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Sustainable nutrient recovery from animal manure: A review of current best practice technology and the potential for freeze concentration

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A R T I C L E I N F O

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A B S T R A C T

Current trends of livestock expansion and associated mass production of manure bring a net import of nutrients that have led to a significant excess in many areas. The implementation of an efficient and more economical technology solution to recover and re-use nutrients from raw or digested wastes is essential and will reduce the need for fossil-fuel based fertilizers. From a waste management standpoint, the identification of nutrient recovery technologies is considered one of the main challenges within a circular economy context. Several traditional techniques exist for manure treatment such as, gasification, thermochemical conversion, composting, hydrothermal carbonization, and liquefaction. However, these technologies face many challenges related to energy consumption and recovered nutrient quality. In this context, freeze concentration (FC) is an emerging technique that can be applied to recover water and concentrate nutrients from waste liquid effluents. This technology brings advantages such as high concentration factor and low energy usage. However, freeze concentration technology is only semi-industrialised and for most applications remains at the development stage. Many studies have been conducted to design and develop processes and applications that target the improvement of both productivity and efficiency, which makes freeze concentration an attractive research subject to the scientific community. Combination of freeze concentration technology with another technology, such as membranes, to generate a more efficient hybrid process must also be considered. This approach of resource recovery from animal manure would ultimately create a more sustainable and circular economy. This paper evaluates the current state-of-the-art and processing strategies related to the treatment of livestock waste materials and contains an up-to-date and critical review on nutrient-rich effluent valorization technologies; focusing on the latest technological progress to recover nutrients from animal manure and introduces the potential that freeze concentration offers, which has only been marginally explored to date. This work makes a comparative analysis of the different processes in terms of their efficiency, cost, energy consumption, operational management, and the results obtained from both bench and large-scale experiments; making it possible to determine the current best practice procedures for the treatment of animal manure.

Abbreviations: AD, Anaerobic digestion; ZLD, Zero liquid discharge; BMED, Bipolar membrane electro-dialysis; FD, Freeze desalination; FC, Freeze concentration; MD, Membrane distillation; EFC, Eutectic Freeze Crystallization; SS, Suspended solids; SFC, Suspension freeze concentration; TDS, Total dissolved solids; PFC, Progressive or layer freeze concentration; BFC, Block freeze concentration; INP, Ice nucleation protein; VOCs, Volatile components; RO, Reverse osmosis; IPCF, Indirect progressive contact freezing; LNG, Liquefied natural gas; MDC, Membrane distillation-crystallization.

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1. Introduction

1.1. Livestock waste production and the resulting environmental impact

According to a recent report, world population has doubled in the last 40 years and reached 7.7 billion in 2019; with a current growth rate of 1.08% per year the population is estimated to reach 9 billion by 2037 (Max et al., 2019). Undoubtedly, this rapidly growing human population generates pressure on livestock production and subsequent consumption to satisfy the demand for protein. This has led to the development of intensive animal production, generating large quantities of livestock manure and turning the disposal of such waste into a serious worldwide environmental issue. Numerous national and international environmental control regulations have been developed to reduce the disposal of these wastes, however, large volumes of gas, organic materials, and other substances are still generated by manure, posing a significant risk factor for natural resources degradation (Van Dijk et al., 2016). A common example would be the ground or surface water pollution caused by the discharge of waste effluents on soils or into water bodies.

The damage caused by various livestock wastes generation and unsuitable management have been frequently enormous and even tragic. In Europe, pollution cost derived from manure management is estimated to be over 12,300 M euros per year (Bernal et al., 2015; Leip et al., 2011). The high moisture content (around 95–98%) of these wastes negatively affects any strategy for direct application in the surrounding areas and negates any plan to export them to other regions with low-nutrient soils as a source of biofertilizers to boost soil fertility and quality. Therefore, the challenge for EU countries, and other areas, is to integrate manure management and treatment into overall farm management plans (IAEA, 2008). In 2011, the EU reported a total of 1400 Mt of livestock waste production, out of this, 600 Mt are in the form of liquid manure from cattle and pig and about 300 Mt from solid cattle manure; the rest is produced by other livestock groups much of which is deposited on land by grazing animals (Buckwell and Nadeu, 2016). Statistical data in 2016 showed that over 50 Mt of livestock population is raised in Spain followed by France and Germany with around 40 Mt each (Fig. 1) (Eurostat, 2019a). In Spain and the United Kingdom, livestock generated over 118,697 and 139,248 thousand t/yr, respectively (Foged et al., 2011). France, Germany, Spain, and the United Kingdom recorded the highest total numbers of livestock units with 22.1, 18.2, 14.4, and 13.3 Munits/km^2 respectively. In contrast Malta, with 32,470 livestock units, recorded the lowest number of livestock units in the same year (Eurostat, 2019b). According to Petit-Boix and Leipold (2018), the implementation of long term strategic environmental policies to base cities evolution on circular economy, which leads to the growth of a more efficient and innovative economy, may rely on the production of bio-products and other materials from renewable resources. In this regard, anaerobic digestion of animal manure has opened a window of opportunity to mitigate the environmental burden derived from animal waste generation and enhance the production of bio-based products (Neshat et al., 2017). This valuable technology has been widely recognized as a powerful alternative for bioenergy production and effective to reduce greenhouse gas emissions (Achinas and Willem Euverink, 2019; Fagerström et al., 2018). The sustainable management of manure to recover nutrients is of paramount importance to create new, high-quality fertilizers and depends significantly on infrastructure and handling. Ammonia evaporation, nutrient leaching, and pathogen contamination are some of the major drawbacks of direct application of manure into the soil (Bernal et al., 2015). Therefore, before introduction of livestock manure as fertilizer into the market, it should be treated and undesirable compounds and minerals must be removed or recovered (Bernal et al., 2015). Different management strategies and treatment technologies have already been tested and scaled-up during recent times to optimize the volume reduction, nutrient concentration and final transportation or exportation of livestock wastes (Martinez et al., 2009). However, an overall nutrient recovery strategy is still required to meet

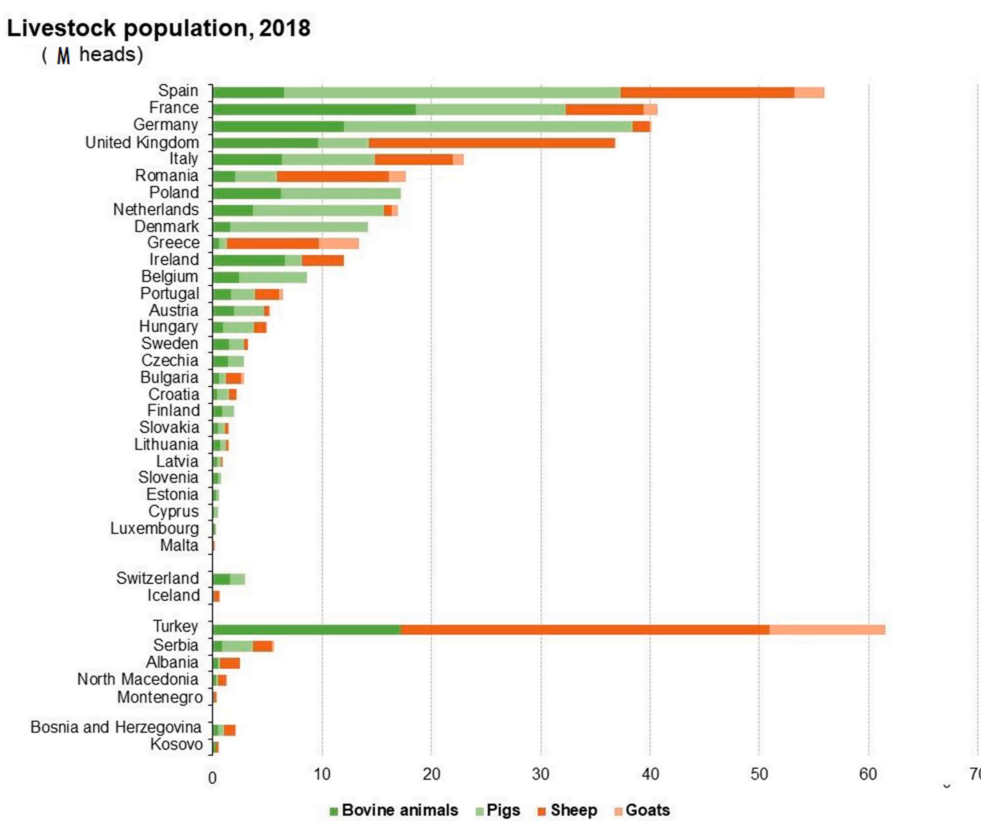

Fig. 1. Quantitative livestock production across Europe (Eurostat, 2019a).

environmental, economic and market needs. Operational problems, regulatory constraints, and the instability of market prices associated to the production of fertilizers, in terms of both quality and quantity, makes setting a global strategy difficult. The key goal would be to find an appropriate technology, or combination of technologies, to optimize the treatment of livestock waste and generate recycled value-added materials in a true circular economy.

1.2. Current fertilizer trends

Fertilizers are the largest piece of the agricultural market input and the consumption of fertilizers has increased remarkably over the last decades, driven by the need to feed a rapidly expanding global population (FAO, 2017). The global market demand for fertilizer was 186.6 and 194.4 Mt in 2016 and 2018 respectively and is expected to grow nearly 2% annually, reaching 201.7 Mt by the end of 2020 (FAO, 2019b). Evidence from many studies clearly indicates that the continuous application of chemical fertilizer causes serious environmental problems; such as groundwater pollution, deterioration of soil, and loss of nutrients (Chandini et al., 2019). The replacement of chemical fertilizers by biowaste-based materials (such as bio fertilizer) has received much attention and is currently known as the green revolution (Chandini et al., 2019). Biofertilizers are not only rich in organic carbon and valuable nutrients, but also supply productive microbes to the soil following application. This helps to reduce the ecological disturbance caused during application when compared to chemical fertilizers (Chojnacka et al., 2020).

