Review

Factors influencing pressure-driven membrane-assisted volatile fatty acids recovery and purification-A review

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HIGHLIGHTS

- Approaches to volatile fatty acids recovery from organic waste sources are critically analyzed.
- A comprehensive discussion was provided on volatile fatty acids recovery using various techniques.
- Pressure-driven membrane filtration is revealed as the most promising way for recovery.
- The recovery efficiency is strongly affected by the membrane and medium properties.
- Operational strategies must be evaluated to boost volatile fatty acids purified concentration.

GRAPHICAL ABSTRACT
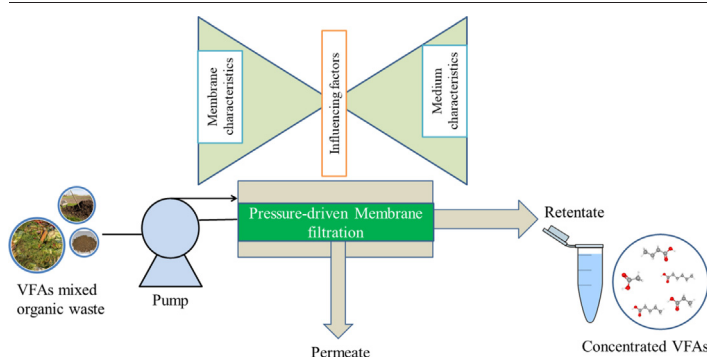
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ABSTRACT

Volatile fatty acids (VFAs) are building block chemicals that can be produced through bioconversion of organic waste streams via anaerobic digestion as intermediate products. Purified VFAs are applicable in a wide range of industrial applications such as food, textiles, cosmetics, pharmaceuticals etc. production. The present review focuses on VFAs recovery methods and technologies such as adsorption, distillation, extraction, gas stripping, esterification and membrane based techniques etc., while presenting a discussion of their pros and cons. Moreover, a great attention has been given to the recovery of VFAs through membrane filtration as a promising sustainable clarification, fractionation and concentration approach. In this regard, a thorough overview of factors affecting membrane filtration performance for VFAs recovery has been presented. Filtration techniques such as nanofiltration and reverse osmosis have shown to be capable of recovering over 90% of VFAs content from organic effluent steams, proving the direct effect of membrane materials/surface chemistry, pore size and solution pH in recovery success level. Overall, this review presents a new insight into challenges and potentials of membrane filtration for VFAs recovery based on the effects of factors such as operational parameters, membrane properties and effluent characteristics.

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1. Introduction

Globally an enormous amount of waste is generated on daily basis posing a serious threat to the stability socio-economic, environmental and health systems (Nizami et al., 2017). A considerable portion of these wastes is composed of organic compounds rich in nutrients which are conducive to be recovered in order to replace virgin resources. Therefore, in recent years, the scientific and industrial communities have shown great interest in different nutrient recovery approaches complying with the principles of a circular economy to build sustainable societies by turning waste into value-added products (Skaggs et al., 2018).

One of the established proficient nutrient recovery and waste remediation pathways is the anaerobic digestion (AD) process that has long been used for biogas and fertilizer production via microbial conversion of organic residues (Wainaina et al., 2020; Xu et al., 2018). Recent research has demonstrated that compounds of higher value and application diversity such as volatile fatty acids (VFAs), intermediate products of anaerobic digestion, could be accumulated and recovered as final products of AD rather than biogas. This is the basis of a potential platform for developing environment friendly and renewable biobased chemical precursors from waste rather than fossil sources (Chen et al., 2013). As an example, Liu et al. (2018) have reported that there is nearly 3-times higher net profit in VFAs (9.12 USD/m^3) production from sewdge sludge than biogas (3.71 USD/m^3). VFAs are short-chain saturated carboxylic acids containing six or fewer carbon atoms such as acetic, propionic and butyric acids. Other than fossil-based routes, VFAs can be produced in the midst of the anaerobic digestion process (acidogenesis and acetogenesis) using a wide range of municipal and industrial by-products, residuals and waste streams rich in organics such as food waste, agricultural residues, swedge sludge, microalgae etc. (Fig. 1a) (Parchami et al., 2020, Wainaina et al., 2019a, 2019b). VFAs are considered as very important chemical compounds for the synthesis of olefins, esters, alcohols and aldehydes. Besides, VFAs can be applied in many research areas and industrial sectors such as bioenergy production, bioplastic synthesis, microbial fuel cells, textiles, food, cosmetics, pharmaceutical industries and as a potential carbon source for biological nutrient removal in wastewater treatment plants (Bhatia and Yang, 2017; Luo et al., 2019; Mengmeng et al., 2009; Uyar et al., 2009; Zheng et al., 2010). Following these commercial applications clearly demonstrates the high market value and need for VFAs now and in the future (Fig. 1b).

Although, waste-based sources are one of the feasible platforms nowadays to achieve VFAs, it should be realized that anaerobically digested waste effluent is an unpurified complex mixture of VFAs, microorganisms, salts, proteins, lipids etc. that limit the application of the obtained solution as a purified VFA source. Moreover, in most cases, the high water content of the AD effluent results in a dilute VFAs solution (Battista et al., 2020; Da Ros et al., 2020; Zhang and Angelidaki, 2015). Besides, acid accumulation in anaerobic fermentation tends to inhibit the further production of VFAs, which means that the VFAs produced should be separated in real time (Yuan et al., 2019). In general, extraction of VFAs at high rate and yield from these mix liquid effluent is a challenging task. These obstacles can be tackled either through enhancement in productivity, product yield and concentration by fermentation and upstream optimizations or the application of practical and efficient separation and concentration processes (Masse et al., 2008).

The downstream or as called "post effluent treatment" for VFA recovery has gained substantial attention recently. Till present, several techniques such as adsorption (Reyhanitash et al., 2017), distillation and evaporation (Horiuchi et al., 2002), extraction (Katikaneni and Cheryan, 2002), electrodialysis (Strathmann, 2010) and pressure-driven membrane processes (Zacharof and Lovitt, 2012) have been implemented for VFAs recovery. Among them, membrane-based purification processes including microfiltration, ultrafiltration, nanofiltration, reverse osmosis and forward osmosis are regularly used to recover, purify and concentrate VFAs from mixed solutions. Membrane filtration offers many benefits such as the ease of customization and scalability, the possibility to apply for different effluents, low energy demand, minimal phase change during separation, separation specificity etc. Moreover, membrane separation technology can be integrated into any stage from fermentation to polishing the final product. The AD can be performed using anaerobic membrane bioreactors (AnMBRs) using pressure-driven membrane modules
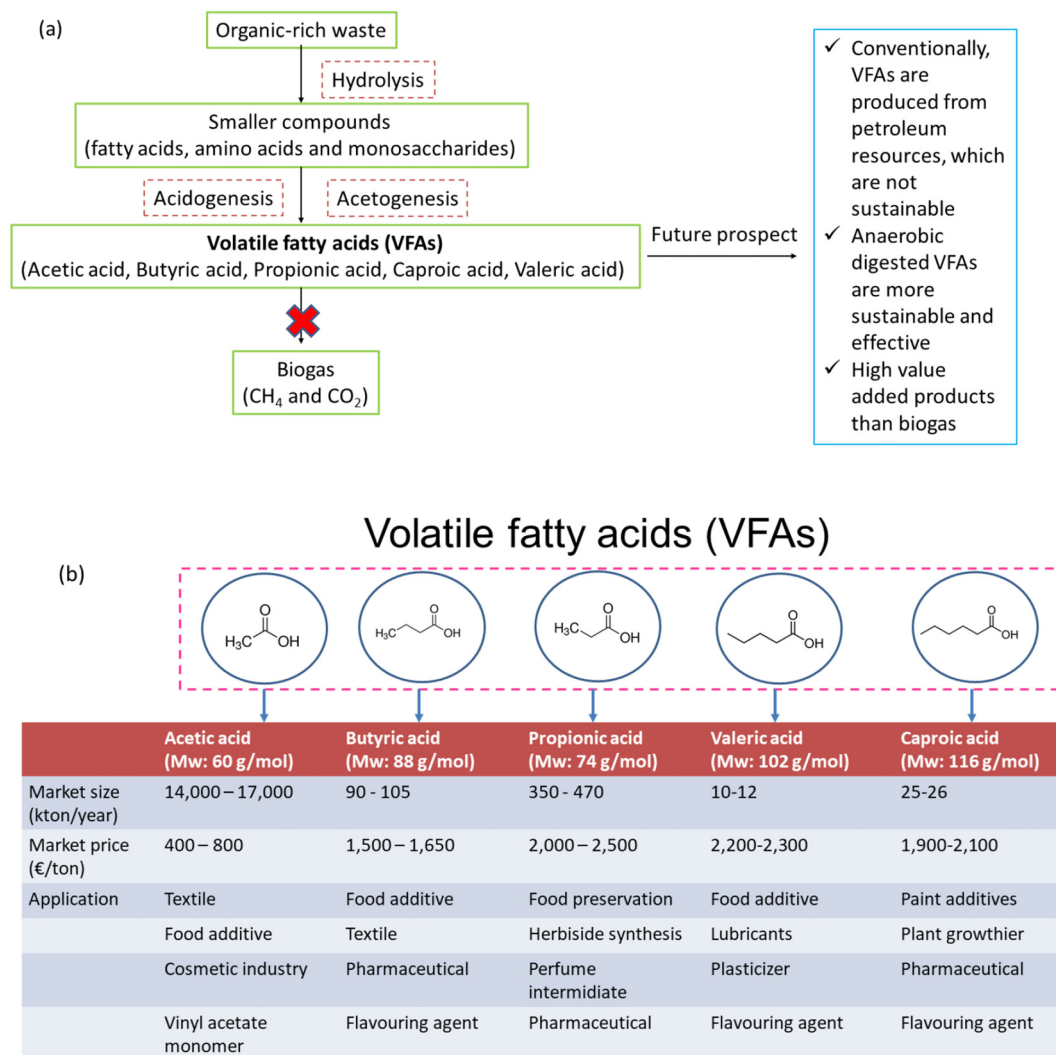
Fig. 1. (a) Production pathway of VFAs (Wainaina et al., 2019a, 2019b) and (b) application of VFAs with their market feasibility (Atasoy et al., 2018).

in order to produce microorganism and particle free VFAs-rich permeates (Trad et al., 2015; Wainaina et al., 2019a, 2019b). Following that, ultrafiltration can be used to remove macromolecules such as lipids and proteins (e.g. enzymes) from the effluent, and nanofiltration and reverse osmosis can be employed to remove monovalent and divalent salts, and excess water (Song et al., 2018, Van der Bruggen et al., 2003a, 2003b). However, membrane fouling and cleaning have always been the major hurdle in the industrial scale application of membrane separation processes.

In order to have a functional cascade of integrated membrane separation processes that assist optimal recovery, purification and concentration of VFAs from waste-derived AD effluents, it is of great importance to have a thorough knowledge of the factors affecting filtration operations. In order to guarantee long-term application of a pressure-driven membrane filtration system aimed at VFAs separation from complex media the effect of parameters such as membrane material, pore size, charge, hydrophilicity/phobicity, operating temperature, separation driving force (pressure, concentration and electrical potential), and effect of medium characteristics such as medium pH, solid content, EPS and SMP content and medium viscosity should be clearly understood and defined (Masse et al., 2010, Odey et al., 2019, Rajabzadeh et al., 2012, Zhou et al., 2013a, 2013b). However, to the knowledge of the authors, there are limited reports specifically on the factors influencing VFAs recovery from AD effluents.

Therefore, the main aim of this review is to present different techniques applied in VFAs recovery and purification, including a comprehensive analysis of the effect of determining factor involved in the membrane filtration

of AD effluents for VFAs recovery. In this regard, the advantages and shortcomings of different existing VFAs separation methods are discussed. Particularly, pressure-driven membrane filtration was found to be the most effective and sustainable technique for VFAs recovery and purification. Accordingly, individual parameter such as membrane material, pore size, charge, operating temperature, medium pH and solid content, and their extent of impact on VFAs recovery have been analyzed in a systematic manner, which is the first time in the literature according to the author's knowledge.

2. VFA recovery methods

2.1. Adsorption

Adsorption has been employed to capture the protonated form of diluted VFAs (neutral) from complex aqueous solutions. Usually, this process occurs through the interaction between a solid adsorbent surface (resin, activated carbon etc.) and adsorbate (VFAs molecules etc.) (Tung and King, 1994). In a recent study, Tonucci et al. (2020) synthesized hybrid imprinted polymer-based adsorbent and tested it for the adsorption of VFAs from anaerobic effluents. Results showed that maximum VFAs adsorption capacity of $Q_e \sim 50$ mg/g (Qe is the amount of VFAs adsorbed) was obtained and efficiently regenerated after the selective adsorption process. In this regard, the adsorbent's surface structure plays a critical role for the uptake of VFAs. The basic groups of adsorbent surfaces (alkaline surfaces) have

more affinity to carboxylic acids because of the acid-base interactions but opposite results were achieved for acidic groups (El-Sayed and Bandosz, 2004). Therefore, several different types of materials, such as ion exchange resins and activated carbon, have been used for the adsorption of VFAs (Fufachev et al., 2020; Talebi et al., 2020).

Ion exchange materials could be applied together with the adsorption process in order to facilitate the recovery process whereby making a strong bond between the functional group of the ion exchange material and the ionized acid (Garcia and King, 1989; López-Garzón and Straathof, 2014). Among them, ion exchange resins have been using frequently due to their high surface area and the macroporous surface where the functional groups are mainly located. This could assist the diffusion mechanism during the adsorption process which is connected to the mass transfer characteristics scenario. In general, the adsorption process is regulated by the diffusion rate and the corresponding resin surface can act as supporting material (Fargues et al., 2010; Kawabata et al., 1981). The rate of diffusion is mainly controlled by three scenarios such as adsorption of VFAs molecules on the surface, penetration of VFAs molecules in the surface and diffusion through the resin surface (Anderson et al., 1968; Smithells et al., 1936).

