Maximizing Biochemical and Energy Recovery from Wastewater Using Vapor-Gap Membranes

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ABSTRACT

Carbon, nutrients, and heat are available in vast quantities in wastewater. However, technologies that can effectively extract chemicals and energy are needed to realize wastewater as a sustainable resource. Recent advances in wetting-resistant porous membranes, termed vapor-gap membranes (VGMs), have demonstrated that they are well-suited to facile, selective, and cost-effective recovery of volatile resources and energy from wastewater. In this review, we examine the promise and limitations of VGM-based processes with a particular focus on the two types of resources from wastewater: dissolved volatile compounds and low-grade heat. We begin by discussing driving forces and selective mechanisms required for extraction of different resources through VGMs. Then, the current status and challenges on the recovery of volatile compounds using VGMs are presented. We also analyze the resource potential of thermal energy in wastewater and its recovery using VGMs. Based on the membrane capabilities, process requirements, and resource availability, we assess the feasibility of the wastewater valorization using VGMs and identify the research needs to achieve high recovery efficiency, long-term reliability, and scalability.

KEYWORDS

Resource recovery; wastewater; vapor-gap membranes; volatile compounds; waste heat; circular economy.

1. INTRODUCTION

Wastewater treatment has become an indispensable process for public health and environmental protection. Treatment of municipal wastewater is, however, an energy-intensive process. Exerting approximately 25-45% of the operations and maintenance cost in wastewater treatment facilities,[1] the energy consumption of wastewater treatment accounts for 3-4% of national electricity demand in advanced countries and up to 30-40% of local municipalities' energy expenditure.[2–4] The electricity demand for biosolid management in these facilities, which currently exerts ~20% of the treatment cost, is likely to further increase due to the ever-more stringent regulations.

The advancement in anaerobic processes has greatly expanded the potential of wastewater as a fuel and organic chemical source that can largely offset the energy demand and lead to value-added chemical production.[5–8] As one of the major chemicals, the amount of ammonia carried in domestic wastewater (primarily ammonium) is equivalent to ~19% of the ammonia production from the Haber-Bosch process which consumes 1-2% of global primary energy.[7,9] In addition to chemicals, wastewater can also be viewed as a source of valuable heat energy. The temperature of wastewater is in general higher than the ambient, presenting a vast thermal energy capacity. For example, wastewater effluent temperatures in England were on average 2.2 °C higher than the receiving river temperatures and could meet 3.6% of the UK's annual heat demand if recovered.[10] Other wastewater streams, such as those from power plant discharge, contain vast amounts of low-grade heat; for example, thermal power plants in the US discharge a total amount of 19 billion GJ yr^{-1} of thermal energy in the form of residual heat.[11] Overall, the chemical and thermal energy contained in wastewater is considered to be greater than the energy requirement for conventional wastewater treatment processes.[7]

Membrane-based separation is increasingly adopted for resource recovery from wastewater streams owing to its modularity, compactness, and process controllability.[7,12] In particular, vapor-gap membranes (i.e., non-wetting porous membranes, VGMs) have been extensively explored in recent decades, primarily for desalination[13–16] and CO_2 capture.[17–20] The remarkable advancement in surface wettability control enabled the development of highly wetting-resistant VGMs,[21–26] and even membranes of dual functionalities such as simultaneous wetting and fouling resistances.[13,27–32] These advances offer an ample opportunity for recovering dissolved volatile compounds [e.g., CH_4, NH_3, and volatile fatty acids (VFAs)] as well as capturing thermal energy from challenging wastewater by VGMs, beyond desalination and CO_2 capture.

VGM-based recovery of volatile compounds is mainly operated in the membrane contactors (MCs). A VGM placed between a feed (containing target compounds) and a permeate stream (receiving the compounds) forms an air gap (or pocket) in the membrane pores.[33] Volatile target compounds are then transferred across the membrane driven by a chemical potential difference of the compounds resulting from a difference in temperature, pressure, concentration and/or solubility between the two streams. Harnessing thermal waste heat using VGMs uses a similar configuration, where a temperature difference drives a pressured flow of water through the membranes which can be converted to mechanical or electrical energy.[34,35]

Effective and sustainable recovery of the dissolved compounds and thermal energy by VGMs is largely dependent on the membrane properties, as well as the chemical properties and availabilities of the target compounds. By definition, VGMs must offer a reliable wetting resistance against wastewater feed containing various contaminants (e.g., organic solvents and surfactants) to maintain the air gap in the membrane pores. Recovery of certain target compounds may require particularly high wetting resistance when a non-aqueous permeate stream of low surface tension is implemented or the hydraulic pressure of feed/permeate streams needs to be altered.[32,36,37] In addition, the long-term exposure of VGMs to wastewater feed requires resistance or reasonable tolerance to membrane fouling. Meanwhile, the chemical properties of target compounds including the volatility, solubility, and reactivity sets the type of driving forces to be employed for their selective recovery. Further, the technical maturity of waste-to-resource conversion processes (e.g., anaerobic processes), the complexity of post-processing of recovered compounds for higher purity, as well as the market value and demand of the compounds largely impact the types and availability of specific compounds in the feed and the efficacy of their recovery process. This complexity in turn influences the determination of suitable VGMs and process specifications.

This review aims to examine the suitability, requirement, and limitations of VGM-based recovery with a particular focus on the two types of resources from wastewater: dissolved volatile compounds and low-grade thermal energy. Water recovery (i.e., desalination) by a temperature-driven vapor flow, known as membrane distillation (MD), is excluded from the focus of this review. Interested readers may be referred to existing excellent reviews.[13,14,38,39] In this paper, first, the working principles of resource recovery by VGMs are presented. Second, we discuss the characteristics of representative and volatile compounds recoverable from wastewater, the current stage of recovery process efficacy, and challenges. Third, we cover the resource potential of thermal energy in wastewater and discuss the past progress in its recovery using VGMs. The following section identifies the research needs to overcome the challenges for achieving high recovery efficiency, long-term reliability, and scalability. The interferences of resource production pathways, the disparities between the available and recoverable amounts, and the necessity of decision-making are also discussed.

2. FUNDAMENTALS

2.1 Working mechanisms

MC generally encompasses all membrane processes that bring two different phases into contact through either selective or non-selective membranes. In this review, however, the term MC is restricted to the process that transfers volatile or gas species across the air gap created in VGMs. A volatile target compound is driven down the partial pressure gradient, generated by a difference in temperature, concentration, pressure and/or solubility between the feed and permeate streams (Figure 1). With wastewater as the feed (liquid), the permeate can be either gas (liquid-gas MC, Figure 1c) or liquid (liquid-liquid MC, Figure 1d–f). The partial pressure of the volatile solute compound at the feed-membrane interface can be estimated by Henry's law (Figure 1c–e). When the solvent is the target compound, the liquid-vapor equilibrium determines its partial pressure at the interface (Figure 1f). Likewise, the partial pressure of the compound is set low in the permeate bulk (for liquid-gas MC) or at the permeate-membrane interface (for liquid-liquid MC).

Liquid-gas membrane contactors. Sweep-gas MC (SGMC) and vacuum MC (VMC) are the two main types of liquid-gas MC for volatile compound recovery (Figure 1c). In SGMC, a sweep-gas flow (e.g., air) is introduced as a permeate stream, while vacuum is applied in VMC. The sweep-gas pressure in SGMC is much lower than that in conventional degassing units (e.g., bubble column, tray aerator), thus its energy consumption is relatively low. However, the resulting gas mixture is highly diluted by the sweep gas, requiring downstream processes to concentrate the target compound.[40] Additionally, SGMC exhibits a trade-off between the recovery value (i.e., fraction of the recovered mass of the compound to its total mass in the feed) and the target compound fraction in the resulting gas mixture.[41–43]

Although VMC does not incur the dilution issue, the applied vacuum in the permeate induces a considerable transmembrane flow of water vapor.[44–46] This water flow has rarely been discussed in the literature, but a recent study employed VMC for CO_2 removal from anaerobically treated wastewater and reported >95 v/v% of water vapor in the desorbed gas mixture.[47] Owing to the water vapor or other gas impurities, VMC also exhibits a similar trade-off relation to SGMC.[48]

