Initial work for Ammonia Separation

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Contents

Abstract

Ammonia removal is an important problem to resolve as whilst it has been responsible for the ability to sustain an 8 Bn population due to increased food production, it has also resulted in widescale degringolade of global waterways via eutrophication and leaching. It requires 2% of the world's energy to produce it and a further 0.5-1% of energy to remediate it. Less than 10 mg/L can harm fish. If left untreated, ammonia poses a serious risk of eutrophication of local water sources. Several processes exist for ammonia treatment and compared with DZ Mb Stripping approach. It is seen from the results that due to an inherently large surface area to volume ratio of the microbubbles, there is a strong increase of the rate of mass exchange or gas exchange for ammonia from the liquid into the air bubble.

An operational lab scale rig was used to characterise the system and partial results have been presented in this study as part of the initial work on ammonia separation. Results obtained have a similar general trend to previously reported work by Desai and Zimmerman. The KD (mass transfer coefficient) obtained has been found to be 3-4 orders of magnitude higher than what has been reported in literature and as industrial comparators. This greatly enhances the process (at least on a prima facie basis) and is higher than previously reported due to use of better and more accurate sensors.

Background

With increasing public scrutiny and environmental restrictions, industrial endusers are under unprecedented pressure to sustainably process waste in a sensitive and economically favourable manner.

Anaerobic digestion (AD) of organic waste draws significant investigation as a sustainable method for processing organic waste into biogas and reducing waste. A rise in food waste has led to increasing greenhouse gas emissions. Anaerobic digestion (AD) has the potential to manage solid waste efficiently and generate renewable energy. In addition to its highly decomposable organic compounds and moisture content, food waste poses health hazards and challenges to waste management systems due to its rapid decomposition, microbial propagation and odour generation. It has been demonstrated that the anaerobic digestion of food waste is an effective method of reducing the amount of waste sent to landfills, thereby reducing pollution and odour problems associated with the disposal site (Ebrahimi-Nik et al., 2018; Midgley et al., 2021).

AD produces ammonia from organic nitrogen, and anaerobic digesters can benefit from the presence of ammonia, which acts as a buffer. Ammonia, in limited amounts, is an essential nutrient for many organisms in the digester. It also functions as a buffer for the digestion process. Nevertheless, high concentrations of ammonia can inhibit digestion, resulting in a loss of capacity and slow production (Procházka et al., 2012; Serna-Maza et al., 2017). A number of studies have found that digestion failures are frequently associated with high levels of free ammonia nitrogen (FAN) (Gerardi, 2003). Ammonia build-up leads to the accumulation of volatile fatty acids (VFAs) within the digester, resulting in decreased biogas production. This can ultimately lead to the failure of the digester. AD failure can be due to the presence of high concentrations of ammonia, inhibiting methanogenic metabolism, hence methane production while triggering the accumulation of VFA in the digester (Nielsen and Angelidaki, 2008). In general, anaerobic microbes consume carbon at a rate 20 to 30 times greater than nitrogen. Therefore, to ensure an efficient biogas production process, feedstocks should have a carbon to nitrogen (C/N) ratio between 20 and 30 (Haque and Haque, 1970). When feedstocks with lower C/N ratios are digested, the concentration of ammonia produced will be higher, resulting in greater ammonia inhibition.

There have been numerous studies conducted to improve and increase the production of biogas from AD. The seeding (by sparging or injection) of carbon dioxide into anaerobic digesters had previously been suggested as a method to improve the methane production from anaerobic digestion of organic waste and sludge (Bajón Fernández et al., 2014, 2015; Al-Mashhadani et al., 2016; Alibardi et al., 2017) using various approaches such as bubble mediated injection, microbubble injection , and carbon dioxide rich atmosphere. It was observed, on a prima facie basis, that the surface area of carbon dioxide interaction was directly corelated to increased biogas production. For example, in comparison with the control reactor, carbon dioxide injection increased methane production by 20% (Bajón Fernández et al., 2015; Koch et al., 2015). In another study, a significant increase (about 100 - 110%) in the production rate of methane had been observed by AD of food waste sparged with pure carbon dioxide gas microbubbles (Al-Mashhadani et al., 2016). However, until the paradigm shifting work by Desai and Zimmerman, (Desai and Zimmerman, 2023) , wherein structures of self-assembled microbubble based scaffolds were utilised to improve mass transfer and gas exchange ,as high as 11-fold improvements in rate of biogas production was not observed. The mechanism for such an interaction is discussed in the paper and is based on **Desai Artificial Lichen**, also known as **Desai Microbubble Scaffolds**.

