system. The fundamental principal of operation rests upon the hydrophobic property of the membrane, causing it to retain liquid and the contaminants dissolved within biosolids, but allowing water vapor to leave. The system is analogous to membrane distillation (MD) (Khayet and Matsuura (2011); Curcio and Drioli (2005); Lawson and Lloyd (1997)). Unlike conventional membrane separation processes that are driven by hydrostatic pressure, in MD a vapor pressure gradient drives the water vapor through the pores, from higher to lower vapor pressure. This vapor pressure gradient is typically provided by a temperature difference between the feed and the permeate sides of the membrane (Khayet and Matsuura (2011); Curcio and Drioli (2005)). Even a small temperature difference can create this gradient in vapor pressure and the consequent water vapor transport. The MD process has been shown to be fairly robust in a variety of applications, including desalination of process waters (Godino et al. (1997); Gryta (2002a); Gryta (2002b); Curcio and Drioli (2005);

Mariah et al. (2006)) and concentration of aqueous solutions of juice or nonvolatile organics (Calabro, Jiao and Drioli (1994); Khayet, Velázquez and Mengual (2004)).

The objective of this study is to evaluate the feasibility of biosolids drying while enclosed in a breathable hydrophobic membrane. For this purpose, a laminated hydrophobic membrane is evaluated. One side of the membrane is glued to a fabric that is in direct contact with biosolids, and the other side is attached to a similar fabric in contact with stagnant water or air. The fabrics protect the hydrophobic membrane from abrasion and accidental puncture. Knudsen and ordinary molecular diffusion likely dominant transport of water vapor across the membrane, while molecular diffusion is expected to control transport through the fabrics. Important questions addressed in this study are (1) Can biosolids drying reduce moisture content from near 95-98% to less than 20% while enclosed in laminated hydrophobic membranes? (2) Can the drying process be readily modeled accounting for surface area and environmental conditions (i.e., temperature and relative humidity)? (3) And is the barrier created by the laminated membrane sufficient to hinder significant transport of fecal coliforms? In addition to these questions, the influence of air movement on the atmospheric side of the laminated membrane and the buildup of solids on the laminated membrane on biosolids drying are also examined. This work is the first step in the evaluation of breathable laminated hydrophobic membranes for enhancing biosolids drying in applications where conventional dewatering/drying technologies may not be feasible, e.g. container-based toilets in low income areas of less developed countries.

2. Drying model

2.1. Overview of drying process

To model moisture loss from biosolids through a laminated hydrophobic membrane, resistance to water vapor movement through the laminate must be described. Biosolids drying takes place in two distinct phases (Ramalho (2012); Chen, Lock Yue and Mujumdar (2002); Benali and Kudra (2002)). The initial phase is a constant-rate drying period where evaporation of free water occurs. Free water is water in biosolids that is not bound to the particles (Kopp and Dichtl (2001); Chen, Lock Yue and Mujumdar (2002)). During the constant-rate period, biosolids surfaces are wet and the rate of evaporation is independent of biosolids properties, e.g., moisture content, and drying rate is limited by mass transfer through the laminate. The drying rate will remain constant if temperature and relative humidity are constant and free water is present at the biosolids-gas phase interface (Tsang and Vesilind (1990); Vesilind (1994); Chen, Lock Yue and Mujumdar (2002); Benali and Kudra (2002); Deng et al. (2011)). Once the biosolids moisture content decreases below a critical level, further evaporation occurs at a falling-rate, as free water is not present at the biosolids-gas phase interface and resistance to moisture transfer within biosolids is important (Ramalho (2012); Rich (1961); Bennamoun (2012); Deng et al. (2011); Lowe (1995)). This critical moisture content marks the transition from the constant- rate to falling-rate period. During the falling-rate phase, the moisture content of biosolids changes nonlinearly with time (Ramalho (2012); Rich (1961); Bennamoun (2012); Deng et al. (2011); Lowe (1995)). This falling rate period may be subdivided further into regions where water is removed from interstices between sludge particles (interstitial water), from sludge surfaces (surface water), and finally the short interval where bound water is removed (Bennamoun (2012)).

A schematic of biosolids drying in a laminated hydrophobic membrane is shown in Fig. 1. The hydrophobic membrane is sandwiched between two woven fabrics that protect the

membrane. When water (component A) evaporates from biosolids into gas (component B), water vapor passes through three layers at the biosolids-laminate interface. The first layer is an air film within the hydrophobic fabric in contact with both biosolids and the membrane (film I). The second layer is the air that fills the membrane pores (film II), while air on the gas-side of the membrane filling the outer fabric forms the third layer (film III). Depending on the wettability and pore size of the fabric containing film I and the pressure of water in wet biosolids, liquid water from the biosolids may penetrate pores of the fabric, thus reducing the thickness of film I or eliminating it. In this work, the fabric was hydrophobic and water pressures were never high enough to penetrate the fabric.