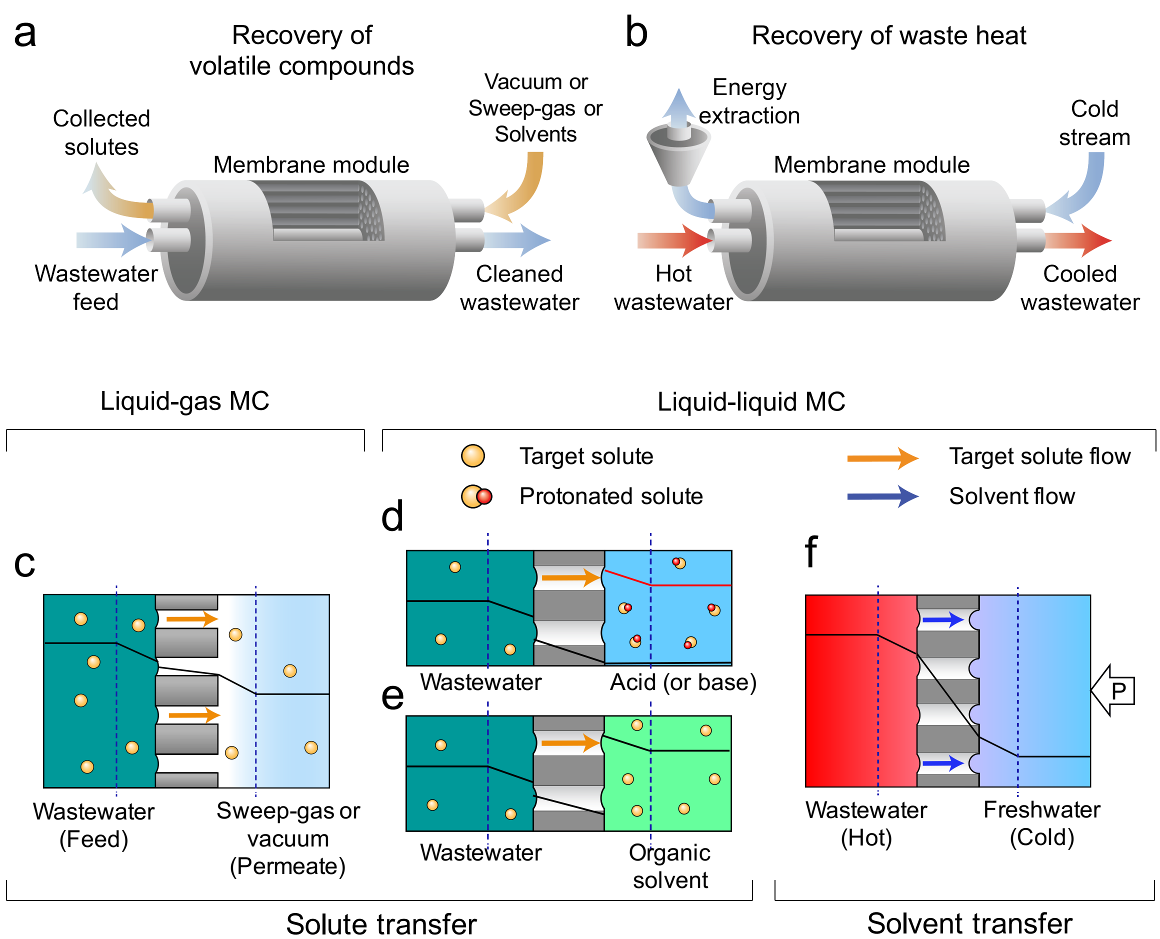

Figure 1. (a) Schematic of membrane contactor module used for recovery of target compounds. (b) Heat recovery using a membrane contactor module. (c-f) Mechanisms of target molecule transfer through vapor-gap membranes in membrane contactor (MC). (c) Liquid-gas MC in which sweep-gas or vacuum serves as the permeate stream having the target species of a low partial pressure (or low concentration). Liquid-liquid MC in which the partial pressure (or concentration) gradient in the pores is developed by (d) chemical reaction (e.g., acid-base reactions) in the permeate, (e) solubility difference, or (f) temperature difference, termed thermo-osmotic energy conversion (TOEC). In TOEC, the vapor pressure of the solvent is elevated by the hot feed temperature and reduced by the permeate pressure. The temperature difference must be sufficiently large to ensure the feed-to-permeate solvent vapor flow. The solid lines indicate the concentration profiles of the target solute species (c–e) and the temperature profile (f). The vertical dotted lines indicate concentration boundary layer (c–e) or temperature boundary layer (f).

Liquid-liquid membrane contactors. In liquid-liquid MC, a low partial pressure of the target compound at the permeate-membrane interface is maintained by the compound's chemical reaction with the permeate, its high solubility, or simply its low concentration in the permeate. An acid-base reaction is commonly employed for this purpose (Figure 1d).[49,50] A solvent that has a high solubility toward the target compound has also been used for the dissolved methane recovery from anaerobic effluent[36,51] and butanol from a fermentation mixture[52,53] (Figure 1e). As a kind of liquid-liquid MC, the thermo-osmotic energy conversion (TOEC) process utilizes a vapor pressure difference of water, induced by high-temperature feed and low-temperature permeate streams (Figure 1f).[34,35]

Transfer resistances and governing parameters. A volatile solute compound transfers from the feed to the permeate by overcoming a series of mass transfer resistances. In liquid-liquid MC, the mass flux of the compound (J in kg m^{-2} s^{-1}) may be expressed[51,54–56]:

$$J = M \frac{P_f - P_p}{R_f^* + R_m^* + R_p^*} \tag{1}$$

Here, M is the molar mass of the compound (in kg mol^{-1}); $P_f \equiv C_f/H_f$ and $P_p \equiv C_p/H_p$ are equivalent to the partial pressures (in Pa) of the compound at its equilibrium with feed and the permeate bulk solutions, respectively; C_f and C_p are the concentrations (in mol m^{-3}) in the feed and the permeate bulk, respectively; H_f and H_p are Henry's constants (in mol m^{-3} Pa^{-1}) with respect to the feed and the permeate solutions, respectively; R^*_m represents the mass transfer resistance of the compound across the membrane (in kg mol^{-1} m s^{-1}), determined by membrane structural properties; R^*_f and R^*_p are hydrodynamic resistances (in kg mol^{-1} m s^{-1}) corrected by the Henry's constant, associated with the compound's diffusion across its concentration boundary layers in the feed and permeate streams, respectively. For the simplest, flat sheet configurations, $R_m^* = R_g T/k_m$ where R_g and T are the universal gas constant (= 8.31 J mol^{-1} K^{-1}) and temperature (in K), respectively. The mass transfer coefficient of the membrane may be modelled as $k_m = \varepsilon D_{eff} / \tau l$, with the diffusion coefficient of the target compound in the air gap (D_{eff}), the porosity (ε), the tortuosity (τ), and the thickness (l) of the VGM. Also, $R^*_{f\ (or\ p)} = 1 / k_{f\ (or\ p)} H_{f\ (or\ p)}$, where various models are available to express the mass transfer coefficient $k_{f\ (or\ p)}$. For other membrane configurations (e.g., tubular module, hollow fiber module), geometrical factors of the modules must be incorporated.[57,58]

The relative magnitude of transfer resistances is influenced by the solubility of the compound, represented by the Henry's constant. In the case of lowly soluble gas (e.g., CH_4), the membrane resistance (R^*_m) is relatively small, and the diffusion resistances in the boundary layers of liquid streams (R^*_f or R^*_p) become more dominant. For liquid-gas MC, P_p is substituted by partial pressure of the target compound in the permeate bulk.

When the solvent (in the form of water vapor) transfers in TOEC process, there is no resistance in the feed or permeate, and the mass flux may be expressed as:

$$J = M\,\frac{P_f^{vap}\left(T_{f,m}, P_{h,f}\right) - P_p^{vap}\left(T_{p,m}, P_{h,p}\right)}{R_m^*} \tag{2}$$

The equilibrium vapor pressure at the feed (or permeate)-membrane interface, $P^{vap}{}_f$ (or $P^{vap}{}_p$), is determined by the temperature at the interface, $T_{f,m}$ (or $T_{p,m}$), and the hydraulic pressure of the feed, $P_{h,f}$ (or permeate, $P_{h,p}$). Different from the solute transfer, the hydrodynamic influence is manifested in the temperature polarization, resulting in the decrease (or increase) of $T_{f,m}$ (or $T_{p,m}$) from the bulk stream temperatures. The vapor pressure elevation by the hydraulic pressure increase is minor compared to the temperature increase. Accordingly, the TOEC process allows for the large pressure development in the permeate without substantial impacts on the mass flux and utilizes the permeate pressure for downstream energy generation.

2.2 Wetting resistance

Pore wetting of VGMs results in an unwanted loss of selectivity and productivity, eventually leading to failure of the process. VGMs must therefore be designed to avoid pore wetting. Below, we review pore wetting phenomena related to membrane design, operating conditions, and feed solution characteristics.

Liquid entry pressure related to membrane properties and pressure. To maintain the air gap and prevent pore wetting, the applied external pressure should be lower than the liquid entry pressure (LEP). Assuming a smooth and straight pore, the Young-Laplace equation describes[59,60]:

$$\mathrm{LEP} = \frac{-2\beta\gamma_{LV}cos\theta_{eq}}{a} \tag{3}$$

Where β is a correction factor for pore entrance shape (β=1 for cylindrical pore), γ_{LV} is the surface tension of the liquid and vapor interface, θ_{eq} is the intrinsic contact angle at equilibrium, and a is membrane pore radius. While most resource recovery applications do not have extremely high pressures in feed or permeate streams, a hydraulic pressure can inevitably form to maintain a crossflow. Furthermore, in TOEC, high pressures are required for system operation. To prevent wetting, membranes should be designed with pores that are sufficiently small and hydrophobic to enable a high LEP.

Pore wetting due to low surface tensions liquids, surfactants, and fouling. In addition to hydraulic pressure, pore wetting can be caused by low surface tension liquids, surfactants, and foulants. This type of fouling is especially common in wastewater resource recovery applications since feed streams often contain industrial organic solvents, wastes from oil processing, food concentrates, and biological organic matter. With high surface tension liquids (e.g., water), the unwetted state is thermodynamically favorable as long as a membrane material is hydrophobic (low surface energy) and the criteria in Equation 3 is met. However, the unwetted state with the hydrophobic material is no longer stable when the surface tension of a liquid is low. With low surface tension feed solutions, forming the solid-liquid interface becomes more energetically favorable than the solid-air interface, and thus, the hydrophobic pores will be fully wet with low surface tension liquids.[24,61]

Introducing re-entrant structures can enable to maintain meta-stable states before reaching the fully wetted global equilibrium state, allowing unwetted pores to be sustained even with low surface tension liquids and surfactants (Figure 2). Re-entrant structures introduce an energetic penalty for expanding the liquid-vapor interface. This energetically unfavorable phenomenon can outperform the favorable wetting progress (growth of solid-liquid interface), and thus, an energy barrier occurs for the transition from the unwetted state to the wetted state. Consequently, membranes that have re-entrant structure, when coupled with a low surface energy, can enable robust omniphobicity that resists wetting by water, low surface tension liquids, and surfactants.[36,62,63] Fibrous mat or microporous substrates feature typical re-entrant structures in VGMs.

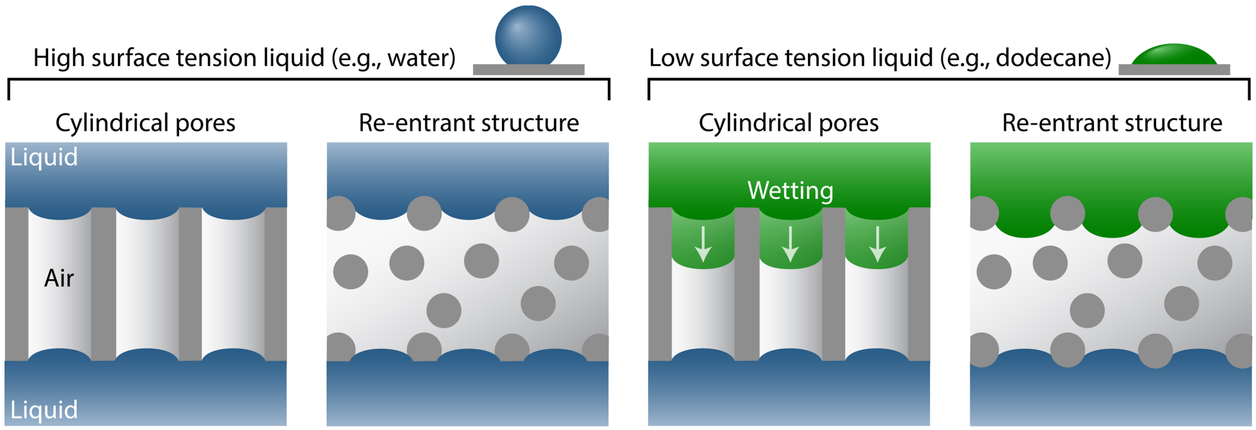

Figure 2. Schematic diagram of the effect of hydrophobic membrane structure on wetting. Hydrophobic membranes with cylindrical pores and hydrophobic nanofiber membranes with re-entrant structures are shown. Blue represents a high surface tension liquid, and green represents a low surface tension liquid.

