ea engenharia agrícola
Journal of the Brazilian Association of Agricultural Engineering
ISSN: 1809-4430 (on-line)

REVIEW PAPER

HYDROPHOBIC MEMBRANE TECHNOLOGY FOR AMMONIA EXTRACTION FROM WASTEWATERS

Doi:http://dx.doi.org/10.1590/1809-4430-Eng.Agric.v36n2p377-386/2016

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ABSTRACT: Total Ammoniacal Nitrogen - TAN (NH_3 + NH_4^+) in wastewaters cause environmental degradation concerns due to their negative impacts on air, soil and water. Several technologies are available for TAN removal from the wastewaters. One emerging technology is the use of hydrophobic membrane as non-destructive NH_3 extraction. In this paper the authors discuss the uses of gas permeable membrane (GPM) and its physicochemical characteristics that influence gas mass transfer rate, diffusion and recovery mechanisms of NH_3 from liquid sources (e.g. animal wastewater). Several aspects of NH_3 extraction from liquid manure and other TAN generation sources using GPM technology as well as its applicability for NH_3 mitigation from liquid effluents and possible recovery as a nutrient for plant growth are also discussed in this review.

KEYWORDS: ammonia, separation, wastewater, nutrient recovery.

MEMBRANAS HIDROFÓBICAS PARA EXTRAÇÃO DE AMÔNIA DE ÁGUAS RESIDUÁRIAS

RESUMO: A presença de nitrogênio amoniacal total - NAT (NH_3 + NH_4^+) em águas residuárias é motivo de preocupação ambiental devido a seus impactos negativos sobre o ar, solo e água. Várias tecnologias estão disponíveis para remoção de NAT. Uma tecnologia emergente é a utilização de membrana hidrofóbica para extração de NH_3. Neste trabalho, os autores discutem o uso de membranas permeáveis a gases (MPG) e suas características físico-químicas que influenciam a taxa de transferência de massa do gás, bem como a difusão e os mecanismos de recuperação de NH_3 de efluentes (por exemplo, águas residuárias animais). Vários aspectos envolvendo a extração de NH_3 de dejetos líquidos e de outras fontes de geração de NAT, usando a tecnologia MPG, bem como sua aplicabilidade para recuperação de NH_3, com vistas à potencial aplicação como fonte de nitrogênio para uso como fertilizantes também são discutidos nesta revisão.

PALAVRAS-CHAVE: amônia, efluentes, recuperação de nutrientes.

INTRODUCTION

Nutrients such as nitrogen (N) and phosphorus (P) are present at high concentrations in animal manure and processing industries' wastewaters and other industrial waste effluents. The concentration of nutrients in these effluents varies due to their inherent N and P compositions, management or treatment processes (NUNES et al., 2012; FERNANDES et al. 2012; BILOTTA & KUNZ, 2013). Nitrogen in liquid phase may be present in different forms from a much reduced state (-3) e.g., NH_3 to a highly oxidized form (+5) e.g., NO_3^-. The presence or absence of these forms is a function of oxidation reduction potential (ORP) of the effluent and also pH. Under anaerobic conditions at low ORP, the reduced N form is prevalent as NH_4^+ and/or NH_3 (depending on pH), expressed as total ammoniacal nitrogen - TAN (WANG et al., 2013).

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Recebido pelo Conselho Editorial em: 2-7-2014
Aprovado pelo Conselho Editorial em: 30-3-2015

In effluents from a livestock production, N is primarily present in the organic form (e.g., amino acids, proteins, and urea). The degradation of this organic N at low ORP results in NH_4^+/NH_3, also known as N mineralization (KUNZ et al., 2009). Among these processes, a quick and important conversion of organic N to inorganic form occurs in animal waste effluents by urea hydrolysis generating NH_3 due to catalysis by an enzyme called urease (Equation 1)

$$H_2NCONH_2 + H_2O \rightarrow 2\underset{urease}{NH_3} + CO_2 \qquad (1)$$

TAN in effluents must be handled correctly to avoid environmental degradation. When effluents with TAN are disposed into water bodies, the first adverse effect is the dissolved oxygen depletion in the water due to its high nitrogenous oxygen demand for TAN oxidation to NO_3^- (Equation 2 and 3) (KUNZ et al., 2012).

$$NH_4^+ + 1.5O_2 \rightarrow NO_2^- + H_2O + 2H^+ \qquad (2)$$

$$NO_2^- + 0.5O_2 \rightarrow NO_3^- \qquad (3)$$

Oxidized species of N; nitrite (NO_2^-) and NO_3^- also may negatively impact the environment due to the toxicity of these ions as nitrosamines generation and as a methaemoglobinemie. Eutrophication of water bodies is another deleterious effect on the natural environment due to N and other nutrient overload causing algal blooms that can dramatically change the characteristics of water bodies (VIDAL & CAPELO NETO, 2014).