Ammonia and Ammonium- basis for separation

AD systems, like most biological systems produces ammonia as a by-product of its metabolic processes, which is typically 500 mg/L- 3,000 mg/L. Ammoniacal nitrogen, NH4-N (a measure for the total amount of ammonia in both dissociated and undissociated forms) is produced by the decomposition of proteins. The nitrogen originates from proteins which begin to degrade during digestion. During anaerobic digestion, the insoluble biological polymers are converted to soluble sugars, amino acids, long-chain fatty acids and glycerol. These are then hydrolysed and fermented into ammonia or ammonium (depending on the pH and temperature of the digester) amongst many products via ammonification. Ammonium is found in high concentrations typically ranging from 500 mg/L- 5,000 mg/L.

Ammonia exists in leachate in equilibrium with water:

$$NH3 + H2O \leftrightarrow NH4+ + OH-$$

Using air to strip NH4-N out is the most common method of treatment (Renou et al., 2008) (Abbas et al., 2009). Stripping occurs when a gas (usually air or biogas), unsaturated with ammonia, passes through the liquid which contains undissociated NH3 available for desorption. The rate at which desorption occurs is affected by the concentration of undissociated ammonia, gas-liquid interfacial area, mass transfer coefficient, partial pressure of ammonia in the gas phase and degree of turbulence. All of these are in turn affected by the process and environment conditions *i.e.* temperature and pH (Kjeldsen et al., 2002) (Srinath and Loehr, 1974).

Mass Transfer Theory

Fick’s Law of Diffusion

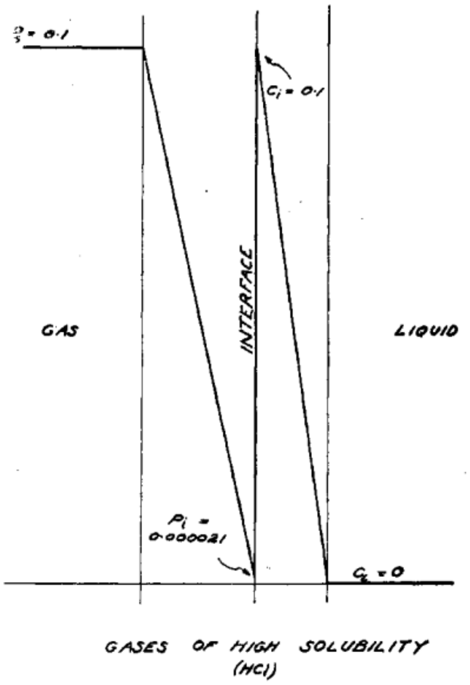

Figure 1. 'Two-film' theory from the original paper published by Lewis and Whitman (1924).

Several theories exist for the mass transfer across the phase interface. Lewis and Whitman (1924) theorised that mass transfer must overcome a resistive layer in each phase at the interface where turbulence dies and mass transfer is governed by Fick's first law of diffusion - Equation 1 - this is known as the 'two-film' theory in Figure 1.

Equation 1

$$J_A = -D_{AB}\frac{dC_A}{dx}$$

Where: JA = molar flux (mol/m2/s)

DAB = diffusion coefficient (m2/s)

CA = concentration (mol/m3)

x = position or length (m)