3. RECOVERY OF VOLATILE COMPOUNDS

Anaerobic processes are widely applied to treat various wastewaters due to the significantly lower energy requirement than aerobic processes, the low amount of produced sludge, as well as generation of byproducts as potential resources. While biogas (or biomethane) is the most common byproduct of the biological breakdown of carbonaceous compounds, nitrogenous compounds end up producing ammonia during anaerobic treatment. Several other valuable compounds such as volatile fatty acids, nitrous oxide, and biohydrogen are also produced during the complex microbial anaerobic processes. All of these byproducts represent potential resources that can be harvested using VGMs.

3.1 Methane

In the US, approximately 1 billion kWh of electricity was produced by biogas from waste treatment facilities in 2021.[64] In Canada, injecting biomethane into the natural gas grid is increasingly practiced, and the Canadian Gas Association aims to replace 10% of natural gas supply with biomethane by 2030.[65] Likewise, China targets the annual biogas production exceeding 30 × 10^{27} m^3 by 2030, which can reduce ~50 million tons of annual coal consumption.[66] The recent demonstrations of upflow anaerobic sludge blanket (UASB) reactors and anaerobic membrane bioreactors (AnMBRs) at ambient or even lower temperatures have shown great promise in converting the large fraction of dissolved organics into methane[67,68] However, the higher gas solubility at lower temperature and supersaturation renders significant fractions of produced methane to exist as dissolved gas (up to 88% at 25 °C) in the anaerobic effluent, resulting in its loss to the environment upon the effluent discharge.[3,69]

Dissolved methane recovery by VGMs has been explored mainly via Liquid-gas MCs. Both SGMC and VMC have shown high recovery values of methane (>79%-98.9%) from synthetic wastewater and real anaerobic effluents over a wide temperature range (12.7-31.5 °C) (Figure 3). Nevertheless, the reported methane fluxes and recovery values vary significantly; in particular, the initial methane flux differs in orders of magnitude, revealing the combined impact on methane flux or recovery by a number of variables such as feed flow velocity, gas flow velocity (or vacuum pressure), and membrane wetting resistance. Still, the overall trend shows a higher methane flux in VMC than in SGMC, suggested by the lower hydraulic retention time to achieve >80% methane recovery. An operation mode also impacts the methane recovery value in hollow fiber modules. The feed flowing through the lumen channel leads to a higher mass transfer coefficient but can cause a clogging of the lumen channel when treating an effluent containing suspended solids of high concentrations (e.g., UASB effluent). Conversely, a flow through the shell side of the module can prevent fiber clogging but lead to a slower methane transfer.

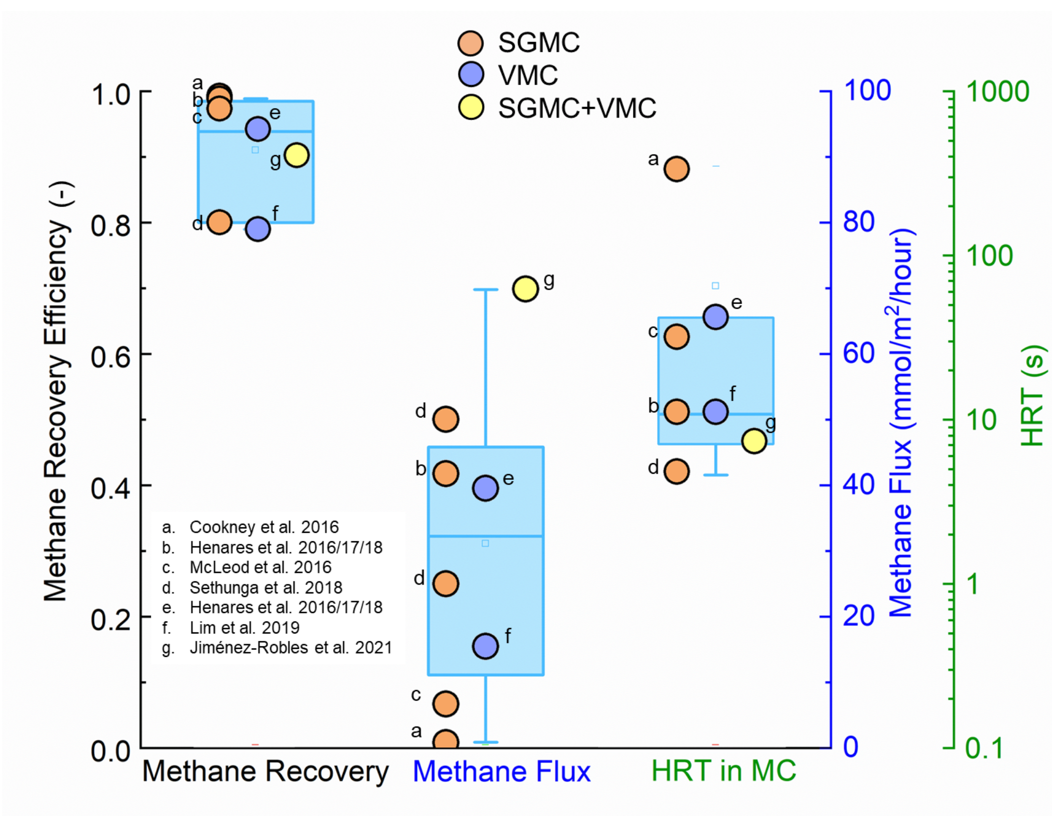

Figure 3. Experimental data of methane recovery, methane flux, and hydraulic retention time for VGM-employed MCs in three configurations (SGMC, VMC, and a hybrid of SGMC and VMC).[42,43,70–75] Summary is provided in Table S1, supplementary material. Box chart is constructed using data from all three MC configurations.

Liquid-liquid MCs (Figure 1e) have been investigated only recently to extract dissolved methane by solubility difference in disparate solvents.[36,63] In these studies, a highly methane soluble, non-volatile, non-polar organic solvent was used as the permeate stream and an omniphobic VGM that is wetting-resistant to the permeate was employed. Methane recovery values of ≥90% were obtained over a psychrophilic to mesophilic temperature range (15 – 35 °C). Importantly, a transmembrane water vapor flow was effectively blocked, potentially lowering the energy requirement compared to VMC (liquid-gas MC) by obviating a dehydration process. It also minimizes a convective flow of foulants toward the membrane surface and alleviates fouling.

A highly diluted biogas (<1 v/v% methane) generated in SGMC is hardly usable and its methane content needs to be increased, as a combined heat and power (CHP) system requires at least 30 v/v% methane.[76] The inevitable trade-off between methane recovery value and methane mass fraction in biogas is a challenging constraint. In the literature, biogas consisting of >30 v/v% methane was acquired only when a methane recovery value was less than 80%.[43] The dilution of biogas can be avoided in VMC. However, the water vapor flux across the membrane becomes significant (resembling a vacuum membrane distillation[44] or isothermal membrane evaporator[77]), and dehydration of the biogas will compromise the energy balance of the entire recovery process. Liquid-liquid MC using a non-polar solvent as a permeate solution minimizes the water content in the acquired biogas. However, the re-dissolved methane in the permeate must be released to obtain methane as a gas product and regenerate the permeate. The technical feasibility of the downstream degassing has yet to be validated. Other volatile species (e.g., CO_2, H_2S, NH_3 and siloxane) also transfer across the VGM. In particular, the concentration of dissolved CO_2 in anaerobic effluent at pH 6.5–7.0 is generally much higher than that of dissolved methane.[3] Therefore, anaerobic effluent of pH >8 is desired to lower the CO_2 fraction in recovered biogas.[63] H_2S content in the anaerobic effluent is generally low. However, its high solubility in water will likely make the H_2S concentration nearly equivalent to the dissolved methane. When used for combustion, biogas containing H_2S can cause corrosion of combustion engine components.

3.2 Ammonia

Ammonia is widely used for fertilizer, ammunition, and chemical production, and is considered as a promising hydrogen carrier for fuel cell vehicles.[78,79] Conventional wastewater treatment facilities are directed to the conversion and removal of ammonia compounds (NH_3/NH_4^+) as dinitrogen (N_2) commonly via the nitrification-denitrification or the more recently proposed Annamox process.[80,81] For ammonia compound recovery, struvite precipitation to produce the slow-release fertilizer has been pursued with some commercial success. For instance, Ostara Pearl(TM)'s struvite reactors are operated in over 18 wastewater treatment plants across North America and Europe.[82,83] However, the NH_4^+ capturing capacity is limited by stoichiometry, as the phosphorus concentration in wastewater is typically much lower than NH_4^+.[84–86] Adsorption of NH_4^+ ions by ion-exchange resins[87–89] has shown significant NH_4^+ recovery, but suffers from the slow diffusion of NH_4^+ ions and the need for regenerant chemicals in large quantities.[90] Microbial fuel cells[91,92] have also been explored for simultaneous ammonia and energy recovery, albeit with low NH_4^+ recovery.[93] Ammonia acquired by air-stripping generally exists in unusably low concentration.[94–96]

Employing VGMs, both liquid-gas MCs and liquid-liquid MCs rely on the pH-dependent ammonia species conversion.[97] At a sufficiently high pH, with pKa of 9.3, ammonia primarily exists as a dissolved gas species, and can be extracted across the VGM. Liquid fraction of both manure and anaerobically digested waste/sludge are suitable sources, as they not only have high concentration of NH_3/NH_4^+ but also of relatively high pH (7.0-8.5).[95,98,99] To further increase the pH, alkali chemicals such as caustic soda are commonly added to the feed.