Anasthas et al. (Anasthas and Gaikar, 2001) used quaternary amino functional groups based ion exchange resin for the adsorption of acetic acid in non-aqueous phases. The sorption process interacted through the H-bonded complex formation between the functional group of resin and proton molecule of acid with the adsorption isotherm data being well fitted to the Langmuir model. Reyhanitash et al. (2017) recovered VFAs from the fermented artificial wastewater by employing four types of resin-based adsorbents, which can be categorized as follows primary, secondary, tertiary amine-functionalized, and nonfunctionalized structured polystyrene-divinylbenzene resins. Initially, they conducted a batch adsorbent screening tests and found that non-functionalized adsorbent endows better adsorption capacity, while the functionalized adsorbents showed higher affinity to mineral acids. Later, they investigated a column test in the presence of non-functionalized adsorbent to determine the concentration profile of VFAs. Total recovery profile of butyric acid was enhanced ranging from 0.25 wt% to 91 wt% evaporated through a temperature-based fractionation of VFAs molecules. In the case of regeneration performance, nonfunctionalized adsorbent could be stable up to four adsorption − desorption cycles, which are of economic importance when large scale VFAs recovery using the adsorption process is targeted (Fig. 2). Rebecchi et al. (2016) explored the VFAs recovery efficiency from an actual VFAs-rich effluent using ion exchange resins based adsorbents. Four different resins were used for the adsorption experimental purposes including primary amine, tertiary amine and quaternary amine. Results showed that tertiary amine (Ambelyst A21) resin prevails adsorption capacity from grape pomace effluent (76%) and synthetic VFAs mixtures (85%), respectively, and interestingly this resin has a lower price compared to others. The adsorption

capacity of Ambelyst A21 resin was also successfully investigated in previous studies (Fargues et al., 2010; Li et al., 2009; López-Garzón and Straathof, 2014). The desorption results were carried out with ethanol and NaOH solvents evaporation technique and ultimately around 97% of the VFAs were recovered on the desorbed surface. This work indicated that the Amberlyst A21 resin could be used for further VFAs adsorption studies from anaerobically digested effluent in an innovative approach.

In a comparative study considering activated carbon and resins for the adsorption of VFAs, the resin demonstrated a superior mixed acid adsorption capacity (74%) over activated carbon (63%) (Da Silva and Miranda, 2013; Uslu et al., 2010; Yousuf et al., 2016). The reason can be explained that the hydrophobic nature of the resin Amberlite IRA-67, attracts more acids under the pKa value of the acids, which makes them better adsorbent than activated carbon (Nielsen et al., 2010). Similarly, Eregowda et al. (2020) conducted VFAs adsorption experiment in a batch system through anion exchange resins and compared it with that of activated carbon. This study utilized 11 anion exchange resins. Results showed that Amberlite IRA-67 adsorption capacity was fitted by the Freundlich model, which means the adsorption process is related to multilayer adsorption. On the other hand, both the Langmuir and Freundlich model fitted the Dowex Optipore L-493 adsorption, resulting in a monolayer and multilayer related adsorption process, respectively (Saadi et al., 2015) These studies indicate that the resin-based adsorbents are preferential for VFAs adsorption purposes.

Although the adsorption process has been considered as an efficient technique for the recovery of VFAs, cost associated with the adsorption process are considerable when commercial adsorbents are needed to be recycled or regenerated (Bélafi-Bakó et al., 2004). Additionally, the presence of competing ions such as phosphate, sulphate and chloride are responsible for poor adsorption efficiency in VFA solutions resulting from the fermentation of complex organic wastes.

2.2. Distillation

Distillation is a fundamental physical separation and purification process of the components or substances from a liquid mixture. This process occurs when a liquid sample is evaporated using various boiling points and turned into a condenser through a distilled column to capture the produced vapors. Usually, the components or substances may be fully or partially separated and purified from the mixture (Lei et al., 2003; Qi et al., 2019). The distillation technique has been used commonly to separate VFAs from various media (Demiral and Ercengiz Yildirim, 2003; Petersen et al., 2018). Regarding the application of distillation for VFA recovery, it is recommended to use distillation for the separation of VFAs from low concentration effluents as the efficiency of the VFAs separation deteriorates as highly concentrated VFA solutions reach the azeotropic point (Huang
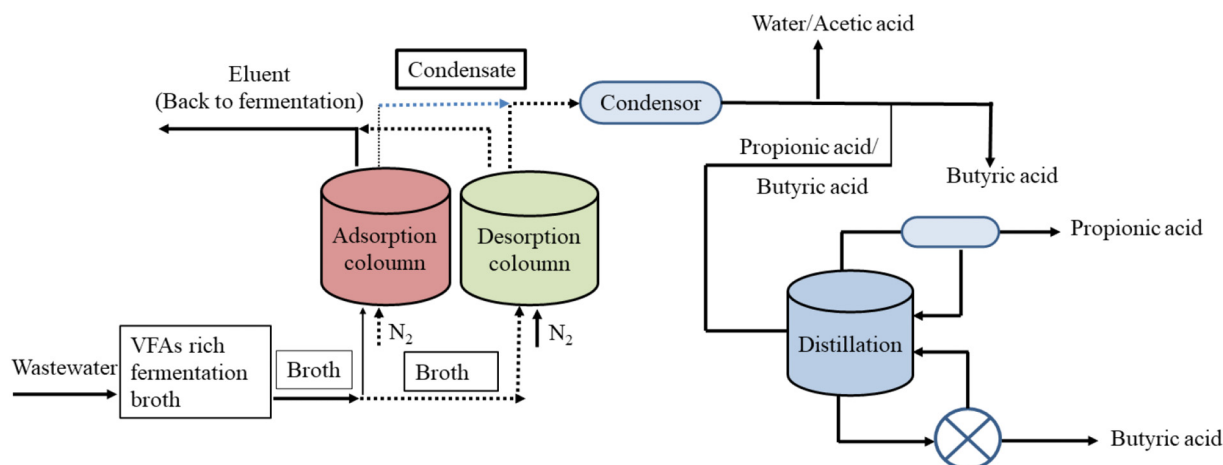

Fig. 2. VFAs recovery by adsorption process (Reyhanitash et al., 2017).

et al., 2008). For VFAs, the boiling point is considerably higher than water due to the fact that the structure of VFAs contains electrophile such as carbonyl groups (Reyhanitash et al., 2019). Various distillation approaches have been implemented to recover VFAs. Demiral et al. (Demiral and Ercengiz Yildirim, 2003) used the extractive distillation process for VFAs recovery from waste streams. They employed two solvents (adiponitrile and sulfolane) with a higher boiling point as the distillation process requires solvent-based substances to become more effective. So far, reactive distillation approach has identified as a promising technique for acid recovery (Gangadwala et al., 2008; Komesu et al., 2015; Painer et al., 2015). In this regard, Singh et al. (2007, 2006) studied the effect of various parameters on acetic acid recovery reaching an experimental recovery of 80%. Regarding reactive distillation, Saha et al. (2000) suggested that the recovery efficiency of acetic acid could be even further enhanced in the presence of an esterification distillation column. Butyric acid has reported being successfully recovered up to 89% through an ionic liquid induce short-path distillation process. This process effectively removed all the salt content resulting in the final product being mainly acids (Blahušiak et al., 2012).

Generally, the distillation process requires two steps: the effluent stream dewatering and organic acid concentration. However, as the final polishing step, the remaining acid may be recovered by evaporation (Wasewar et al., 2002). Literature shows that the distillation based purification can open new processing possibilities for VFAs recovery. This process also has some disadvantages, like water is the main component of the fermentation broth with less boiling point than VFAs components, requiring a higher amount of energy, which is not economical. In addition, the concentration step is also time-consuming in the distillation process.

2.3. Precipitation

Precipitation is a conventional method that is usually applied to separate compounds from a mixture. Among them, the calcium-based precipitation process is commonly used to recover VFAs and four steps are required to obtain the final product including, a certain amount of $Ca(OH)_2$ or $CaCO_3$ added to the filtered liquid of fermentation broth under mixing conditions, then, calcium salts of VFAs have filtered away from the original aqueous liquid and exposed to H_2SO_4 in order to release the desired amount of VFAs and finally purification added for obtaining a pure form of VFAs components (Min et al., 2011). Although environmental issues accompany the calcium precipitation process, the processing costs are higher since a large amount of H_2SO_4 and lime needed during the operation eventually increased the production budget (Wasewar et al., 2003). Followingly, King et al. (King, 1992) conducted a three-step process including extraction by solid solvent, dewatering to precipitate the acids and the collection of the acid conversion products in order to remove VFAs from water. VFAs can also be separated by using ammonia-based titration precipitating agents (Berglund et al., 1999). Moreover, the production of unwanted byproduct such as solid waste of calcium sulphate during the precipitation process has also hindered its application (Zacharof and Lovitt, 2013).

2.4. Esterification

Another widely used VFAs recovery method is esterification. The benefits of esterification have been harvested in applications such as biodiesel production, VFA-ester compounds for fragrance and scents industry, and production of ammonium sulphate as fertilizer (Ishihara, 2009; Katikaneni and Cheryan, 2002; Plácido and Zhang, 2018). The esterification process produces ester products using an equilibrium reaction between two reactants, such as alcohols and acids. This process generally occurs in the presence of an acid catalyst and heat. Recovery of carboxylic acids using the esterification process has long been a popular method. Neumann et al. (Neumann and Sasson, 1984) used methanol esterification method for the recovery of dilute acetic acid as methyl acetate in a chemorectification column with an organic solid polymer catalyst. The reaction process was kinetically fitted with second-order for acetic acid and zero-order for methanol. One study showed that the addition of salt ($CaCl_2$) promoted the reaction yield when the polystyrene-supported

solid acid catalyst was used under ethanol esterification (Yagyu et al., 2013). Accordingly, it was found that diarylammonium salt could catalyze the direct esterification process, which helps suppress solubility in water due to its hydrophobic nature and facilitates acetic acid recovery. Moreover, the esterification process occurred in a flow reaction system that can be pertinent to the industrial-scale operation (Igarashi et al., 2012). On the other hand, Bianchi et al. (2003) implemented two types of experimental protocols for acetic acid esterification with alcohol: a complete evaporation condensations reaction and distillation. They have reported that the latter method was most favorable for a high amount of acetic acid recovery. Recovery of acetic acid by methyl esters esterification from the real anaerobic fermentation broth was examined by Plácido et al. (Plácido and Zhang, 2018) and stated that 50% of acetic acid could be recovered with a VFA concentration of 800 gVFA/L while less than 10% observed at 500 gVFA/L. Besides, they noticed that ammonium sulphate salt was produced as a byproduct due to acidification of the fermentation broth that influenced plants economic growth. However, the downside is that esterification can be inhibited in the presence of a large amount of water. The adjustment of pH also can affect esterification potential as it is reported that the recovery of acetic acid without pH control (pH at about 6.5) resulted in low recovery yields of 5–20% (Horiuchi et al., 2002).

2.5. Extraction

Extraction is a well-established technique whereby two different immiscible liquids, such as water (polar compound) and an organic solvent (nonpolar), are in contact with one another phases during the operation upon their relative solubilities. Besides, a net transfer reaction is observed from one phase to the other driven by chemical potential. After completing the transfer process, the solvent product is enriched with solute(s), which is known as extract substance (Kertes et al., 2009a, 2009b). The extraction process has been known for more than a century to recover VFAs compounds effectively (Kertes et al., 2009a, 2009b; Mostafa, 1999).

Benefiting from the extraction techniques, three extraction processes, including solvent extraction, reactive extraction and ionic liquids extraction, have been accomplished to recover VFAs so far (Fig. 3). Solvent extraction is an efficient and economical process and uses various kinds of solvents in the aqueous phase. For instance, the use of trioctylphosphine oxide (TOPO) solvent allows an acetic acid recovery yield of 75%. TOPO can make a strong hydrogen bond with carboxylic acids in the organic phase due to its low solubility, high boiling point and good stability, and as a consequence, result in an extract phase with a higher amount of carboxylic acids (Golob et al., 1981). In another variation of recovery sources of VFAs within the solvent extraction approach, Shin et al. (2009) introduced waste-derived streams sources along with an organic solvent (2-ethylhexyl alcohol) and successfully, 96.3% of acetic acid was recovered. Consistent with the above studies, several studies are also recovered acetic acid using a synthetic solvent (Cebreiros et al., 2017; Chen et al., 2016; Kim et al., 2016). However, recovery of VFAs by a green solvent in lieu of synthetic toxic solvents is of interest from the point of sustainable development goals (SDGs), for this, a recent study (Rodríguez-Llorente et al., 2019) proposed a sustainable route for the recovery of VFAs from aqueous solution using natural solvents. Results showed that valeric and butyric acids extraction yield percentages were higher. Additionally, the authors conducted solvent stability studies by using an alkali medium.

On the other hand, the reactive extraction process also separates VFAs compounds from aqueous solutions using several extractants. Rasrendra et al. (2011) investigated the VFAs extraction efficiency from the phase splitted pyrolysis oil aqueous solution in the presence of tri-n-octylamine (TOA) and yield of 84% for acetic acid, while the extraction efficiency of acetic acid increased to more than 90% using aliphatic tertiary amines reactive extraction process (Mahfud et al., 2008). Furthermore, there is considerable research that uses triisooctylamine (Yang et al., 2013), tri-butyl phosphate (Eda et al., 2017), trioctylamine/Octanol (Ahsan et al., 2013) and tributyl phosphate (Mukherjee and Munshi, 2020) in the reactive extraction process as effective extractants for a high amount of VFAs recovery.
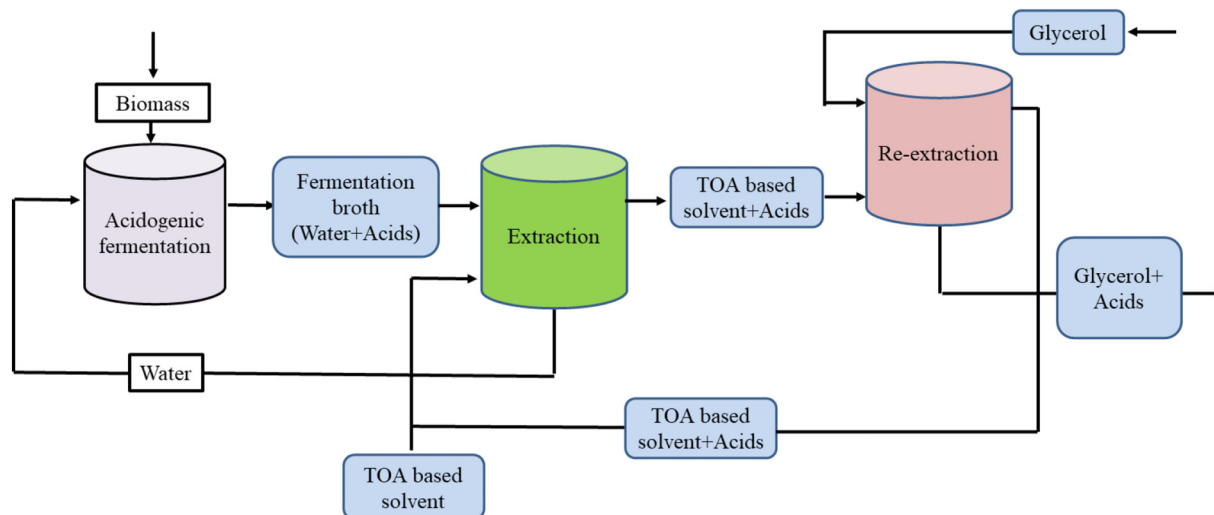
Fig. 3. VFAs recovery by the solvent extraction process (Aghapour Aktij et al., 2020).