Research has demonstrated that manure-based fertilization is an alternative to chemical fertilization, not only achieving high crop yields and improved soil quality, but also promoting sustainability and efficiency of agricultural ecosystems in the long term (Altieri et al., 2017; Geng et al., 2019). The use of chemical fertilizer is prohibited, especially for organic farming. Thus, the application of manure serves as an important resource to replenish the organic matter content of cultivated soils and also supplies plant nutrients (Song et al., 2017). Financial valuation of manure would be dependent on the market value of the required plant nutrients that the manure will replace (Chojnacka et al., 2020). For intensive farming areas where tonnes of animal wastes are used daily, oversaturation of nutrient can occur and a proper management strategy to recover nutrients from manure prior to application is essential (Rayne and Aula, 2020).

In some areas there are legal regulations that require the recovery of nutrients. For example, the German sewage sludge ordinance for wastewater treatment facilities mandates the recovery of phosphorus from sewage sludge in this country. The recovery of phosphorous from wastewater treatment plants has also become mandatory in Sweden, Switzerland and Austria (Günther et al., 2018). This trend is highly likely to extend to animal waste. Previous reviews for nutrient recovery processes have predominantly focused on conventional treatment methods for food wastes and limited information is available on current nutrient recovery technologies from animal wastes (Font-Palma, 2019; Mehta et al., 2015). The aim of this review is to highlight the technologies currently available for nutrient recovery and introduce the potential for nutrient recovery using freeze concentration as an innovative technology for the treatment of animal manure.

1.3. Method

In this paper, we review the rapidly growing body of academic literature on available nutrient recovery technologies for animal manure. We go beyond the sole focus on conventional methods and highlight the most recent environmentally friendly and cost-effective technologies. The purpose of this work is to clarify the processes that are currently being applied for the treatment of livestock waste. In addition, the generation of new value-added products, such as natural fertilizers, the development of sustainable production lines and areas of environmental best practice is discussed. Therefore, studies related to new and emerging technologies, as well as their combination, are analysed and detailed in this work in order to emphasize current best practice. The databases Web of Science, Scopus, and Google Scholar were used as a basis for the literature search. Subsequently, the articles generated from the initial search were checked manually mainly by reading through the abstract. We excluded studies that did not focus on livestock manure. This pool of literature was further developed by checking the references of the articles yielded by the initial search.

2. Composition of animal manure

The characterization of livestock manure would be advantageous to optimally select appropriate treatment or valorising technologies (Malomo et al., 2018). Determining the chemical composition of animal manure is difficult due to high variability and changes in the concentration of nutrients. Many factors could affect the chemical and nutrient composition of animal manure such as environmental factors, animal species and classes of animals, feeding pattern, stage of animal growth (nutrient intake, digestion, and absorption) as well as storage time of the manure (Celi et al., 2017). Table 1 highlights the useful components of manure that can be obtained from animal wastes.

2.1. Factors affecting manure composition

2.2.1. Animal feeding patterns

Nutrients required by livestock depend on many factors such as age, gender, animal type or production stage, resulting in different feeding patterns and manure composition. For instance, different quantities of nutrients are required by young animals or lactating and mature or gestating animals (FAO, 2012). Some nutrients are retained in livestock, while they gain weight, also transferred to eggs or milk produced by the animal (Erickson and Kalscheur, 2020). Those nutrients that are not retained or absorbed by the animal are excreted ending up in the manure. Other contributing factors include the amount of milk produced or the feed composition. About 50–90% of the nitrogen and phosphorous content in the animal feed is not absorbed in the livestock digestive system and even less for fully grown animals (not gaining weight or producing milk) with almost 100% of nutrient excretion (FAO, 2012). A more accurate adjustment of feed composition and quantity (based on the location, age, and type of animal) will provide an opportunity to decrease the quantity of nutrients contained in the resulting manure (Šebek et al., 2014). For example, a correct amino acid and protein balance in animal feed would cause a significant reduction of nitrogen in both the urine and faeces (NRC, 1989).

2.2.2. Manure storage

The storage design and structure of a slurry pit specify the surface area which is exposed to air, the amount of rainfall that enters storage, evaporation losses and volatilization of ammonia gas. Covering manure storage greatly reduces evaporation losses and consequently increases moisture content. Liquid manure storage with smaller surface areas is

Table 1
Useful components contained in manure (Manitoba, 2015).

Manure component	Use	Benefit
Energy	Bio oil and Biogas	Supplementary source of energy; Significant reduction of relying on fossil fuels
Fiber	Building material and paper	AS an environmentally friendly source turned into lucrative goods
Organic matter	Soil relief	Reclaim of soil construction and improving water holding capacity
Nutrients	Compost, fertilizers, animal feed	Cost effectiveness strategy to replace chemical fertilizers and revenue output from sales of manure

also effective in reducing nitrogen losses (Government, 2019).

2.2.3. Weather

Seasonal changes in precipitation and temperature can alter the nutrient content of slurries through volatilization, dilution, and evaporation, especially in uncovered storage systems. Year to year variations in weather (e.g. wet or dry, warm or cool) affect manure composition (Manitoba, 2015). For instance, higher temperatures enhance the enzymatic production of ammonia from urea and generates higher ammonia emissions, which contributes to nitrogen loss from manure.

2.2.4. Water usage

The amount of water consumed by the animal will affect the resulting manure composition. This amount depends on the stage of growth, animal species, feed intake and barn temperature (FAO, 2019a). For example, during winter a mature cow consumes about 80 L of water every day, nonetheless this consumption may increase above 140 L/d in summer. The feeding equipment and the method used for cooling and washing animals could also influence the amount of water in manure (FAO, 2019a).

2.2. Nutrient content of animal manures

Livestock manures are valuable sources of organic matter and nutrients and are used as fertilizers to improve crop yield. However, knowledge about the specific nutrient content of the manure is of paramount importance for this task (Bhogal et al., 2018; Lpelc, 2019). Table 2 shows typical compositions and characteristics of different animal manures. Considering the high variability in animal manure composition observed from one location to another, preference should be given to locally derived manure characteristics (Malomo et al., 2018). Nonetheless, the information presented in Table 2 can serve as an initial guide to the expected composition of animal slurries that can be further processed for nutrient recycling within crop production systems.

2.2.1. Nitrogen in manure

Nitrogen is present in manure in two forms, namely organic and inorganic. The inorganic form of nitrogen (consisting primarily of ammoniacal nitrogen NH_4–N) is a fast release form and is immediately available for plant growth. In contrast, organic nitrogen is slowly released and provides a longer term mineralized form of nitrogen for plants (Lpelc, 2019). During field application and storage, inorganic nitrogen is susceptible to losses in the form of NH_3 through volatilization. Rapid integration of manure into the soil may cut down these nitrogen losses. In addition, the organic form or nitrogen is slow release, i.e. generally not accessible to the plant during the year of application. The quantity of unavailable nitrogen has no value for the plant, whereas nitrogen that is likely to be directly available to the plant has value as a fertilizer. According to Soliman et al. (2017), following about 4 h post collection, the ammonia content of manure increases six fold, as a result of uric acid breakdown.

2.2.2. Potassium (K) and phosphorus (P) in manure

P and K are present in manure and are easily accessible for plant uptake. The quantity of these elements required in a fertilizer is based on crop nutrient needs as specified by yield goals and soil testing (Szogi et al., 2015). The solubility of these nutrients can also be reduced in defecation, especially for P as phytate (stable molecule) in cereals for animals feeding (Szogi et al., 2015). As an example, pigs and chickens are not able to absorb phytate; and this form of phosphorus is excreted through the faeces. Generally, the nutrient content in animal manure also vary widely depending on the place of breeding. Harada et al. (1993) reported that nutrient content such as calcium, magnesium, potassium and nitrogen in Japanese animal manure is in the following order: poultry > swine > cattle.

2.2.3. Micronutrients in manure

Besides containing significant amounts of potassium, nitrogen, and phosphorous, animal waste is a good source of other nutrients such as calcium, magnesium and sulphur. Micronutrients are needed in very small amount by plants and the addition of micronutrients is profitable when a deficiency exists or when certain crops have a high requirement for a specific micronutrient. Uniquely two micro elements that may not be fully supplied by manure are boron and zinc (Shahid et al., 2016).

3. Conventional nutrient recovery technologies

3.1. Biological technologies

3.1.1. Anaerobic digestion

The concept of biological treatment is the use of microorganisms, which are naturally present in the animal waste or are added artificially, to reduce the biological oxygen demand of the waste and release nutrients. Anaerobic digestion (AD) is a common biological treatment technology for the hydrolysis of organic solids, pathogen reduction, stabilization of wastes and energy recovery through biogas generation from livestock manure (Logan and Visvanathan, 2019). This process is driven by anaerobic microorganisms in an anaerobic digester and converts organic phosphorus to the soluble form and organic matter into ammonium, carbon dioxide, methane, hydrogen sulphide and other volatile compounds (dependent on the digester operational conditions) (Anukam et al., 2019). This method is widely used across the world especially in USA and EU to treat manure and wastewater generated from dairy industry or farming (Font-Palma, 2019). At the end of the AD process, the released nutrients from digested wastes will turn into inorganic forms or get adsorbed onto the solid surface of digested materials (Nag et al., 2019). Studies demonstrate that in the AD process, phosphorus is mostly released in the organic form, however, less than 10% of this organic form remains soluble after digestion (Hongjian et al., 2015). Open AD causes a reduction of the nitrogen content through ammonia volatilization (Grant et al., 2013). The removal of ammonia from slurry manure could be carried out using steam stripping. The steam stripping method is a platform for production of chemicals and

Table 2
Typical nutrient composition of various animal manures (Lpelc, 2019).

Animal manure	Total Nitrogen (kg/t)	Total Phosphate (kg/t)	Total Potash (kg/t)	Ammonium Nitrogen (kg/t)	Nitrate Nitrogen (kg/t)	Dry Matter (%)
Broiler/turkey litter	30	25	18	6.2	0.2	60
Duck (fresh)	6.5	5.5	7.5	1.6	0.0	25
Duck (old)	6.5	5.5	7.5	0.9	0.1	25
Sheep (fresh)	7	3.2	8.0	1.4	0.0	25
Sheep (old)	7	3.2	8.0	0.6	0.1	25
Pig (fresh)	7	8.0	6.0	1.8	0.0	25
Pig (old)	7	8.0	6.0	0.9	0.1	25
Cattle (fresh)	6	8.0	3.2	1.2	0.0	25
Cattle (old)	6	8.0	3.2	0.5	0.1	25
Horse	7	5.0	6.0	0.6	0.1	30
Layers	19.0	14.0	9.5	5.6	0.2	35
Goat	6	3.2	8.0	0.5	0.1	25

base fertilizers (Zarebska et al., 2015). This method is expensive and should only be implemented if the ammonia recovery is of commercial benefit. For example, the ammonia released could easily absorbed by sulfuric acid to produce ammonium sulphate (Zeng et al., 2006). This technology, coupled to an AD process, is an economically promising technology to regulate the concentration of ammonia in the digester since this compound inhibits the activation of the methanogenic bacteria.