VMC has shown >70 % NH_3 recoveries at pH of >7 and a wide range of temperatures (40-75 °C) for various initial ammonia concentrations in feed wastewater (100-1500 mg NH_3-N L^{-1}).[100–104] The low selective nature of the VMC process is somewhat advantageous to NH_3 recovery in the presence of dissolved CO_2 in the feed.[100] The removal of CO_2 from the feed elevates pH, resulting in an increased NH_3 flux. SGMC tends to be less prone to membrane wetting than VMC.[105] The overall reported NH_3 recovery using SGMC varies from 15 % at feed pH of ~8-9 and temperature of 35 °C[106] to >97% at pH 11.5 and temperature of 75 °C.[105] Importantly, ammonia in the product gas from both VMC and SGMC is heavily diluted by a carrier gas (in SGMC) or water vapor/other extracted gases (in VMC), impractical for direct use.[54]

Most liquid-liquid MCs for NH_3 recovery employ an acidic solvent (e.g., sulfuric, nitric, and acetic acids) as the permeate. The reaction of the transferred NH_3 with the acid at the membrane-permeate interface converts the gaseous NH_3 to NH_4^+, which subsequently diminishes the partial pressure of ammonia in the VGM pore at the interface (Figure 1d) and drives the ammonia transfer across the pore. The ammonium salts in the permeate can be used as fertilizers.[107] A high NH_3 recovery (up to 99%) has been achieved from various types of wastewater as the feed, including municipal wastewater,[108–110] radioactive wastewater,[111] leachate,[103] swine manure,[98,99,112] and human urine[113–115].

Figure 4 presents the initial ammonia concentrations and temperatures tested in the literature employing liquid-liquid MC and VMC, and the estimated mass transfer coefficient. Over the wide range of initial feed ammonia concentrations (50-25,000 mg NH_3-N L^{-1}), the mass transfer coefficient (and therefore ammonia flux) hardly shows any correlations with the initial concentration. Conversely, a higher feed temperature generally results in a higher mass transfer coefficient. For example, with the permeate at ambient temperature, the average mass transfer coefficient (5.35×10^{-5} m s^{-1}) with the feed at 50 °C (study f, in Figure 4) was nearly four times larger than that (1.4×10^{-5} m s^{-1}) at 32 °C (study e, in Figure 4). The increased diffusivity of the gas as well as the reduced gas solubility at higher temperature facilitates the extraction of ammonia.[116] However, elevating the feed temperature not only requires thermal energy, but also generates a feed-to-permeate water vapor flow. This flow dilutes the permeate[110] and requires post-treatment of the solution to obtain a concentrated product. These limitations can be circumvented by operating liquid-liquid MCs at isothermal conditions. However, even at the same bulk temperature of feed and permeate solutions, a reverse water flow from the permeate to the feed may occur, owing to the exothermic ammonia-acid reaction at the membrane-permeate interface and the consequent temperature increase at the interface.[117] This reverse water flow can be advantageous to produce a more concentrated permeate solution, at the cost of a reduced ammonia flux due to the dilution of the feed by water.

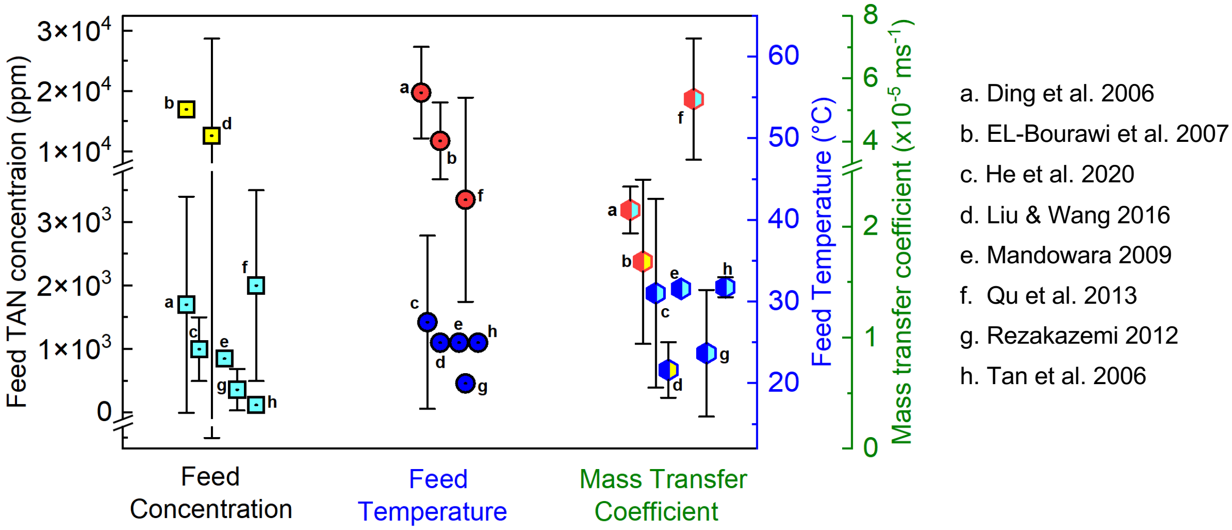

Figure 4. Effect of feed conditions on ammonia recovery performance using VGMs (summarized in Table S2). All shown data are from liquid-liquid MC, employing an acid as permeate.[54,97,103,104,111,118–120] The left column indicates Total ammonia nitrogen (TAN) concentrations of the feed, representing TAN in various wastewaters (50-25,000 mg L^{-1}). The center column indicates tested feed temperatures. The right column indicates estimated mass transfer coefficients of NH_3. Error bars represent the maximum deviation from the average of each parameter (feed concentration, temperature, and mass transfer coefficient) reported in each study.

Several issues exist before achieving an economically feasible recovery.[110] Ammonia-rich wastewater streams are essential and a high pH of the feed needs to be maintained. Electrochemical techniques can potentially replace the chemical addition to maintain a high feed pH, thereby reducing the chemical cost, but fouling control remains a challenge.[121] In addition, the use of excess acid in the permeate solution yields a highly acidic (pH<4) ammonium salt solution, unusable for direct land application as fertilizer.[122] Downstream processes such as precipitation or electrochemical separation will then be required.[123,124]

3.3 Volatile fatty acids (VFAs)

VFAs are carboxylic acids containing six or fewer carbon atoms and are primary intermediate products of anaerobic digestion or fermentation of organic-rich wastes.[125–127] Conventional anaerobic digestion mostly recovers energy compounds in the form of methane. However, the recent interest in organic acid recovery promotes increased VFA production by eliminating the methane-forming phase in the anaerobic digestion or fermentation process.[128] VFAs are excellent feedstock in the biorefinery industry for producing value-added chemicals such as alcohols, esters, aldehydes, ketones, and olefins,[127,129] as well as for biofuels and bioplastics.[127,128] VFAs can also be used as a carbon source for biological nitrogen/phosphorus removal processes treating wastewater, and as a raw material for biological hydrogen production.[127,128,130]

Among VFAs, acetic acid's global demand was 12.1 Mt in 2014, and its market revenue is forecasted to reach $11.4 billion by 2024.[131] Butyric acid's market size is estimated to be USD 405 million in 2027[132] for its demand in the cosmetic, food, and pharmaceutical industries.[133,134] At a growth rate of 7.8%–9.6%,[135] propionic acid market is projected to reach USD 2.9 billion by 2026.[136] In the Netherlands, the market supply potential of VFAs by their recovery from wastewater was estimated to be 1% for acetate, 17% for propionate, and 6% for butyrate of the national demand.[7]

Available technologies for VFA recovery include gas stripping with absorption,[137,138] adsorption,[139,140] electrodialysis,[134,141] solvent extraction.[142,143] Adsorption and electrodialysis generally incur high cost and energy demand, while solvent extraction requires further process for extractant regeneration. The volatile nature of VFAs renders VGMs well-suited for VFA recovery from wastewater,[144] in particular via liquid-liquid MC with a high pH permeate. Analogous to ammonia recovery, the deprotonation of VFAs upon their transfer to the permeate side generates a low partial pressure of VFAs in the membrane pore, driving the feed-to-permeate VFA transfer. Recovery of several VFAs from synthetic solutions and a fermented organic solid waste leachate has been demonstrated, where valeric acid showed the highest selectivity.[128] It was also demonstrated that the recovery efficiencies of VFAs from their mixtures were approximately the same for acetic, propionic, butyric, valeric, and caproic acids.[127] A liquid-liquid MC incorporating temperature difference as the driving force (analogous to membrane distillation for desalination) was also shown to recover acetic acid from the fermentation broth,[145] although the water vapor transfer caused the dilution of the permeate.

Similar to ammonia recovery, VFA recovery using VGMs requires a pH adjustment of the feed below ~4 to ensure VFAs in the volatile (protonated) form.[127,128] Such acidification of feed wastewater along with the base addition (e.g., NaOH) in the permeate stream adds chemical cost. To minimize the chemical cost, an electrically conductive VGM has recently been attempted. The conductive layer of the membrane serves as an anode, producing protons in the vicinity of the VGM. The locally increased pH protonated VFAs and allowed their transfer across the VGM.[146] A challenge outside the separation process stems from the biological production of VFAs, as the mixed culture fermentation results in a wide spectrum of end products. In particular, carbohydrate fermentation leads to various products including VFAs and larger fatty acids, alcohols, carbon dioxide, and hydrogen.[7,125] Attaining optimal operating conditions for the targeted microbial community must be first achieved to ensure increased VFA production, prior to a VFA recovery using VGMs.

3.4 Other potential resources

Nitrous oxide. Nitrous oxide (N_2O) is produced as a byproduct of the denitrification process during wastewater treatment.[147,148] As a potent greenhouse gas, N_2O emissions can contribute up to 78% of the carbon footprint of wastewater treatment facilities.[149,150] However, N_2O is a highly energetic oxidant, which boosts combustion reactions and is therefore used to supercharge the engines of high-performance vehicles and in hybrid rockets.[151] Recovering N_2O has emerged as a promising pathway for simultaneously removing nutrients and producing a renewable energy source from wastewater, particularly by the coupled aerobic-anoxic nitrous decomposition operation (CANDO).[152] This process entails (i) partial nitrification of ammonium to nitrites, and (ii) partial anoxic denitrification of nitrites to N_2O. Thermal energy can be produced by its conversion to nitrogen via catalytic decomposition of N_2O to N_2 and O_2. Also, the N_2O can be used to oxidize biogas (CH_4), producing more heat than biogas oxidation with oxygen.[152,153] Compared to the conventional biological nitrogen removal process, CANDO requires less organic addition for nitrogen removal, demands less oxygen for aeration, produces less biomass, and allows for increased energy production through N_2O recovery.[152,154] However, N_2O is highly soluble (~17x than CH_4), making the extraction of N_2O from the mixed liquor slow. Although gas stripping is commonly practiced for N_2O recovery or removal, the heavily diluted N_2O is not adequate for combustion.[155]

VGMs are suitable to recover N_2O gas produced from the CANDO process.[155] Weißbach et al. demonstrated 77% recovery efficiency of N_2O harvesting from aqueous N_2O solution using an SGMC process, along with potential recovery efficiency of up to 90%.[156] VGMs in a hollow fiber module were also recently applied to separate N_2O from its mixture with air.[157]

While the high solubility of N_2O in water slows down the recovery process, large membrane areas available in hollow fiber module can readily compensate the recovery rate. However, as the energy potential is relatively low, high-strength wastewater would be required as a proper source of N_2O. Besides, the complex operation and the instability of N_2O production through the CANDO process hinder large scale implementation.[155] A stable coupling of nitrous denitritation and nitritation must be realized. Also, as the recovery of N_2O competes with ammonia recovery, the economic feasibility of harvesting either chemical needs to be assessed.