Finally, the extraction process has been amplified by the use of ionic liquids (ILs)-based extractants towards VFAs recovery due to their outstanding separation efficiencies, good stability and, more importantly, well fitted for the environment (Cevasco and Chiappe, 2014; López-Porfiri et al., 2020; Şahin and Kurtulbaş, 2020; Sprakel and Schuur, 2019; Wang et al., 2017). After that, Schuur's group published some potential results of acetic acid recovery by using ionic liquids medium, and they also confirmed that the extraction process is influenced hydrogen bond (Reyhanitash et al., 2019; Reyhanitash et al., 2015; Reyhanitash et al., 2016). Recently, oliveira and co-workers (Oliveira et al., 2012) have proved that phosphonium-based ILs are more amendable than traditional organic solvents in terms of VFAs recovery performances. Despite the progress of the extraction process, this process depends on several factors, including operational parameters, raw materials availability and selection of appropriate solvents, etc. These issues must be amended constructively in order to achieve maximum output.

2.6. Gas stripping

Gas stripping is a relatively simple process, which involves gas sparging via the fermentation broth to transfer volatile compounds from the liquid state to the gaseous state to obtain the recovered products. This process has maintained Henry's law related to liquid and gas-phases concentration behaviour (Atasoy et al., 2018; Qureshi and Blaschek, 2001).

The recovery of VFAs compound from glucose fed meat anaerobic digester using gas stripping has been investigated by Li et al. (2015). This work conditioned their inoculum (anaerobic sludge) under an acidic medium (pH kept below 4.8), which favors VFAs accumulation since, at this pH, all VFAs compounds are found in undissociated form, and it is reported that undissociated form of VFAs must be present in the stripping process to be effective (Ramos-Suarez et al., 2021). Results showed that at an acidic medium, the compounds of VFAs were recovered in the form of their salts with an amount of 80% butyrate and 20% acetate while only a minor portion of propionate and valerate. Interestingly, this study also demonstrated that the reduction of pH trend promoted lactic acid conversion to other valuable VFAs compounds during the fermentation process. Furthermore, a recent study (Huang et al., 2016) has shown that the gas stripping process can also be employed in another way rather than ammonia stripping directly from VFAs-riched liquid digested of swine manure using the solid-liquid separation technique. Afterwards, the total concentration of VFAs was 94.4 mg-COD/g-VS, which is promising in terms of the marketable demand for VFAs. However, the gas stripping process faces some challenges, such as needed a large volume of gases for circulation and subsequent recovery steps for adsorbents/condensers (Qureshi et al., 2014).

2.7. Membrane contactor

Membrane processes have attracted significant interest in the field of product recovery from waste-based resources due to their advantages over other extraction approaches in efficiency, time and energy-saving and sustainability (Abels et al., 2013; He et al., 2012, Hube et al., 2020, Mahboubi et al., 2016, Pervez et al., 2020a, Pervez and Stylios, 2018, Pervez et al., 2020b, Shi et al., 2018a, 2018b). Various membrane processes including membrane contactor, forward osmosis, membrane distillation, pervaporation, electrodialysis, microfiltration, ultrafiltration, nanofiltration and reverse osmosis have been utilized at different stages of VFAs recovery (He et al., 2012; Rongwong et al., 2018).

Membrane contactor (MC) is an effective technique when it comes to the recovery of valuable compounds. This method realizes the separation of compounds into two phases while preventing their mixing. In other words a membrane brings two phases in contact through which the mass transfer between the phases occurs (Asfand and Bourouis, 2015). This method is mainly affected by the liquid phase characteristics (aqueous or organic) and the membrane surface properties (hydrophobicity, pose size or charge) (Drioli et al., 2005). In general, hydrophobic membranes are more preferential for resource recovery because of their good thermal stability and chemical resistance, also low energy required and large scale mass transfer ratios (Rongwong and Goh, 2020; Zhang et al., 2009). The application of MC for the recovery of VFAs has been practiced for a century. Tugtas (2014) used a membrane contactor for VFAs recovery in the presence of a flat membrane. Results highlighted higher selectivity of acetic acid (1.599) over water with separation occurring at a mass flux of 12.23 g/(m²h). In MC system, separation of VFAs can be affected by the medium pH. In this regard, Yesil et al. (2014) conducted the VFAs recovery experiments using polytetrafluoroethylene (PTFE) membrane contactor and they reported that higher selectivity (permeation fluxes) of VFAs could be obtained at lower pH. Among the VFAs considered, caproic acid showed better selectivity compared to other acids. Membrane contactors can be also utilized to bring gas-liquid or gas-gas phases in contact. A recent study by Aydin [145] used vapour permeation membrane contactor (VPMC) to recover mixed VFAs. They utilized three VPMC configurations including air-filled PTFE membrane with the other two being extractants (TOA or TDDA) filled PTFE membrane. The recovery percentage of VFAs was reported higher for extractants filled PTFE membrane except for acetic acid. In addition, TOA-filled membrane has also demonstrated a higher selectivity towards caproic acids, butyric acids and valeric acids (Rongwong and Goh, 2020). However, MC VFAs separation can be challenged by medium properties. It is noteworthy that the feed containing suspended particles result in lower VFAs recovery percentages (Yesil et al., 2014) and

therefore suspended particles-free feed solution are the target of MC VFAs separation.

2.8. Membrane distillation

Membrane distillation (MD) is a thermally driven process whereby the vapour molecules are transported across a hydrophobic membrane. The hydrophobic property of the membrane used in the MD process allows passing gases and vapors through the membrane surface while preventing liquid transport (Fig. 4) (Song et al., 2007). MD has various advantages over other thermal membrane separation processes such as high concentration of compounds could be retained as retentate, lower temperature requirements, larger membrane pore size and operation at atmospheric pressure. More importantly, fouling issues of the MD process could be minimized using suitable cleaning protocols since it is in direct contact with fermentation broth, while the processes such as membrane filtration or PV needed liquid steam pretreatment procedure to prevent the fouling scale (Gryta, 2008; Guillen-Burrieza et al., 2014).

An anaerobic membrane distillation bioreactor with polyvinylidene fluoride (PVDF)-based hydrophobic membrane has been utilized to treat synthetic wastewater that contained acetic acid, propionic acid, butyric acid and isovaleric acid (Yao et al., 2019). The results showed that propionic acid, butyric acid and isovaleric acid were successfully recovered in terms of higher concentrations obtained (1.3, 20.3 and 15.6 mg/L) in the permeate solution than feed solution (1.1, 16.7 and 12.6 mg/L) except for acetic acid, which is associated with higher boiling points than acetic acid that may facilitate transfer rate through the membrane surface during the operation, resulting in a higher recovery rate. Besides, they found that higher temperature showed a negative effect on recovery rate and membrane fouling phenomena. As it is known that the MD process is a thermal membrane separation process, so using low temperature during the operation possibly increased their reliable uses from the point of economic and energy-intensive perspective (Fasahati and Liu, 2014). Another study also used low temperature (35 °C) to separate arsenic from glycerol fermentation broth in the MD process. They utilized polypropylene membranes and exhibited satisfactory resistance to wettability during the processing stages. However, fouling issues are still noticed in their MD process, and surely this is one of the potential challenges for the future progress of MD (Gryta et al., 2013).

2.9. Electrodialysis

The use of electrodialysis (ED) has been widely investigated in the effluent treatment process to recover valuable compounds. The ED process is formed of a separation an ion-exchange membrane where an electric field is supplied for the migration of the anions and cations to the anode and cathode chambers (Fig. 5) (Vertova et al., 2009; Ward et al., 2018). It is often suggested that the charged form of VFAs are more suitable for recovery processing stages, therefore, a series of charged membranes are placed in between two electrodes to prevent the Donnan repulsion in the ED process (Huang et al., 2007; Wang et al., 2006).

Generally, the conventional electrodialysis (CED) process has been effective for acetic acid recovery than other VFAs compounds (Jones et al., 2015; Pan et al., 2018). Later a study carried out by Tao and co-workers who showed that MF pretreatment could enhance the CED process recovery efficiency with an amount of 92% acetic acid and 85% n-valeric acid. They also found that concentration increased from 11.73 g/L to 19.82 g/L (Tao et al., 2016), which agrees with other studies that also showed that higher concentrated VFAs compounds were achieved after electrodialysis (Bermeo et al., 2003; Scoma et al., 2016). These results attributed to the lower concentrations of VFAs in the initial feed solution, but these concentrations are very low in view of the practical aspects, and it has been proposed to use 200–500 g/L of VFAs concentration as a feedstock in the feed solution (Ramos-Suarez et al., 2021).

Electrodialysis with bipolar membrane (EDBM) demonstrated as an environment-friendly and energy-efficient technology. Bipolar membranes are typically ion-exchange membranes together with anion and cation exchange through the use of a junction layer in their laminated structures (Huang and Xu, 2006; Pärnamäe et al., 2021). In some cases, cation exchange membranes are not needed in the direct recovery system using the EDBM process; applying only an anion exchange process, it is possible to recover up to 70% of acetic acid that may save energy consumption and operating costs (Yu et al., 2000). On the other hand, the efficiency of the EDBM process could be increased using a two-stage operation, around 87% of VFAs were recovered from real pig manure hydrolysate (Shi et al., 2018a, 2018b).

Another important design is the placement of the ED unit in direct contact with the fermentation broth for in-situ recovery of VFAs. Dai et al. (2019) have discussed the feasibility of this alternative integrated system. By coupling the ED unit into fermentation broth, the recovery rate of acetic
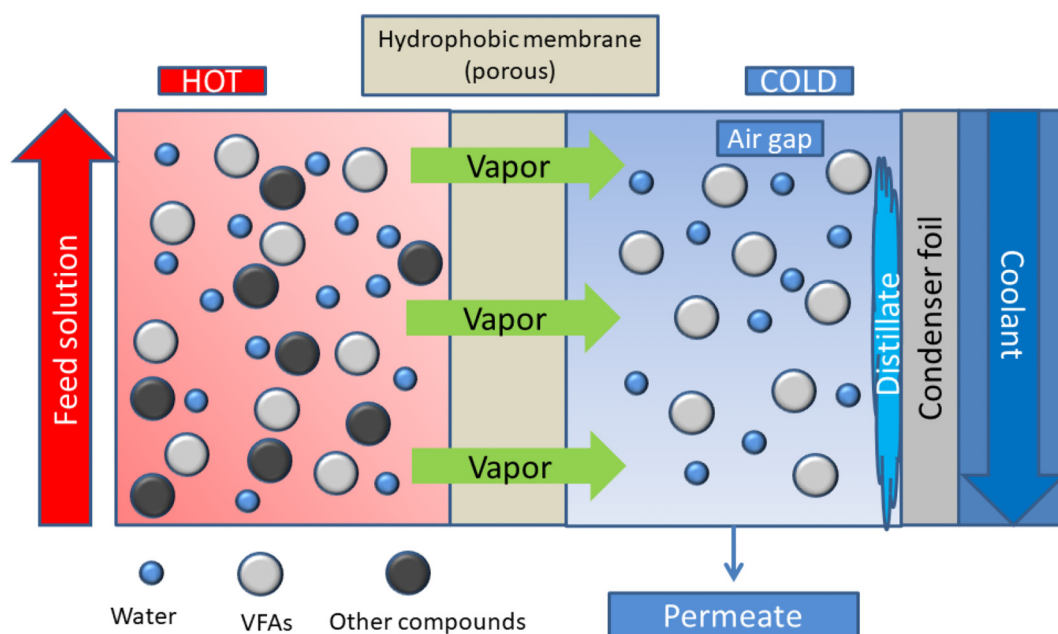

Fig. 4. Tentative MD process scheme for VFAs recovery (Aghapour Aktij et al., 2020).
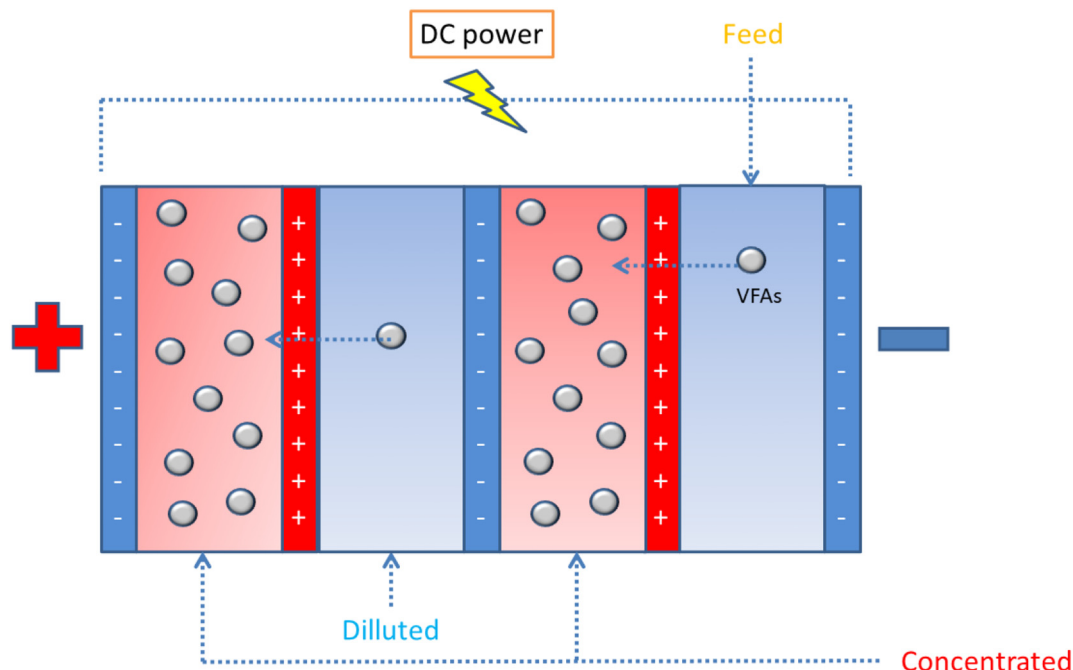
Fig. 5. Tentative CED process scheme for VFAs recovery (Aghapour Aktij et al., 2020).

acid was faster than the alone ED unit due to the use of direct electricity sources. Interestingly, another benefit was observed after combining the unit can simultaneously enhance hydrogen production, potentially influencing the passage of VFAs compounds with 95% and 69% recovery efficiency (Arslan et al., 2017; Zhang and Angelidaki, 2015). However, some drawbacks have been noticed in all ED processes such as membrane operation cost, non-VFAs anions (such as Cl^-) removal favoured over VFAs anions, requiring additional salts that are not practicable towards further downstream applications (Chalmers Brown et al., 2020; Tao et al., 2016; Zhang and Angelidaki, 2015).