REVIEW

Chemical equilibrium implications of TAN on free ammonia generation

In wastewaters, the chemical equilibrium between NH_3 (free ammonia) and NH_4^+ and the prevalence of ionic or nonionic specie is completely dependent on pH (Figure 1) and temperature (equation 4 to 7), directly impacting N bioavailability and removal processes (WELLINGER et al., 2013).

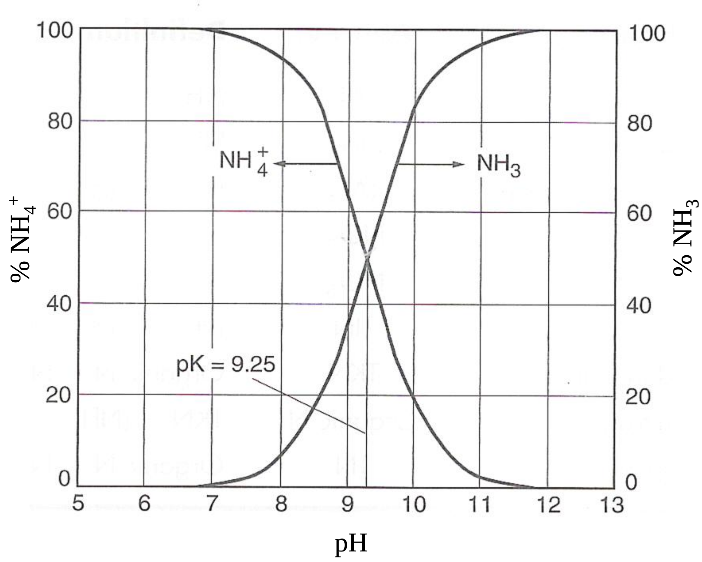

FIGURE 1. Ammonium and ammonia distribution as a function of pH.

In water, without other reactants and impacted by its alkaline characteristic (Table 1) the chemical equilibrium is shifted to the left of [eq. (4)].

$$NH_4^+ + OH^- \underset{k_b}{\overset{k_a}{\rightleftharpoons}} NH_{3(aq)} + H_2O \qquad (4)$$

TABLE 1. Properties of ammonia (NH_3) in water (at 25 ^{0}C)

Property	values
Molecular weight (g mol^{-1})	17.03
Solubility (%)	34
Dissociation constants, kb	1.74×10^{-5}
ka	5.67×10^{-10}
Kinetic diameter (Å)	2.6
Dipolar moment (D)	1.47
Henry's constant, H_{NH3} (mol L^{-1} Pa^{-1})	5.53×10^{-4}

The basic and acidic equilibrium constants, (Kb and Ka), respectively can be described by eqs (5) and (6).

$$K_b = \frac{[NH_4^+][OH^-]}{[NH_3]} \tag{5}$$

$$K_a = \frac{[NH_3][H^+]}{[NH_4^+]} \tag{6}$$

In liquid effluents (e.g. animal wastewater), free ammonia (FA) can be determined using [eq.(7)].

$$FA\ (NH_3, mg/L) = \frac{17}{14} \times \frac{[\text{total ammonia as N}] \times 10^{pH}}{e^{[6344/(273+T(^\circ C))]} + 10^{pH}} \tag{7}$$

Toxicity from free ammonia may reduce the biological activity of microorganisms due to NH_3 permeability into the microbial cellular membrane (SCHNEIDER et al. 1994). This is an important aspect to be considered in biological N treatment processes due to its impact on deammonification processes for biological removal of ammonium from wastewater. Thus, it is important to control pH to avoid the increase of FA to inhibitory concentrations (DE PRA et al., 2012).

Volatilization of FA to the atmosphere can cause environmental pollution and aids in acid rain deposition when combined with air pollutants namely SO_x and NO_x, emitted in the atmosphere from industrial processes and other anthropogenic activities (LEDDA et al., 2013). For liquid effluent, NH_3 can be removed by biological, chemical and physical processes (Figure 2).