Reduction of solids handling costs for AD processes requires the solid digested parts to be dewatered to produce a by-product rich in nutrients (mainly K and N). This nutrient-rich by-product can be a substrate for nutrient recovery purposes and the remaining materials (particle bound nutrients), considered as bio-solids, have value as nutrient amendments for agricultural applications (Buckwell and Nadeu, 2016). The AD process is not a nutrient valorization technique but a technology used to produce local energy for farms. Nutrient recovery could take advantage of other techniques rather than AD process to recycle nutrients in a sustainable and environmentally safe manner.

3.1.2. Bioleaching

Bioleaching is a low-cost technology based on nutrient solubilisation from solid substrates by a leaching microorganism, either through direct or indirect metabolism. Some potential microorganisms which have been identified for bioleaching are: *Acetobacter, Acidibiobacillus ferrooxidance, Pencillium, Fusarium, Sulfobacillus thermosulfidoxidans*, and *Aspergillus* (Mercier et al., 2006; Zaosheng et al., 2011). These microorganisms have the ability to grow in acidic conditions and perform oxidation of sulphur/iron compounds and the release of heavy metals and nutrients (Pathak et al., 2009a; Zaosheng et al., 2011). This technology is a cost effective process due to capability of using chemically bound metals which are already present in sufficient quantities in wastes. The only disadvantage of the bioleaching process is the slow release of phosphorus and nitrogen compared with undesirable heavy metals, generating a requirement for further separation processes (Pathak et al., 2009b).

3.1.3. Biodrying/composting

The application of bio-thermal drying processes is an interesting option to stabilize the solid fraction of manures. As an example, applying a composting intensive process (at 70 °C), such as biodrying, to the solid fraction of pig manure takes only a few days and has been carried out frequently for pasteurization purposes (David et al., 2017; Shi et al., 2018) or biofuel production. The final dried product can be used as an organic fertilizer and application of these materials enhance the soil structure (Lin et al., 2019; Monfet et al., 2018). Certain physicochemical properties of manure are not suitable for this process and could limit the efficiency of the composting, for instance, high moisture content, high N/C ratio, and high pH in certain manures (Wright and Inglis, 2002).

Composting of livestock manure would produce a fertilizer with less N, as this would be lost through volatilization during the composting process, although fully sanitized, stabilized and more mineralized than a bio-dried material. Many strategies have been applied to control the cost, time, avoid N volatilization, and improve the quality of compost. For instance, Bautista et al. (2011) used alum or zeolite as amendments and could reduce the emission of ammonia from pig manure compost to about 92%. This means more N remains in the final compost compared with the unamended control (Bautista et al., 2011). The main advantages of animal manure composting, prior to direct addition to the soil without treatment is the significant reduction of moisture content, odour removal, sanitation, and easier transportation (Millner et al., 2014). There are some drawbacks associated to the application of composting, namely: large areas for operation and storage, cost of installation and additional cost for amendments (Sweeten and Auvermann, 2008). The application of composting technologies to valorise animal manure adds value to the high-quality final product obtained, which better meets the requirements of the fertilizer market.

3. 2Physicochemical technologies

3.2.1. Compaction

This is a physical technique, based on baling and pelletizing processes, that boosts the handling and storage of solids contained in manure (Bernhart and Fasina, 2009). During the pelletizing process, the density of animal manure increases significantly. For example, McMullen et al. (2005) reported the fourfold increase for the bulk density of poultry litter to reach 790 kg m^{-3} after pelletizing with vegetable oil (McMullen et al., 2005). Optimisation of the energy requirement and moisture content resulting from the compaction process is essential. Due to the high equipment cost and energy demand required for compaction, this technology is economically unfeasible in some activities such as poultry farming. For this reason, innovative methodologies are being developed, as lower energy alternatives to pelletizing, such as the combined process of wrapping and compression (Szogi et al., 2015).

3.2.2. Chemical amendments

An alternative to recover target nutrients from digested manure is to form struvite (precipitation of substrates) in order to get a slow release of fertilizing compounds into the soil (Shi et al., 2018). Struvite formation is strongly affected by temperature, suspended solids, ion concentration and pH. For example, if magnesium ion concentration in the digestate is insufficient, magnesium chloride or magnesium sulphate must be supplemented to induce struvite formation. A high concentration of magnesium ions would negatively affect the calcium ion precipitation and would adversely impact the recovery yield of struvite (Tervahauta et al., 2014). Both pH and temperature have impact on solubility and crystal morphology of nutrients respectively and consequently affect struvite formation. For instance, during the recovery of P and N from digestate using insufficient magnesium creates a possibility of PO_4^{3-} precipitation when pH increases from 5 to 7.5, while the optimum pH is in the range of 8.5–9.5, and consequently this would cause a secondary pollution by permeation into groundwater (Shi et al., 2018). Another alternative that has been widely used in the USA to reduce or control the ammonium release from manure is the application of sodium bisulphite (Szogi et al., 2015). Various pre-treatment methods have been applied to increase the concentration of target macro and micronutrients in their soluble form such as ultrasonic, microwave, acidification and heating; however, these chemical amendment techniques are costly, especially at large scale (Cerrillo et al., 2015; Kataki et al., 2016).

3.2.3. Thermochemical treatments

Thermochemical treatments such as incineration, gasification, hydrolysis, pyrolysis, and hydrothermal carbonization could convert biomass into gases and ash residues and reduce the bulk volume of wastes (Liu et al., 2018; Shi et al., 2018). Direct combustion of solid organic wastes in the presence of air have been performed at full scale as an energetic valorization process of municipal sludge in Europe (Vaneeckhaute et al., 2017). Lynch et al. (2013) reported the potential of poultry litter incineration to recover phosphorous. They illustrated that after incineration of poultry litter, the average P content was about 110 g kg^{-1} in the ash, while no N content was recoded as reduction to gaseous emissions occurred during the incineration process. Incineration produces gaseous pollutants which are introduced to the atmospheric environment, this is a particular issue for the incineration of sewage sludge which is loaded with both organic and inorganic pollutants. Moreover, removal of these gaseous pollutants will create additional cost for the waste treatment process. Gasification and hydrolysis at reaction temperatures of 800 °C and 400 °C produce synthesis gases (Panigrahi et al., 2003), bio-oil, and biochar respectively (Jadhav et al., 2019). Hydrolysis is a promising technology to manage animal manure and add value by producing biochar as a fertilizer (Cantrell et al., 2012). Hydrolysis requires large sums of energy to evaporate moisture from manure while generating a low gas output. Therefore, from an energetic point of view, hydrolysis is frequently unfeasible, although in specific cases hydrolysis is an adequate solution as with the hydrolysis of spent plastic mulch wastes with swine manure that produces a combustible gas (higher value than natural gas) along with biochar (Ro et al., 2014).

Hydrothermal carbonization is a less energy intensive technology and has been applied to treat digested and fresh manure through a first hydrolysis of the biomaterial at 170 °C followed by carbonization at 250 °C. The final product is a valuable solid char (hydrochar) and is used as a sustainable sorbent for pollutants. As the evaporation of water is avoided, this technology requires less energy than other thermal treatments and the technology has received increasing attention in the last decade. Reza et al. (2016) observed that most of the K and almost half of the N were mobilized after processing cow manure through hydrothermal carbonization. Compared to pyrolysis method, the liquid fraction produced by hydrothermal carbonization process can be applied as a liquid fertilizer because of the high ammonium concentration, while in pyrolysis technique a pre-drying of materials to be hydrothermally carbonized is not required. Lucian and Fiori (2017) analysed the techno-economical aspects of the hydrothermal manure carbonization process in detail and estimated the production cost of pelletized hydrochar to be 157 euro per tonne.

3.2.4. Membrane filtration

Membrane technologies target treating those effluents containing compounds or elements that may be either retained or pass through a thin physical barrier depending on molecular or particle size, concentration of certain compounds, operational temperature and applied pressure (Logan and Visvanathan, 2019; Van-Beek et al., 2018). Many parameters should be considered to apply membrane filtration such as the chemical composition of the effluent to be treated and the interactions between membrane surface and components in the feed flow (Logan and Visvanathan, 2019). According to the literature, the utilization of membranes have been proven effective to concentrate and purify nutrients (Ainscough et al., 2017; Massias et al., 2015). Microfiltration and ultrafiltration membranes are basically aimed to remove particles, while reverse osmosis and nanofiltration membranes can be applied as nutrient recovery techniques (Shi et al., 2018). Zacharof et al. (2019) reported the potential of nanofiltration to recover phosphorous and ammonia using membranes at pilot scale and achieved the retention of 31.8 and 13.4 mmol L^{-1} of P and ammonium respectively. In a similar work conducted by Gerardo et al. (2015), over 90% of N recovery was achieved treating dairy manure digestate through membrane technology. They reported that the separation and filtration of P and N are correlated to pH, membrane electric charge, and ionic speciation. Molinuevo-Salces et al. (2018) investigated the performance of two anaerobic digesters coupled to gas-permeable membrane technologies, targeting the recovery of N from liquid manures in a concentrated stable ammonium solution and achieved ammonia and organic matter removal efficiencies of 96% and 69%, respectively; the final product could be used as a stable fertilizing salt solution. A recent study conducted by Shi et al. (2020) assessed the application of three bipolar membrane electro-dialysis (BMED) units configured for nutrient recovery from animal manure. The base- BMED presented an insufficient technique for recovery of ammonia (only 60% was recovered), while the integration of two operation modes i.e. acid-BMED and base-BMED demonstrated a significant increase in the concentration of ammonia to 16 gL^{-1} compared to the feed solution with 5.1 gL^{-1} ammonia. One major drawback associated with membrane separation technologies is membrane fouling, which negatively affects the process efficiency, application and durability due to the increasing hydraulic resistance (Fierascu et al., 2019). The fouling issue is a complex phenomenon which mainly depends on the feed stream composition and causes flux decline. Fouling reduces the membrane performance and productivity (regardless of its type) and requires additional costs for cleaning and maintenance. Membrane technology is the main separation technique used for industrial and for commercial applications as the technique provides high recovery rates of over 98% with comparatively low capital cost (Komesu et al., 2017).