2.10. Pervaporation

Membrane-based pervaporation (PV) is an emerging separation process for the recovery of value-added chemicals from waste streams. In the pervaporation process, liquid mixtures are separated using the solution flow and diffusion of the components through the membrane. The components subjected to separation are absorbed into the membrane and the permeate is collected over the membrane in the vapour-phase (Fig. 6) (Feng and Huang, 1997).

This technique has the potential for VFAs recovery as it is environment-friendly and economical. Pervaporation membranes are highly preferable for acetic acid selection over water. Selectivity performance could be improved by adjusting the membrane pore size (0.2–0.5 nm) and layer mechanism. One study developed a new kind of composite membrane by casting sodium alginate solution onto an N_2 plasma modified polypropylene membrane and further crosslinked by Ca^{2+} and Al^{3+}. They showed that N_2 plasma significantly increased their pore size and hydrophilicity, while the crosslinking mechanism improved the stability and separation factor of acetic acid/water compared to the original membrane (Zhang et al., 2014a, 2014b), which is in line with other works that have been discussed the benefits of using a hydrophilic pervaporation membrane prepared by
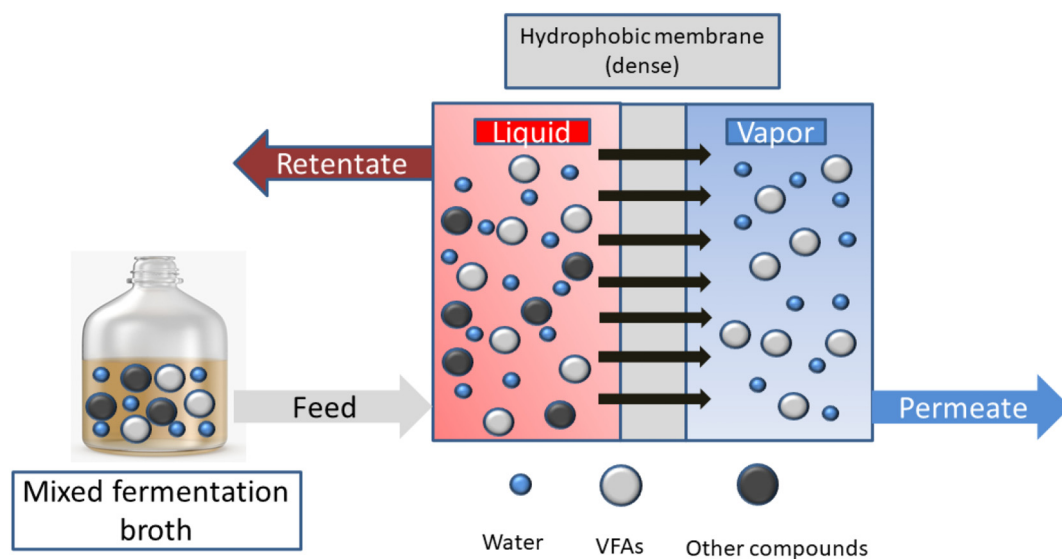

Fig. 6. Tentative PV process scheme for VFAs recovery (Khalid et al., 2019).

the use of amine-functionalized metal-organic framework (Su et al., 2015), graphene oxide (Dave and Nath, 2016) and molybdenum disulphide (Choudhari et al., 2015). On the contrary, some researchers synthesized hydrophobic pervaporation membranes through the use of common polymers polydimethylsiloxane (Li et al., 2004), zeolite (Bowen et al., 2003) and silicalite (Sano et al., 1997) and exhibited their promises on VFAs recovery because of their high affinity towards organic compounds. Besides, the corresponding permeate fluxes were also double after incorporating such fillers.

Moreover, other than conventional dense hydrophilicity/phobicty membranes, another membrane such as supported liquid membrane has been used for VFAs recovery in the pervaporation technique. A study by Yesil and coauthors utilized the pervaporation method for recovery of mixed VFAs from the fermentation broth using three types of membrane; polytetrafluoroethylene (PTFE), tridodecylamine (TDDA) filled PTFE and composite silicone rubber/PTFE membranes. Results showed that the highest selectivity of VFAs, flux, separation factor and permeance was obtained in the presence of TDDA filled PTFE liquid membrane (Yesil et al., 2020), This is because of lower aqueous − organic interfacial tensions between liquid membranes and VFAs compound, which agrees with a previous report (Qin and Sheth, 2003). However, previous researchers did not focus on product capture from the vapour phase in the commercial method, which is very sensitive in terms of product loss during the pervaporation process, and it should be carefully monitored.

2.11. Forward osmosis

Forward osmosis (FO) is another new approach evaluated for the treatment of the effluents using the semi-permeable membrane panels in which the target solutes from the feed (dilute) solution are transferred to the draw solution (concentrated) over osmotic pressure (Fig. 7). The FO process does not require any external energy input since it is driven by osmotic potential except a small amount of energy needed to circulate feed and draw solutions (Khan et al., 2021). This process has a couple of practical advantages such as low membrane fouling, low or no pressure requirements and high retentate efficiency (Cath et al., 2006).

Concentration of VFAs derived from waste streams through FO has been recognized as an emerging technology in recent years (Cagnetta et al., 2017; Garcia-Aguirre et al., 2020). During the FO process, the rejection performances of VFAs compounds appeared to be pH-dependent rather than membrane orientation (Khan et al., 2020). With the increase of pH values, the recovery percentages of individual VFAs components were increased. Around 90% recovery of VFAs was achieved at pH 7.5, while only 30% for pH 4, as investigated by Blandin et al. (2019). These results can be described by focusing their ionization constant (pKa) values, as all VFAs exhibits pKa around 4.8, thus the rejection rate at pH 7.5 was modulated by charged effects. At higher pH, VFAs compounds and membrane surface charge both became negatively charged, resulting in high retention behaviour due to the formation of electrostatic repulsion (Verliefde et al., 2008). On the other hand, recovery percentages at pH 4 were explained by a size-dependent mechanism, meaning higher molecular weight compounds are better retained than smaller molecular weight compounds (Bellona et al., 2004). Besides, permeate flux is also affected by pH solution, higher flux is obtained when solution pH was higher (Jung et al., 2015). The combination of the FO process with other recovery techniques such as membrane distillation, nanofiltration and reverse osmosis could be more efficient. This can be applied through pre-treatment (NF/RO-FO) and post-treatment (FO-MD) integrated set-up, intending to recover the water and concentrated acid from fermentation broth simultaneously (Cho et al., 2012; Zhang et al., 2014a, 2014b).

Moreover, some studies proposed a dynamic model for the concentration of VFAs under the FO process. The motivation to develop this dynamic model for controlling the FO operational parameters systematically. By using this model, recovery efficiency and flux behaviour over time can be optimized. The model was based on the Levenberg-Marquardt algorithm to determine all process variables. After investigating the results, it was found that the concentration percentage of acetic, butyric and valeric acids were enhanced, as complying with the predictions made by the model (Ruprakobkit et al., 2016; Ruprakobkit et al., 2017; Ruprakobkit et al., 2019).

Moreover, the above discussion stated that the FO process has some potential challenges, such as reverse solute diffusion, internal concentration polarization, and membrane fouling. These phenomena should be resolved to obtain a high amount of VFAs from liquid waste streams.

2.12. Pressure-driven membrane filtration

Pressure-driven membrane filtration process is a classical separation method and has significant aspects in purifying mixed waste effluents. These processes can be categorized into four types based on the membrane pore size and driving force (transmembrane pressure) during the operation, including microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) (Fig. 8). Pressure driven membrane process can be arranged in two ways, such as dead-end and cross-flow membrane filtration. In the dead-end membrane filtration, the feed solution flowed through the vertical direction of the membrane surface with one stream of the membrane module, while the feed solution flowed tangentially towards the membrane surface with two streams (one for retentate and one for
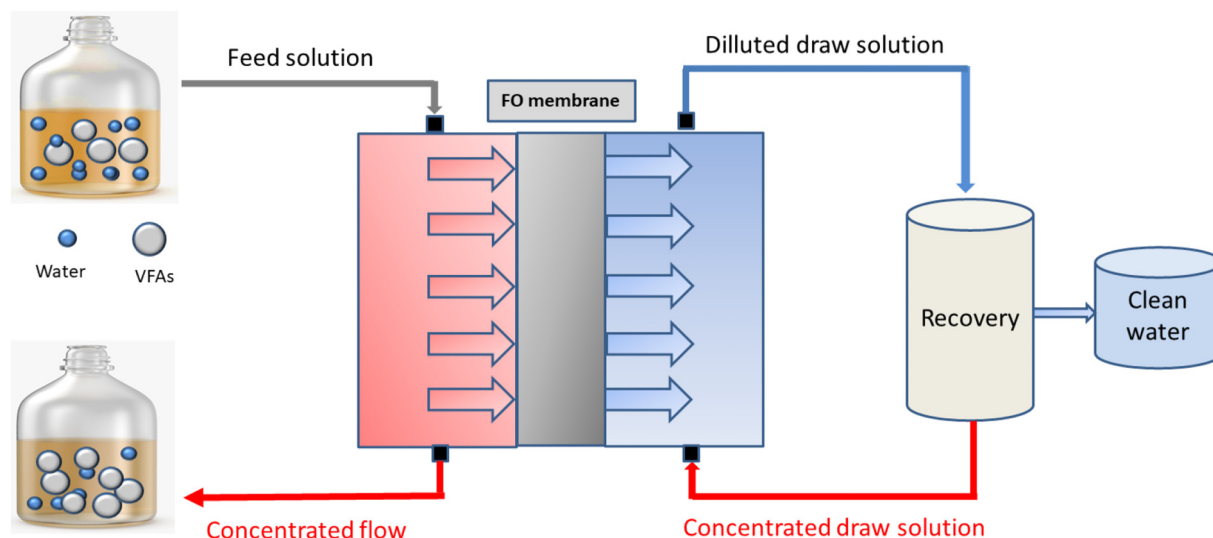

Fig. 7. Tentative FO process scheme for VFAs recovery (Zhang et al., 2020).
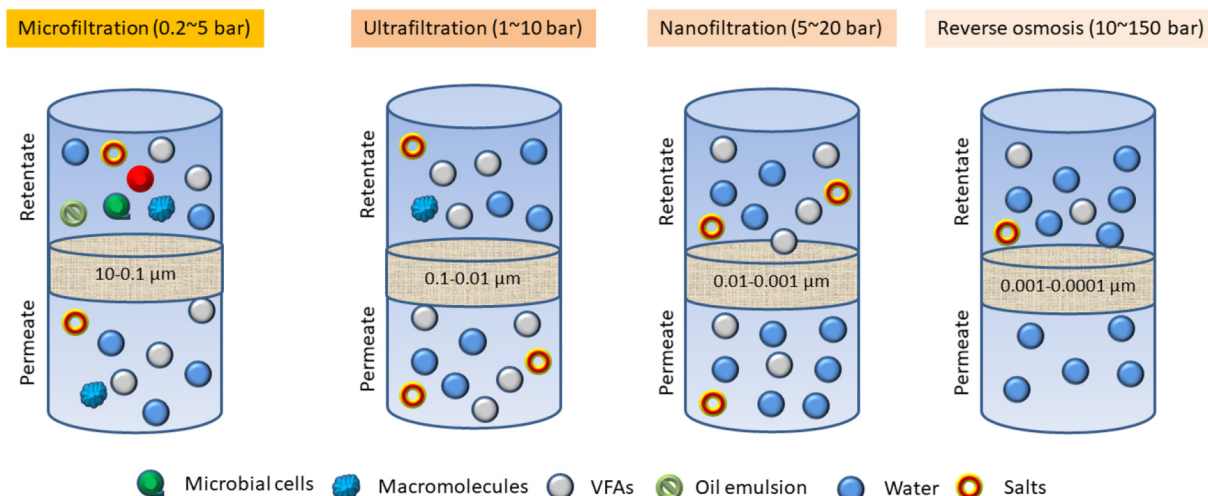
Fig. 8. Pressure driven membrane filtration process for VFAs recovery (Liao et al., 2018).

permeate) for cross-flow membrane filtration. Moreover, various membrane modules, including tubular, hollow, flat sheets and spiral wounds, are typically utilized for pressure-driven membrane operation (Van der Bruggen et al., 2003a, 2003b).

In the MF process, the membrane was used with pore sizes ranging from 10 to 0.1 μm and operating hydraulic pressure applied from 0.2 to 5 bar, leading to a higher flux. In general, larger particles such as bacteria/protozoa, suspended particles and emulsified components than pore size can be removed are in the MF process from the feed solution due to the size-exclusion separation mechanism. Phase-inversion, stretching, track etching and sintering techniques are mostly used for the production of hydrophobic MF membranes. Polymeric, ceramic and metallic membranes are frequently applied in the MF operation. On the other hand, the UF process used smaller pore size membranes (varied from 0.1 to 0.01 μm) and higher transmembrane pressure ranges between 1 and 10 bar. Similar to MF, the UF process retained the larger particles such as proteins and sugars by following the size exclusion mechanism. Mainly, the phase inversion technique was selected for the synthesis of polymeric UF membranes. Some polymers are commonly blended during the processing to increase the membranes' hydrophilicity. Notably, both MF/UF membranes are structurally microporous.

In the NF process, membranes can be either porous or dense with a pore size of about 0.01–0.001 μm and molecular cut-off (MWCO) about 300–-500 dalton (Da). This makes NF membranes suitable for retaining low molecular weight acids and divalent inorganic ions (Mohammad et al., 2015). The separation mechanism of the NF membrane process is governed by the combination of the Donnan effect, steric and dielectric poles (Ernst et al., 2000). Usually, the NF process operates at applied pressures in the range from 5 to 20 bar. On the other hand, reverse osmosis membranes are generally known as dense non-porous membranes with a pore size between 0.001 and 0.0001 nm and MWCO about 100 Da. During the RO operation, this filtration process requires relatively high pressure (10–150 bar). Mostly, polymeric/organic membranes have been utilized for NF/RO applications because of their cost-effectiveness and high efficiency.