If free water is present and in contact with the fabric on the biosolids side, any resistance to water transfer within the biosolids will not affect biosolids drying. This is the situation with initially wet biosolids positioned against the fabric. The relative humidity (RH) will remain 100% at the interface between film I and the biosolids. As drying progresses, the biosolids moisture content decreases below a critical value, such that free water is no longer in contact with the fabric. Until this critical biosolids moisture content is reached, though, biosolids drying is limited by diffusion through films I-III. The model that follows describes this initial drying stage: water vapor diffusion through the three films is the limiting process, and transport is modeled as through a single film, whose properties are a composite of films I-III.

2.2. *Mathematical model*

Mass flux of water vapor across the laminated hydrophobic membrane can be described by (Alkudhiri, Darwish and Hilal (2012)).

$$J_A = C_l \Delta p_A \quad (1)$$

where J_A is the molar flux of component A (water vapor) (moles/m²/s), C_l is a laminate coefficient that describes mass transfer resistance (moles/m²/Pa/s) through the three air films, and Δp_A is the difference in water vapor pressure between the biosolid-side and the gas-side of the laminate (Pa). The laminate coefficient is a function of fabric and membrane properties, most importantly pore size and thickness. For steady-state mass transfer through the laminated membrane, C_l can be expressed as

$$C_l = \frac{\delta_l}{\frac{\delta_{if}}{C_{if}} + \frac{\delta_m}{C_m} + \frac{\delta_{of}}{C_{of}}} \quad (2)$$

where δ is the layer thickness (m), C is a resistance coefficient (moles/m²/Pa/s), and subscripts l , if , of , and m indicate laminate, inner fabric in contact with biosolids, outer fabric in contact with air, and membrane, respectively. Thicknesses of the laminate and fabrics can be readily measured. Estimating the resistance coefficients for each material is more difficult. For water vapor transport through the fabrics, the resistance coefficient typically describes molecular diffusion alone, if pore diameters are greater than ≈ 100 times the mean free path of water vapor in air or 0.14 μm at 20 °C and 1 atm (Alkhudhiri, Darwish and Hilal (2012)). For transport through the hydrophobic membrane where pore sizes may be sub-micron, both molecular diffusion and Knudsen diffusion may be important (Alkhudhiri, Darwish and Hilal (2012)).

To model water vapor flux through a laminated hydrophobic membrane covering biosolids, it is useful to assess the importance of Knudsen and molecular diffusion. In this study, the GORE Wrap Cover Laminate (W.L. Gore & Associates, Inc., DE, USA) was selected for biosolids testing. Using representative pore sizes and measured thicknesses for the inner and outer fabrics and membrane for the GORE Wrap Cover Laminate reported in Table 1, the contributions of Knudsen diffusion to C_{if} , C_{of} , and C_m are estimated (Khayet et al. (2004)). For the GORE Wrap

Cover Laminate, C_l is estimated following this approach and molecular diffusion is predicted to dominate. The error in C_l is less than 4% if Knudsen diffusion is neglected. For this laminate, mass transfer resistance through the relative thick inner and outer fabrics dominates the system. Details of this analysis are found in Supplementary Material (SM).

For laminated hydrophobic membranes where molecular diffusion dominates transport, steady-state diffusion of water through stagnant air filling the laminate can be described with the following equation (Bird, Stewart and Lightfoot (2007))

$$J_A = \frac{P}{R(T_{avg} + 273.15)} \frac{D_{eff}}{\delta_l} \ln \left(\frac{P - p_{A1}}{P - p_{A2}} \right) \quad (3)$$

where P is the average total gas pressure in the films and is often equal to atmospheric pressure (Pa), R is the ideal gas constant, T_{avg} is the average temperature across the films ($^{\circ}\text{C}$), and p_{A1} and p_{A2} are the water vapor pressures on the biosolids and air side of the laminate, respectively (Pa). D_{eff} is an effective diffusion coefficient (m^2/s) describing water vapor diffusion through the laminate and can be expressed as $D_{eff} = D_{AB} \varepsilon / \tau$ (Poling et al. (2001)), where D_{AB} is the diffusion coefficient of component A (water vapor) in component B (air), and ε and τ are the dimensionless porosity and tortuosity of the laminate, respectively. D_{AB} is a function of temperature and for the diffusion of water vapor in air can be estimated with the Fuller equation

$$D_{AB} = (2.23 \times 10^{-5}) \left[(T_{avg} + 273.15) / 273.15 \right]^{1.75} \quad (\text{Gibson (2000)}).$$

Equation (3) has been used to describe the diffusive flux of water through membranes in MD (Lawson and Lloyd (1997)). As long as biosolids in contact with the hydrophobic fabric contain free water, $RH = 100\%$ at the laminate-biosolids interface and the vapor pressure at this interface is at the saturated state and

can be estimated with Antoine's equation $p_{A1} = \exp[23.238 - 3841 / (T_1 + 228.51)]$, where T_1 is the biosolids temperature ($^{\circ}\text{C}$) (Poling et al. (2001)).