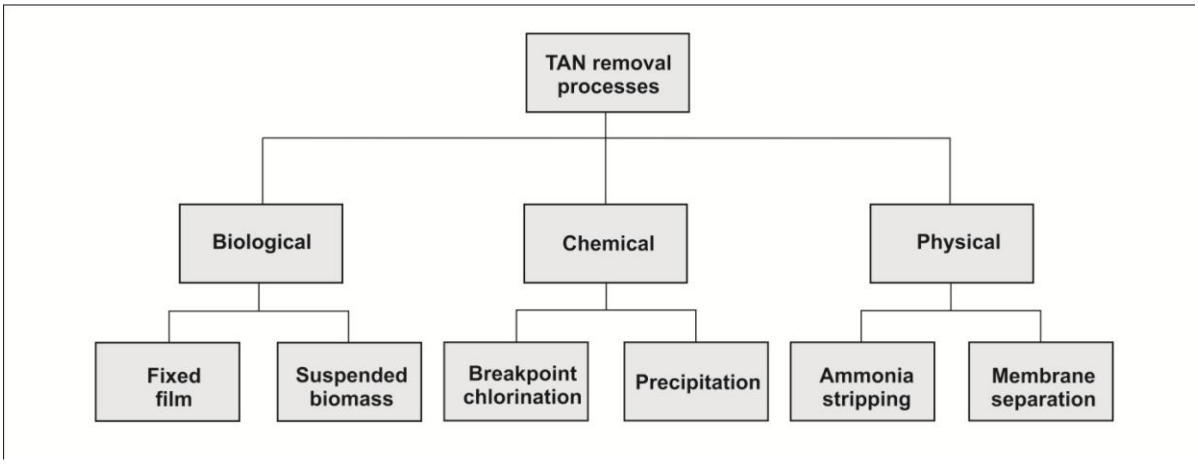

FIGURE 2. Different strategies for TAN removal from effluents.

Biologically it can be removed, for example, by using nitrification/denitrification, chemically by struvite precipitation or chlorination, and physically by stripping or membrane technologies. The use of physical methods to remove NH_3 is not as effective as biological or chemical processes but it presents the possibility of NH_3 extraction and possibly for agronomic use as fertilizer. This is especially an interesting consideration for effluents with high TAN such as animal wastewater.

Although TAN can be separated from liquid using membrane technologies as microfiltration, dialysis and reverse osmosis, these processes require a good pre-treatment to avoid reduced efficiency due to material deposition (fouling) on the membrane surface that can reduce membrane life (LI et al., 2008; MOOK et al., 2012; NIEUWENHUIJZEN & VAN DER GRAAF 2012).

Membrane Uses

Membrane technologies have improved significantly in the last two decades resulting in new materials and better chemical and physical proprieties. Some applications include medical use (e.g., blood filtration), industrial processing (e.g. juice separation) and environmental mitigation (LI et al., 2008).

For water and wastewater treatment main applications of membranes include microfiltration, ultrafiltration, nano-filtration and reverse osmosis using pressure difference as a driving force to particle separation using modest to high pressure (69 – 10343 kPa). These treatment methods can generate filtered products of better chemical quality but require expensive or complex process controls to avoid loss of efficiency due to fouling. For gas separation, such as NH_3, the membrane technology may be more appropriate than filtration thus avoiding the use of pressure and other sophisticated process controllers due to lesser mass transfer resistance in gas phase compared to liquid phase (IMAI et al., 1982).

According to the chemical composition of membrane, it can be divided in inorganic and organic categories with organic being more suitable for effluent treatment processes. Based on their morphology, membranes are generally classified as dense or porous (symmetric or asymmetric). For dense membranes, the transport of a substance is by dissolution and diffusion and for porous membranes it is by continuous fluid phase exchange. Common membrane configurations are flat sheets, tubes, hollow fibers and spiral-wound cylinders, the last two types have higher gas-liquid interfacial area (m^2/m^2) when compared to the other configurations.

For gases separation and recovery, organic hydrophobic gas permeable membranes, e.g., polypropylene (PP), polyvinilidenefluoride (PVDF) and polytetratfluorethylene (PTFE) are preferred due to lesser mass transference resistance (ROTHROCK et al., 2010; TAN et al., 2006). For NH_3 extraction, PTFE $(C_2F_4)n$ and its expanded form, ePTFE, are preferred due to their hydrophobic characteristics, excellent organic resistance, and chemical stability with organic solvents, acidic and alkaline solutions (WEF, 2006; GUO et al., 2007; ROTHROCK et al., 2013).