4. Freeze concentration technique

The valorization of animal manure via biological or physical-chemical technologies is rare; this is mainly due to perceived high cost (either capital investment or operational cost) or the availability of space to conduct the process. Freeze concentration (FC) has become a technology of interest due to the low temperature of the process (Sánchez et al., 2009). The use of FC has been reported by many sectors including: concentration of liquid foods, seawater desalination, purification of organic chemicals, and treatment of hazardous wastewater (Chen et al., 2019; Htira et al., 2018). Regardless of the application, the freezing temperature should be below the freezing point of the treated solution. The performance of newly developed FC methods is evaluated according to energy consumption and the feasibility of the method when compared to the existing in-place technology. FC is advantageous as energy required to evaporate water is seven times greater than that to freeze water (2500 kJ kg^{-1} vs 335 kJ g^{-1}) (Pazmiño et al., 2017). This does not take into account the possibility of energy recovery in both processes, where the economic potential benefits of using FC remain high. The experience with full-scale FC in a variety of industries shows that the process is superior in terms of efficiency to state-of-the-art mechanical vapor recompression and thermal vapor recompression evaporators (Pazmiño et al., 2017). Evaporation shows outstanding results in terms of concentration obtained compared to membrane technology which is limited by the osmotic pressure limit (Hubbe et al., 2018). Perhaps, the potent advantage of FC are the low energy and temperature requirements that will significantly reduce energy consumption when compared to other technologies. Similarly, low temperature operation leads to low-cost materials of construction for the process equipment. Use of plastics rather than steels has a major improvement to the carbon footprint of the resulting equipment and, with the advent of bio-plastics, this could improve even further with time. All of these aspects offer a potentially significant environmental improvement over other technologies.

Thus, FC is an inexpensive technology with minimal corrosion and scaling problems. For desalination, crystallization, juice concentration and general water removal, the initial solution to be frozen does not need a pretreatment step, thus chemicals typically required for pretreatment are avoided. There is no fouling as with membrane separation and low ecological impact. In the next sections, the following key aspects of FC will be presented and discussed: fundamentals of the process, classification, industrial applications and applications for animal manures. The discussions will clarify where the application of FC technology is appropriate to treat or valorise animal manure and will emphasize the potential environmental improvement.

4.1. Principle and the general concept of freeze concentration

Crystallization processes can be divided into crystallization from melts and crystallization from solutions (Fig. 2). In the case of melt crystallization, the crystallizing species are the main component (solvent) of a liquid mixture. Supersaturation in melt crystallization is mostly created by cooling or modifying the pressure, although a high-pressure difference is required. In the case of solution crystallization, the crystallizing species are one of the minor components of the liquid mixture (solute). For highly soluble substances, like sugars and highly soluble salts, the difference between solution crystallization and melt crystallization becomes unclear (Pronk, 2006). Some authors suggest that whenever heat transfer dominates the phase change process, this should be called melt crystallization, while in solution crystallization mass transfer dominates the process (Ulrich et al., 1988). Eutectic Freeze Crystallization (EFC) can be considered as a combination of both melt and solution crystallization since water and solute crystallize

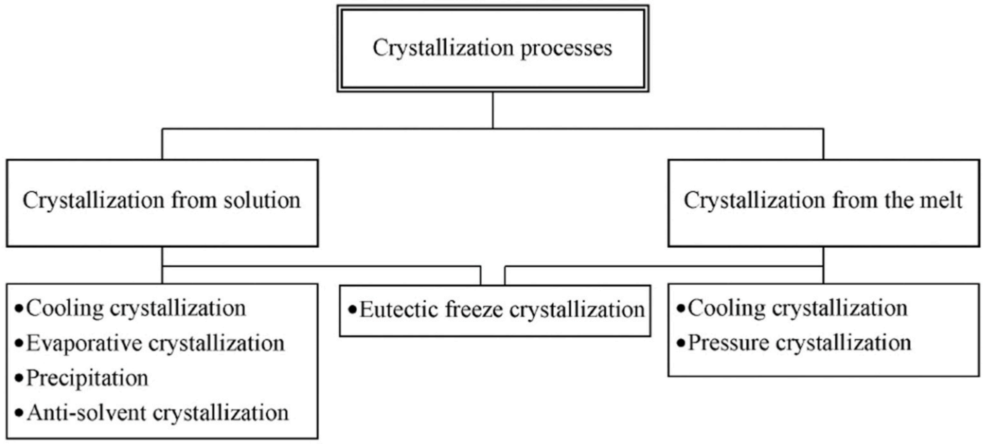

Fig. 2. Overview of crystallization processes (Pronk, 2006).

simultaneously (Williams et al., 2015).

The different types of crystallization can be seen in a typical binary phase diagram of water solution, as shown in Fig. 3. Separation by freezing is based on solid-liquid phase equilibrium and depending on the initial concentration, different products can be obtained in a crystallization process by freezing. If the initial concentration of the solution is higher than the eutectic concentration, the solute will crystallize first. In contrast, if the initial concentration is lower than the eutectic concentration, ice will crystallize first. Finally, if the solution concentration is equal to the eutectic concentration, the crystallization of ice and solute occurs simultaneously. FC is a particular type of cooling crystallization from the melt, in which water is separated from the liquid through ice crystallization at low temperature, followed by a separation step to remove ice from the concentrate (Fig. 4) (Lu et al., 2017). This technology involves lowering the temperature of the product in a sufficiently controlled manner to partially freeze the product, resulting in the formation of ice crystals in a fluid concentrate. If formed under the appropriate conditions, these ice crystals will be very pure. This results in the formation of ice crystals with the lowest possible incorporation of product solute. The ice crystals are then removed whilst maintaining a

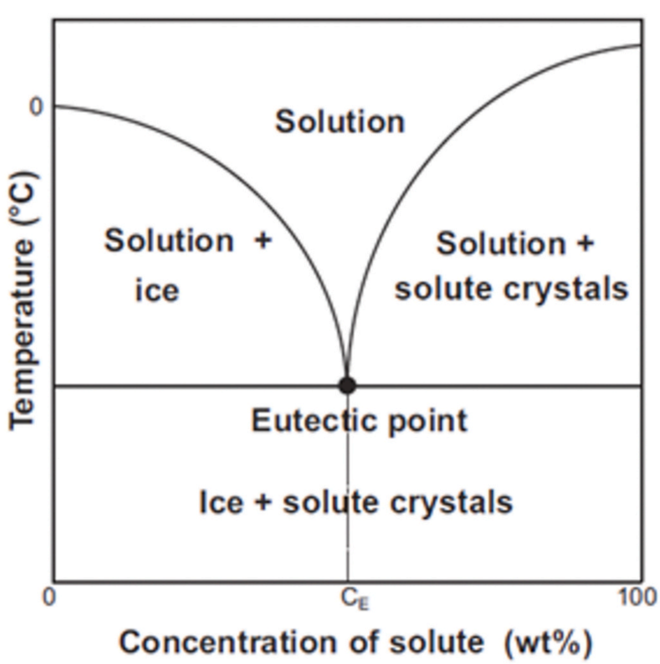

Fig. 3. Binary phase diagram for a solute in water (Randall and Nathoo, 2015).

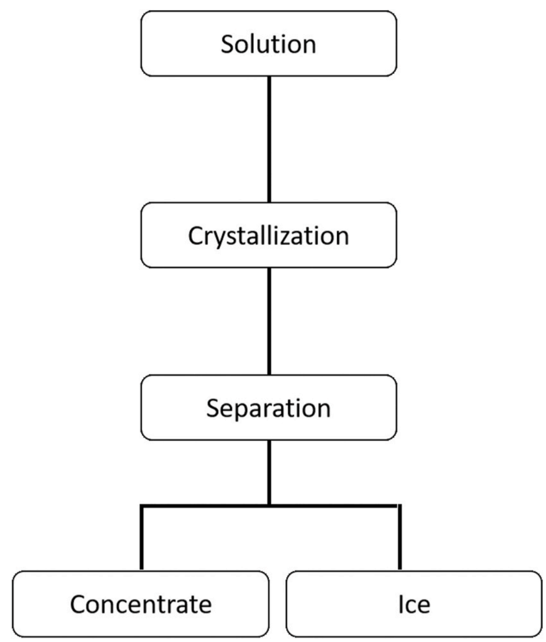

Fig. 4. Schematic for a basic freeze concentration process (Berk, 2009).

minimum of liquid carryover, resulting in a concentrated product. The process of FC is dependent on two main characteristics of the concentrate product. The maximum concentration obtainable is determined by the viscosity of the concentrate and the freezing point (Ruemekorf, 2000). The maximum concentration is reached when the viscosity of the liquid prevents the growth of ice crystals (Van-Beek et al., 2018). The crystal growth rate decreases as viscosity increases and the system requires a longer residence time (and thus larger equipment) to reach a separable crystal size because the capacity of ice separator is inversely proportional to the viscosity (Petzold and Aguilera, 2009). The freezing point depression due to the solute concentration can be so great that the lower temperature limit for the refrigerant may be reached and results in the need for a multi-stage refrigeration system, which is usually too expensive to be feasible. Typically, commercial systems in the food industry operate with up to 45–55% of total dissolved solids (Van Nistelrooij, 2013).

4.2. Classification of freeze concentration

FC processes can be classified by the contact mechanism between the refrigerant and the solution. In the direct freezing, the refrigerant used to cool the solution is mixed directly with the solution and promotes highly efficient heat transfer. Other advantages include the high production rate per unit volume at a low driving force, low power consumption, absence of moving parts, and a compact and efficient unit (Rahman and Al-Khusaibi, 2014). The major drawback is the refrigerant contamination. Also, the use of a flammable and potentially explosive refrigerant, such as butane, creates a major safety concern (Randall and Nathoo, 2015). In contrast, indirect freezing is obtained without direct contact and occurs via some form of heat exchanger device. One of the advantages of this system is no interaction between solution and refrigerant, which allows the separation of ice crystals and concentrate solution in the same equipment. The main disadvantage is the large heat transfer resistance of the wall and ice layers, which result in the requirement for a large heat transfer area with low heat transfer rate and long crystallization time (Rahman et al., 2007).