Penetration Theory

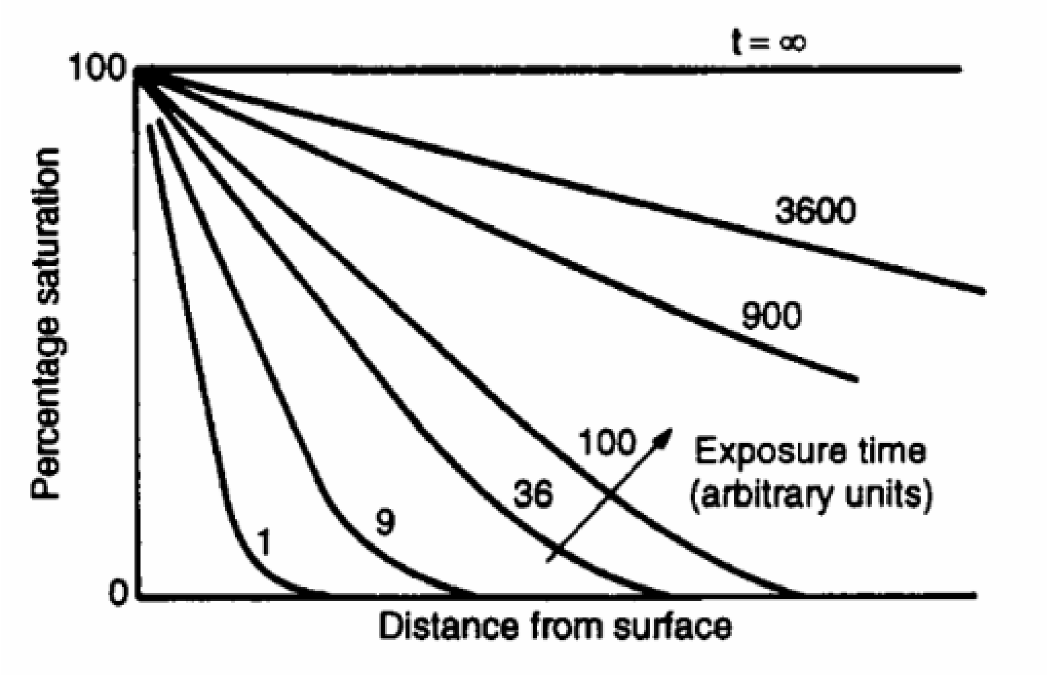

Figure 2 The penetration theory (Coulson et al., 1999).

Higbie (1935) proposed the 'penetration' theory where turbulent eddies transport fluid to the interface where it is then exposed to the alternative phase for a time until being remixed into the bulk. The fluid at the surface will diffuse into the alternative phase until equilibrium is established, or it is carried away by turbulent eddies. The term 'penetration' comes from the measure of how far the solute has diffused into the bulk solvent away from the interface with respect to time, as shown by Figure 2, in terms of absorption.

Film Penetration Theory

Toor and Marchello (1958) proposed the 'film-penetration' theory which combines aspects of the 'two-film' and 'penetration' theories. A laminar film is present, similar to the two-film theory, where turbulent eddies die out, however, mass is transported at irregular intervals to the film by eddy currents. Mass transfer then occurs across the interface as according the Fick's second law of diffusion (Equation 2).

Equation 2

$$\frac{\partial C_A}{\partial t} = D_{AB}\frac{\partial^2 C_A}{\partial x^2}$$

Where, for unsteady-state mass transfer:

$$\frac{\partial C_A}{\partial t} = D_{AB}\frac{\partial^2 C_A}{\partial x^2} - u_f\frac{\partial C_A}{\partial y}$$

Where, uf, is the bulk velocity. When a steady concentration gradient as been established, mass transfer more closely follows Fick's first law of diffusion as in the two-film theory. The film-penetration theory can be summarised as:

Equation 3

$$\pi \leq \frac{L^2}{Dt} < \infty \qquad (N_A)_t = (C_{Ai} - C_{A0})\sqrt{\frac{D}{\pi t}}$$

Equation 4

$$0 < \frac{L^2}{Dt} < \infty \qquad (N_A)_t = (C_{Ai} - C_{A0})\frac{D}{L}$$

Where: NA = number of moles transferred.

Equation 4 will most closely represent mass transfer for droplets or bubbles (Coulson et al., 1999). Many more theories have been proposed which describe mass transfer across an interface, however, focussing on ammonia, as previously shown it dissolves in water and forms ammonium ions as follows:

$$NH3 + H2O \leftrightarrow NH4+ + OH-$$

Only ammonia gas, and not ammonium ions, can be removed by stripping. The amount of undissociated ammonia in water is a function of the pH, and the ionisation constants of aqueous ammonia, kb, and water, kw which are in turn a function of temperature. Most N in the centrate exists as NH4+ and NH3 depending on the pH(Srinath and Loehr, 1974). Srinath and Loehr (1974) showed that the fraction of undissociated ammonia-nitrogen can be found from Equation 5.