General separation mechanism

Membrane separation mechanism is based mainly on chemical or electrical potential gradient (concentration or pressure gradients) under isothermal conditions and can be expressed as gradients of pressure, concentration or partial pressure. For NH_3 separation using a polymeric dense membrane, the mass transfer between both sides of the membrane wall will proceed due to a concentration difference across membrane (IMAI et al., 1982). The theory that better describes this process is the solution-diffusion model that assumes that the pressure within a membrane is uniform and the chemical potential gradient is related to the concentration gradient (WIJMANS, 1995). The flux (J) of the substance across the membrane can be represented by Fick´s law (Equation 8) (HUGG, 2008).

$$J = -D * \Delta c / \Delta x \qquad (8)$$

where,

D = diffusion coefficient ($m^2 s^{-1}$);

c = concentration ($mol\ m^{-3}$);

x = position or length (m).

The diffusion coefficient (D) is a combination of Knudsen diffusion and molecular diffusion. The Knudsen diffusion occurs when collision between molecule and the pore wall is more frequent than the molecular interactions (membrane pore diameter - $d_p < 0.1$ µm). When the interaction between molecules become more significant ($d_p > 10$ µm) the corresponding diffusion coefficient can be calculated from the kinetic gas theory and the molecular diffusion mechanism is predominant. However, both processes can occur between the two pore sizes of the wall (SCHOFIELD et al., 1990).

Influence of membrane wetting characteristics on TAN mass transference

For TAN separation, the wetting characteristic of the membrane is very important and can directly affect this process. A membrane is considered wet when the pores are completely filled with a solvent. Different liquids that are separated by a hydrophobic membrane cannot mix together unless the pressure across membrane wall is more than the membrane-surface expels pressure (IMAI et al., 1982). This is an important phenomenon of mass transference because the diffusion coefficient of a molecule in the gas phase is greater as compared to a molecule in the liquid phase. The wetting or saturation of membrane pores depends upon molecular forces within the liquid (cohesive), and between the membrane surface and the liquid (adhesive) contacting it. The surface wetting can be predicted from the contact angle formed between liquid drop and the membrane surface.

A contact angle (Θ) greater than 90^0 indicates that the liquid dissociates from the solid surface having a non-wetting trend. Conversely, when the angle (Θ) is equal to zero, complete wetting of the porous membrane occurs. The wetting pressure (ΔP_c) can be obtained by using La-place Young equation (HUGG, 2008).

$$\Delta P_c = \frac{2\gamma_L \cos\theta}{r_p} \tag{9}$$

where,

γ_L = surface tension at liquid/gas interface ($N\ m^{-1}$);

Θ = contact angle between liquid phase and membrane (^{0});

r_p = membrane pore radius (m).

Liquids with low surface tension tend to increase membrane wetting, thereby increasing resistance for mass transference across membrane walls. In this way, increasing membrane hydrophobicity helps to reduce membrane wetting (LI & CHEN, 2005).

Ammonia removal mechanism from wastewater

Ammonia in aqueous phase can be removed following the steps listed below:

1 – TAN in *solution A* (Figure 3) must be converted to free NH_3 (FA). As discussed above NH_4^+/NH_3 equilibrium in liquid solution is pH dependent (Figure 1) and an alkaline medium is necessary to shift the chemical equilibrium to the NH_3 side (Figure 3).