4.2.1. *Direct systems*

There are three main direct FC methods, namely:

a) *Direct contact or triple point (vacuum freezing):* direct primary or triple point systems operate near to the triple point of a solution, which is the temperature and pressure at which solid, liquid, and vapor of a mixture occur simultaneously. The water itself acts as the refrigerant because as vaporization occurs, heat is removed which facilitates crystallization of the solution. The crystals formed are washed in a column and melted (Roos et al., 2003).
b) *Direct secondary:* in the direct secondary freeze method, a volatile liquid refrigerant is introduced into the solution. This refrigerant vaporizes at a higher temperature, cooling the remaining fluid and promoting the formation of crystals. The refrigerant applied in these systems must conform to several prerequisites to be successful, i.e. must be non-toxic and chemically stable during the process, immiscible in water, and available at low cost (Rahman et al., 2006). Control of the refrigerant, as well as contact with the solution, must be optimized to ensure that the final product is free of impurities (Kalista et al., 2018). Typical refrigerants include butane, propane, and carbon dioxide.
c) *Direct Clathrate (gas hydrate):* gas hydrates are crystalline solids formed when water and gas come into contact under elevated pressures and low temperatures. Gas molecules are enclosed or trapped within the water molecule lattice connected by hydrogen bonds. The most common hydrate structure can also be found by CO_2 and CH_4 hydrates. Since the gas hydrate can be formed above 0 °C, energy demands are lower than that in regular freezing. Hydrate technology has been received much interest in applications in the field of gas storage, CO_2 capture, and desalination. The state of the art of gas hydrate desalination processes is summarized in (Kalista et al., 2018). In the food industry, the use of this technology was reported for the concentration of liquid foods by the formation of CO_2 hydrates (Claβen et al., 2019).

4.2.2. *Indirect systems*

In the indirect process, there exists a wall separating the solution and refrigerant. The indirect-contact process can be subdivided into suspension freeze concentration (SFC), progressive or layer freeze concentration (PFC) and block freeze concentration (BFC).

a) *SFC:* in this process, small ice crystals grow large in suspension crystallizers and are purified by the Ostwald ripening mechanism (Fig. 5a). The effective ice removal and separation can be done in wash columns specifically developed for this purpose. Using this technique the impurity of the ice crystals are <100 ppm (Van-Beek et al., 2018), which is highly attractive. Although SFC has been used industrially in liquid food applications as a key technology, in practice the Ostwald ripening process needs a long operation time.

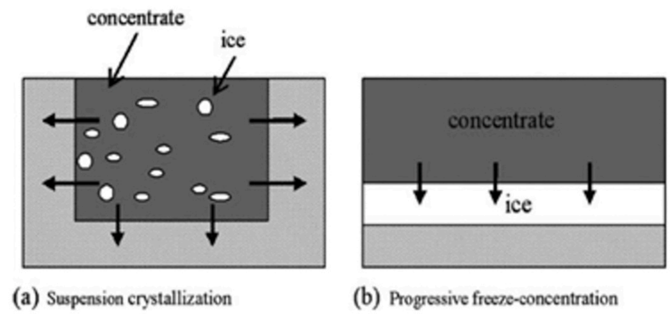

Fig. 5. Freeze concentration methods. a) SFC b) PFC. The direction of arrows represented heat transfer (Petzold and Aguilera, 2009).

Thus, SFC is usually limited to large scale processes in continuous operation mode. For this reason, this technology requires a high initial investment with less operational flexibility, limiting the practical application of SFC.
b) *PFC:* unlike the suspension method, progressive FC consists of the formation of a single ice crystal, which is formed layer by layer on the heat exchange surface (instead of many ice crystals as in SFC). The separation between the ice crystal and the concentrated solution is then easier and can be done in the same equipment, significantly reducing the cost of the operation (Fig. 5b). This process is used for purifying chemicals, but not at large scale. One reason for this is probably because solute inclusion or entrapment in the ice layer is hard to avoid during the growth process (Flesland, 1995). For PFC there exists a range of different equipment at various scales that have been applied to several food liquid applications (Sánchez et al., 2011).
c) *BFC:* in the case of the block system (BFC), also known as FC by freezing-thawing, the fluid is completely frozen and the temperature in the centre of the product is below the freezing point (Aider and de Halleux, 2009). Subsequently, the block is thawed, and a concentration gradient is observed among the thawed fractions with a higher concentration in the initial fractions (Fig. 6). The concentrated fraction is separated from the ice fraction by gravity, combined with techniques to improve solute performance through the application of centrifugal force, vacuum, microwave, annealing, ice nucleation protein (INP), among others. One of the main advantages of this technique is related to the absence of moving parts, like stirrers or pumps, which offers a reduction in production costs. The concentration efficiency of BFC is limited and multistage operations are inevitable (Aider and de Halleux, 2008) to obtain a high level of concentration, which requires high energy consumption. This technique is still at the laboratory stage of development.

4.2.3. *Eutectic freeze crystallization (EFC)*

EFC has been examined to recover water and salts from solutions. The solution is cooled down to a eutectic temperature where both salt

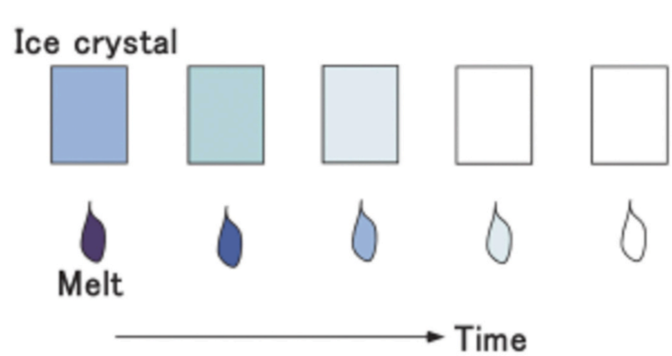

Fig. 6. The principle of partial ice-melting for a frozen block (Miyawaki, 2018).

and ice simultaneously crystallize (Lu et al., 2017). The salt, being denser than the solution and ice, sinks to the bottom of the crystallizer while the ice, being less dense, floats to the top. Theoretically, the technique can handle all kinds of soluble contaminants with removal efficiency close to 100%. The EFC process has used both direct and indirect freezing to reach eutectic conditions (Fig. 7). The advantages of this process are no use of chemicals, low temperatures resulting in less corrosion, and a safe process easily controlled. Reviews on the use of the EFC technique in desalination (Kalista et al., 2018), wastewater treatment (Lu et al., 2017), and reverse osmosis brine (Randall and Nathoo, 2015) are available. In the case of food liquids, the FC process can only be applied to a concentration just below that of the eutectic point, this is because problems arise when separating ice from a very viscous liquid (both increased concentration and low temperature create difficulty) (Deshpande et al., 1984).

4.3. Advantages and disadvantages of the freeze concentration technique

FC is of particular interest due to the low temperatures used in the process and has been explored industrially for food processing, but has merit for various other sectors such as seawater desalination and wastewater treatment. The major advantage for FC is the relatively low energy consumption when compared to evaporative processes and could compete with membrane separations at large scale production volumes (Table 3). Throughout history, many methods have been developed to optimize the efficiency of the FC process and to improve the quality of the final product. Regardless the sector of application FC, also known as Cryo-concentration, the freezing temperature should always be below the freezing point of the treated solution. The performance of novel FC applications are normally evaluated according the energy consumption and the feasibility of the method when compared to the existing technology in use (Raventós et al., 2012a). For example, evaporation shows outstanding results in terms of concentration achieved when compared to membrane technology. However, membrane technology will use far less energy as the requirement for a phase change is avoided. The lower energy requirement for freezing coupled to low temperature operation means that low-cost materials can be used for the process and the usual problems with corrosion and scaling disappear (Samsuri et al., 2016). For desalination, crystallization, juice concentration and water removal, the initial solution to be frozen does not need pretreatment. The chemicals required and costs associated with pretreatment are then avoided. Similarly, there is no fouling of surfaces, which would be the case with membrane separations, and low ecological impact (Shi et al., 2018; Wang et al., 2015).

Another major advantage of FC is the ease of hybridization, especially with membrane systems. The use of a hybrid process will be beneficial due to synergies between technologies. Membrane processes are often limited by the fouling potential of certain salts present in wastewater and also by the osmotic limit of the solution as concentration increases. In contrast, FC can operate at any salt concentration. Also, if EFC is adopted, then salts can be recovered from the wastewater and a hybrid process using membranes can then work at higher water recoveries. Similarly, the volumes of brine created are significantly reduced, which will aid in the unsustainable disposal problems currently faced. Hybrid FC processes are also suggested to have the lowest energy consumption when compared to conventional saline waste stream treatment options (Zikalalaa et al., 2017). Obviously any technology cannot guaranty a complete and perfect solution without limitation (Table 4). The FC process, when compared to evaporation and membranes process, has a higher capital and operating cost. This is mostly related to the ice separation stage and is the most common problem

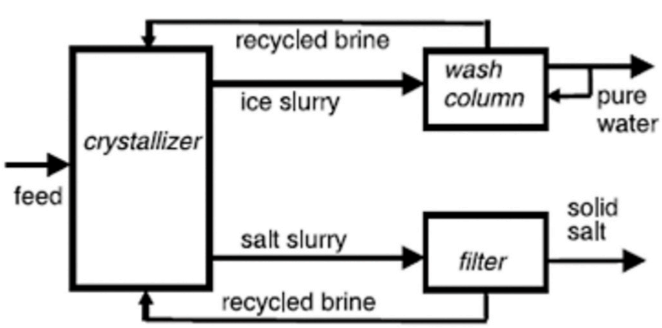

Fig. 7. The basic process for eutectic freeze concentration (Frank et al., 2004).

Table 3
Advantages and applications of freeze concentration reported in the literature.