Biohydrogen ($BioH_2$). Hydrogen can be biologically produced from wastewater via a two-step anaerobic process: hydrolysis and acidogenic fermentation.[7] $BioH_2$ production can be increased by inhibiting methanogenesis,[158] or incorporating in-situ gas extraction using AnMBR.[159] In addition to powering fuel cells,[160] $BioH_2$ can be used on-site as an electron donor for reducing nitrate, perchlorate, and selenate, and for detoxifying a wide range of water pollutants.[161]

VMC was recently investigated for $BioH_2$ recovery in a microbial electrolysis cell.[162] H_2 produced at the cathode was extracted across a PTFE-made VGM, which deterred methanogenesis and consequently allowed for H_2 yield 3-4 times higher than otherwise. In another study, a composite VGM with encapsulated acetogenic bacteria was fabricated to simultaneously generate and capture $BioH_2$ during the treatment process of a high strength wastewater. This VGM exhibited nearly complete H_2 capture efficiency (up to 99%) from the wastewater. The H_2 yield (up to 48.43 ± 9.41 mL H_2 g^{-1} hexose) was comparable to the estimated values from anaerobic digestion sludge as seed cultures (45–92 mL H_2 g^{-1} hexose at 22 °C).[163]

The major challenge of $BioH_2$ process is the low yield,[7] as only ~15% of the energy available from the organic source can be recovered as hydrogen from biomass.[160] In addition, producing $BioH_2$ requires suppression of methanogens, inhibiting methane production. As biogas harvesting from anaerobic digesters is a mature process, it is unlikely that $BioH_2$ production will replace the methane recovery process. As an alternative approach, co-production of hydrogen and methane via two-stage anaerobic digestion has been investigated.[164] $BioH_2$ was first produced in a dark fermenter. Subsequently, the liquid effluent consisting of mainly VFAs and alcohols was introduced into a methanogenic biofilm reactor as a preferred substrate. The total estimated energy recovery in the form of hydrogen was 28% of the initial COD, while the remainder was in the form of methane.

4. RECOVERY OF WASTE HEAT

4.1 Heat energy recovery using vapor-gap membranes

Low-grade heat at temperatures below 100 °C is often found in exhaust gases, waste liquid streams, and cooling water but is difficult to use in practice.[165,166] Power plants in the US discharge a total amount of 18 billion GJ yr^{-1}. Geothermal wells and solar collectors also have the potential to deliver huge quantities of heat. State-of-the-art technologies for converting heat to electricity are limited in their ability to efficiently use low-grade heat sources since the small temperature difference does not provide a sufficient driving force for efficient power generation.[166–168] Common heat engines rely on the steam Rankine cycle which conventionally requires temperatures above 550 °C, and variations of the Rankine cycle that use organic working fluids typically run at temperatures of >100 °C.[169,170] Thermo-electric generators have been developed over a decade to directly convert waste heat to electrical power, yet low efficiencies and high cost of the current materials have restricted their implementation for large-scale power generation.[167,171] Metal complexation reactions and electrochemical redox potentials created more opportunities for low-grade heat recovery, yet the efficiencies of these devices have been lower than 2% of the Carnot efficiency.[172]

VGMs were recently used to recover energy from low-grade heat.[34] Power generation is accomplished by using a temperature difference across a VGM to produce a partial vapor pressure difference that drives the working fluid across the membrane from a feed reservoir at ambient pressure to a pressurized permeate reservoir (Figure 1f). This flow increases liquid volume in a pressurized tank, and this pressurized liquid flow can be converted to electricity by depressurizing the flow in a hydro-turbine. Since the process relies on a vapor pressure difference, low temperature differences can be utilized so long as they generate sufficient driving force for water permeation. The process of converting low-grade heat to electricity has been called both thermo-osmotic energy conversion (TOEC) or pressure-retarded membrane distillation (PRMD) in the literature.[173] Throughout this review, we use the TOEC terminology.

Waste heat recovery systems using VGMs can be operated in both closed- and open-loop systems, both of which have potential advantages over conventional technologies.[34,174] While any working fluid can be used in principle, most research has focused on utilizing water as a working fluid for the process. The relatively high surface tension of water (72 mN m^{-1} at 25 °C) and the large availability of water sources make it well-suited for TOEC. Closed-loop processes circulate a working fluid within a system to generate power from an available temperature difference (Figure 5a). Closed-loop systems can potentially outcompete other systems for low-grade heat power generation since they can operate with low temperature heat sources (less than 100 °C), tolerate fluctuations in the heat source temperature, and because no toxic chemicals or materials are required for operation.[175] Furthermore, TOEC systems can utilize existing engineered components including pressure-tolerant membrane modules, pressure exchangers, and heat exchangers. Open-loop systems share many of the advantages of closed-processes but can operate such that both energy and water are recovered from the feed stream (Figure 5b). The open-loop system enables wastewater to be used as the feed stream. Non-volatile contaminants are rejected from the feed as the water passes through the membrane, producing purified permeate water.

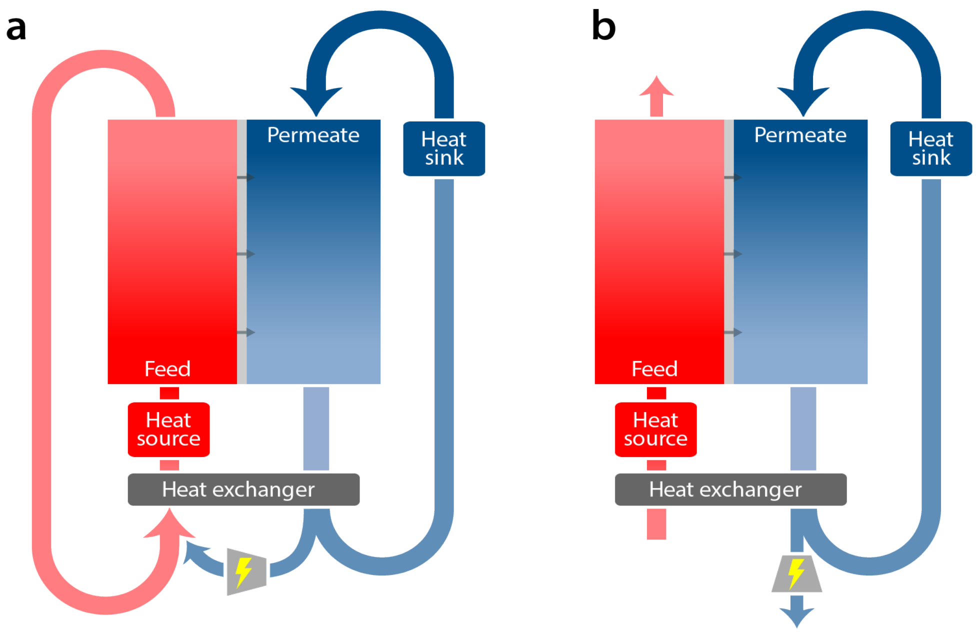

Figure 5. Thermo-osmotic energy conversion using (a) closed-loop and (b) open-loop configurations. Darker red and blue indicates hotter and colder working fluids, respectively.

4.2 Achievable power output and energy conversion efficiency

The competitiveness of VGM processes with existing power generation systems will ultimately be determined by their ability to extract energy more effectively than established alternatives. Studies have predominantly focused on two metrics to describe the performance of membrane-based power generation systems: power density and energy efficiency.[176] Power density is defined as the amount of power that can be extracted per area of the membrane. High power densities decrease the cost in the system by enabling higher power production with a smaller membrane area. The other key metric in power generation is the heat-to-electricity energy conversion efficiency, which can simply be defined by the work output of the process divided by the thermal energy input. The efficiency represents how effectively the available heat is converted to useful work. The upper limit of efficiency is defined by the Carnot efficiency.

Research efforts in low-grade heat power generation have largely focused on improving the power density achievable in small-scale membrane testing (Figure 6a).[173,177,178] In laboratory settings, the power density achievable is typically calculated as the product of the water flux and the pressure generated across the membrane. Therefore, membranes that can achieve high water fluxes and high operating pressures are considered desirable. Equation 3 shows that obtaining a high LEP requires (1) decreasing the pore size of the membrane and (2) increasing the hydrophobicity. Figure 6 summarizes the power densities and LEPs reached in the literature thus far. These data show that increasing the operating pressure generally leads to higher power densities.

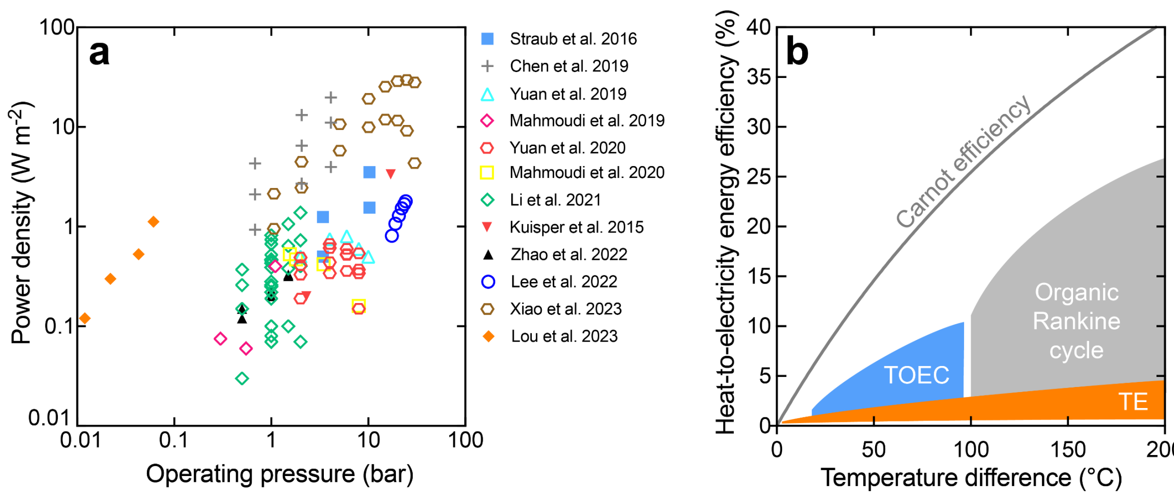

Figure 6. (a) Measured experimental power density of TOEC in the literature shown as a function of the operating pressure.[34,173,184,185,174,177–183] (b) Estimated heat-to-electricity energy conversion efficiency of thermo-osmotic energy conversion (TOEC), thermoelectrics (TE), and the organic Rankine cycle as a function of the temperature difference with a heat sink at 20 °C.[167,170,194–198,186–193] The Carnot efficiency, which represents the theoretical upper bound, is also shown.