Equation 5

$$F = \frac{\text{undissociated ammonia} - \text{nitrogen}}{\text{total ammoniacal nitrogen}} = \frac{10^{pH}}{\frac{k_b}{k_w} + 10^{pH}}$$

Therefore, the ability to remove ammonia via stripping increases with temperature and pH. This backs Le Chatelier's principle and in turn, DZ Mb Stripping, in that an increase in the pH and therefore the hydroxide concentration drives the reaction/equilibrium towards undissociated ammonia production.

Effects of Temperature and pH on ammonia removal

Figure 3 illustrates the effects of temperature and pH on the fraction of undissociated ammonia.

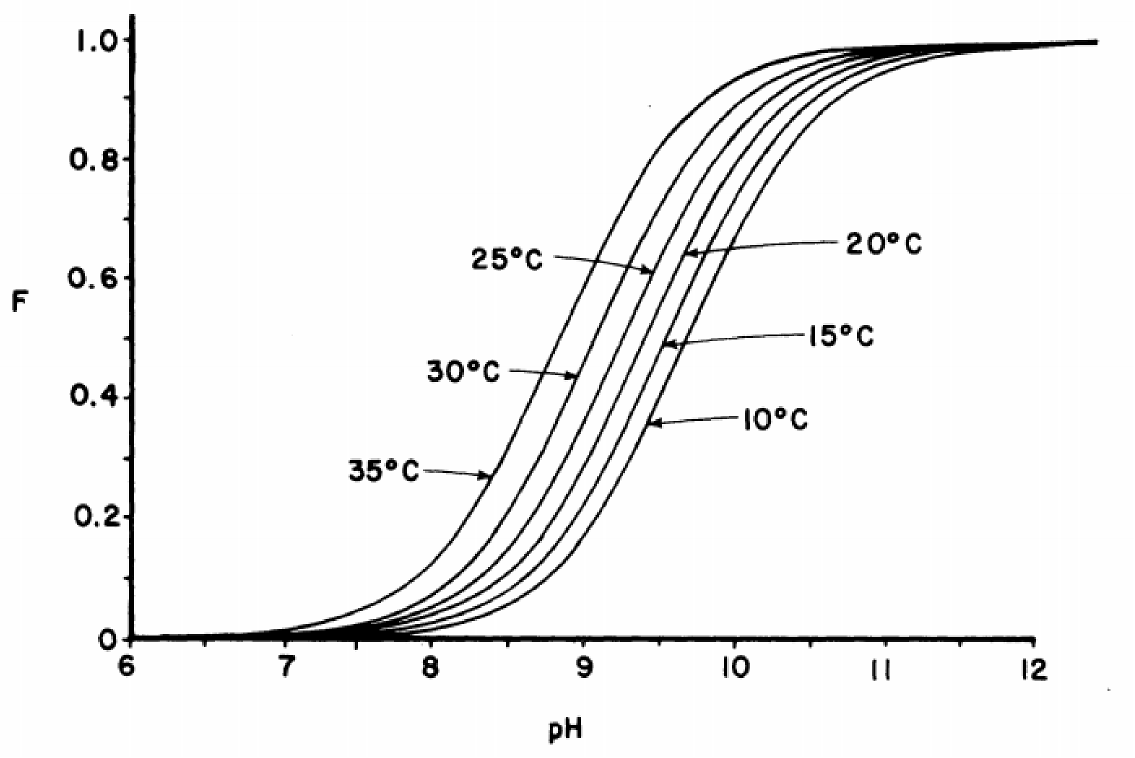

Figure 3. Effect of pH and temperature on the fraction of undissociated ammonia (Srinath and Loehr, 1974).

Besides temperature having an effect on the ionisation constants, it also has an effect on the solubility of ammonia in water. The solubility of a gas is influenced as per Van't Hoff's law of mobile equilibrium, that is, if the temperature of a system at equilibrium is raised, a change will occur to absorb the heat. Usually, when a gas is dissolved in a solution, heat will be generated, therefore as the temperature rises, the solubility decreases (an exception to this is light gases e.g. H2, O2, N2, CH4).