Advantages	Method	Sector	References
1- Less energy consumption	• Direct freezing with n-butane	Desalination	Madani (1992)
	• Direct freezing with vapor compression	Desalination	(Rane and Padiya, 2011)
2- Atmospheric pressure operation	• Direct freezing with air	Desalination	Çerçi (2003)
	• Indirect freezing with scrapped surface heat exchanger SSHE	Desalination	Habib and Farid (2006)
	• FC system with heat pump, FCSwHP	Food industry	Rane and Jabade (2005)
	• Indirect freezing with fluidized bed Heat exchanger	Desalination	Cheng et al. (1987)
3- High mass transfer coefficient	• Vacuum freezing multi-phase transformation	Desalination	Rane and Jabade (2005)
	• Indirect freezing with fluidized bed Heat exchanger	Food industry	Habib and Farid (2006)
	• FC system with two stage compression using tubular heat exchanger	Desalination	(Rane and Padiya, 2011)
4- Eliminate difficulty of ice brine separation	• FC system with heat pump, FCSwHP	Desalination	Rane and Jabade (2005)
	• FC system with two stage compression using tubular heat exchanger	Desalination	(Rane and Padiya, 2011)
5- Eliminates refrigerant and compressor	• Vacuum absorption vapor compression	Desalination	Cheng et al. (1987)
	• Vacuum freezing ejector absorption		El-Nashar (1984)
6–75%–90% reduction of the energy required by conventional thermal process	• Freezing melting separation	Desalination	Heist (1979)
7- The advantage of a low operating temperature, which minimizes scaling and corrosion problems	• Freezing melting separation	Desalination	Rahman et al. (2007)
8- The quality of the product obtained due to the low temperatures used	• suspension and film FC	Food industry	Qin et al. (2006)
9- Heat transfer coefficient with phase change is about 3–5 times greater than that without phase change.	• scraped-surface heat exchanger SSHE	Food industry	Qin et al. (2006)
10- Minimal loss of volatile compound	• Cryoconcentration	Food industry	Raventós et al. (2012b)

Table 4
Disadvantages of freeze concentration reported in the literature.

Disadvantages	Method	Sector	References
1- Difficult ice brine separation	• Direct freezing with n-butane	Desalination	Madani (1992)
	• Direct freezing with vapor compression	Desalination	Cheng et al. (1987)
	• Direct freezing with air	Desalination	Çerçi (2003)
	• Indirect freezing with scrapped surface heat exchanger SSHE	Desalination	Habib and Farid (2006)
	• Vacuum absorption vapor compression	Desalination	Cheng et al. (1987)
	• Vacuum freezing ejector absorption	Desalination	El-Nashar (1984)
	• Vacuum freezing multi-phase transformation	Desalination	Cheng et al. (1987)
2- Batch process results in thermal cycling loss	• FC system with heat pump, FCSwHP	Desalination	Rane and Jabade (2005)
	• FC system with two stage compression using tubular heat exchanger	Desalination	(Rane and Padiya, 2011)
3- Higher capital costs and higher operating costs during the ice separation	• Freezing melting process	Desalination	Olowofoyeku et al. (1980)
4- Retention of undesirable flavors	• Freezing melting process	Food industry	(Braddock and Marcy, 1987)

among the various forms and methods of freezing technologies (Kalista et al., 2018). To optimize the separation step, the use of a compressor has been implemented, However, this also represents an expensive method of furnishing the energy requirements of the system (Sánchez et al., 2011). Quality improvement of solutions requires high quality energy, required for crystallization, when compared to low quality energy that can be used in many evaporation processes (Table 5). The points raised in this section suggest that the FC technique has significant potential as a cleaner and more sustainable technology when directly compared to other technologies for the treatment of animal manures.

4.4. Industrial applications of the freeze concentration process

The three applications where the FC process has gained popularity at industrial scale are the concentration of fruit juices, purification of organic chemicals and treatment of hazardous wastes (Rahman and Al-Khusaibi, 2014). The main reasons for these successes are due to the development of more efficient high capacity processes and products of high purity or quality. The choice of a separation system depends upon the type of solution to be concentrated and several options may exist. Therefore, an economic analysis of the FC system integrated into an application should be performed (Englezos, 1994). The status and the prospects of these applications will now be presented.

4.4.1. Food industry

Several authors have published applications of FC in the food industry. From these works, the conclusions can be made that FC is highly effective to concentrate juices (Raventós et al., 2012a), wine, beer, milk (Sánchez et al., 2011), coffee, and tea; with all flavour and aromatic components retained (Miyawaki, 2018). Concentration processes normally reduce the cost of transportation, handling, and storage once those costs are based on the final product mass.

The SFC system is the only technique applied at a commercial level by the companies GEA Group and Sulzer Ltd. Both companies have created a mature and efficient technology to obtain clean ice (impurity $<$ 100 ppm) and highly concentrated effluents (45–55% w/w dissolved solids). Nevertheless, some disadvantages can be cited: technical complexity, high initial capital cost (€ 2 M for 10 m^3 h^{-1} capacity), and high energy consumption (35–40 kWh t^{-1}) (Van-Beek et al., 2018). These drawbacks limit application of FC to products with high added value as these products are usually produced in much lower quantities. With the purpose to overcome these inconveniences, a new generation of equipment has been developed, namely the Icecon™ by GEA Group and Multiblok™ by Sulzer Ltd. The major difference between traditional design and latest Icecon™ technology is the production of ice crystals and ice crystal growth in the same vessel, avoiding the construction of an auxiliary vacuum vessel and significantly reducing the equipment footprint. This improvement reduces the capital cost of the system by around 35% and reduces energy consumption by 20% (Van Nistelrooij, 2013). The Multiblok™ system uses an increased crystallizer volume and simplifies the technology by combining two processes in one. Dividers were added to the crystallizer to enable effective radial and axial mixing, which increases average crystal size

Table 5
Reported energy consumption of various technologies that could be used for nutrient recovery from animal waste.

Treatment technology	Performance	Energy consumption (kWh/m^3)	Efficiency (%)	Reference
Freeze desalination	Heat pump operated layer freezing based technology	9–11	99	Rane and Padiya (2011)
PCE	Cryo-concentration of orange juice	1500	97	Sánchez et al. (2010)
Reverse osmosis	Depending on system size and salinity	6 to 8	99	Lee et al. (2011), Max et al. (2019)
Freeze desalination	Recorded the salt removal	7.45	98	Mtombeni et al. (2013)
Nano-filtration and reverse osmosis membranes	dissolved organic matters were removed from livestock manure	7	80	Carretier et al. (2015)
Seawater freeze desalination	Cold energy provided by regasification of liquefied natural gas (LNG)	0.28	91	Ong and Chen (2019)
PFC	Extracting sucrose from solution	1100	95	Moussaoui et al. (2018)
Hybrid system (freezing desalination combined with a solar evaporator)	fresh water and micronutrients recovery from wastewater	13780	80	Madani (1992)
Bio electrochemical systems	Ammonia recovery rate of 7.1 g N m^{-2} d^{-1} from wastewater	5700	83	Qin et al. (2017)
Air stripping process	Ammonia recovery rate of 0.76 kg N m^{-3} d^{-1} from wastewater	11000	91	Maurer et al. (2003)
Nitrification & denitrification	0.14–0.58 kg N m^{-3} d^{-1} removal	14000	90	Erisman et al. (2008)
Eutectic Freeze Crystallization	7.8 ton day-1 35 w% of NaNO3 solution	2200	98	Van der Ham et al. (1998)

and prevents crystal agglomeration.

Ding et al. (2019) considered that the future of FC in food industry applications will guide the development of new PFC equipment to replace the SFC system due to the simplicity of the separation process. The main challenges for the PFC technique to be commercialised are obtaining high purity ice, the implementation of a continuous process and optimizing the energy consumption. Recent research combining the PFC with controlled partial thawing is available (Miyawaki et al., 2016; Zambrano et al., 2018). F.L. Moreno et al. (2014) combined falling film FC technology, fractionated thawing and BFC to achieve an industrially viable system for coffee extract production. The integration of these techniques results in a concentration efficiency of 99.2% and solute yield of 95%, which is comparable to industry standards in freeze-concentrated coffee extract production. Zambrano et al. (2018) studied the integration of two FC techniques as an alternative to obtain potable water and a salt removal efficiency of 98.5% was achieved. MEIWA CO. Ltd. Successfully applied PFC to brewing, winery, fragrance production, dairy applications and pharmaceutical manufacturing. The progress made is the result of more than 5 years of joint research between the Ishikawa Prefecture University and the Ishikawa Prefecture Industrial Research Institute. The equipment is available from a laboratory-scale of approximately 5 L, to a practical scale of approximately 50 L and higher capacity equipment at 250 L is under development. Apple juice was effectively concentrated from 12.8 to 21.0 °Brix with 79.0% yield, which was improved to 90% by recovering 30% of the initially melted fractions using the ice partial melting system.

4.4.2. Chemical industry

FC processes in the chemical industry focus mainly on the organic melt crystallization technology (Wynn, 1992). The key advantage of melt crystallization over distillation is in the separation of substances with very close boiling points like isomers (Rahman et al., 2007). Crystallization is often the best alternative when distillation is difficult or even impossible. High purity products are manufactured from close boiling or azeotropic mixtures of components that are thermally unstable at their boiling point. The capital investment is high, but the process is affordable for a high-value product. Some typical applications include the purification of acetic acid, acetonitrile, adipic acid, benzene, caprolactam, durene, ethyl lactate, hexamethylenediamine, ionic liquids, lactic acid, methylene diphenyl isocyanate, methacrylic acid, o-phenyl phenol, p-diisopropylbenzene, p-dichlorobenzene, p-chlorotoluene, p-nitrochlorobenzene, p-xylene, phenol and trioxane (Ahmad et al., 2018; Rahman et al., 2007).

4.4.3. Industrial wastewater

Crystallization is the widely used due to the technologies maturity and acceptable energy consumption. Other crystallization techniques have unique advantages in specific applications. For example, pre-concentration with FC is one possibility for hazardous wastewater treatment (Lemmer et al., 2001). This technique is applicable when incineration is required and the hazardous material is a mix of non-volatile and volatile components (VOCs) (Van Nistelrooij, 2013). FC and incineration costs are likely to be of the same order of magnitude as the viable oxidation and bio-treatment alternative. FC has several advantages over bio-treatment such as the elimination of large vessels, higher flexibility and reliability relative to changes in both feed flow rate and composition and the avoidance of shut downs caused by the presence of random toxins (Kalista et al., 2018). FC has a lower environmental impact and improved inherent safety. For this application, EFC may be suitable to reduce saline loads to bio-treatment. EFC is a sustainable and environmentally friendly technology offering zero waste potential and is capable of cleaning water while producing valuable products, leading to a low cost process with realisable potential for a circular economy.