Substituting the expression for the effective diffusion coefficient into equation (3), the model for water vapor flux across a laminate is

$$J_A = \frac{P}{R(T_{avg} + 273.15)} \frac{D_{AB}(T_{avg})}{\lambda} \ln\left(\frac{P - p_{A1}}{P - p_{A2}}\right) \quad (4)$$

where $\lambda = \delta_i \tau / \varepsilon$ (m) is an effective diffusion length that accounts for the thickness, tortuosity, and porosity of the laminate and is a composite measure of the inner fabric, outer fabric, and membrane properties. A helpful and simplifying result from this model is that the overall resistance imposed by the laminate is characterized by λ and is nonspecific to the experimental conditions, allowing general prediction of performance based on laminate area, temperature, and water vapor pressures on either side of the three-component laminate. The utility of equation (4) for describing biosolids drying was tested in the experimental portion of this work.

3. Materials and methods

The GORE Wrap Cover Laminate was used to contain biosolids. The laminate consisted of three separate pieces: an inner microporous expanded polytetrafluorethylene (ePTFE) breathable hydrophobic membrane, with mean pore size of 0.2 μm , and two outer woven hydrophobic fabrics with fabric thread diameter of approximately 200 μm sandwiching the membrane.

In all experiments anaerobically digested biosolids were used. Biosolids samples were collected from the Hay Road Wastewater Treatment Plant in Wilmington, DE (USA). In all

experiments, a virgin, unused laminate was placed in direct contact with biosolids and transport of water vapor and fecal coliforms across the laminate were monitored as the biosolids dried. After selected biosolids drying experiments, the laminate was examined for evidence of fouling.

3.1. *Hydrophobic fabric wettability*

Hydrophobicity of the inner and outer fabric layers was quantified by contact angle measurements using the sessile drop method (Bachmann, Ellies and Hartge (2000); Leelamanie and Karube (2009); Leelamanie, Karube and Yoshida (2010)). A goniometer fitted to a microscope (NRL CA Goniometer model 100-00-155, Ramé-hart, Inc., Mountain Lakes, NJ) measured the contact angle of 7 μL droplets of deionized (DI) water placed on each of the hydrophobic fabric layers. The *RH* and temperature during all measurements were 35% and 22.2°C, respectively.

3.2. *Biosolids drying experiments*

Three laboratory setups were used to measure drying of biosolids covered with the laminated membrane. The experimental setup for Type I experiments is shown in Fig. 2 and consisted of a $10.0 \times 10.0 \times 1.0$ cm (length $\times$ width $\times$ thickness) “envelope” with one side made of the opaque GORE laminate (surface area, $SA = 100. \text{ cm}^2$) and the other a clear impermeable polyethylene plastic sheet. The laminate and polyethylene plastic were sealed with thermal glue along the outer edges, with the exception of a small hole.

At the start of each experiment, 100 mL of wet biosolids were injected through this hole with a syringe and the filling hole quickly sealed with thermal glue. The polyethylene plastic side of the envelope was placed either on a laboratory bench or on a 10×10 cm Thermo Scientific

Cimarec model HP130915Q hot plate (Cincinnati, OH) with $\pm 1^\circ\text{C}$ precision to heat the biosolids to $+2 \pm 0.5$ or $+10 \pm 0.5^\circ\text{C}$ above room temperature. The temperature gradients between the biosolids and outside the membrane were chosen to be -2 ± 0.5 , $+2 \pm 0.5$, and $+10 \pm 0.5$ to evaluate the drying rate process under no heat, low heat, and relatively high heat conditions, respectively.

Ambient temperatures were maintained at $35 \pm 0.5^\circ\text{C}$ for Type I experiments in a climate control room and monitored inside and outside the envelope with an Omega RHXL3SD Handheld Thermometer (Stamford, CT) with $\pm 0.5^\circ\text{C}$ precision. The temperature of biosolids inside the membrane was approximately 2°C below air temperature for unheated samples, or either 2 or 10°C above air temperatures, depending on the hotplate setting. *RH* was measured in the climate control room with a DATAQ Model EL-USB-2 RH meter (Akron, OH) with accuracy of $\pm 3\%$. Periodically, the envelope was weighed with a Mettler Toledo XP4002S Balance (Columbus, Ohio) to within $\pm 0.01\text{ g}$ to record evaporative moisture loss through the laminate until biosolids gravimetric moisture content (water mass/total mass of biosolids) decreased to 12-30%, depending on the experiment. Six Type I experiments were conducted, two at each temperature condition to evaluate reproducibility (see Table S1 in SM for list of all experiments).