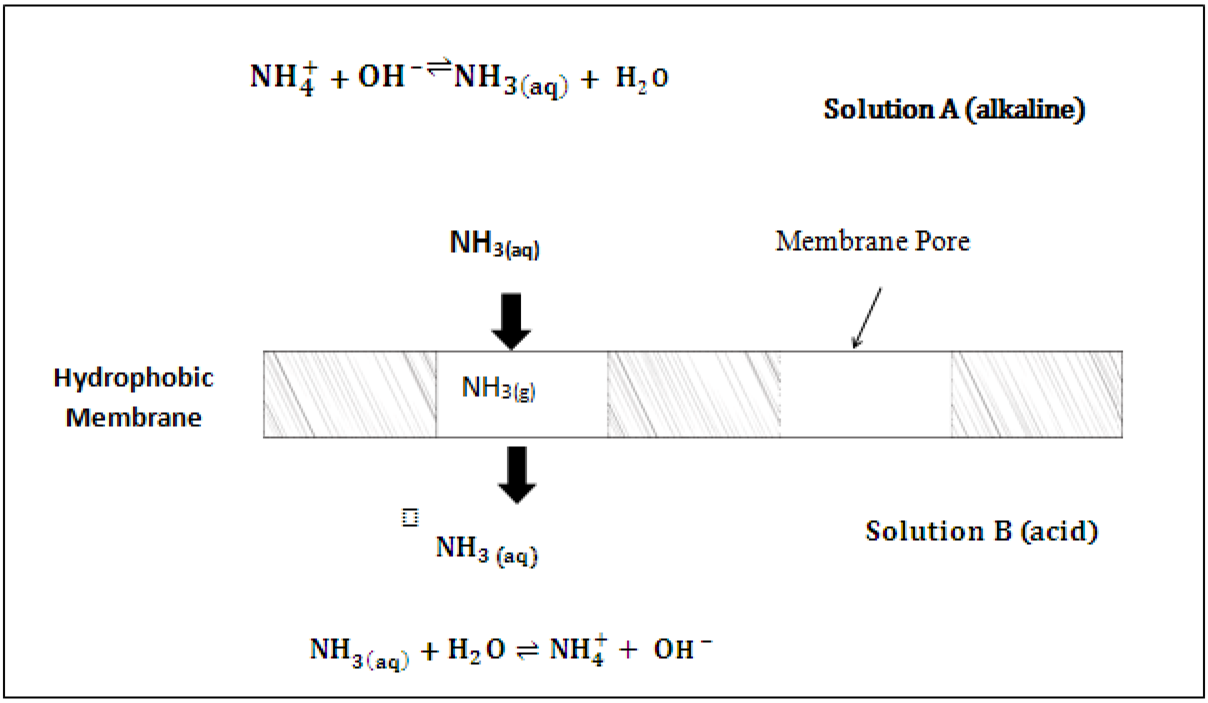

FIGURE 3. Representation of gaseous NH_3 permeation through membrane pore.

To keep the chemical equilibrium shifted to alkaline side and increase the process efficiency, pH must be controlled and buffered. This is important because during NH_3 extraction using membranes, pH of the NH_3 side (Alkaline solution) decreases due to accumulation of H^+ ions that will consume alkalinity (ZHU et al., 2005).

2 – According to Henry´s law, FA in liquid solution can be converted to gas phase following the chemical equilibrium between NH_3 in liquid and gaseous forms, according to [eq. (10)].

$$NH_3(g) \overset{H_{NH_3}}{\rightleftharpoons} NH_3(aq) \tag{10}$$

This chemical equilibrium depends on temperature and can be represented by the van ´t Hoff equation [eq. (11)].

$$H_{NH_3} = \frac{[NH_3]_{aq}}{[NH_3]_g} = 5.53\ .10^{-4}\exp\left[4092\left(\frac{1}{T} - \frac{1}{298.15}\right)\right] \tag{11}$$

In the liquid phase, as represented in [eq. (4)], NH_4^+/NH_3 are in equilibrium and can be described by Ka and determined according to [eq. (12)].

$$K_a = \frac{[NH_3]_{(aq)}.[H^+]}{[NH_4^+]} = 5.67\ .10^{-10}\exp\left[-6286\left(\frac{1}{T} - \frac{1}{298.15}\right)\right] \tag{12}$$

Both equilibrium processes (in liquid and in liquid/gas) are reversible and have impact on volatilization of NH_3. Therefore partial pressure of $[NH_3]_g$ (pNH_3) can be obtained from these NH_3 chemical equilibriums (Equation 13 and 14).

$$p_{NH_3} = \frac{[NH_3]_{aq}}{H_{NH_3}} = \frac{Ka}{H_{NH_3}} \frac{[NH_4^+]}{H^+} \tag{13}$$

$$pNH_3 = \frac{5.67\ .\ 10^{-10}\, exp\left[-6286\left(\frac{1}{T} - \frac{1}{298.15}\right)\right]}{5.53\ .10^{-4} exp\left[4092\left(\frac{1}{T} - \frac{1}{298.15}\right)\right]} \frac{[NH_4^+]}{[H^+]} \tag{14}$$

The concentration of NH_3 in gas phase can then be determined according the ideal gas law as described in [eq. (15)].

$$[NH3]_{(g)} = \frac{p_{NH_3}\ .\ M_{NH_3}}{R\ .T} = \frac{2.75\ .10^6}{T} exp\left(\frac{-1.04\ .\ 10^4}{T}\right).\tau \tag{15}$$

where,

$[NH_3]_g$ = gaseous ammonia concentration (kg m^{-3});

p_{NH3} = NH_3 partial pressure (Pa);

M_{NH3} = molecular mass of NH_3 (kg mol^{-1}, R = universal gas constant (8.314 Pa m^3 K^{-1} mol^{-1});

T = Temperature (K);

τ = NH_4^+ and H^+ concentration ratio in solution (dimensionless).