There is a broad consensus that the pressure-driven membrane processes have better capability in recovering VFAs compounds from different waste streams (Aghapour Aktij et al., 2020, Zhu et al., 2021a, 2021b). For instance, Kim et al. (2005) investigated the microfiltration performance (ceramic MF membrane) of organic sludge for the recovery of VFAs. Results showed that bacteria were completely removed while more than 80% of VFA could be recovered through the permeate. However, the recovery percentage could be increased to 90% using a modified polyethersulfone microfiltration membrane module (a KrosFlo Research Iii System) (Tao et al., 2016). In recent years, some studies demonstrated that using MF together with MBR may open a new possibility for continuous in-situ

recovery of VFAs from various waste effluent streams (Wainaina et al., 2019a, 2019b), food waste slurry and excess sewage sludge (Parchami et al., 2020), chicken manure (Yin et al., 2021) and cow manure (Jomnonkhaow et al., 2021). The transformation from batch to continuous production and recovery using a newly developed immersed membrane bioreactor eventually led to a sustainable VFAs recovery strategy in the MF process. Similarly, UF has also been applied for VFAs recovery, as the pore size of the membranes is normally less that could assist in retained low molecular weight VFAs compounds. For example, Longo et al. (2015) recovered VFAs from sewage sludge fermentation with the use of an ultrafiltration setup. They successfully recovered a high amount of VFAs, especially acetic acid and propionic acid.

However, MF/UF still possesses some limits, such as larger pore size, which are not beneficial for VFAs recovery effectively. In this context, NF/RO membranes with smaller pore sizes have been demonstrated as a potential route for improving the recovery efficiency of VFAs compounds. For instance, Han et al. (Han and Cheryan, 1995) screened several different types of nanofiltration and reverse osmosis membranes for acetic acid recovery from synthetic solutions. At high pH, the recovery percentage of acetic acid increased reaching an average of 40%. In another work, Xie et al. (Xie and Liu, 2015) concentrated 60% of acetic acid from paulownia hot water wood extracts using a nanofiltration module. They suggested that during nanofiltration, multiple parameters such as membrane hydrophobicity affect acetic acid filtration efficiency. Besides, the nanofiltration efficiency was directly influenced by the intermolecular interactions in the biomass, meaning the solution pH plays an important role during the nanofiltration process. For the separation of acetic acid from lignocellulosic substrate, Weng et al. (2009) reported and acetic acid recovery percentage of 90% in a xylose solution using NF at medium pH of 2.0 and 24.5 bar. Similarly, Malmali et al. (2014) could reach a complete separation of acetic acid from biomass hydrolysate using NF. They have clearly noted that the recovery of acetic acid is pH-dependent while using NF. It has been reported that a sequential filtration can enhance the recovery percentage of acetic acid considerably when using NF (Lyu et al., 2015). NF can also be used in combination with RO to increase recovery percentage. Recovery of acetic acid by NF and RO from pulp mill spent sulphite liquor was conducted by Afonso et al. (Afonso, 2012). It was observed that although the NF membrane flux was higher than that of the RO membrane, the retention percentage of acetic was better for RO membrane (89%) compared to the NF membrane (77%).

Considering the fact that pressure-driven membrane filtration process shows a potential scope in recovering a high amount of VFAs compounds compared to the other techniques. The most reliable feature of this process is membrane properties that have a strong influence on VFAs recovery. In this paper, the pressure-driven membrane filtration process is discussed in

detail since it is simple, unique and high efficiency, and various factors play a vital role, hence it is worth investigating.

3. Factors affecting pressure driven membrane-assisted recovery of VFAs

In order to guarantee a long-term sustainable membrane filtration for separation of VFAs from different streams a synchrony between different membrane-, medium- and operation-related factors is essential. In pressure driven membrane VFAs separation the main parameters affecting the success level of the process can be listed as; membrane properties (membrane material/composition, pore size, charge, hydrophilicity/phobicity and permeability) and medium characteristics (Feed concentrations, pH, temperature, pressure and ionic strength).

3.1. Effect of membrane properties on VFA recovery

3.1.1. Membrane material

The membrane filtration efficiency is greatly influenced by membrane characteristics such as membrane materials (Choi and Ng, 2008; Sadeghi et al., 2018). Up to present, various qualities of membranes such as metals, ceramics and polymers have been employed for VFAs recovery purposes in pressure-driven membrane filtration. Metallic membranes are used successfully in the MF/UF filtration units. It has been reported that metallic membranes have more strength to high-temperature oxidation and better endurance, which assist with fouling reduction (Zhang et al., 2005). On the other hand, ceramic membranes have been used widely on a commercial scale for MF/UF systems as they offer high flux, corrosion resistance, low fouling tendency and backwashability (Ersu and Ong, 2008). Moreover, ceramic membranes provide a high flux because of the fewer interactions between the membrane surface and foulants (Baker, 2012). Ceramic membranes have therefore been used for VFA recovery from liquid organic sludge. For example, Kim et al. (2005) used a cross-flow microfiltration unit and showed that ceramic membranes could be used to recover more than 80% of VFAs in a solution. Moreover, ceramic membranes (made of α-Al$_2$O$_3$) have been successfully applied to remove large particles from the anaerobic digestion streams with low VFAs retention (Zacharof and Lovitt, 2014). However, as both ceramic and metallic membranes have shortcomings when it comes to shaping and modularity, weight and price, polymeric membranes have been applied on a large scale in the pressure-driven membrane filtration system (Ng et al., 2013; Van der Bruggen et al., 2003a, 2003b).

As presented elaborately in Table 1, variety of polymeric membranes have been used for microfiltration and ultrafiltration in VFA recovery systems. As an example, polyvinylidene fluoride (PVDF)-based membranes have been used in the cross-flow microfiltration for the extraction of VFAs in a solution with concentrations of up to 52 g/L (Trad et al., 2015). In addition, surface modification of polymeric membranes (e.g. modified polyethersulfone) has been reported to result in better recovery percentage of VFAs (around 90%) (Tao et al., 2016). Another commonly used

membranes for NF and RO are composite polyamide(PA)-based membranes. Typically composite membranes are anisotropic in nature and composed of two or more highly porous supporting materials. The structure of composite polyamide membranes is characterized by three layers: a top polyamide layer (size around 100 nm), the second layer made of a microporous polysulfone and finally, a non-woven supporting material to provide mechanical durability (Tang et al., 2009). The top layer is considered as the crucial part since it determines the selectivity performance of the membrane. Categorized based on the structural monomer group, fully aromatic PA membranes although fully commercialized, it possesses a rough surface that challenges smooth operation. Alternatively, semi-aromatic poly (piperazinamide)-based PA membranes exhibited a smooth surface and are often used in membrane filtration process. However, fully aromatic based membranes are still considered as a mainstream membrane because of their high recovery efficiency than poly(piperazinamide)-based membrane (Choi et al., 2008; Tang et al., 2009). As summarized in Table 2, although fully aromatic based XLE (Polyamide, Dow Filmtec) nanofiltration membrane effectively retained acetic acid (97%), butyric acid (99%) and propionic acid (99%) in a ternary solution, the retention percentages for piperazinamide-based NF-45 (Polyamide, Dow Filmtec) membrane was only 68%, 88% and 72%, respectively (Zhu et al., 2020a, 2020b). There are several more commercial fully aromatic-based membranes such as NF 90 (Polyamide, Dow Filmtec), NF 97 (Polyamide, Alfa Laval), ES 10 (Aromatic polyamide, Nitto Denko) that have exhibited better VFAs recovery compared to the piperazinamide-based membranes. In addition, fully aromatic membranes performance can be improved by the application of a cross-linker in the parent molecule. In this regard, Zacharof et al. (2016) used polyvinyl alcohol-aromatic cross-linked polyamide membrane LF10 (Polyvinyl alcohol/polyamide, Nitto Denko) in treating agricultural wastewater digestate rich in carboxylic acids. They found that the recovery percentages of acetic and butyric acid were maximal for LF10 (Polyvinyl alcohol/polyamide, Nitto Denko) membrane compared to the piperazine-based polyamide membranes such as NF 270 (Polyamide, Dow Filmtec), HL (Polyamide/polysulfone, GE Osmonics) DL (Polyamide/polysulfone, GE Osmonics) DK (Polyamide/polysulfone, GE Osmonics). Similar trend was also noticed for reverse osmosis membranes, such as fully aromatic-based SWC5 (Polyamide, Hydranautics) and SWC6 (Polyamide, Hydranautics) RO membranes, capable of recovering more than 80% of acetic acid and 100% of isobutyric acid, while the recovery percentage increased to 95.2% in the presence of coated fully aromatic flat sheet SW 30HR (Polyamide/polysulfone, Dow Filmtec) membrane (Table 3). However, the recovery percentage decreased to 60% when the piperazine based polyamide membrane SG (Polyamide, GE Osmonics) used. This indicates that fully aromatic membranes are the most suitable for satisfactory recovery percentages of VFAs.

Moreover, applied membranes for VFAs recovery were mostly flat sheets and spiral wound modules, as presented in Tables 1–3. It was noticed that the recovery percentages of VFAs are quite similar in both type of membranes, but the use of flat sheet membrane is higher than spiral wound

Table 1
Effect of membrane characteristics on VFAs recovery (MF-UF).

Filtration module	Membrane module	Manufacturer	Membrane material	Membrane area (m^2)	Pressure	MWCO/pore size	VFAs recovered	Ref
Cross-flow MF	Tubular, monolith	Nihon Gaishi, Japan	Ceramic	Tubular: 0.035 Monolith: 0.12	TMP 30–200 kPa	Monolith: 0.1, 0.2, 0.5 μm Tubular: 1.0, 2.0, 5.0 μm	87%	(Kim et al., 2005)
Cross-flow MF	Membralox model, monolith	Pall Corporation, UK	Ceramic	0.22	–	0.2 μm	21.08 mM acetic acid 15.81 mM butyric acid	(Zacharof and Lovitt, 2014)
Tangential flow MF	mPES MiniKros™, hollow fiber	KrosFlo® Research Iii, USA	Modified polyethersulfone	0.26	2 bar at inlet	2 μm	92.8%	(Tao et al., 2016)
Cross-flow MF	Hollow fiber	GPC, France	Polyvinylidene fluoride	0.155	0.4 bar at permeate side	0.2 μm	Total VFAs concentration 52 g/L	(Trad et al., 2015)
Cross-flow UF	MO P13U 1 m, tubular	Berghof Group, Germany	Polyvinylidene fluoride (PVDF)	0.31	10 kPa	MWCO: 15 kDa	VFAs concentration 7453 mgCOD/L	(Longo et al., 2015)

Table 2
Effect of membrane characteristics on VFAs recovery (NF).