In addition to a high operating pressure, high water fluxes are also critical to maximizing the achievable power density. Membrane properties must be tailored to achieve high water fluxes.[177] Generally, membranes with high porosity and low tortuosity are favorable for increased vapor permeability. Membranes must also be thermally insulating to prevent detrimental heat transfer across the membrane. Heat transfer results in temperature polarization that decreases the temperature difference across the membrane, ultimately reducing the flux and efficiency. The membrane thickness must therefore be optimized to be thin enough to maximize the water flux while also being thick enough to provide thermal insulation; simulation studies have found that an optimum membrane thickness is around 10–30 μm.[35] As the membrane resistance is dominated by diffusive barriers in TOEC (Equation 2), larger pore sizes (greater than 200 nm) are desired for high water fluxes. However, the benefit of increasing pore size to increase the vapor permeability must be balanced against the need for small pore sizes to maximize the LEP of the membrane (Equation 3). An ideal structure for TOEC is, therefore, an asymmetric membrane with two layers: a thin skin layer with a small pore size (less than 50 nm) to maximize LEP and a thicker support layer with a larger pore size to maximize the vapor permeability.[199]

Heat-to-electricity energy conversion efficiency largely depends on how heat is managed in the system.[35,183] Employing a heat exchanger is an effective strategy to recover the heat of vaporization and conductive heat transferred across the membrane. Under a hydraulic pressure of 50 bar, 60 ºC heat source, and 20 ºC heat sink, TOEC systems have been simulated to obtain peak energy conversion efficiency of 3.1% (34% of Carnot limit) with ideal heat recovery by heat exchanger (Figure 6b).[35] At the peak efficiency, the system has a low power density (1 W m^{-2}), thus, some energy efficiency needs to be sacrificed for high power density. Parasitic loads in the system such as the energy used for pumping also decline net energy output. In addition, minimizing temperature difference across the module and increasing hydraulic pressure difference are both necessary to improve the conversion efficiency. TOEC system has a higher projected energy efficiency than thermoelectric devices operating at low temperatures and can operate at lower temperatures than the more efficient organic Rankine cycle.[175]

4.3 Prospects and challenges for efficient power generation

Several challenges in terms of membrane design and heat management must be addressed for the closed-loop TOEC system to operate effectively. State-of-the-art commercial hydrophobic VGMs typically wet at applied pressure below 5 bar due to their relatively large pore sizes and pore size distributions.[34] Since power density and thermal efficiency both rely on high operating pressures, the low pressure tolerance limits process performance. Novel membranes with small, uniform pores and high liquid entry pressures need to be further developed. Low vapor permeability of the membranes can be enhanced by using an asymmetric design, increasing porosity, and lowering tortuosity. Recent advances in high flux and pressure tolerant materials for membrane distillation indicate promise in this area.[200] In addition, novel modification techniques are desired to lower surface energy, tailor membrane surface with anti-wettability, and improve its mechanical strength. Innovative heat recovery methods with heat exchangers or stacked modules are suggested to minimize heat losses and achieve high energy efficiency.[179]

Open-loop TOEC systems face similar challenges as closed-loop systems, but the use of saline water in feed reservoirs poses new challenges related to scaling and fouling.[185] Pre-treatment processes such as coagulation, filtration, and acidification can be integrated with TOEC system to remove fouling and scaling precursors. Antifouling coatings and specially designed membrane modules can also play a role in minimizing fouling.

5. RESEARCH NEEDS

5.1 Membrane wetting

Wetting is a universal challenge for processes using VGMs. The air layer created in the VGM can be compromised by pore wetting, severely reducing the selectivity of the system. VGMs for chemical and heat recovery experience harsh conditions that increase wetting propensity, such as exposure to low surface tension liquids (e.g., liquid-liquid MC for CH_4 recovery) or high pressure (e.g., heat recovery in TOEC). In comparison, VGMs for desalination through MD treat aqueous streams at near-ambient pressure. The principles of wetting were explained in Section 2.2. Below, we discuss the key mechanisms of pore wetting and possible mitigation strategies.

Wetting from low surface tension liquids and surfactants is of particular importance in resource recovery processes. Wastewater from a variety of sources contains low surface tension liquids and surfactants that are used to disperse oils or as cleaning agents.[201] Low surface tension liquids are known to cause instantaneous pore wetting.[24] Surfactants, on the other hand, have been shown to induce progressive pore wetting where surfactants decrease the surface tension of the feed solution and the wetting front advances through the pore.[202] In surfactant-induced wetting, the kinetics of pore wetting are determined by the rate at which surfactants can be transported to the wetting frontier.

To prevent pore wetting from surfactants and low surface tension liquids, pretreatment can be used to remove chemicals in the feed solution that cause pore wetting.[203] Pretreatment to remove surfactants can be carried out using traditional water treatment processes such as microfiltration/ultrafiltration, coagulation, and sand filtration. Such pretreatment is already employed in other membrane-based processes (e.g., reverse osmosis) but is considered undesirable since it adds to the cost and footprint of a resource recovery. However, continued research is needed to identify the cost-effectiveness of pretreatment for mitigating pore wetting in specific applications.

VGM modification using omniphobic coatings is also a promising method that has already shown success in preventing pore wetting for a variety of applications.[204–209] As was discussed in Section 2.2, surface omniphobicity can be attained via combining (1) a low surface energy and (2) a re-entrant structure.[24] Fabricating VGMs with robust omniphobic coatings has been the subject of active research in the membrane distillation field, and a variety of approaches have been used including nanoparticle coating and nanotexturing. The use of re-entrant structures can, in theory, prevent wetting with liquid of surface tensions down to 21 mN m^{-1}.[22] Experiments in the literature have been able to operate with low surface tension liquids including ethylene glycol, mineral oil, ethanol, dodecane and surfactants such as sodium dodecyl sulfate without observing pore wetting.[22,24,210]

Long-term loss in wetting resistance of VGMs remains a challenge. Chemical and physical changes during long-term operation can decrease the hydrophobicity of membranes.[211] Surface hydrophilization by the addition of polar functional group and morphology changes have been observed, resulting in decreases in the observed contact angle of water on the membrane surface.[212,213] However, long-term loss of wetting resistance has been poorly studied thus far in the literature, and more work is required to identify material properties that allow for long-term robustness and maintenance strategies that prevent long-term pore wetting.[214]

5.2 Membrane fouling

When exposed to suspended solids and organic compounds, VGMs are inevitably prone to fouling. The blockage of the membrane surface and pores results in a dramatic reduction of the flux of the target compound.[37] For instance, experiments of dissolved methane recovery from AnMBR and UASB effluent showed a 60-90% reduction of CH_4 flux within 40h of operation.[215] Membrane fouling can even induce pore wetting, potentially failing the recovery process.[24,216,217] The hydrophobic-hydrophobic attraction between the membrane and the organic compounds accelerates organic fouling.

Adding a hydrophilic layer onto VGMs (often called Janus membranes) has been investigated to reduce the foulant-membrane attraction.[15,24,27,218,219] Janus membrane fabrication can be achieved via several techniques such as dip-coating, plasma treatment, surface initiated radicalpolymerization, and initiated chemical vapor deposition[15] where a hydrophilic antifouling layer is grafted onto the wetting-resistant substrates. The tested hydrophilic materials are mainly polymeric or nanomaterials, including polyethylene glycol,[220,221] hydrogels,[25,222] polydopamine,[223–226] zwitterionic polymers,[27,227,228] graphene oxide,[229,230] and metal-oxide nanoparticles.[231–233]

While effective in slowing down the fouling and fouling-induced wetting, Janus membranes made of conventional hydrophobic substrate may still be susceptible to pore wetting when exposed to low surface tension liquids or surfactants. As discussed in Section 5.1, omniphobic membrane substrates maximize the benefit of the Janus membranes.[219] Nevertheless, the opposite wettabilities of the antifouling hydrophilic layer and the wetting-resistant substrate bear a potential risk of delamination in long-term MC operation.[234] Additionally, the added transfer resistance may slow down the recovery rate.[27,218] Future research needs to be directed to developing a stable Janus membrane without delamination, assessing its fouling resistance in a long-term operation, and optimizing the fouling-resistant layer thickness without compromising the flux of target compounds. As most studies on Janus membranes focus on membrane distillation for desalination, Janus membrane performance for recovering non-solvent resources also needs to be investigated.

In addition to engineering surface hydrophilicity, fouling mitigation has been attempted through the control of operating conditions. The impact of transmembrane water vapor flow on the fouling of vapor-gap membranes is noteworthy. Recent studies on dissolved methane and ammonia recovery showed that an absence of transmembrane vapor flows leads to remarkably fouling-free membrane surfaces, as convective flows that carry foulants toward the feed-membrane interface are obviated (Figure 7).[63,235] These observations render liquid-liquid MCs in an isothermal mode quite attractive in fouling control, at the cost of reduced flux compared to the non-isothermal mode. It should be noted that in the TOEC process transmembrane water vapor flow is inevitable and desirable, like in the MD desalination process. In that case, pretreatment of feed solution for foulant removal and surface-engineering to prevent fouling of the VGMs will be beneficial.