This is why, (Collivignarelli et al. 1998), reported that for an initial ammonia-rich liquor (2000 mg/L) at a pH of 12, required 8h of processing time, with a 50% of removal rate. This limits approaches for use with conventional air stripping. Companies such as Nijhuis require pure CO2 to be used as the carrier gas in order to remove the NH3.

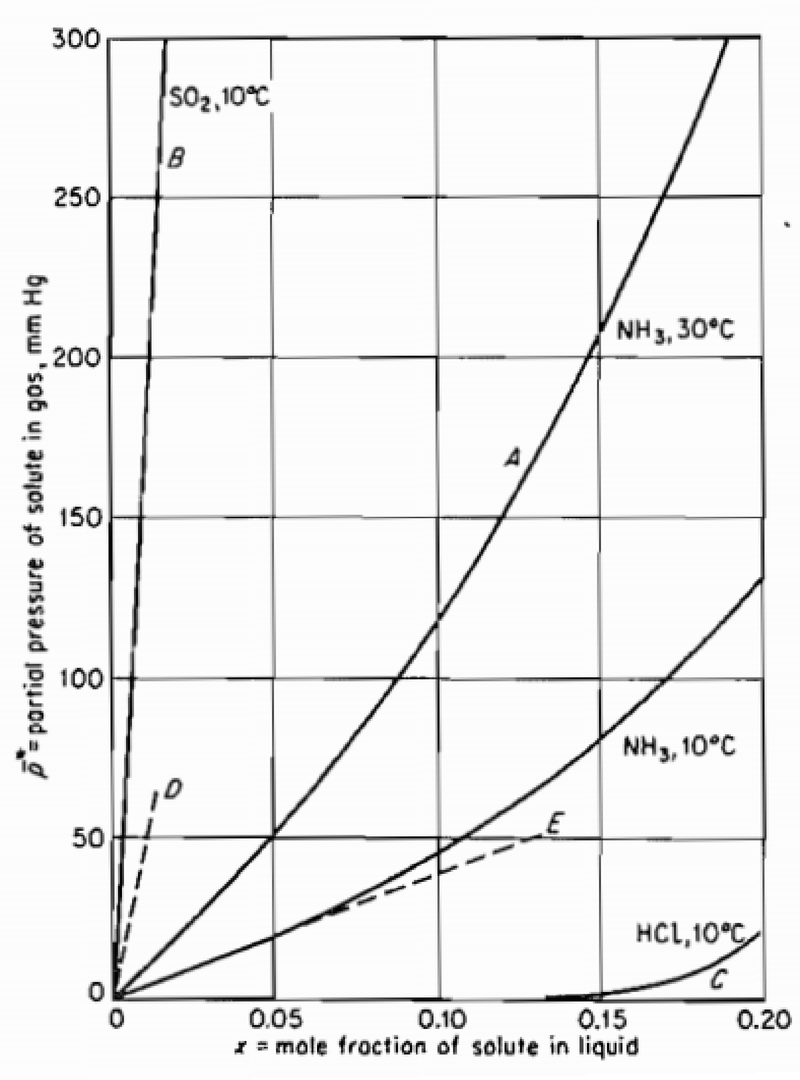

Figure 4. Solubility of gases, including ammonia, in water (Treybal, 1980).

Equation 6 (Srinath and Loehr, 1974) shows that the change of concentration of ammoniacal nitrogen, ΔC, in the ammonia-rich liquor is proportional to the interfacial area, Ai, time that is spent where the gas is in contact with the liquid, t, the concentration of undissociated ammonia in the centrate, CF, and the mass transfer coefficient. It shows that the rate of ammonia stripping is first order and that stripping is most effective at higher concentrations of NH4-N (Kabdasli et al., 2000). It should be noted, however, that NH3 saturation in the bubble has been found to occur within the first few millimetres of ascent (Smith and Arab, 1988), hence, the effect of time on the desorption into the bubble can rapidly become insignificant depending on the bubble rise velocity which may be found using the Hadamard-Rybczynski equation (Hadamard, 1911), (Rybczynski,1911).