Filtration module	Membrane module	Manufacturer	Membrane material	Membrane area (m²)	Pressure	MWCO/pore size (µm)	VFA recovered	Ref
Dead-end NF	NTR729, flat sheet	Nitto-Denko, Japan	Polyvinylalcohol, polyamide	0.00145	250 psig	MWCO: 700 Da	Acetic acid, 40%	(Han and Cheryan, 1995)
Dead-end NF	MX07, flat sheet	GE Osmonics, USA	Polyamide	0.00145		MWCO: 300–500 Da	Acetic acid, 38%	
Crossflow NF	HL, flat sheet	GE Osmonics, USA	Polyamide, polysulfone	0.0042	15 bar	MWCO: 100–300 Da	Butyric acid, 8%	(Cho et al., 2012)
Crossflow NF	Duraslick NF, flat sheet	GE Osmonics, USA	–	0.0042		MWCO: 100–300 Da	Butyric acid, 6%	
Dead-end NF	Desal DL, flat sheet	GE Osmonics, USA	Polyamide, polysulfone	0.0138	8 bar	MWCO: 150–300 Da	Acetic acid, butyric acid,100% propionic acid, 30%,	(Xiong et al., 2015)
Dead-end NF	Desal DK, flat sheet	GE Osmonics, USA	Polyamide, polysulfone	0.0138		MWCO: 150–300 Da	Butyric acid, 100% propionic acid, 50%,	
Dead-end NF	NF97, flat sheet	Alfa Laval, Denmark	Polyamide	0.00132	26 bar	MWCO: 200 Da	Acetic acid, 78%	(Afonso, 2012)
Dead-end NF	NF99 HF, flat sheet	Alfa Laval, Denmark	Polyamide	0.00132		MWCO: 200 Da Da	Acetic acid, 15%	
Dead-end NF	NF200, flat sheet	Dow Filmtec, USA	Polyamide	0.00132		MWCO: 200–400 Da	Acetic acid, 22%	
Dead-end NF	NF270, flat sheet	Dow Filmtec, USA	Polyamide	0.00132		MWCO: 200–400 Da	Acetic acid, 24%	
Dead-end NF	NP010, flat sheet	Microdyn-Nadir, Germany	Polyethersulphone	0.00132		MWCO: 1000 Da	Acetic acid, 9%	
Dead-end NF	NP030, flat sheet	Microdyn-Nadir, Germany	Polyethersulphone	0.00132		MWCO: 400 Da	Acetic acid, 29%	
Dead-end NF	HL, flat sheet	GE Osmonics, USA	Polyamide, polysulfone	0.00146	1500 kPa	MWCO: 150–300 Da	Acetic acid, 22%, butyric acid, 30%,	(Zacharof et al., 2016)
Dead-end NF	DL, flat sheet	GE Osmonics, USA	Polyamide, polysulfone	0.00146		MWCO: 150–300 Da	Acetic acid, 45%, butyric acid, 30%,	
Dead-end NF	DK, flat sheet	GE Osmonics, USA	Polyamide, polysulfone	0.00146		MWCO: 150–300 Da	Acetic acid, 57.2%, butyric acid, 45.2%,	
Dead-end NF	NF270, flat sheet	Dow Filmtec, USA	Polyamide	0.00146		MWCO: 150–200 Da	Acetic acid, 52.6%, butyric acid, 69.7%,	
Dead-end NF	LF10, flat sheet	Nitto Denko, Japan	Polyvinyl alcohol-polyamide	0.00146		MWCO: <150 Da	Acetic acid, 72.2%, butyric acid, 69.7%,	
Dead-end NF	HL, flat sheet	GE Osmonics, USA	Polyamide, polysulfone	0.0127	25 bar	MWCO: 100–300 Da	Acetic acid, 91.1%	(Ecker et al., 2012)
Dead-end NF	DL, flat sheet	GE Osmonics, USA	Polyamide, polysulfone	0.0127		MWCO: 100–300 Da	Acetic acid, 94.8%	
Dead-end NF	DK, flat sheet	GE Osmonics, USA	Polyamide, polysulfone	0.0127		MWCO: 100–300 Da	Acetic acid, 96.1%	
Dead-end NF	HT, flat sheet	Hydranautics, USA	–	0.0127		MWCO: 720 Da	Acetic acid, 77.7%	
Dead-end NF	NF270, flat sheet	Dow Filmtec, USA	Polyamide	0.0127		MWCO: 270 Da	Acetic acid, 92.7%	
Dead-end NF	KO, flat sheet	Koch, USA	–	0.0127		MWCO: 1000 Da	Acetic acid, 71.1%	
Dead-end NF	XN45, flat sheet	Microdyn-Nadir, Germany	Polyamide	0.0037		MWCO: 500 Da	Acetic acid, 36%	(Jänisch et al., 2019)
Dead-end NF	NF 90	Dow Filmtec, USA	Polyamide	0.0037		–	Acetic acid, 28%	
Organophile NF	M3#	PolyAn, Germany	PV composite polymer	0.0037	–	–	Acetic acid, 74%, propionic acid, 84%	
Dead-end NF	NF-45	Dow Filmtec, USA	Polyamide	0.003	20 bar	MWCO: 200 Da	Acetic acid, 68%, butyric acid, 88%, propionic acid, 72%	(Zhu et al., 2020a, 2020b)
Dead-end NF	XLE	Dow Filmtec, USA	Polyamide	0.003		MWCO: 100 Da	Acetic acid, 97%, butyric acid, 99%, propionic acid, 99%	
Cross-flow NF	NF-90, flat sheet	Dow Filmtec, USA	Polyamide	0.01387	550 kPa	MWCO: 200 Da	Acetic acid, 85%	(Bellona and Drewes, 2005)
Cross-flow NF	NF-200, flat sheet	Dow Filmtec, USA	Polyamide	0.01387		MWCO: 300 Da	Acetic acid, 83%	
Cross-flow NF	NF90, flat sheet	Dow Filmtec, USA	Polyamide	0.015	20 bar	MWCO: ~200–400 Da	Acetic acid, 50%	(Yasin et al., 2020)
Cross-flow NF	NF270, flat sheet	Dow Filmtec, USA	Polyamide	0.015		MWCO: ~200–400 Da	Acetic acid, 10%	
Crossflow NF	ES10, flat sheet	Nitto Denko, Japan	Aromatic polyamide	0.006	280 ± 3 kPa	MWCO: 100 Da	Acetic acid, 98%, propionic acid, 98%	(Choi et al., 2008)
Crossflow NF	NF270, flat sheet	Dow Filmtec, USA	Piperazine-based polyamide	0.006		MWCO: 200–300 Da	Acetic acid, 78%, propionic acid, 82%	
Crossflow NF	NF90, flat sheet	Dow Filmtec, USA	Polyamide	0.036	10 bar	MWCO: 200–400 Da	Acetic acid, 50%	(Nguyen et al., 2015)
Crossflow NF	NF270, flat sheet	Dow Filmtec, USA	Polyamide	0.036		MWCO: 200–400 Da	Acetic acid, 4.5%	
Crossflow NF	NF245, flat sheet	Dow Filmtec, USA	Polyamide	0.036		MWCO: <300 Da	Acetic acid, 6.7%	
Crossflow NF	DK, flat sheet	GE Osmonics, USA	Polyamide, polysulfone	0.036		MWCO: 150–300 Da	Acetic acid, 4.9%	
Crossflow NF	Desal-5 DK, spiral wound	GE Osmonics, USA	Polyamide, polysulfone	0.2	24.5 bar	MWCO: 150–300 Da	Acetic acid, 90%	(Weng et al., 2009)
Crossflow NF	PA 100, flat sheet	Permeonics Pvt. Ltd., India	Polyamide	0.014	8 bar	MWCO: 100 Da	Acetic acid, 30%	(Maiti et al., 2012)
Crossflow NF	PA 150, Flat sheet	Permeonics Pvt. Ltd., India	Polyamide	0.014		MWCO: 150 Da	Acetic acid, 5%	
Crossflow NF	PA 400, flat sheet	Permeonics Pvt. Ltd., India	Polyamide	0.014		MWCO: 400 Da	Acetic acid, 10%	
Dead-end NF	PES 150, spiral wound	NovaSep, France	Poly ether sulphonate	0.278		MWCO: 150 Da	Acetic acid, 12%	
Dead-end NF	NF90, flat sheet	Dow Filmtec, USA	Aromatic polyamide	0.00146	400 psi	MWCO: 200–400 Da	Acetic acid, 75%, propionic acid, 80%, isobutyric acid, 95%, valeric acid, 84%	(Zhang et al., 2018)
Crossflow NF	DL, spiral wound	General Electric Co., USA	Polyamide, polysulfone	0.32	10 bar	MWCO: 150 Da	Acetic acid, 30%	(Lyu et al., 2015)
Crossflow NF	DK, spiral wound	General Electric Co., USA	Polyamide, polysulfone	0.32		MWCO: 300 Da	Acetic acid, 38%	
Crossflow NF	DK, flat sheet	GE Osmonics, USA	Polyamide	0.0042	10–20 bar	MWCO: 150–300 Da	Acetic acid, 9%	(Lyu et al., 2016)
Crossflow NF	DL, flat sheet	GE Osmonics, USA	Polyamide	0.0042		MWCO: 150–300 Da	Acetic acid, 6%	
Crossflow NF	NF270, flat sheet	Dow Filmtec, USA	Polypiperazine amide	0.0042		MWCO: 200–400 Da	Acetic acid, 9%	
Crossflow NF	NF90, flat sheet	Dow Filmtec, USA	Polyamide	0.0042		MWCO: 200–400 Da	Acetic acid, 30%	
Crossflow NF	XN45, flat sheet	Microdyn-Nadir, Germany	Polyamide	0.0042		MWCO: 300–500 Da	Acetic acid, 3%	
Crossflow NF	TS40, flat sheet	Microdyn-Nadir, Germany	Polypiperazine amide	0.0042		MWCO: 200–300 Da	Acetic acid, 7%	

membrane because of their easy cleaning operation and steady water flux (Corzo et al., 2017).

3.1.2. Membrane pore size and molecular weight cut-off

The pore size of the membranes has been called as the backbone of the MF, UF and NF and is well-understood that membrane pore size has a fundamental relationship with the retentate and permeate solution characteristics (Dizge et al., 2011). The selectivity of MF membranes are determined by indicating the pore size, while for pore sizes for UF, NF and RO membranes are usually presented in MWCO (expressed as Da or kDa). Generally, the pore sizes of the MF and UF membrane are relatively large (Section 2.12) that allows removing larger particles, microorganisms and macromolecules, while small MWCO of NF and RO membranes assist with the retention/removal of low molecular weight compounds/ions.

In addition, membrane permeability is an effect of pore size, which describes the passage rate of a compound through the unit area of the membrane per unit time and pressure applied. Membrane has the most important role in defining permeability as its selectivity acts as a resistance against the flow of all compounds through the membrane. In general, there is a trade-off between membrane permeability and its selectivity as higher permeability tends to lower the selectivity and vice versa (Park et al., 2017). When separation of VFAs from the anaerobic digestion effluent is to be conducted, considering the complex composition of the effluent high selectivity towards VFAs is sought that may challenge membrane permeability and process productivity.

Table 1 shows that MF and UF membranes have been actively used for clarification of VFAs containing solutions rather selective retention of VFAs (Longo et al., 2015; Trad et al., 2015). Although, some studies have reported that a marginal amount of VFAs could be retained while applying MF or UF, surprisingly, there are reports on negative retention percentages for VFAs using MF or UF (Jänisch et al., 2019; Zacharof and Lovitt, 2014). As such, the acetic acid concentration of permeate solution (1.265.85 mg/L) was higher than retentate solution 1.083.30 mg/L in the presence of a Membralox ceramic microfiltration membrane (Pall Corporation). Whereas the same trend was observed for ultrafiltration membrane (UH050, Microdyn-Nadir), having a higher acetic acid concentration in the permeate solution (3.45 g/L) compared to that of the original hydrolysate solution (3.33 g/L). These results are achieved due to the larger pore size of MF/UF membranes. As the pore size range provided by MF and UF does not satisfy selective removal of VFAs, smaller pore sizes in the range of NF and RO are required for this purpose.

Similarly, membrane permeability in MF and UF has a minimal influence on VFAs recovery (Zacharof and Lovitt, 2014). Although ceramic membranes have been reported to show higher permeability than their polymeric counterparts, their VFAs recovery percentage is inferior to that of polymeric membranes (Kim et al., 2005; Zacharof et al., 2016). For example, MF ceramic membranes were used to recover about 87% of VFAs, while polymeric ones could reach up to 92.8% (Table 1). A recent study has suggested the application of high permeability ceramic membranes for the fractionation of higher molecular weight compounds and polymeric membranes with lower permeability for the removal of lower molecular weight substances (Luiz-Santos et al., 2020).

Applying nanofiltration, experimental results indicated that 200 Da MWCO membrane can provide a high recovery percentage for acetic acid (78%) compared to 200–400 Da and 1000 Da membranes that exhibited only 22% and 9%, respectively (Table 2) (Afonso, 2012). There is a general consensus on the direct relation between MWCO and VFAs recovery percentage (Bellona and Drewes, 2005; Zacharof et al., 2016). For example, it has been reported that when the MWCO was increase from 200 to 300 Da, acetic acid recovery percentage of dropped from 85% to 83%. Based on our own unpublished results, the percentage of VFAs recovery dropped to almost half when 150–300 Da (Acetic acid, 22%, Butyric acid, 30%) membrane was used compared to that of <150 Da membrane (Acetic acid, 72.2%, Butyric acid, 69.7%). The highest amount of VFAs recovered (>90%) has been obtained in using NF membranes with about 100 Da MWCO, which is attributed to the average molecular weight of VFAs is

around 100 g/mol or larger, leading to higher retention in the 100 Da membrane (Choi et al., 2008; Ecker et al., 2012; Wainaina et al., 2019a, 2019b; Zhu et al., 2020a, 2020b).

For NF membrane, as shown in Table 2, direct relation between permeability and selectivity exist as the water permeability reduced VFAs recovery increases. The permeability rate of HL, DL, DK and LF 10 membranes were found to be 118.43, 56.02, 44.60 and 15.95 L/(m²h), respectively, leading to an ascending order recovery percentages of VFAs of 22%, 45%, 57.2% and 72.2%. The LF 10 that provided a higher recovery percentage at low permeability, practically requires higher filtration pressures that may hinder the economic feasibility of the filtration process (Zacharof et al., 2016). NF 90 and NF 270 are among the most popular NF membranes used for VFAs recovery. Among the two, NF90 membrane, although low in permeability, has had better VFAs recovery performance. Similarly, the ES10 flat sheet membrane with permeability of 1.67 L/(m²h) performed better in the recovery of acetic and propionic acids compared to NF 270 with a permeability around 3.88 L/(m²h) (Bellona and Drewes, 2005). Moreover, as reported by Afonso et al. (Afonso, 2012), the recovery percentage of acetic was more than 3 times higher in the case of NP030 (29%) compared to NP010 membrane (9%) as the former possesses lower permeability. This was the common trend for other commercial membranes as the lowest permeability demonstrated the best recovery amount of acetic acid. For example, PA 100 (Polyamide, Permeonics Pvt. Ltd) flat sheet membrane had the permeability of 2.8 and exhibited 30% acetic acid recovery, while PA 150 (Polyamide, Permeonics Pvt. Ltd), PA 400 (Polyamide, Permeonics Pvt. Ltd) and PES 150 (Poly ether sulphonate, NovaSep) membranes had lower recovery percentages between 2 and 12% (Maiti et al., 2012). Lyu et al. (2016) also confirmed that the lower permeability rate of TS40 (Polypiperazine amide, Microdyn-Nadir) flat sheet membrane help 7% acetic acid recovery while for the higher permeability membrane XN45 (Polyamide, Microdyn-Nadir) this was only 3%.

Based on the expected MWCO, reverse osmosis membranes are similar or better in performance compared to nanofiltration membranes in VFAs recovery. The literature available lacks presentation of MWCO for the RO membranes used for VFAs recovery. Considering NF and RO membranes researched, it was noticed that when a membrane with MWCO of about 100 Da is used butyric acid recovery of about 78% can be expected while MWCO of higher than 150 Da membrane can drop the recovery to 62–68% (Table 3) (Cho et al., 2012; Ozaki and Li, 2002). Interestingly, Yasin et al. (2020) found that two membranes with the same MWCO range (200–400 Da) showed different recovery percentages for acetic acid of 50% and 10%. These types of results confirm that the presence of various functional groups on the membrane surface can strongly affect the recovery percentages of VFAs in NF and RO processes.

As shown in Table 3, the effect of permeability on concentration VFAs using RO membranes has also been investigated. Liu et al. (2020) concentrated higher percentage of acetic acid in using a SWC5 membrane (81.92%) than with SWC6 membrane of higher permeability. The flat sheet membrane ES 20 demonstrated twice permeation flux than other commercial membranes and therefore, attributed to lower recovery percentages (Ozaki and Li, 2002). The performance of RO membrane from Alfa Laval also is in agreement with these findings. In this regard, RO 98pHt (membrane permeability of 3.15) recovered around 44.21% of acetic acid present in the feed, while a recovery percentage of 47.51% was achieved using RO 99 membrane (2.60 permeability) (Zhou et al., 2013a, 2013b). However, in contradiction to the general trend, it reported that XLE membrane (permeability of 100 L/(m²h)) succeeded to retain 78% of butyric acid while the lower permeability membrane LE (70 L/(m²h)) could only provide 62%. On the other hand, a series of membranes such as CPA3 (2.6 L/(m²h), acetic acid 40.1%), CPA2 (3.1 L/(m²h), acetic acid 43.5%), ESPA2 (5.8 L/(m²h), acetic acid 54.5%), SG (2.36 L/(m²h), acetic acid 60%), BW30 (2.92 L/(m²h), acetic acid 68%) BW30FR (3.98 L/(m²h), acetic acid 70%) showed linear outcome (Table 3) (Lyu et al., 2016; Nguyen et al., 2015). These results indicate the importance of building the synchrony between higher selectivity and higher permeability in

Table 3
Effect of membrane characteristics on VFAs recovery (RO).