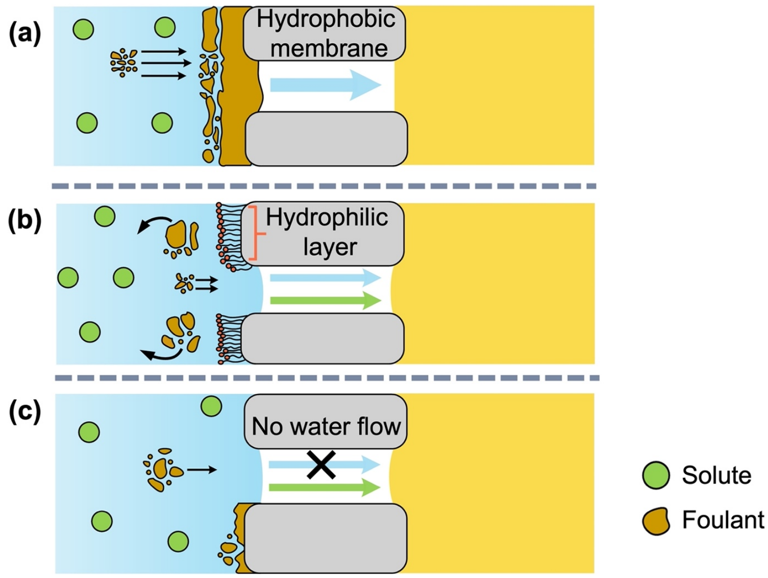

Figure 7. VGM fouling and mitigation mechanisms (a) Fouling occurring in the presence of organic foulants in feed water on a hydrophobic/omniphobic membrane surface. Two mechanisms for fouling tolerance of VGMs in MCs: (b) hydrophilic surface modification or coating on the membrane (Janus membrane) to impede the attachment of organic foulants, and (c) minimizing transmembrane water vapor flow under isothermal condition, obviating a convective transfer of organic foulants towards the membrane surface.

5.3. Recoverable resource quantities and qualities

Despite the effective demonstration of VGMs for volatile compound recovery, the biological production of target compounds in the wastewater interferes with and/or excludes other compounds in complex biological processes (Figure 8). For instance, anaerobic digestion aimed at CH_4 production from the carbonaceous organic matter also produces NH_3 from the biological breakdown of nitrogenous compounds. However, the NH_3 concentration needs to be maintained low due to its inhibitory effect on CH_4 production.[236] Similarly, CH_4 production must be suppressed for VFA and H_2 production in an anaerobic digester,[237] as hydrogen-producing microbes or acidogens need to dominate methanogens. Further, undissociated VFA molecules inhibit H_2 production.[237] Likewise, the production of N_2O via the CANDO process excludes NH_3 production. Therefore, the biological pathway taken in wastewater treatment determines the specific end products, and VGMs in a suitable MC configuration for the target resource will need to be implemented. On the other hand, thermal energy in wastewater can be extracted via TOEC independently from the dissolved chemical compounds, but higher temperature effluent is favorable for energy recovery efficiency.[34]

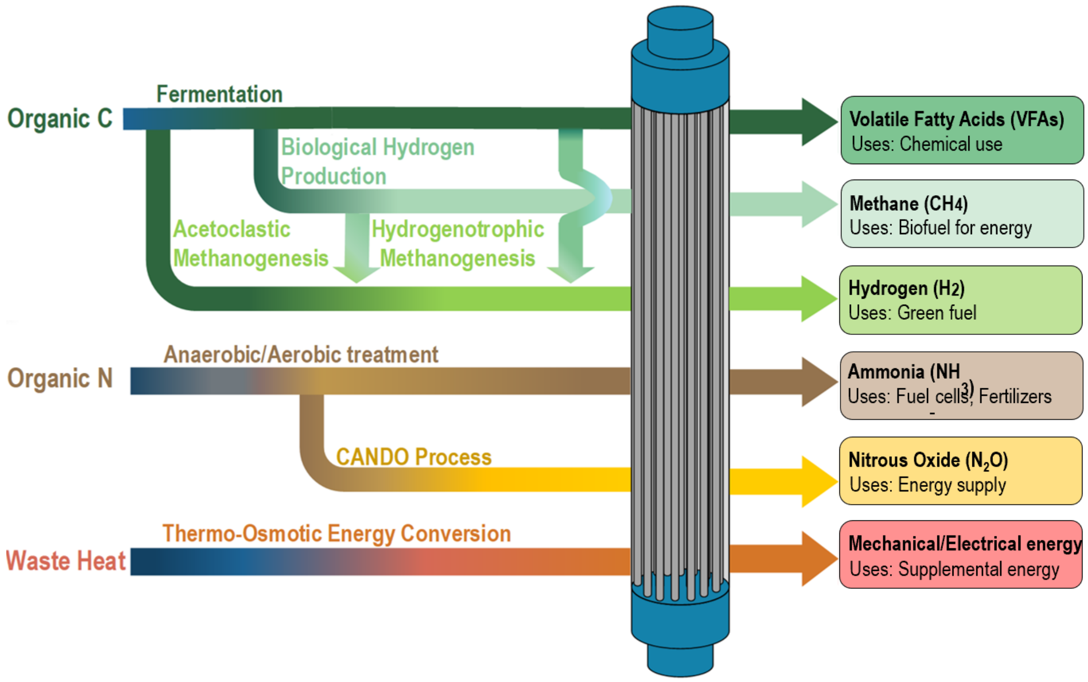

Figure 8. Schematic of possible resources that can be recovered from wastewater via different pathways. The complexity of resource removal from wastewater can be understood by the different biological or physiochemical pathways that limit the recovery of other resources listed in this study. The complexity of the anaerobic process can be understood by the various pathways that can be undertaken under the treatment scheme. The source of each product (left of contactor) and the final desired product post recovery (right of contactor) have been labelled schematically.

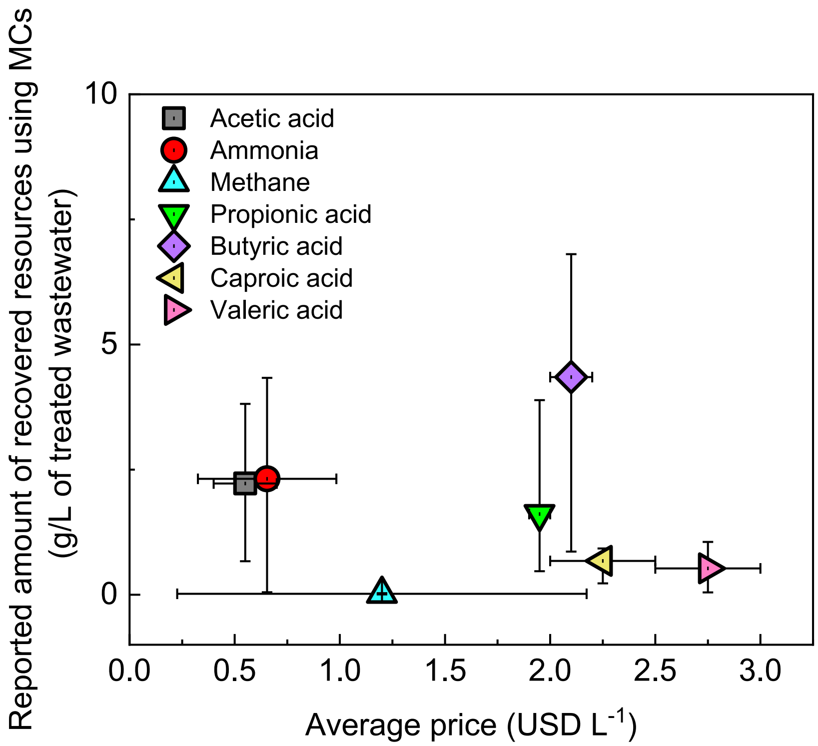

Figure 9. Reported amounts of resources recovered from a unit volume of wastewater using VGMs and their average market price per volume (liquid) based on the literature. The error bars in the market price indicate the lowest and highest values. The full list of references and detailed information on the reported recovery efficiency and feed wastewater concentration values[36,42,75,97,103,104,110,111,116,118–120,43,127,128,215,238,239,51,63,70–74] along with the market price data[240–242] are provided in supplementary material, Table S4.

The market potential and recoverable amounts are critical factors in determining the target resources and the recovery processes.[243] Municipal wastewater can potentially supplement up to 14% of the nitrogen fertilizer in Flanders, Belgium, and 1% of the energy market in the Netherlands by CH_4 recovery. An estimated total global market demand for acetic, butyric and propionic acids for the year 2020 was 18.5 Mt.[130] VFAs recovered from municipal wastewater can satisfy a market potential of up to 17% in the Netherlands.[7] Figure 9 shows recovered amounts of different dissolved compounds per given volume of wastewater using VGMs in the literature. The recovered methane is quite low in amount, as the methane solubility in water is low and dissolved methane accounts for a minor fraction compared to the biogas in the headspace of anaerobic digestors.[244] However, anaerobic treatment of low strength wastewater (e.g., municipal wastewater) at ambient temperature is an active area of research. Such processes will not only reduce the energy requirement for heating significantly, but also increase the total amount of recoverable methane due to its supersaturation and large volume of wastewater.[3,36,69,245] VFAs generally have a higher market price than methane, and high yield VFA production requires suppressed methanogenesis.[246] However, co-production of VFAs and biogas is also possible via continuous extraction of excess VFAs, while allowing for stable biogas production in anaerobic digestors.[247]

Ammonia in domestic wastewater, expected to increase to >35 Mt in its amount by the year 2050,[9] can be a supplementary source for ammonia production. However, the technologically mature Haber-Bosch process already produces ammonia at 0.3-0.5 USD L^{-1}, assuming its transportation as liquid.[9,248] Additionally, feed water of high ammonia concentration (>2000-3000 mg NH_4^+-N L^{-1}) is needed for profitable recovery processes.[9] Nevertheless, domestic wastewater and industrial wastewater of higher ammonia concentrations can serve as a more stable and sustainable source in comparison to the Haber-Bosch process, which largely depends on natural gas extraction and incurs 1-2% of global CO_2 emissions. The end products using VGMs are typically ammonium salts. Recovering ammonia as a gas through an additional downstream process may be more profitable owing to the twice higher market price than ammonium salts (ammonia price varies from 0.325-0.983 USD L^{-1}), and to its potential application as a hydrogen carrier.[9,240,249]

The total quantity of low-grade heat available motivates further development of VGM-based energy recovery from heat. Power plants in the US generated waste heat from cooling towers and flue gases with an estimated amount of 3,100 TWh yr^{-1}.[11] Geothermal energy can be exploited using temperature gradients varying from different geographical locations and geological formations. Despite the large reservoir of geothermal energy from Earth’s crust, the amount of power generation worldwide is limited to only 570-1200 TWh yr^{-1}.[250–252] Other sources of low grade-heat from manufacturing, solar collectors, and other sources have been more difficult to quantify, but can potentially supply a massive amount of additional energy. It is notable that the Carnot efficiency decreases with very small temperature differences, and thus lower quality heat sources will have a less efficient conversion to electricity.[35]

High recovery of the dissolved compounds requires large membrane surface/contact areas or multiple passes. Hollow-fiber membrane (HFM) modules offer a large interfacial area in a given volume, and have been tested for NH_3[104,111,116,253] and CH_4 recovery,[254] and for acetic acid at much smaller scales.[255] Nevertheless, pilot-scale or larger scale studies are scarce, and have yet to demonstrate the resource extraction in profitable amounts from wastewater streams.[3] While highly wetting-resistant membranes are ideal,[12,243] hollow-fiber omniphobic VGMs are not commercially available. A large-scale manufacturing of omniphobic VGMs is necessary to achieve fast and sustainable resource recovery in compact modules.[33] Some recent studies explored different approaches to fabricate hollow fiber Janus VGMs, using hydrophilic surface coating,[224] co-extrusion method,[256] and nonsolvent phase separation technique.[257] However, the fabrication of such membranes in HFM configuration as well as their performance evaluation for resource recovery via MC are yet to be investigated.