Equation 6

$$-\Delta \mathrm{C} = \mathrm{KA_i CF \Delta t}$$

Which can be integrated for a batch reactor to give:

$$\ln\left[\frac{C_1}{C_2}\right] = K_D F(t_2 - t_1)$$

Without pH control, an ammonia-rich liquor undergoing aeration will experience a reduction in pH as the undissociated ammonia is stripped and the alkali leachate begins to absorb the carbon dioxide from the air to produce carbonates from hydroxides. Srinath and Loehr (1974) show the final form of the equation:

$$\ln\left[\frac{C_2}{C_1}\right] = \frac{K_D(L_2 - L_1)(t_2 - t_1)}{(pH_2 - pH_1)\ln 10}$$

Where: Ln = ln(10pHn + kb / kw)

Alternatively, using Henry's law, this can be written as follows to allow for the prediction of how much air is required to achieve a desired ammonia removal assuming constant pH.

$$\ln\left[\frac{C_1}{C_2}\right] = \left(\frac{A\rho_a}{VH}\right) F(t_2 - t_1)$$

Where: A = air flowrate

ρa = density of ammonia

V = volume of liquid

H = Henry's constant

All of the equations show that the rate of desorption is equivalent to interfacial area. In practical terms, the desorption into bubbles has been found to be secondary to the liquid surface area. Furthermore, the bubbles have been found to serve only to create turbulence and agitation at the liquid surface hence causing an increase in surface area (Cheung et al., 1997) (Smith and Arab, 1988). This is not dissimilar to non-aerated systems which can achieve ammonia removals of 50% through desorption solely at the surface, however, this performance is also improved through aeration and surface mixing. The approach proposed by DZ Mb Stripping relies on microbubbles removing the ammonia using similar principles like quantum computing. A larger bubble (like the green one in figure 5) can remove a relatively high amount of ammonia compared to a smaller bubble (like the red bubble in figure 5). However, for the same volumetric throughput, a thousand red bubbles can fit in the same volume as the green bubble but contains 1000-fold higher surface area. As discussed previously, surface area is important for ammonia removal or process improvement. A large number of smaller bubbles can accomplish the same as that of a single larger bubble.

The second principle of DZ Mb Stripping is based on Ludwig-Soret Effect , which postulates that different particles behave differently under a temperature gradient, ie. concentration gradient is driven faster by a temperature gradient. In one of the pieces of work, Desai has made advances to the separation of gaseous vapours as a separation mechanism on the basis of the Dufour effect (Dufour, L., 1872), which is based on evaporative cooling.