Filtration mode	Membrane module	Manufacturer	Membrane material	Membrane area (m²)	Pressure	MWCO/pore size (μm)	VFA recovered	Ref
RO	Spiral wound	Sempas Membrantechnik, Germany	Polyamide	0.7	10 bar	–	Acetic acid, 45% Propionic acid, 60% Butyric acid, 80% Valeric acid, 83% Isobutyric acid, 100% Isovaleric acid, 100%	(Hausmanns et al., 1996)
Crossflow RO	SWC5, spiral Wound	Hydranautics, USA	Polyamide	0.003116	4 MPa	–	Acetic acid, 81.92%	(Liu et al., 2020)
Crossflow RO	SWC6, spiral Wound	Hydranautics, USA	Polyamide	0.003116		–	Acetic acid, 81.32%	
Crossflow RO	ES20, flat sheet	Nitto Denko, Japan	Polyamide	0.006	3 kg/cm²	MWCO: <200 Da	Acetic acid, 68%	(Ozaki and Li, 2002)
Crossflow RO	RO98pHt	Alfa Laval, Sweden	Polyamide	0.0174	20 bar	–	Acetic acid, 90%	(Zhou et al., 2013a, 2013b)
Crossflow RO	CPA2, flat sheet	Hydranautics, USA	Polyamide	0.036	20 bar	–	Acetic acid, 43.5%	(Nguyen et al., 2015)
Crossflow RO	CPA3, flat sheet	Hydranautics, USA	Polyamide	0.036		–	Acetic acid, 40.1%	
Crossflow RO	ESPA2, flat sheet	Hydranautics, USA	Polyamide	0.036		–	Acetic acid, 54.5%	
Crossflow RO	XLE, flat sheet	Dow Filmtec, USA	Polyamide	0.036		MWCO: ~100 Da	Acetic acid, 79.8%	
Crossflow RO	SG, flat sheet	GE Osmonics, USA	Polyamide	0.036		–	Acetic acid, 60.5%	
Dead-end RO	XLE, flat sheet	Dow Filmtec, USA	Polyamide	0.0037	–	MWCO: ~100 Da	Acetic acid, 88%	(Jänisch et al., 2019)
Dead-end RO	SW 30HR, flat sheet	Dow Filmtec, USA	Polyamide, polysulfone	0.0037		MWCO: ~100 Da	Acetic acid, 95.2%	
Dead-end RO	SW 30, flat sheet	Dow Filmtec, USA	Polyamide, polysulfone	0.0037		MWCO: ~100 Da	Acetic acid, 94.7%	
Dead-end RO	SWC4, flat sheet	Hydranautics, USA	Polyamide	0.0037		MWCO: 100–200 Da	Acetic acid, 99.2%	
Dead-end RO	SWC5, flat sheet	Hydronautics, USA	Polyamide	0.0037		MWCO: 100–200 Da	Acetic acid, 98.8%	
Crossflow RO	SE, flat sheet	GE Osmonics, USA	Polyamide	0.0042	40 bar	–	Acetic acid, 47%	(Lyu et al., 2016)
Crossflow RO	SG, flat sheet	GE Osmonics, USA	Polyamide	0.0042		–	Acetic acid, 60%	
Crossflow RO	BW30, flat sheet	Dow Filmtec, USA	Polyamide	0.0042		MWCO: ~100 Da	Acetic acid, 68%	
Crossflow RO	BW30FR, flat sheet	Dow Filmtec, USA	Polyamide	0.0042		MWCO: ~100 Da	Acetic acid, 70%	
Dead-end RO	RO90, flat sheet	Alfa Laval, Sweden	Polyamide, polyester	0.00146	40 bar	–	Acetic acid, 41%	(Malmali et al., 2014)
Dead-end RO	RO98, flat sheet	Alfa Laval, Sweden	Polyamide, polypropylene	0.00146		–	Acetic acid, 50%	
Dead-end RO	RO99, flat sheet	Alfa Laval, Sweden	Polyamide, polyester	0.00146		–	Acetic acid, 55%	
Crossflow RO	XLE, flat sheet	Dow Filmtec, USA	Polyamide	0.0042	15 bar	MWCO: ~100 Da	Butyric acid,78%,	(Cho et al., 2012)
Crossflow RO	LE, flat sheet	Dow Filmtec, USA	Polyamide	0.0042		MWCO: 150 Da	Butyric acid,62%,	
Crossflow RO	RO98pHt, flat sheet	Alfa Laval, Sweden	Polyamide, polypropylene	0.0174	30 bar	–	Acetic acid, 44.21%	(Zhou et al., 2013a, 2013b)
Crossflow RO	RO99, flat sheet	Alfa Laval, Sweden	Polyamide, polyester	0.0174		–	Acetic acid, 47.51%	

order to build a robust membrane filtration process satisfying both productivity and purity of the final VFAs rich stream (Werber et al., 2016).

Moreover, the applied pressure is also integrated with membrane pore size and permeability rate, which has played a vital role in the pressure-driven membrane filtration process for the transport of solutes onto the membrane surface and influences the recovery percentage of VFAs compounds. Substantial VFAs recovery percentages are obtained at increased pressure with a higher polarization layer of the membrane at low flow velocity during the filtration process (Tables 1–3) (Abidi et al., 2016). It is recommended to control the operating pressure so that there is no very high initial flux that causes the membrane to be performed insufficiently altogether (Choi et al., 2008). As presented in Table 2, when pressure is increased from 8 to 24 bar during NF and RO, a minimal increase is obtained for the recovery percentage of acetic acid and butyric acid. The reason can be explained in two ways; firstly, at higher pressures, the water flux through the membrane is higher than that of the solute and secondly, charge effects influence concentration polarization behaviour of solute transport eventually determining the recovery rate of the solute (Gherasim et al., 2013).

3.1.3. Membrane charge

The membrane surface charge is considered as a crucial parameter towards the effective membrane filtration process (Moritz et al., 2001). Membrane surface charge can be determined by measuring the zeta potential values and it provides a clear understanding of electrostatic interaction between the feed components and the membrane's surface. Suppose the membranes surface charge is positive, and feed components charge are negative. In this case, strong electrostatic attraction forces are observed between the membrane surface and media components leading to reduced retention percentage. On the other hand, electrostatic repulsive forces occur between the negatively charged membrane surface and the negatively charged feed components, consequently, enhancing retention percentages according to the Donnan theory. The Donnan effect is referred to the interaction between charged molecules that are solubilized and charged membrane surface at their equilibrium state, as described by the British chemist Frederick George Donnan (Donnan, 1995; Rho et al., 2020). MF and UF membranes have require chemical surface modification to produced charged on their surface as they are uncharged initially (Bowen et al., 2005). Most thin-film composites membranes of NF and RO are synthesized with a negatively charged surface at alkaline and neutral conditions and positively charged at acidic conditions. Commonly, NF membranes possess a higher negative charge compared to the RO membranes, therefore making them a favorable choice to retain the negatively charged VFAs. In addition, stoke diameters of compounds plays a role in the membrane recovery process (Cho et al., 2012). Membrane surface charge also depends on the solution chemistry (Childress and Elimelech, 1996). It is presented in the literature that NF 90 membrane is more negative at neutral and alkaline conditions than NF-200, resulting in higher retention percentage of acetic acid (85%) by NF 90 than NF 200 (Bellona and Drewes, 2005). Tables 1–3 represent the recovery percentages of VFAs with respect to various membranes, considering that nearly all membranes are negatively charged.

3.1.4. Membrane hydrophilicity/phobicity

Membrane hydrophilicity is one of the key influencing factors in membrane filtration performance. Membrane hydrophilicity or -phobicity is defined by the extent of membrane surface affinity to water molecules (Rana and Matsuura, 2010). The identification of the surface hydrophilicity or hydrophobicity can be determined through contact angle measurements. If the water droplet contact angle with the membrane surface is more than 90°, the membrane can be termed as hydrophobic, while values less than 90° indicate a hydrophilic surface (Law, 2014). Membranes with a hydrophobic surface are more prone to fouling by the deposition and adsorption of microorganisms, proteins and suspended particles. On the other hand, hydrophilic membranes are less affected by the above mentioned medium component, sustaining there retention-permeation performance (Kumar and Ismail, 2015; Salgin et al., 2013). As presented in Table 1, membranes

of different hydrophobicity and hydrophilicity have been used form VFAs recovery in MF/UF filtration systems.. In this regard, many studies have focused on the enhancement of the hydrophilicity of PVDF membranes through various surface modification techniques in order boost their filtration performance (Hashim et al., 2009; Liang et al., 2013). The quality of the hydrophilic membrane also plays a great role, e.g. the recovery percentage of VFAs dropped from around 90% to 80% when hydrophilized PES membrane was replaced with a hydrophilic ceramic MF substitute (Tao et al., 2016). The effect of hydrophilicity becomes even more pronounced when it comes to NF and RO (Zhao and Ho, 2014; Zhu et al., 2017). It is a common practice to make the active layer of the NF and RO membranes using polyamide and poly(piperazinamide)-based materials that are hydrophilic in nature due to the presence of extra carboxylic acid groups, and amine and ketone groups (Cho et al., 2012). Therefore, the contact angles of the NF and RO membranes are usually low (hydrophilic surface), which assist them in recovering a higher amount of VFAs during the filtration process. As shown in Table 2, the nanofiltration membrane DK flat sheet can retain a high amount of VFAs than a DL flat sheet membrane just due to its higher hydrophilicity (Vieira et al., 2018). Similarly, the contact angle value of NP030 (Polyethersulphone, Microdyn-Nadir) flat sheet membrane is lower than NP010 (Polyethersulphone, Microdyn-Nadir) flat sheet membrane (Vieira et al., 2018); as a result, higher retention percentage of acetic acid has been reported using NP030. However, in general, both membranes (NP010 and NP030) recovery percentages are very low because of their parent raw material, polyethersulfone, which is hydrophobic in nature (Zhao et al., 2013). Recent studies have been shown that upon surface modification of the membrane by adding hydrophilic groups as supporting material in their reaction chain induces. Among them, polyvinyl alcohol (PVA)-based thin-film composite membranes show hydrophilic properties due to the inherent hydrophilic nature of PVA (Zhu et al., 2020a, 2020b). As shown in Table 2, NTR 729 and LF 10 membranes exhibited premium VFAs recovery percentage as these membranes are made of PVA blended PA membrane.

Among all hydrophilic RO membranes show the best recovery percentage (Table 3). For instances, the contact angle of the XLE membrane is lower than LE RO membrane therefore this led to a higher recovery percentage of butyric acid (around 78%) from the fermentation broth using XLE. A study by Hurwitz et al. (Hurwitz and Hoek, 2006) showed that SG membrane have a higher contact angle in the pH range of 2 to 12 compared to XLE membrane, making a less suitable for the recovery of VFAs. The recovery percentage of acetic acid was around 43.5% for CPA 2 flat sheet membrane and only 40% for CPA 3 flat sheet membrane due to their water contact angle difference (Dolar et al., 2012; Yin et al., 2017). On the other hand, it was noticed that hydrophobic membranes such as RO 90, RO 99, RO 98 could recover only 50% of acetic acid, these differences might have result from their high contact angle (>90°) (Malmali et al., 2014). As expected, the recovery percentage was significantly high (above 95% of acetic acid) when using seawater hydrophilic RO membranes (SWC4, SWC5, SW30, SW30 HR) (Baek et al., 2012). Moreover, brackish water hydrophilic RO membrane (BW 30) showed a better recovery percentage of acetic acid (68%) than ESPA 2 flat sheet membrane (54%) as a result of hydrophilicity (Simon and Nghiem Long, 2014).

3.2. Effect of medium characteristics on VFAs recovery

3.2.1. VFAs concentrations

The concentration of feed solution also influences membrane filtration performance and consequently the VFAs recovery percentage (Tables 4–6). Feed concentrations may vary with respect to the source of VFAs. Generally, microfiltration and ultrafiltration process cannot effectively retain VFAs, and there are no substantial concentration changes observed for initial and filtered VFAs solution. On the other hand, the accomplishment of NF/RO processes revealed a significant VFAs concentration change in the retentate and permeate solution; therefore, the concentration of VFAs in feed solution plays a crucial role in the recovery percentage of NF/RO process (Zhu et al., 2020a, 2020b).

The initial concentration of VFAs in the feed can affect final recovery results. For example, Han et al. (Han and Cheryan, 1995) used three concentrations of 1%, 5% and 7.5% of acetic acid and showed that 40% of acetic acid was recovered at 1%, while the recovery percentage dropped to 20% when the initial VFAs concentration was increased to 7.5%. This might be due to the ion exchange capacity of the membrane surface (Han and Cheryan, 1995). When the feed concentration is high, the excess ions can pass through the membrane easily and lowering the apparent recovery percentage of the VFAs compounds. Moreover, permeability also decreases at a higher concentration because water and the permeating ions compete for a specific membrane area (Han and Cheryan, 1995). It has also been reported in the literature recovery percentage stays unchanged regardless of the initial concentration of VFAs when a NF 90 membrane fully aromatic active layer is used for filtration (Nguyen et al., 2015).

In contrast, the use of RO membrane supports higher recovery percentage at higher initial concentrations. As presented by Zhou et al. (2013a, 2013b), the recovery percentage of acetic acid gradually increased from 50% to 55% as the initial feed concentration of acetic acid increased from 10 to 50 g/L using RO 98pHt membrane. Following these results, Liu et al. (2020) conducted the acetic acid concentrated process through the use of the reverse osmosis membrane and found that there are no considerable changes in the recovery percentage observed at varying concentrations. The similar results are also documented by a previous study (Zhou et al., 2013a, 2013b).

3.2.2. Medium pH

In the pressure-driven membrane filtration process, solution pH is a key parameter on the percentage of VFAs recovery, controlled by the acidic/basic medium. Previous literature indicate that the recovery percentages of VFAs is significantly enhanced at higher pH ranges and vice versa (Zhu et al., 2021a, 2021b). Here, acidity constant (pKa) values of VFAs are considered as crucial in recovery success. The pKa values of VFAs compounds are at about 4.75 (acetic acid), 4.82 (butyric acid), 4.88 (propionic acid), 4.84 (valeric acid), 4.8 (caproic acid), which implies that higher recovery percentages will be observed at beyond pKa values as acids convert to their dissociated forms. For instance, this concept can be verified through the experimental study carried out by Han et al. (Han and Cheryan, 1995), who recovered more than 75% of acetic acid at pH 6.8, but the recovery amount significantly decreased to 10% when the pH was 2.7. This means that at higher pH, the NF membrane becomes negatively charged and solute (acetic acid) transferred to acetate, which is also negatively charged, thus electrostatic repulsion occurred and retained percentages increased eventually. On the other hand, at lower pH solute became positive in charge and is attracted to the negative membrane surface, thereby decreasing the retention percentages. Although all VFAs compounds recovery percentage showed higher at neutral to alkaline pH medium, interestingly, a study by Xiong et al. (2015) reported that higher recovery percentage of butyric acid is observed even in highly acidic medium of pH 3 and continues to neutral pH. The relation between the recovery percentage and pH in NF/RO filtration has been presented in references listed in Tables 5–6.

It has been reported that size-exclusion was not the only mechanism dominating the retention percentage. Maiti et al. (2012) used 150 Da PA, 400 Da and 150 Da PES membranes for acetic acid recovery and found

that increased recovery percentages were achieved at higher pH ranges. It should be pointed out that the Stokes radius of acetic acid is 0.206 nm which is 0.5 times smaller than 100 and 150 Da membranes and 0.33 times smaller than 400 Da membrane. Therefore, the higher recovery of acetic acid in an alkaline medium is influenced by other mechanisms such as Donnan effect. Moreover, Zhang et al. (2018) confirmed that the charge effects primarily governed a higher recovery percentage of acetic acid at pH 8 under the nanofiltration process. While the solution-diffusion mechanism additionally influences the recovery percentages of VFAs in RO filtration. Generally, the structure of the reverse osmosis membrane is composed of a nonporous dense layer that plays a vital role in the recovery percentage of uncharged molecules (Lyu et al., 2016; Zhou et al., 2013a, 2013b).