Type II and Type III experiments were conducted to evaluate the drying of a larger volume of wet biosolids (300 cm^3) or DI water through the laminate and the influence of external mass transfer resistance on biosolids drying. For Type II and Type III experiments, experiments with DI water were used to assess water evaporation alone through the laminated membrane. Experimental setups for both Type II and Type III experiments are shown in Fig. 3a. A cylindrical polyethylene container 13 cm diameter and 4 cm in height with one end open was

filled with either wet biosolids or DI water. The hydrophobic membrane ($SA = 133 \text{ cm}^2$) was then attached to the open end of the cylinder using thermal glue, and the container rotated 180° so that biosolids or DI water rested against the membrane. The container was suspended in air in a climate controlled room ($35 \pm 0.5^\circ\text{C}$), and if biosolids or DI water were to be heated the hotplate was placed against the polyethylene base as shown in Fig. 3a in the Type II configuration. The configuration was identical for Type III experiments, except a small fan was positioned 4 cm below the membrane to circulate room air against the outer fabric surface. Type III experiments were used to evaluate the significance of mass transfer resistance in air adjacent to the outer fabric surface. Similar to Type I experiments, the temperature of biosolids inside the membrane was approximately 2°C below air temperature for unheated samples, or either 2 or 10°C above air temperatures, depending on the hotplate setting. Air and biosolids temperature, the *RH* of the air, and mass of the biosolids or DI water-filled container were measured periodically during the experiments using the same instruments as for Type I experiments until biosolids gravimetric moisture content dropped below 12-30%, depending on the experiment. For Type I experiments the laminated membrane was on top of biosolids, while for Type II and III experiments the membrane was positioned at the bottom of biosolids to maintain membrane/biosolids contact while drying these larger biosolids samples. Five Type II experiments and two Type III experiments were conducted, with one replicate Type II experiment to evaluate reproducibility (see Table S1 in SM).

3.3. *Transport of Fecal Coliforms*

In principle, only evaporated moisture passes through the hydrophobic membrane and pathogens within biosolids are retained in the laminate-enclosed biosolids as they dry. Type IV

experiments were designed to test this hypothesis. The experimental apparatus is shown in Fig. 3b and consisted of two, 13 cm diameter glass columns (Ace Glass Inc., NJ) separated by the laminate and connected with a horseshoe clamp. The bottom glass column was 13 cm in height with a glass bottom and submerged in a cold water bath maintained at approximately 20 °C. The bottom column was prefilled with pathogen-free DI water at the start of the experiment and mixed with a stir bar to maintain uniform temperature and solute concentrations. The top glass column was filled with approximately 750 mL of biosolids, covered with a plastic cap to inhibit evaporation, and stirred with a mechanical mixer. Heat tape wrapped around the upper column was used to maintain a constant temperature of 22 $\pm$ 0.5 °C or 32 $\pm$ 0.5 °C, which created water vapor pressure gradients across the laminate for two experiments in this apparatus. Omega TJ36-CPSS Thermocouples (Stamford, CT) were positioned adjacent to the laminate in the bottom and top columns to monitor the temperature gradient across the membrane.

At the start of each experiment, water was transported from the biosolids through the laminate to the bottom water-filled column. Biosolids samples from the top column and water samples from the bottom column were collected through time to determine changes in biosolids moisture content and transport of fecal coliforms, indicator organisms for pathogens, across the laminate. Samples from the top column were collected from the well-mixed biosolids, while samples from the bottom column were removed from the water collection tube shown in Fig. 3b. The water level in the water collection tube slowly increased with time due to water vapor transport across the laminate. Fecal coliform concentrations were determined in biosolids and water samples using EPA Method 1680 (USEPA (2002)). Two Type IV experiment were performed.

Membrane fouling

As biosolids dry, non-volatile constituents in biosolids may accumulate on the inner fabric of the laminate, eventually restricting pores and reducing biosolids drying in subsequent usage. Scanning electron microscopy (SEM) images of the inner fabric in contact with biosolids were obtained with a Hitachi S4700 FE-SEM at 20 Kv at the conclusion of Type II biosolids drying experiments. Images were compared with similar images obtained for virgin material.

4. Results and discussion

4.1. *Hydrophobic fabric wettability*

Hydrophobicity of the inner and outer fabric layers was quantified with contact angle measurements using the sessile drop method. Contact angle was invariant with time for measurements < 180 s and was 102.6 ± 5.8 (95% confidence interval) for the inner fabric adjacent to biosolids and 97.8 ± 4.9 for the outer fabric. Thus, in addition to the hydrophobic membrane sandwiched between the two protective fabrics, the fabrics themselves were hydrophobic (contact angle $> 90^\circ$), which is an important assumption required to neglect Knudsen diffusion in the drying model developed above. Unless liquid pressures are sufficiently high to penetrate the hydrophobic fabric, biosolids drying is likely limited by molecular diffusion through the thick fabrics ($\delta_{if} = \delta_{of} = 0.42$ mm) rather than the diffusion through the thinner inner hydrophobic membrane ($\delta_m = 0.03$ mm).