Treatment of acetone containing industrial wastewater, by static progressive freezing on a cold wall, has also been studied. The lowest impurity concentration (3.92 g L^{-1}) was obtained by applying the lowest ice growth rate (0.1 mm h^{-1}) (Htira et al., 2018). However, this concentration was not in agreement with the standards set for direct discharge of the water into the natural environment of France. As a solution, the authors suggested adding a sweating step to drain out the liquid trapped in the ice and to improve the resulting ice purity. PFC may also provide an effective, economical, and feasible alternative for recovering Tetrahydrofuran from Grignard reagent wastewater (Chen et al., 2019). Results obtained from pilot-scale tests showed an optimal COD removal efficiency of 98.1% and a COD as low as 680 mg L^{-1} in the melted ice solution. Similar results regarding COD removal have been reported (99.1%) when the FC technique was applied to milking wastewater using a rotary ice-making machine (Dai et al., 2018). Ab Hamid and Jami (2019) also studied the effect of the coolant temperature and stirring speed on the efficiency of the PFC process to concentrate organic matter and concluded that the best conditions were found at a moderate coolant temperature of −10 °C and a maximum stirring speed of 500 rpm. This combination of operating conditions resulted in the lowest effective partition constant ($K = 0.486$) and highest solute recovery (0.9 g of glucose obtained per 1 g of initial glucose).

A prototype of indirect contact freezing was also developed to demonstrate the application of FC to purify landfill leachate (John et al., 2020). Average removal efficiencies over 95% were achieved for both organic and inorganic matter including heavy metals (Szpaczynski et al., 2017). The first 30% of the melted liquid volume contained over 90% of all impurities and a significant agglomeration of solid particles was also noted. The results revealed that the application of this process at full scale is feasible.

4.4.4. Desalination

In this section, the term desalination should be understood in a broad sense, not only salt removal to obtain potable water, but also to treat brines from reverse osmosis processes or certain types of industrial wastewater. Technologically all the FC methods used in the food industry could be used for desalination purposes. The only real consideration needed is the economic analysis of the FC process for desalination since water is a low-value product compared to food (Rahman and Al-Khusaibi, 2014). To date, FC technology is mainly adopted when there are no other alternatives available for the desalination application. FC is gaining interest as a technological option to treat brine (Randall and Nathoo, 2015). In contrast to membrane systems, EFC does not have the feed composition limitations and produces high purity solid salts (>90% purity).

A comprehensive review of different types of freeze desalination technologies and development has been made previously (Williams et al., 2015). The general conclusion from these studies is that the research and development of FC for desalination should focus principally on the following aspects (Randall and Nathoo, 2015): design of new equipment to reduce costs and improve the efficiency; reduction of energy consumption, use of low-grade energy and renewable energies for cooling and integration with existing technologies such as membrane systems (Kalista et al., 2018). Continuous efforts are being carried out for the reduction of energy consumption in FC processes. The freezing process was proposed as a method of seawater pre-treatment for reverse osmosis (RO) membranes (Baayyad et al., 2015). An evaluation of the energy consumption shows energy savings of approximately 25% when compared to the conventional RO desalination. For seawater desalination, Chen et al. (2020) presented the SWDIM (super-cooled water dynamic ice making) with an energy consumption of 58% when compared to the indirect progressive contact freezing (IPCF) system. Whereas Cao et al. (2015) presented a new suggestion to apply the liquefied natural gas (LNG) cold energy in the freezing desalination. Due to the low temperatures of the processes (−162 °C), the recommendation was made to use intermediate refrigerant to transfer energy to the process. The calculations show that the consumption of 1 kg equivalent of LNG cold energy can obtain about 2 kg of ice meltwater (Cao et al., 2015).

Lin et al. (2017) presented a seawater freeze desalination prototype system applying R410 as a secondary refrigerant. The prototype was able to reach the freshwater capacity of 150 $L\ h^{-1}$ and the salt removal rate was around 50%. Thus, more cycles of freeze desalination, or freeze desalination assisted with RO, are needed to produce drinking water.

Likewise, membrane distillation (MD) and FC were evaluated as alternative RO concentrate treatment options (Naidu et al., 2018). A direct contact MD (DCMD) could obtain 60% water recovery with chemically pre-treated RO concentrate. FC in the three-stage freeze/-thaw approach was able to achieve 57% water recovery with no scaling issues. The efficacy of DCMD was compromised by membrane scaling, which implies an additional cost due to pre-treatment for scale removal. The FC was advantageous as a non-scaling and chemical-free process. The FC could be coupled to the LNG refrigerant coolant source (Fig. 8), given that the majority of LNG plants are located in coastal areas. However, the practical industrial application of FC is inherently restricted due to the complexity of the operation that limits capabilities in large-scale configurations. Suspension crystallization has also been successfully demonstrated as a desalination technology (Ahmad et al., 2018). In the study of Lu et al. (2019) a novel freeze desalination and membrane distillation-crystallization (FD-MD-C) hybrid system has been developed at lab-scale and may provide valuable guidance for designing a low-cost desalination system with zero liquid discharge (ZLD). A hybrid RO-Freeze process has been proposed that increased water recovery by 400%. On the other hand, the feasibility of a hybrid system of freeze desalination and vacuum membrane distillation powered by LNG regasification and solar energy for seawater desalination was demonstrated (Chang et al., 2019), with a high-water recovery of 74%.

4.5. Applications of freeze concentration in animal manure

One of the first works in this area was reported by Gao (1998). The “freezability” of the wastewater can be evaluated by quantitative determination of the ice nuclei concentrations. Pig slurry piggery wastewater had the lowest nuclei concentration and therefore the lowest “freezability”, compared to other types of waste effluents. The freezing temperature of piggery wastewater was sensitive to the change in the impurity concentration, volume drop, and pH.

Different studies have been published regarding the application of PFC to treat animal manure. The performance of PFC to concentrate organic matter contained in liquid fraction of digested manure was evaluated using a batch freezing reactor with a working volume of 300 mL (Young et al., 2001). Dissolved organic matter and suspended solids (SS) were further concentrated to 98% and 92% respectively. Also, PFC (falling film type) on a semi-industrial scale has been used to treat different types of wastewater (Rodriguez, 2015). The equipment used was an evolution of the design presented by Raventós et al. (2007). In the case of poultry manure, an 82% reduction in total dissolved solids (TDS) and conductivity was achieved in a single treatment stage and a TDS reduction of 94% and conductivity diminishment were reported for a two-stage treatment processes. Rodriguez-Pascual (2016) suggested the scheme as shown in Fig. 9 for the treatment of pig slurry, combining different separation techniques in a semi-commercial facility at 1 $m^3\ h^{-1}$. Reduction volumes greater than 80% and energy consumption in FC of 25–40 $kWh\ m^{-3}$ water removed were reported (Rodriguez, 2015).

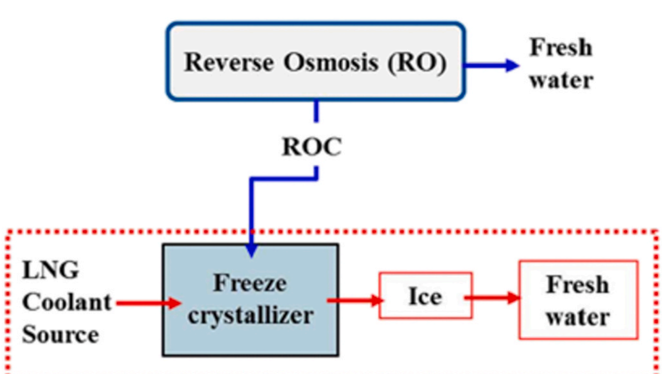

Fig. 8. A holistic approach for fresh water production, combining reverse osmosis with freeze concentration and coupling to a liquefied natural gas terminal.

In a recent work conducted by Cantero et al. (2019), FC is mentioned as an option for the physical pre-treatment of manure, to convert the raw material into a more efficient form in terms of storage, transport, and capability of being employed in further treatment. Moisture content contained in solid residues derived from livestock waste was reduced by 50%. Since the management of the liquid fraction of animal manure has certain similarities with the management of human urine, some references in this area can also be cited. Gulyas et al. (2004) conducted laboratory-scale batch FC of yellow water with a stirred vessel and a falling film freeze concentrator. The results indicated that multistage processes are necessary, i.e. the melted ice phase must be purified (and the concentrates must be further enriched) in a second or even in a third stage to decrease the ice contamination observed. Ganrot et al. (2007) investigated a freezing methodology to recover nitrogen and phosphorus in a hybrid system that also included struvite precipitation and nitrogen adsorption on zeolite and activated carbon. The freezing-thawing method concentrated 60% of the nutrients in 40% of the initial volume and significantly improved the N reduction. The P recovery was 95–100%, mainly as struvite.

Recently, EFC was considered as means of volume reduction (Chipako and Randall, 2020). When applied to urine, up to 99% of the nitrogen bound in urine could be recovered at a temperature of −30 °C. An estimated 95% water recovery was possible when using EFC for urine treatment. However, due to the low-temperature requirements, and considering that most of the energy is required for ice formation, EFC works better when the stream is already significantly concentrated. As an alternative, the volume reduction of wastewaters could be achieved using RO as a pre-treatment step and followed by EFC.

The information presented to this point highlights the possibilities and challenges for the FC process. Cost is the main limiting factor hindering application of the technology, requiring the development of new systems targeting a reduction in capital and energy cost of the process. The technology may potentially save 55–90% of energy compared to the existing concentration technologies such as evaporation and regular FC.