3.2.3. Operating temperature

The operating temperature can also affect the recovery percentage of VFAs during pressure-driven membrane filtration. From the literature, it is evident that increasing the temperature causes decrease in recovery percentages of VFAs (Tables 4–6). The lower recovery percentages of VFAs compounds are attributed to the higher solute diffusion at an increased temperature, which assists in the transport of solute over the membrane surface, enhancing the mass transfer rate obtained (Nilsson et al., 2008; Snow et al., 1996). Additionally, the polymer structure in the active layer and pore size of the membrane surface significantly changes at a higher temperature, resulting in a low recovery of VFAs at a higher permeate flux. Regarding nanofiltration, Lyu et al. (2016) reported that the recovery percentage of acetic acid was reduced to 20% (at 45 °C) from 40% of the initial recovery rate at 15 °C by NF 90 membrane. Other types of membranes such as DK, DL, NF270, XN45 and TS40 showed low acetic acid recovery percentages (8–4%) at varied temperature (15–45 °C). The same trend was also observed for reverse osmosis membranes, as the recovery percentage of acetic acid was calculated >80% at 20 °C, but reduced to below 70% at 35 °C (Liu et al., 2020).

3.2.4. Ionic strength

Effluent with high ionic strength, e.g. inorganic salts can alter the trend for the selective separation of VFAs during membrane filtration (Choi et al., 2008; Zhu et al., 2020a, 2020b). Previous studies have reached different conclusions regarding the recovery percentage of VFAs when a mixed effluent is used as feed (Tables 4–6). For instance, Xiong et al. (2015) achieved limited recovery percentage of acetic and butyric acids when salts were added in the actual digestion liquor. Adding salt increases the osmotic pressure in the solution, resulting in a higher retention scheme of VFAs. Zacharof et al. (2016) reported that elevation in ionic strength improved the recovery percentages of acetic acid and butyric acid. They used four types of salts (sodium carbonate, sodium bicarbonate, sodium chloride and calcium chloride) at two different concentrations (50 mM and 100 mM). Synthetic VFAs mixtures containing salt solution showed increased recovery percentages for both concentrations. Although all salts provided satisfactory results, among them, sodium bicarbonate and sodium chloride were the dominant co-existing ions in terms of VFAs retention percentage. This was also experimented on raw agricultural wastewater effluent containing salts where an enhanced effect on butyric acid retention over acetic acid was observed (Zacharof et al., 2016).

Table 4
Effect of medium characteristics on VFAs recovery (MF-UF).

Filtration mode	Feed media	Initial VFAs concentrations	pH	Temperature	VFAs recovered	Ref
Crossflow MF	Liquid organic sludge	–	5–6	35 °C	87%	(Kim et al., 2005)
Crossflow MF	Agricultural wastewater + synthetic VFAs mixture (acetic and butyric acid)	24.38 mM acetic acid, 18.91 mM butyric acid	8.25	–	21.08 mM acetic acid, 15.81 mM butyric acid	(Zacharof and Lovitt, 2014)
Tangential flow MF	Activated sludge	11.73 g/L total VFAs	6	37 °C	92.8% total VFAs	(Tao et al., 2016)
Crossflow MF	Glucose or agrowaste (straw)	–	6	35 °C	Total VFAs 52 g/L	(Trad et al., 2015)
Crossflow UF	Sewage sludge	315.6 mg COD/gTVS	5–10.5	35 ± 1 °C	Total VFAs 7453 mgCOD/L	(Longo et al., 2015)

Table 5
Effect of medium characteristics on VFAs recovery (NF).

Filtration mode	Feed media	Initial VFAs concentrations	pH	Temperature	VFAs recovered	Ref
Dead-end NF	Acetic acid solution	1% acetic acid	5.6	50 °C	Acetic acid, 40%	(Han and Cheryan, 1995)
Cross flow NF	Synthetic fermentation broth	0–5 g/L butyric acid	3	37 °C	Butyric acid, 8%	(Cho et al., 2012)
Dead-end NF	Lignocellulosic Whole willow biomass	0.7–1.2 g/L acetic acid	7	25 ± 0.5 °C	62% Acetic acid	(Xiong et al., 2015)
		1.6–2.4 g/L propionic acid			50% Propionic acid	
		1–2 g/L butyric acid			100% Butyric acid	
Dead-end NF	Condensate of eucalyptus spent sulphite liquor	6.8 g/L acetic acid	Natural pH 2.4	25 °C	77% acetic acid	(Afonso, 2012)
Dead end NF	Digested agriculture wastewater	Acetic acid 21.08 mM, 15.81 mM butyric acid	8.5	50 °C	72.2% acetate and 69.7% butyrate	(Zacharof et al., 2016)
Dead end NF	Silage juice from pilot plant	19 g/L acetic acid	3.9	25 °C	96.1% acetic acid	(Ecker et al., 2012)
Dead end NF	Sugar beet fermentation broth	3.3 g/L acetic acid			Acetic acid, 74%	(Jänisch et al., 2019)
Dead end NF	Grass cut fermentation broth	6.83 g/L acetic acid			Propionic acid, 84%	
Dead end NF	Grass cut fermentation broth	14.28 g/L acetic acid				
		2.05 g/L propionic acid				
		0.35 g/L valeric acid				
Dead end NF	Corn-triticale fermentation broth	8.40 g/L acetic acid				
		2.81 propionic acid				
		6.23 g/L butyric acid				
		2.54 g/L valeric acid				
		1.56 caproic acid				
Dead end NF	Organic and organic salts	100 mM acetic acid	8	20 ± 7 °C	68% acetic acid	(Zhu et al., 2020a, 2020b)
		100 mM propionic acid			79% propionic acid	
		100 mM butyric acid			86% butyric acid	
Cross flow NF	Organic acids	10–15 mg/mL acetic acid	7		85% acetic acid	(Bellona and Drewes, 2005)
Cross flow NF	Dimethyl terephthalate process wastewaters	14,611 ± 565 mg/L acetic acid	7	25 ± 1 °C	100% acetic acid	(Yasin et al., 2020)
Cross flow NF	Domestic waste waters	100–500 mg/L	6.7–8.7	25–35 °C	98% acetic acid	(Choi et al., 2008)
		40 mg VFA/L			98% propionic acid	
Cross flow NF	Lignocellulosic hydrolysate	5 g/L acetic acid	10–11	20 °C	50% acetic acid	(Nguyen et al., 2015)
Crossflow NF	Model solution of xylose and acetic acid	5 g/L acetic acid	9.1	25 °C	90% acetic acid	(Weng et al., 2009)
Cross flow NF	Rice straw hydrolysate	2 g/L acetic acid	9	30 °C	60% acetic acid	(Maiti et al., 2012)
Cross flow NF	Hydrothermal liquefaction wastewater	2500 mg/L acetic acid	8	25 °C	75% acetic acid, 80% propionic acid,	(Zhang et al., 2018)
		2000 mg/L propionic acid			95% isobutyric acid, 84% valeric acid	
		350 mg/L isobutyric acid				
		300 mg/L valeric acid				
Cross flow NF	Hydrothermal liquefaction of rice straw	1.809 g/L acetic acid	–	35 °C	38% acetic acid	(Lyu et al., 2015)
Crossflow NF	Hydrothermal liquefaction hydrolysate	2000 mg/L acetic acid	2.78	25 °C	41.57% acetic acid	(Lyu et al., 2016)
Crossflow NF	Hydrothermal liquefaction hydrolysate	2000 mg/L acetic acid	9.52	25 °C	97.73% acetic acid	

Table 6

Effect of medium characteristics on VFAs recovery (RO).

Filtration mode	Feed media	Feed characteristics/initial VFAs	pH	Temperature	VFAs recovered	Ref
RO	Model solution of acetic acid	20 mmol/L acetic acid	2–3.6	18 ± 22 °C	Acetic acid, 45% Propionic acid, 60% Butyric acid, 80% Valeric acid, 83% Isobutyric acid, 100% Isovaleric acid, 100%	(Hausmanns et al., 1996)
Crossflow RO	Model solution of acrylic acid and acetic acid	1.5% acetic acid	–	25 °C	Acetic acid, 81.92% Acetic acid, 81.32%	(Liu et al., 2020)
Crossflow RO	Organic compounds	10 mg/L acetic acid	9	25 ± 2 °C	Acetic acid, 68%	(Ozaki and Li, 2002)
Crossflow RO	Model solution of monosaccharides and acetic acid	5 g/L acetic acid	9.88	25 °C	Acetic acid, 90%	(Zhou et al., 2013a, 2013b)
Cross flow RO	Lignicellulosic hydrolysate Sugar beet fermentation broth Grass cut fermentation broth	5 g/L acetic acid 3.3 g/L acetic acid 6.83 g/L acetic acid	10–11	20 °C	Acetic acid, 79.8% Acetic acid, 99.2%	(Nguyen et al., 2015) (Jänisch et al., 2019)
Cross flow RO	Grass cut fermentation broth Corn-triticale fermentation broth	14.28 g/L acetic acid 2.05 g/L propionic acid 0.35 g/L valeric acid 8.40 g/L acetic acid 2.81 propionic acid 6.23 g/L butyric acid 2.54 g/L valeric acid 1.56 caproic acid				
Cross flow RO	Hydrothermal liquefaction hydrolysate	2000 mg/L acetic acid	2.78	25 °C	Acetic acid, 70%	(Lyu et al., 2016)
Cross flow RO	Cornstover hyrolysate	3.445 g/L acetic acid	5.75	42 °C	Acetic acid, 55%	(Malmali et al., 2014)
Cross flow RO	Synthetic fermentation broth	1000 ppm butyric acid	3	37 °C	Butyric acid, 78%,	(Cho et al., 2012)
Cross flow RO	Model solution of monosaccharides and acetic acid	5 g/L acetic acid	9.91	25 °C	Acetic acid, 47.51%	(Zhou et al., 2013a, 2013b)

4. Challenges

Pressure-driven membrane filtration technologies are promising sustainable approaches for the efficient recovery of VFAs from waste streams. However, further improvements are still needed by mitigating challenges involved in the continuous recovery of VFAs.

- Previously, Atasoy et al. (2018) suggested that optimization of operational parameters are the most crucial parts of VFAs recovery, in addition to that, it is seen from this current review that membrane and medium characteristics both play a determining role in the recovery of VFAs which are required to manage in an effective way.
- Membrane properties in terms of membrane materials, charge, pore size and surface wettability behaviour strongly influence VFAs recovery. As shown in the previous literature analysis, chemical structure of membrane play an important role, such as fully aromatic polymeric membranes are more suitable than poly(piperazinamide)-based membranes. Hence, fully aromatic membranes and simultaneously customization of poly(piperazinamide)-based membranes could be continued in order to have a satisfactory VFAs recovery amount. Additionally, if long-term optimal VFAs recovery from complex anaerobic digestion effluents is targeted, remediation of membrane fouling should be prioritized.
- Among the medium characteristics, solution pH has been found to be the dominant factor in VFAs recovery. Moreover, solution pH also interacts with membrane surface charge, thereby most of the nanofiltration membranes are typically negatively charged, leading to a maximum recovery in alkaline pH. In addition to that, the solute substance is also affected by solution pH and membrane surface charge, which readily might interact with each other, and careful consideration of the fundamental materials must be appropriately taken.
- Driving from biogas to VFAs production and recovery is a new concept, and limited economic analysis exists until now in the literature. Especially, purification costs should be as competitive according to the market demand. Achieving a pure form of VFAs is generally the most difficult task since it contains a low concentration, and mainly, for this reason, the overall cost became higher. For example, direct purification cost was found 14.96 USD/m³ while pretreatment leading purification cost increased to 15.5 USD/m³ (Bonk et al., 2015) that means process stages should be minimized such as co-pretreatments could be adopted by keeping the profitability and quality of the final product.

- There is a great recent attraction towards waste-based biorefineries that produce a variety of products from organic waste such as VFAs, whereby the purification of VFAs is one of the major challenges because of their low concentrations in the effluent streams. Currently, there are limited techniques available for VFAs recovery and purification from the bio-based streams, definitely worth exploring on a wide scale. The need of biorefineries for fractionation approaches that are environmentally friendly, economically feasible and scale-up makes MF, UF, NF and RO membrane separation an inseparable part of the future in VFAs recovery.

5. Future perspectives

Volatile fatty acids recovery from organic waste streams, especially food waste, has been evaluated in terms of processing feasibility in this study, as well as techniques and challenges encountered under the scheme of the circular economy. Considering the economic point of view, this aspect appears crucial in future bio-based feedstock renewable sources because of their low-cost market demand. Therefore, in the future, several attempts can be overlooked to enhance the recovery efficiency of VFAs compounds.

- In-line recovery strategy of VFAs could be considered an alternative rapid purification medium because this stage interacts with the metabolic pathways of VFAs production. By applying this method, reduction of processing steps with feasible recovery percentages can be obtained.
- Using an external recovery unit coupled with membrane technology seems to be a promising solution for modulating the recovery process. Besides, the in-situ recovery process shows a significant downstream process without affecting the production of biogas. Overall, these approaches have demonstrated a potential scope in determining the recovery percentage of VFAs.
- Portal recovery of VFAs is another newly suggested approach for improving the process intensification, in which recirculation during the purification operation could be a great benefit for the elongation of purified VFAs.

6. Conclusion

In this review, various approaches for VFAs separation and concentration has been discussed with the emphasis on membrane filtration technologies. Pressure driven membrane filtration processes have proven to be promising options for the recovery of VFAs in high concentrations and high purity. Membrane properties and feed characteristics play a pivotal

role in terms of membrane VFAs recovery success. Among defining membrane related parameters, VFAs recovery is most sensitive to membrane chemistry and pore size, while temperature and medium pH are feed and operation related parameters of importance. However, a low amount of VFAs with variable organic content in the substrate still remains a challenge for full-scale industrial recovery processes that limits the development of waste-based biorefineries. By controlling effluent composition, filtration operational parameters, and correct selection of membrane, membrane separation can contribute greatly to the range and quality of products in a biorefinery.

CRediT authorship contribution statement

M.N.P., **A.M.**, **C.U.**: Concept, Data analysis, Writing – original draft. **M.N.P.**, **A.M.**, **T.Z.**, **V.B.**, **V.N.** and **M.T.**: Writing – review & editing. **A.M.** and **M.T.**: Conceptualization, Funding acquisition, Project administration, Supervision, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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