4.2. *Vapor transfer during drying*

Typical drying curves for biosolids samples from Type I experiments are shown in Fig. 4 without heating and for two heating conditions. The steady-state temperature difference between

biosolids and room air for the experiment without heating was approximately $\Delta T = T_1 - T_2 = -2\text{ }^{\circ}\text{C}$, where T_1 and T_2 are temperatures ($^{\circ}\text{C}$) inside and outside of the laminate, respectively. Steady-state temperatures for experiments with heating were either $\Delta T = 2\text{ }^{\circ}\text{C}$ or $10\text{ }^{\circ}\text{C}$. For each experiment the normalized mass (biosolids mass/initial biosolids mass) decreased linearly until normalized masses reached approximately 20%. At the end of these biosolids drying experiments, normalized masses ranged from 3.9 to 4.8%. Biosolids drying was fastest at $\Delta T = 10\text{ }^{\circ}\text{C}$ and slowest at $\Delta T = -2\text{ }^{\circ}\text{C}$, which is consistent with the effect of temperature on molecular diffusion (faster at higher temperature) and saturated water vapor pressure at the laminate-biosolids interface (higher at higher temperature). Results for replicate experiments at the same temperature conditions in Fig. 4 are shown in Fig. S1 (SM) and also show the rate of biosolids drying decreasing at a normalized biosolids mass of approximately 20%.

Drying curves like those shown in Fig. 4 have been reported for many materials, including rice (Igathinathane and K. Chattopadhyay (1999), clay Vasić, Radojević and Grbavčić (2012), and biosolids Léonard et al. (2005)). The general pattern observed in most drying curves is a linear region, which represents a constant drying rate period, followed by a nonlinear segment where drying rate decreases with time. In the constant rate period during experiments in this study, water evaporates rapidly from free liquid surfaces within biosolids and is presumed to maintain a near constant $RH = 100\%$ at the laminate-biosolids interface. In this region, the drying rate is controlled by water vapor diffusion through the laminated hydrophobic membrane (Fig. 1). Laminate area, temperature, and water vapor pressures on either side of the three-component laminate are postulated to control the drying rate during this period for a given laminate.

The nonlinear segment of the drying curve is the period of falling drying rates, where either free water is no longer present within biosolids to maintain $RH = 100\%$, and/or internal mass

transfer resistance within the biosolids limits the transfer of moisture to the hydrophobic fabric-biosolids interface such that $RH = 100\%$ cannot be maintained. As a consequence, even under constant external conditions the rate of drying drops as drying proceeds and the moisture content of biosolids decreases (Vaxelaire et al. (2000)).

If the factors postulated above for controlling drying during the constant rate period are valid, equation (4) describes the constant rate period data for different experimental configurations (Type I, II or III). To test the hypothesis that equation (4) describes biosolid drying for the linear region for Type I and II experiments, a mass balance equation for the contents retained by the membrane enclosure, either biosolids or DI water, is written yielding

$$\tilde{M} = \frac{\varepsilon}{\tau} \tilde{t} + 1 \quad (5)$$

where $\tilde{M}$ and $\tilde{t}$ are dimensionless mass inside the membrane enclosure and dimensionless time, respectively, and are defined by

$$\tilde{M} = \frac{M}{M_i} \quad (6)$$

$$\tilde{t} = \frac{M_w SA P D_{AB}(T_{avg})}{M_i RT_{avg} \delta_l} \ln \left(\frac{P - p_{A1}}{P - p_{A2}} \right) t \quad (7)$$

where M is the mass of contents within the laminated hydrophobic membrane enclosure (g) and M_i this mass at the start of the experiment (g), M_w is the molecular weight of water (g/mole), SA is the surface area of the membrane (m^2), and t is time (s). Using the experimentally measured temperature inside and outside of the laminate, RH of the outside air, and assuming $RH = 100\%$ inside the laminate for biosolids or DI water, water vapor pressures, p_{A1} and p_{A2} , were estimated for each experiment. Atmospheric pressure, P , was assumed throughout the laminate, and T_{avg} was computed using the average of T_1 and T_2 to estimate the molecular diffusivity of water

vapor, D_{AB} , within the laminated hydrophobic membrane. With these measurements and assumptions and using measured δ_l , data shown in Fig. 4 with normalized masses $> 19\%$ were scaled using equations (5-7) and are plotted in Fig. 5. In addition, data from three Type I biosolids drying experiments at $RH = 67\%$ and three Type II biosolids drying experiments all with normalized masses $\geq 19\%$ (constant drying period data) were also scaled and these data are also shown in Fig. 5. The data from these nine experiments collapse to nearly a single line, suggesting that differences in drying associated with temperature gradients, RH or laminate surface area ($SA = 100. \text{ cm}^2$ in Type I and $SA = 133 \text{ cm}^2$ in Type II experiments) can be described by equation (4) and scaled using equations (5-7). Data collected for $\Delta T = -2^\circ\text{C}$ show more variability for $\tilde{M} \leq 0.5$, which might be due to greater variability in temperature in experiments at this temperature where a hot plate was not used to control biosolids temperature.