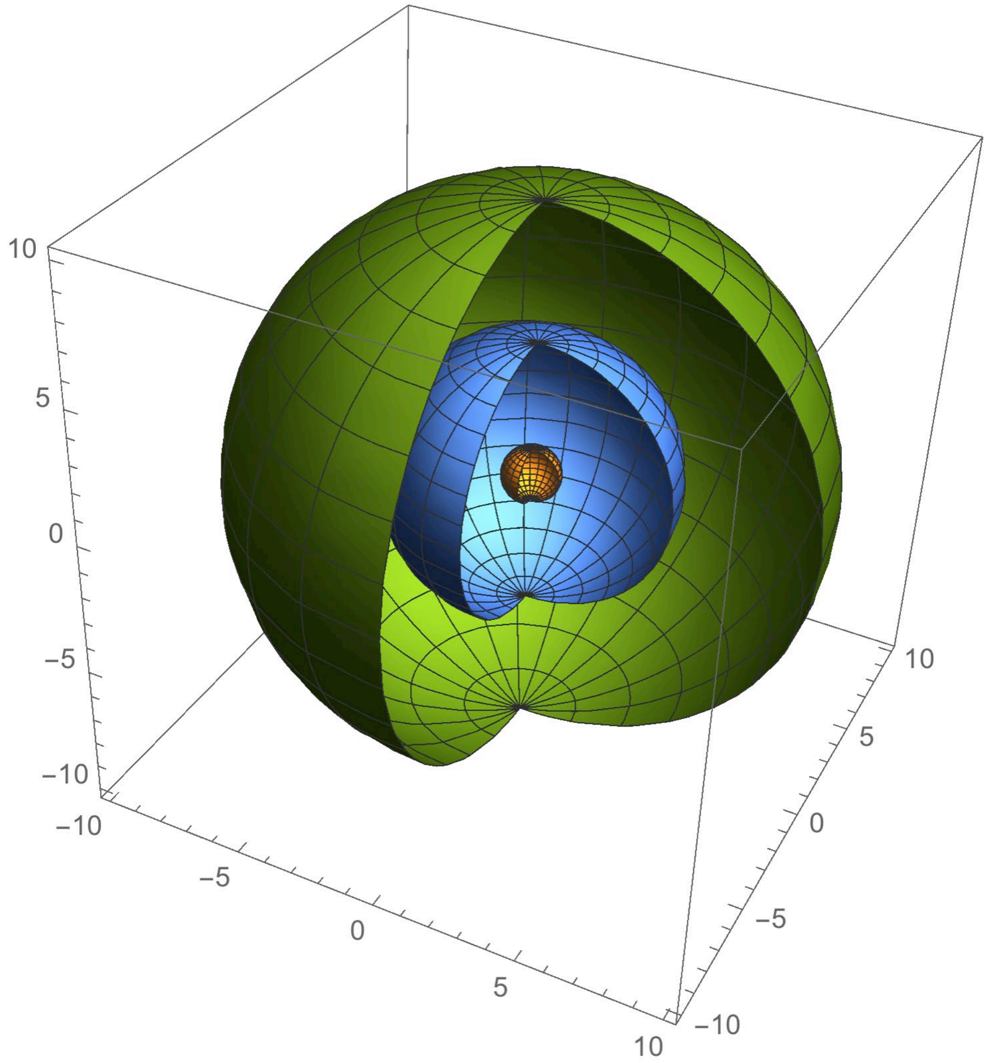

Figure 5 Bubbles of unit size 1, 5, and 10, showing the differences in volume and surface area for various sizes.

Some work has been done analysing the effect of heat on the bulk liquid; (Marttinen et al. 2002) reported stripping at temperatures as low as 6 °C and that a higher efficiency of ammonia stripping can be achieved in a shorter time by using higher temperatures. However, the majority of research has been into determining the optimum air flowrate and pH. There are papers in literature that show some applications of stripping where the optimum pH has been found to be around 11-12, however, an economic analysis is required to balance the cost of alkali against removal efficiency. Figure 3 shows that the increase in F when the pH is increased from 11 to 12 is small compared to 10 to 11. This has also been observed in practice by (Collivignarelli et al. 1998) who observed an increase in removal efficiency from 46 to 50% when the pH was increased from 11 to12. (Collivignarelli et al. 1998) also took a unique approach in analysing the specific ammonia removal with respect to air usage. An increase in air flow from 80 L/h to 200 L/h decreased the specific ammonia removal from 1.13 to 0.8 mg/L air, therefore, better efficiencies of air usage can be achieved at lower flow rates, particularly for higher temperatures.

The effect of hydraulic level has not received much attention due to it being concluded as making no significant difference to removal efficiency as the bubbles reach saturation within a few millimetres, (Collivignarelli et al. 1998). This effect is much more pronounced in DZ Mb Stripping due to much smaller bubble sizes which demonstrate slower rise velocity.

Ammonia stripping has disadvantages in that it can be expensive and prone to scaling and foaming due to the lime used for pH control (Smith and Arab, 1988) (Renou et al., 2008) (Silva et al., 2004). The foaming commonly requires a large column hence incurring added costs (Li et al., 1999). It also produces the hazard of gaseous ammonia escaping into the atmosphere, hence, it is common for the stripped gas to be treated with sulphuric acid or hydrochloric acid (Renou et al., 2008) (Li et al., 1999).

Stripping does have the advantage of being able to treat a wide range of ammonia concentrations. (Li et al, 1999) reported that air stripping is used for NH3-N concentrations greater than 3,000 mg/L which follows from Fick's law of diffusion that a large concentration gradient is required for effective stripping. However, more recent research has shown that leachates with concentrations as low as 74 mg/L can still be treated (Marttinen et al., 2002). DZ Mb Stripping has been used to strip as low as 1 mg/L, down to 0.01 mg/L.

Methods

The MS1 report showcases the experimental set up. The current setup relies on modifications made after that to enable appropriate project operation. Set up has an additional inclusion of 1.5kW process heater, to ensure that room temperature gas could be introduced within the system. The 1.5kW process heater has been designed to enable modular approach to reduce or increase power, so as to be able to be included within the system for pilot use.

The SOP and the forms are based on the standard approach discussed in MS1.