3- Ammonia in gas phase is transferred through the gas-filled membrane pores due to chemical gradient between both liquid sides of the membrane. The mass transfer flux (N) can be expressed by [eq. (16)] (IMAI et al., 1982; ZHU et al., 2005; SCHNEIDER et al., 1994).

$$N = K\ (C_A - C_B) \tag{16}$$

where,

K= overall mass transference coefficient (m s^{-1});

C_A = concentration of NH_3 in alkaline solution side of membrane (kg m^{-3});

C_B = concentration of NH_3 in acid solution side of membrane (kg m^{-3}).

The pressure necessary to expel the gas out of membrane pores depends on pore diameter, surface tension and the angle of contact.

4 – In the membrane side, containing solution B (Figure 3), NH_3 will react readily with acid to form NH_4^+ as nonvolatile specie (equation 4). This chemical reaction suggests that mass transfer of the acid solution resistance is very low and can be neglected (IMAI et al., 1982). It is important to keep the pH of this solution under control avoiding the regeneration of NH_3 that contributes to the reduction of efficiency of mass transfer from side A to B of membrane.

Applications of hydrophobic membrane in ammonia separation

Several studies were conducted for separation of NH_3 in effluents using hydrophobic gas permeable membrane. Different chemical compositions of membrane have been used but most studies used PVDF and PTFE (or ePTFE).

TAN et al. (2006) using an ammonia-rich synthetic effluent and PVDF hollow fiber membranes studied the alkaline medium pH effect on K. They observed that when pH of the effluent was increased from 8 to 10, K increased significantly but only from 1.39 x 10^{-5} to 1.52 x 10^{-5} m s^{-1} when the pH was increased to 11. Initial TAN concentration also did not affect NH_3 extraction rate indicating that the extraction efficiency is highly dependent on pH of solution (ZHU et al., 2005; QI & CUSSLER, 1985). The NH_3 extraction efficiency of PTFE tubular membrane was evaluated in a lab-scale experiment using synthetic solution. It was observed that NH_3 extraction varied with pH under alkaline or with flow rate of the circulating effluent under basic conditions (BLET et al., 1989). Effluent NH_3 concentrations decreased almost exponentially with increase in flow rate. The increase in flow rate of acidic solution can decrease NH_3 concentration gradient in acid phase resulting in an increase in the mass transference to the acidic side due to faster homogenization and neutralization of NH_3.

Membrane configuration can also influence NH_3 separation efficiency. IMAI et al. (1982) tested tubular, flat and spiral PTFE membranes using synthetic effluent (0.14 to 1.4 g L^{-1} of N-NH_3) and observed that the mass transfer coefficient for flat membrane was one order of magnitude greater than the tubular one but similar to spiral configurations. This difference was attributed to greater turbulence at the tubular membrane surface.

AHN et al. (2011), using PTFE membrane and synthetic effluent, studied the effect of suspended solids on membrane mass transfer coefficient. No significant effect was observed until suspended solid concentration was increased to 3 g L^{-1}. This is a very important result for applicability of these membranes in effluents with greater suspended solid concentrations, such as dairy and swine wastewater.

FINAL REMARKS

Nitrogen is present in livestock and poultry wastewater and other effluents at different compositions and concentrations. Additionally, TAN is prevalent in wastewater containing N at low ORP, generating this mineralized specie. Due to its ability to change forms between NH_4^+ and NH_3 as well as its dependence on solution pH, it is possible to generate free NH_3 that can be extracted by using hydrophobic membranes that are chemically resistant and highly selective to certain gases. This process has a potential use in effluents with high NH_3 concentration, e.g., anaerobically digested effluents and animal waste. The NH_3 separation process with a gas permeable membrane system can be used in effluents with high total solids concentration avoiding the use of complex and costly apparatus used in other membrane technologies (e.g., reverse osmosis, ultrafiltration). The recovery as NH_3 using GPM technology has the potential to be used as a fertilizer, supplying an important nutrient to meet the growing needs of plants.

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