Analysis of Covariance (ANCOVA) was used to test the hypothesis that the drying rate, the slope ε / τ in equation (5), is independent of ΔT and SA , using data from Type I ($SA = 100. \text{ cm}^2$) and Type II ($SA = 133 \text{ cm}^2$) experiments with biosolids. The principles of ANCOVA analysis are summarized in SM, and Table S2 describes all ANCOVA testing conducted in this study. At a significance level of $\alpha = 0.05$ this hypothesis that drying rate is independent of ΔT and SA was not rejected with P -value of 0.249. That is, when ΔT and SA are examined together scaled biosolid drying rates were independent of ΔT and SA . This result also indicates that the small difference in hydrostatic pressure against the membrane between Type I and Type II experiments, approximately 3 cm of water, had no significant effect on mass transfer since scaling data only with ΔT and SA accounted for differences in drying rates. A separate ANCOVA testing the hypothesis that the slope ε / τ is independent of ΔT resulted in P -value < 0.05 , indicating that the hypothesis is rejected. ANCOVA using only data at $\Delta T = -2^\circ\text{C}$ and $+2^\circ\text{C}$ (P -

value < 0.05), or data at $\Delta T = -2\text{ }^{\circ}\text{C}$ and $+10\text{ }^{\circ}\text{C}$ (P -value < 0.05), or data at $\Delta T = +2\text{ }^{\circ}\text{C}$ and $+10\text{ }^{\circ}\text{C}$ (P -value = 0.248) indicates that the difference in slope ε / τ due to experimental temperature gradients is associated with data collected at $\Delta T = -2\text{ }^{\circ}\text{C}$.

While ANCOVA results indicate that the slope ε / τ for data at $\Delta T = -2\text{ }^{\circ}\text{C}$ is statistically different from slopes and thus drying rates at other temperature gradients, the slopes differ by small amounts. $\varepsilon / \tau = -0.093 \pm 0.003\text{SE}$ (SE = one standard error) at $\Delta T = -2\text{ }^{\circ}\text{C}$, $-0.072 \pm 0.002\text{SE}$ at $\Delta T = +2\text{ }^{\circ}\text{C}$, and $-0.069 \pm 0.002\text{SE}$ at $\Delta T = +10\text{ }^{\circ}\text{C}$. The slightly faster drying rate at $\Delta T = -2\text{ }^{\circ}\text{C}$ is likely due to inaccuracy in either measuring or maintaining $\Delta T = -2\text{ }^{\circ}\text{C}$ for experiments without a hot plate. Fig. 6 shows measured and predicted $\tilde{M}$ using equation (5) for Type I and II biosolids drying experiments, where the experiments differed primarily in ΔT and SA . The data are distinguished by different ΔT 's, and most outliers are associated with $\Delta T = -2\text{ }^{\circ}\text{C}$. The root mean square error of prediction (RMSE) = 0.058 over all 110 data points. Even with inaccuracies for experiments at $\Delta T = -2\text{ }^{\circ}\text{C}$, the model works reasonably well in predicting the experimental drying data.

While equations (5-7) are well-suited for hypothesis testing, equation (4) may also be used to describe drying in the linear portion of biosolid drying data. Best-fit λ were determined using Solver in Microsoft Excel and standard errors estimated using the macro SolverAid (de Levie, 2012). Results are shown in Table 2 for Type I and II experiments with biosolids. The fitted λ for three Type I and one Type II replicate experiments are within one standard error of the estimated parameters, verifying the reproducibility of the experiments. (Differences in RH between replicate experiments were $\sim 1\%$ and assumed negligible.) Similarly, equation (4) was fitted to biosolids drying or DI water evaporation when a fan circulated outside air in the vicinity of the laminated hydrophobic membrane in Type III experiments, which were otherwise identical to

Type II experiments. These experiments were conducted at $RH = 66\%$ and $\Delta T = -2^\circ\text{C}$. The Type III experimental results are shown in Table 3 along with corresponding Type II experiments when a circulation fan was not used. Fitted λ are similar for experiments with or without a circulation fan, but very different for experiments with biosolids versus DI water.

ANCOVA was used to test the hypothesis that the drying rate, the slope ε / τ in equation (5), is independent of the degree of air circulation outside of the membrane and the type of wet material filling the membrane enclosure, either biosolids or DI water. At a significance level of $\alpha = 0.05$ the hypothesis that external air circulation did not affect ε / τ could not be rejected at $P\text{-value} = 0.271$. Thus, resistance to mass transfer was not affected by transport in the room air adjacent to the laminate. However, at the same significance level, ε / τ and thus drying rates were different if biosolids filled the membrane rather than DI water ($P\text{-value} < 0.01$), implying that drying rates were slower and ε / τ larger for biosolids versus DI water. The slower drying rate of biosolids versus DI water may be due to biosolids blocking some of the laminate pores, or a smaller chemical potential for water in biosolids, which is discussed in SM. Additional study is required to determine the cause of slower drying of biosolids versus water when contained in the hydrophobic laminate.