Determining the system configuration is crucial to realize cost-effective recovery. Even when the permeate is enriched with target compounds, other volatile species are inevitably transferred across the VGM into the permeate. Therefore, single or multiple post-processing steps may be required to finally obtain the target compound of high concentration and purity. For instance, CH_4 recovery in VMC would require scrubbing and condensation to remove CO_2 and water vapor, respectively, from the recovered gas mixture. Recovering CH_4 using a nonpolar permeate in the liquid-liquid MC (Section 3.1 and Figure 1e) would require downstream processing to release the gas from the permeate by applying a vacuum. While it has been estimated that this combination of vacuum and the liquid-liquid MC would be net energy positive,[36] this combined process is yet to be experimentally demonstrated.

During NH_3 recovery by VGMs, the gas solubilizes into the acid solution as a dissolved ammonium salt and requires downstream processing to concentrate the salt content and/or remove the excess acid.[123] These necessities are rarely mentioned in the literature but dewatering and pH readjustment would add significant downstream processing cost. Alternatively, MC in tandem with a bipolar membrane electrodialysis system can replace the chemical addition with renewable electricity and produce NH_3 gas, which has higher commercial value than ammonium salts (more than twice)[122] and has wider applicability, particularly in the hydrogen fuel cell industry.[79] Similarly, VFA recovery through liquid-liquid MC requires a permeate of high pH. After VFA extraction into the permeate, the VFA further needs to be separated from the permeate, and this solution needs to be regenerated for reuse. Electrochemical separation techniques for VFA separation have been applied for other applications such as removal from fermentation broths to enhance biofuel production.[134,258] However, the feasibility of combining these systems with MCs still needs to be investigated. Future techno-economic analyses need to focus on a combined life-cycle assessment (LCA) approach to estimate the cost of producing any of the desired resources from a point source to the finished product.

Existing systems can potentially be optimized to reduce the CAPEX and OPEX. CH_4 recovery using liquid-liquid MC can be optimized by selecting permeate solvents that have a large solubility contrast between CH_4 and other dissolved gases (e.g., CO_2). Additionally, as CH_4 is a poorly soluble gas in water, mass transfer resistance across VGMs is much smaller than those across the feed and permeate. This feature gives additional room for surface modification of VGMs to increase the CH_4 selectivity. It is worth noting that liquid-liquid MC for dissolved CH_4 recovery makes commercial sense only after low-strength wastewater is anaerobically treated at ambient or lower temperatures, as CH_4 produced from high COD wastewater in anaerobic bioreactors is mostly recovered from the headspace as biogas. For NH_3 recovery, it has been estimated that almost 60% of the costs in MCs arise from the chemical usage for pH adjustment.[259] These costs to the OPEX can be reduced by using anaerobically digested wastewater as feed or other high pH industrial wastewater. The acid strength of the permeate also determines the number of modules required for efficient NH_3 recovery and therefore needs to be featured in the CAPEX estimates.[55] VFA, H_2, and N_2O recovery using MCs are at the nascent stage and therefore further research needs to seek the understanding of the impact of VGM structures, MC operating parameters, and feed characteristics on the selectivity targeted resource and post processing costs.

While cost and quantity considerations are critical in determining the type of resource to recover and the process configuration, environmental benefits and regulations need to be factored into decision making. VGM-based recovery processes currently aim to complement sustainable resource management, rather than compete with existing state-of-the-art technologies. For example, it is infeasible to replace the Haber-Bosch process for ammonia. However, ammonia recovery in wastewater treatment plants can lead to both meeting the aquatic life ambient water quality criteria[260] and discharge regulations[261] while reducing the production demand from the Haber-Bosch process that heavily relies on fossil fuels[7,262]. Likewise, capturing dissolved CH_4 will be complementary to conventional anaerobic digesters, and can significantly reduce the GHG potential of wastewater treatment processes. Several actions and plans for intensifying resource recovery are also taking shape. For instance, the European Green Deal launched by the European Commission in December 2019, and the new Circular Economy Action Plan adopted in March 2020, emphasized resource recovery and GHG reduction from wastewater as a key component of sustainable resource management.[263] Various countries offer incentives (e.g., carbon credits) to wastewater treatment plants for emission reduction/methane recovery.[264,265] These drivers and incentives, along with the recovery cost, will need to be collectively considered to determine the priority and feasibility of resource compounds to recover.

6. OUTLOOK

A qualitative comparison among VGM-employed processes for volatile compound and heat recovery is provided in Table 1 (decision criteria provided in Table S3, supplementary material). In most cases, >90% recovery of volatile compounds is achievable. However, they all require downstream processing of varying degrees as the target compound will be captured into a mixture with other liquids, gases or impurities in the permeate. VGMs can potentially recover energy from heat at around 34% of the Carnot efficiency. Such recovery efficiencies are higher than competing low-grade heat technologies but must still be demonstrated experimentally. None of the MCs are robust to wetting in long-term operations, as commercial VGMs are not perfectly wetting-resistant. While membrane fouling is a persistent issue, it can be mitigated by imparting fouling-resistant surface properties onto VGMs or by precluding solvent flows. For scalability, SGMC and VMC modules are already commercially available, but more recently proposed MC processes for CH_4, VFA, and low-grade heat recovery lack pilot testing or suitable VGMs with large area.

The energy intensity of the various configurations (i.e., liquid-gas MC, liquid-liquid MC) for volatile resource recovery varies based on the desired product specifications and the feed stream characteristics. For VGM module operation, SGMC configurations generally exhibit a moderate to a high energy intensity due to sweep gas compression and circulation. The energy intensities in operation of VMC and liquid-liquid MC are generally lower, but an increased transmembrane water vapor transfer, altered form of target compounds in the permeate, or a stronger affinity of the target compounds to the permeate solvent will further add energy or chemical cost for purification in the post-processing step.

On one hand, an improved design of VGMs can certainly improve the wetting and fouling resistance, allowing for long-term process usability. On the other hand, examining the quantity and the quality of available resources determines the economic feasibility of the process and the overall system requirement, and provides incentives to develop suitable VGMs. For system level implementation, the modular nature of VGM-based recovery processes makes them easier to be adapted to the existing wastewater treatment facilities. Future wastewater resource recovery systems may employ multiple VGM modules in series to recover different resource compounds, and thermal energy may be independently recovered.

As wetting and fouling are persistent problems in all VGM-based processes, cost-effective production of omniphobic or hydrophobic/hydrophilic composite membranes (Janus membranes) in commercial scale is required. Also, it should be noted that most of the omniphobic VGMs in the literature are fabricated using perfluorinated chemicals. The tighter regulation on these chemicals motivates the development of non-toxic low surface energy materials. The recent efforts demonstrated a certain level of surface omniphobicity using non-fluorinated chemicals, albeit with less wetting resilience than long-chain fluorinated organosilanes.[266–268] Active research efforts on membrane distillation for desalination will certainly benefit omniphobic, fluorine-free VGM development.

The complex pathways of biological production make it necessary to discern the resource compounds that are profitable and sustainable. The target compounds in turn determine the required VGM characteristics, process configurations, and operating conditions and costs. In addition, nearly all mentioned processes require downstream processing, such as pH swing, to produce the target resource in a usable form. The increasing emphasis on electrified processes renders electrochemical processes, such as bipolar membrane electrodialysis, potentially to replace the chemical input with renewable electricity. A systematic investigation of the combined processes (i.e., MC and downstream process) that connects feed to final products will be necessary to ensure a continuous and energy-efficient recovery process. At the system level, rigorous LCA and total cost assessment (TCA) will guide decision-making on target resource compounds and suitable VGMs-based recovery processes.

ASSOCIATED CONTENT

Table S1: Summary table listing the experimental data of methane recovery via VGM-employed MCs, including information on flux and hydraulic retention time; Table S2: Literature data on VGM-utilized ammonia recovery, and associated feed temperature, total ammonia nitrogen concentrations, and mass transfer coefficient values; Table S3: The decision criteria behind the qualitative comparison of VGM-employed MCs for volatile compounds and heat recovery; Table S4: Reported recovery efficiencies of the volatile compounds and their concentrations in the feed wastewater along with the market prices.

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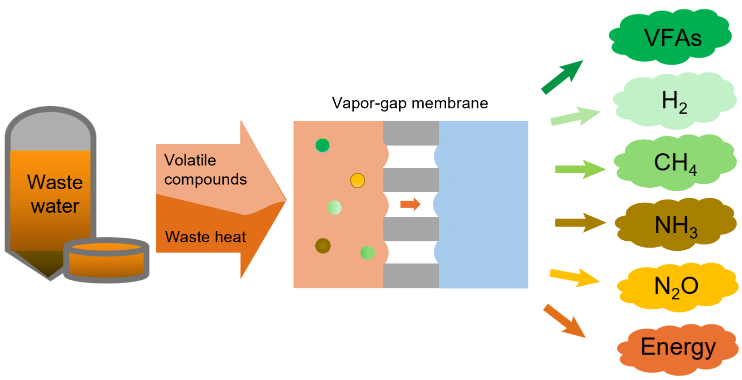

Table 1. A qualitative comparison of vapor gap membrane technologies for the resource recovery. Criteria descriptions are provided in Table S3, supplementary material.

Metrics	**Liquid-Gas MC**		**Liquid-Liquid MC**		
	Sweep Gas MC (SGMC)	Vacuum MC (VMC)	Aqueous liquid-Solvent MC	Aqueous liquid-Aqueous liquid MC (Isothermal)	Aqueous liquid-Aqueous liquid MC (Non-isothermal)
Target Resources	CH_4, NH_3, N_2O	CH_4, NH_3, H_2	CH_4	NH_3, VFA	Low-grade heat
Effectiveness of recovery	★★★	★★★	★★★	★★★	★★
Applicability as standalone process	★	★	★★	★★	★★★
Process robustness to wetting	★★	★	★	★★	★
Process robustness to fouling	★★	★	★★★	★★★	★
Scalability	★★★	★★	★	★★	★★

Legend: ★★★ Good; ★★ Fair; ★ Poor

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