Chemical analysis

Ammoniacal nitrogen concentration

The ammonium concentration in the solution was directly measured using various ammonium ion selective electrodes (ISE) as advised by the American Public Health Association (APHA) Standard Method for the examination of water and wastewater (APHA, 2007) and literature (Kabdasli et al., 2000, Marttinen et al., 2002, Silva et al., 2004, Collivignarelli et al., 1998, Cheung et al., 1997). It is noted that APHA (2007b) states that this method is only applicable up to concentrations of 1,400 mg/L, however, Collivignarelli et al. (1998) used it for concentrations of 2,000 mg/L and the probes itself can operate up to 18,000 ppm (~18,000 mg/L). There were significant issues with the probes being used as they were not providing replicable results on a consistent basis. The electrode is made of a membrane containing an ammonium ion selective exchanger. As the NH4+ of the sample diffuses through the membrane, an electrochemical potential is produced which, when compared to a reference standard, yields the ammonium ion concentration as given by the Nernst equation.

$$E = E_0 + S \log X$$

Where: E = measured electrochemical potential

E0 = reference potential

S = electrode slope

X = level of NH4+ in sample

The level of NH4+ is representative of the effective concentration as only the free ions i.e. not the bonded ions, interact with the probe

$$X = \gamma C_F$$

Where: γ = activity coefficient

CF = concentration of free ammonium ions (= Ct - Cb, where Ct is the total number of ammonium ions and Cb are bound ions)

If the ionic strength of the sample is high relative to the NH4+ concentration (this is achieved using an ionic strength adjuster (ISA) which, for ammonium, is recommended to be NaCl or LiCl) then γ is constant and X is proportional to CF. Therefore, E is proportional to CF and a semi-log plot can be used to convert the measured E to ammonium concentration. This is directly converted based on software provided by Terabithia.

pH measurement

The pH of the solution was directly measured as it is important for ammonia measurement as seen in Figure 3. The probe was calibrated using Phthalate buffer solution (pH 4.0), Deionised water (pH 7.0), & Tetraborate buffer solution (pH 10.0)

UV- Vis Spectrophotometer

The device used for experimentation was a Jenway UV/Visible Scanning Spectrophotometer with a 1.5-nm Bandwidth. The visible region - 400–700 nm – and the spectrophotometry associated with it is used extensively in colorimetry. Ultraviolet–visible spectroscopy or ultraviolet-visible spectrophotometry (UV-Vis or UV/Vis) refers to absorption spectroscopy or reflectance spectroscopy in the ultraviolet-visible spectral region. This means it uses light in the visible and adjacent (near-UV and near-infrared [NIR]) ranges. The absorption or reflectance in the visible range directly affects the perceived colour of the chemicals involved. In this region of the electromagnetic spectrum, molecules undergo electronic transitions.

The system follows the Beer-Lambert Law:

$$A = \log_{10}(I_0/I) = \varepsilon c L$$

where A is Absorbance, I0 – incident light, I is the absorbed light, L is the path length, c is the concentration and ε is the molar absorptivity.

The change in the spectrum can be used to characterise changes to a system as well as quantitatively assess how well does the transmittance/adsorbance changes post processing.

Samples are usually prepared in cuvettes; depending on the region of interest, they may be constructed of glass, plastic (visible spectrum region of interest), or quartz (Far UV spectrum region of interest).

Results and Discussions

[REDACTED]

Conclusions

Initial set of results are showcasing a positive outcome with the application of DZ Mb Stripping for removal of ammonia. This is also observed with the increased KD demonstrated, which is 1,000-10,000 times higher than what has been observed in literature. There is a high variability of results due to the problems faced by older probes and various sensors. Higher temperature of inlet gas has demonstrated better performance for separation. Higher pH, when adjusted, leads to a significant increase in processing and reduction in costs.

This is a work in progress but the initial work is showcasing significant promise and complements work conducted by CCM and Reepel in their respective work packages.

The use of appropriate sensors, high pressure steam cleaning, and recirculation due to pumping has ensured appropriate control strategies are in place and downtime is reduced.

There has been background work on the process wherein CO2 is used for stripping the gas and it demonstrates lower results as compared to air for the same removal rate due to pH shift which is quicker and therefore against Le Chatelier’s principle. This also means that it is a viable candidate for use in situations when there might be an electricity shutdown, which further de-risks the scenario and process.

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