4.3. *Transport of fecal coliforms during drying*

Type IV experiments were conducted in the experimental apparatus shown in Fig. 3b to assess if pathogens cross the laminated hydrophobic membrane during biosolids drying, using fecal coliforms as indicator organisms. Experiments were conducted at two temperature gradients ($\Delta T = 2^\circ\text{C}$ or 10°C). The moisture content of the biosolids decreased with time with drying faster at $\Delta T = 10^\circ\text{C}$ than $\Delta T = 2^\circ\text{C}$, consistent with other experimental results (see

Fig. S2 in SM). However, biosolids drying was slightly nonlinear (Fig. S2) and was attributed to inadequate mixing resulting in nonuniform temperature and moisture in this 750 mL biosolids volume, which was 7.5 and 2.5 times larger than biosolids volumes in Type I and Type II/III experiments, respectively. Fecal coliform concentrations were measured in biosolids and in the distillate during the experiments and results are shown in Fig. S3. Concentrations of fecal coliforms in biosolids decreased three orders of magnitude from the beginning to the end of the experiments, which corresponded to a reduction in moisture content from approximately 97 to 40%. Fecal coliform concentrations in the distillate over the same biosolids drying period were always below the detection limit (0.18 MPN/mL of water). Thus, as expected transport of fecal coliforms across the laminated hydrophobic membrane during biosolids drying was negligible, while significant inactivation occurred for fecal coliforms in biosolids.

4.4. *Membrane fouling*

Biosolids contain organic and inorganic compounds that may form deposits on the laminated membrane, potentially fouling the inner fabric in direct contact with biosolids. Over time such fouling may decrease biosolids drying efficiency. SEM images of the inner fabric of the laminate before and after biosolids drying for a Type II experiment are shown in Fig. S4. While some deposits are visible on fabric exposed to biosolids during drying, there are very few visible deposits in the images, suggesting that the impact of solid deposition on water vapor transport through the laminated membrane is small, at least for this single cycle of biosolids drying. Future work should examine the impact of multiple cycles of biosolids drying on laminate performance.

5. Conclusions

Container-based sanitation (CBS) systems have been proposed as a way to mitigate the challenges associated with biosolids disposal in low-income urban regions of developing countries. A technology that permits biosolids drying during toilet usage might reduce the mass of biosolids for transport, decreasing the frequency and cost of toilet emptying. Alternatively, biosolids drying in temporary storage/treatment facilities after removal from a CBS system may reduce disposal costs. In both situations a breathable hydrophobic membrane that permits biosolids drying but prevents pathogen transport could be beneficial.

Biosolids drying while contained in a three-layer laminated, breathable, hydrophobic membrane was evaluated in this study. Biosolids moisture contents were reduced from 97 to 12-30% with nondetectable transport of fecal coliforms across the laminated hydrophobic membrane. The drying process inactivated fecal coliforms in biosolids sufficiently that dried biosolids met Class A and Class B requirements (USEPA, 2000) for $\Delta T = -2^{\circ}\text{C}$ and $+10^{\circ}\text{C}$ treatments, respectively. Mass transfer was described with a stagnant film model for the initial constant-rate drying period and able to describe time-dependent changes in moisture content for experiments at different temperatures, laminate surface area, and ambient relative humidity. To predict biosolids drying for the constant-rate drying period for other configurations, such as above-ground toilets lined with laminated hydrophobic membranes, the mass transfer model developed here can be used as long as the laminate-dependent effective diffusion length (λ) is known. This effective diffusion length was measured for the GORE Wrap Cover Laminate, but must be measured for other laminated hydrophobic membranes before predicting biosolids drying in such systems. While the mass transfer model developed in this study is only for the constant-rate drying period, in some cases this period may be the most relevant for CBS systems: reductions in moisture and biosolids mass are most rapid and significant in the constant-rate

drying period, which occurs when fresh biosolids are added to toilets or transferred to biosolids storage/treatment facilities. Modeling the entire biosolids drying process will likely require modeling heat transfer as well. Drying is enhanced when biosolids are warmer than ambient air, and laminated hydrophobic membranes help retain heat generated by microbial processes in biosolids or perhaps provided by solar energy.

Anaerobically digested biosolids were used as a surrogate for fecal biosolids drying in this study. Solutes from these biosolids might alter the chemical potential of water differently, potentially modifying the equilibrium water vapor pressure such that drying rates differ even during the constant rate drying period. While we believe such differences in drying rates will be small, future research should test this hypothesis.

This work constitutes an initial investigation of the use of laminated hydrophobic membranes for allowing evaporative biosolids drying while limiting pathogen exposure to humans and transfer to the environment. Future work is needed to evaluate the incorporation of these laminates in toilets or as components of a biosolids storage/treatment facility for enhancing biosolids mass loss before subsequent application. Because laminated hydrophobic membranes permit the transfer of all gas vapors, their implementation must necessarily consider the transport of other constituents in addition to water vapor, most notably odorous compounds. Finally, while SEM images of a used laminate in this study suggests that dried biosolids are unlikely to foul the laminated hydrophobic membrane, future work must substantiate this claim.

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Supplementary Material

An analysis of the importance of Knudsen diffusion for water vapor transport through the Gore Wrap Cover Laminate, details of the Capillary Flow Porometry measurements, biosolids drying data for replicate Type I experiments, biosolids drying fecal coliform data for Type IV experiments, and summaries of all experimental data and ANCOVA analyses are presented in Supplementary Material.

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Table 1. Properties of fabrics and membrane in GORE Wrap Cover Laminate.

Material	Membrane or Fabric Thickness ¹	Pore Diameter
	(mm)	(μm)
Inner fabric	0.42	2 - 20 ²
Membrane	0.03	$0.236 \pm 0.002\text{SD}^3$
Outer fabric	0.42	2 - 20 ²

¹ Measured with micrometer

² Estimated range from SEM images of fabric

³ Mean flow pore size estimated from Capillary Flow Porometry measurements. SD = one standard deviation based on 3 samples.

Table 2. Best-fit effective diffusion length, λ , for Type I and Type II biosolids drying experiments at various temperature gradients in laminated membrane. SE = one estimated standard error.

	<i>RH</i> = 68% Type I		<i>RH</i> = 67% Type I		<i>RH</i> = 68% Type II	
ΔT (°C)	λ (m)	SE	λ (m)	SE	λ (m)	SE
-2±0.5	0.0102	0.0010	0.0104	0.0007	0.0121	0.0007
2±0.5	0.0134	0.0014	0.0120	0.0016	0.0129	0.0011
10±0.5	0.0138	0.0035	0.0111	0.0051	0.0131	0.0023

Table 3. Best-fit effective diffusion length, λ , for biosolids and DI water drying in laminated membrane at $\Delta T = -2$ °C and $RH = 66\%$ in the absence (Type II) or presence (Type III) of circulation fan. SE = one estimated standard error.

Sample	Configuration	λ (m)	SE
Biosolids	Type II	0.0129	0.0012
Biosolids	Type III	0.0120	0.0014
DI Water	Type II	0.0095	0.0009
DI Water	Type III	0.0093	0.0010

Fig. 1. Diffusional moisture transfer from biosolids into air through boundary films. p_{A1} and p_{A2} are water vapor pressures on the biosolids and air side of the laminated membrane, respectively. The air film thicknesses in equation (2) are indicated in the illustration.

Fig. 2. Setup for Type I experiments. a. Filling of biosolids into enclosure with top side laminated hydrophobic membrane and bottom impermeable polyethylene (PE) plastic. b. Heating for selected experiments at either + 2 °C or + 10 °C above ambient conditions.

Fig. 3. a. Setup for Type II and III experiments. Biosolids or DI water filled polyethylene (PE) container capped with laminated membrane. Heating for selected experiments at either + 2 °C or + 10 °C above ambient conditions. b. Setup for Type IV experiments. The bottom column inner diameter and height are 13 cm. Inner diameter and height of the top column are 13 and 21 cm, respectively.

Fig. 4. Biosolids drying curves illustrating percent normalized biosolids weight versus drying time. The percent normalized weight is calculated as the biosolids instantaneous mass divided by the initial weight. The results are associated with three Type I experiments. ΔT is the temperature gradient across the laminate and is obtained as $\Delta T = T_1 - T_2$, where T_1 and T_2 are the temperatures inside and outside the laminate, respectively. $T_2 = 35^{\circ}\text{C}$ and $RH = 67\%$ for all experiments. **Estimated standard errors for data are < 0.1 .**

Fig. 5. Biosolids drying data associated with Type I and II experiments. The plot shows the dimensionless mass (equation 6) versus the dimensionless time (equation 7). ΔT is the temperature gradient across the laminate and is obtained as $\Delta T = T_1 - T_2$, where T_1 and T_2 are the temperatures inside and outside the laminate, respectively. $T_2 = 35^{\circ}\text{C}$ and $RH = 67\%$ or 68% for all experiments. Error bars representing $\pm$ estimated 95% confidence intervals are as so small as not to be visible.

Fig. 6. Plot of predicted dimensionless biosolids mass (equation 5) versus experimentally observed dimensionless biosolids mass. The diagonal line indicates predicted $\hat{M}$ is equal to the observed one and the dashed lines represent one order of magnitude uncertainties. The data are associated with Type I and II configurations, $\Delta T = -2^\circ \text{C}$, $+2^\circ \text{C}$, and $+10^\circ \text{C}$, and RH s 67% or 68%. For all data $T_2 = 35^\circ \text{C}$.