4.6. Energy consumption of the freeze concentration process

Evaluating the economic aspects of emerging resource recovery processes is critical for uptake and establishment in the agricultural sector. The freeze concentration process is considered to have high energy consumption due to the nature of feed solutions that are being treated (Chourot et al., 2003). Typically, high moisture content (40–80%) of raw materials would require significant energy usage in water solidification. Therefore, the energy demand would reduce significantly in processing systems with less water content to crystalize (Lopez-Quiroga et al., 2016). The capital and energy cost of the FC system might be the most concerning factor and a key challenge that will limit future applications. Recent studies are more focused on reducing the energy and capital cost of FC technology while maintaining the quality of the final product (Van Nistelrooij, 2013). Basically, the energy consumed in FC systems comes from the electrical energy required for the pump, engine, and refrigeration units. The refrigeration system removes heat or energy from the feed fluid; this is the inherent sensible and latent heat of the feed, penetration of heat into the equipment from ambient conditions (assuming ambient temperature is higher than that of the equipment) and energy derived from agitation and heat related

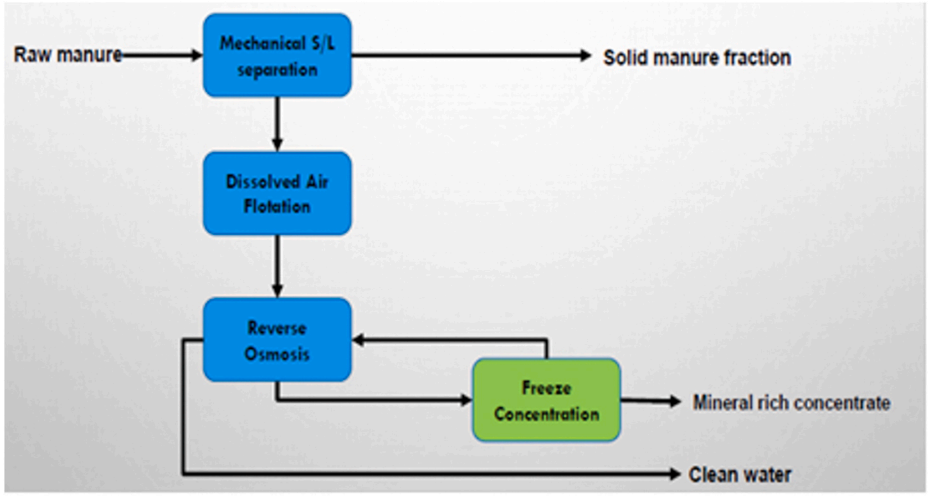

Fig. 9. A suggested pig manure treatment system using different separation techniques (Rodriguez Pascual, 2016).

with ice crystal formation. About 10 and 25% of the total heat generated is estimated to come from mechanical sources and ambient conditions respectively (Hartel and Heldman, 1997). The energy consumption in the FC system is based on the heat used to convert one kg of the raw material into a concentrated solution and ice mixture. As a comparison to membrane technology, FC can be considered as requiring similar or even lower energy expenditure for processing in some cases (Table 5). For instance, the energy required to recover freshwater from seawater using reverse osmosis technique is much higher than other alternative techniques for water treatment and nutrients recovery (Schunke et al., 2020). Conventional techniques consume much more energy, commonly extracted from fossil fuels, which contributes to global warming and greenhouse emissions (Schunke et al., 2020). Sustainable techniques for nutrient recovery from animal waste have become increasingly important. The negative environmental effects produced by some manufacturing processes undoubtedly contribute to this trend. For example, the Haber-Bosch process, by which most synthetic fertilizers are produced, contributes significantly to pollution and global energy consumption (Sutton et al., 2011). The typical energy consumption for the production of NH_3 through the Haber-Bosch process is around 11, 000 kWh m^{-3} (Rafiqul et al., 2005). To make a unit cost comparison among various technologies is difficult as many parameters must be considered, such as the type of technology in use, the type of feed solution, the ambient temperature, the targeted recovery rate, and the cost of electricity in each region. The required investment of each technique could be variable based on the construction cost and production capacity. Pazmiño et al. (2017) reported energy savings up to 30% utilizing a continuous system of FC, treating sucrose solutions, and integrated with the falling film technique, compared to the energy consumption of other conventional methods. They obtained a high concentration index of 4.0 and a concentration efficiency of 98.5% with energy consumption of 10.3 kWh m^{-3}.

In a similar study, the energy usage of 23.3 kWh m^{-3} was reported in the integrated system for treating orange juice and coffee (Rodriguez et al., 2011). Recent researches are based on the improvement efficiency of FC in terms of energy consumption and operation. For instance, the operation of at least two FC units in a parallel mode, would be a good approach to reduce the total energy consumption of the process and consequently increase the economic feasibility of the FC process (Rane and Jabade, 2005). Theoretically, FC has a huge energy-saving potential compared to thermal and evaporating processes. FC uses 304% less energy than evaporation to treat wastewater and consumes less than 62% energy while combined with ice thermal storage technology and precooling method (Ling et al., 2012). Table 6 shows a cost comparison between the freezing, evaporation and membrane processes for various

Table 6
A comparison between the average operational costs (US $ per m^3) for concentration processes.

Industrial application	Freezing	Evaporation	Membrane
Desalination seawater	0.8 [4]	1.8 [1]	1.5 [1]
Fruit juice concentration	2 [3]	5.4 [4]	1 [3]
Sugar production	1.3 [5]	8.4 [3]	–
Digested manure	–	–	2.4 [2]
Brine treatment	1.4 [6]	1.4 [1]	0.76 [6]

[1]Druetta et al. (2014); [2]Gerardo et al. (2015); [3]Moreno et al., 2014; [4] Panagopoulos et al. (2019); [5]Rahman et al. (2006); [6]Williams et al. (2015).

concentration purposes. The membrane processes have the lowest operating costs, although it must be taken into account that FC is an emerging technology with wide possibilities for improvement. For example, the study of He et al. (2018) reported the use of LNG to replace the external refrigeration cycle used in classical hydrate desalination plants. They found that this method could reduce energy consumption to 0.60 to 0.84 kWh m^{-3}, which is equivalent to only 25% of the energy requirement for the RO process. Also, as indicated in previous sections, a good energy option and therefore cost effective process is the combination of FC and membrane process. However, the freezing process is not fully commercially developed and requires further effort. Therefore, future research on FC systems for full-scale industrial applications is necessary to boost the economic argument for the technology when compared with conventional techniques. The studies to date would suggest that the key to achieving a more sustainable method to treat animal manure would be a hybrid process that can be optimized for improved recovery and sustainable operation.

4.7. Future remarks

The future development of FC processes should focus on removal of the current limitations of the technology. Such as improving the level of ice purity, increasing the concentration of the final solution, improving the simplicity by reducing the number of moving parts, and finally design of a continuous FC apparatus in order to optimize the operation of the system. FC processes are varied in terms of the principle of operation and the sector of application. Regardless of the application, the common issues encountered for FC systems are the formation of the ice crystals and the separation of the ice from the concentrated solution. The use of vacuum pressure as a step in the FC process to optimize the separation of the solids has been implemented. The applied vacuum is generated using a compressor which leads to high energy consumption.

This coupled to the increased maintenance of the compressor is seen as a hindrance facing FC in the case of vacuum implementation. The solid separation process can be done through filtration, centrifugation, or in a wash column. However, the cost of centrifugation is prohibitive. Likewise, the filtration process can also be costly when fouling occurs.

To optimally recover a final product using any technology, a clean and efficient separation is crucial. There are various solutions that can be adapted for an ideal FC process, providing both clean ice and a maximized concentration of the solids in the liquid phase. Typically, the ideal process would be generating larger ice crystals, in other words a smaller surface to volume ratio, which leads to a better separation. In order to achieve this ideal situation, slow cooling is the first option to optimize the FC technology. The slower the cooling, the larger the mass of ice crystals formed, which at the same time should conserve the product quality in the concentrated fraction.

For this purpose, EFC can be a good candidate. In particular, for the inorganic fraction recovery of animal manure, it is essential that continuous solid-solid-liquid separation is achieved in order to allow a smooth continuous EFC operation. This requires large ice and salt crystals which easily separate mechanically. FC alone may not be not suitable for animal manure valorization. Hence, combination with another technology such as membrane systems to generate a hybrid process must be considered. In particular, for the inorganic fraction recovery, coupling continuous EFC with RO seems a very reasonable option. Furthermore, EFC produces clean water and potentially pure salts which can be re-used or sold to offset the crystallization costs. This approach of resource recovery from animal manure would ultimately create a more sustainable and circular economy.

Refrigeration is the main component of energy consumption in all FC processes. The use of cleaner and green energy is a trend that is being imposed in the technological processes of many companies. In our opinion the only “c“lean energy” is saved energy, i.e. energy which was not needed to be generated in the first place. Thus, the refrigeration systems for FC must be based on high-efficiency equipment that uses low-cost energy (e.g. regasification of LNG, waste energy). This combination can make the processes economically competitive (Chang et al., 2019). The cooling equipment design must be based on the use of heat pumps (Rane and Padiya, 2011) and thermally activated systems (refrigeration by absorption), especially in NH_3–H_20-based systems.

In the agricultural sector, the use of FC to treat animal manure for nutrient recovery would significantly reduce volumes for transport and would provide a sustainable low cost source of N and P in high quality format. The discussion thus far could be framed within the concept of a ‘rural biorefinery’. Integration of different technologies in such a processing strategy demonstrates that the “zero waste” concept can be realised by converting waste into a new resource (Fig. 10).

5. Conclusion

The increasing demand of animal products worldwide, together with the expanded consumption of fertilizers, is leading to the development and improvement of technologies to valorise solid waste generated in livestock activities and obtain clean water and bio-based fertilizers among other value-added products. To create functional and efficient strategies to obtain these goals, research committed to the evaluation of effective and sustainable strategies that promote circular economy are required. There are several studies that demonstrate the applicability of technologies for waste treatment, however, these often present the requirement for large capital investment and high energy consumption. This paper provides a mechanism to evaluate current and future technologies using a side by side comparison across a range of sustainable measures. Freeze concentration is a technology with great potential for the concentration of nutrient-rich waste effluents with a significantly less environmental burden than other methods. This method has shown several significant advantages at laboratory and pilot scale when compared with other technologies, such as membranes, evaporation or compaction, for volume reduction or nutrient concentration. Application of freeze concentration is not constrained by raw material variability or the presence of solids (a major issue for membranes) and does not produce odors or nutrient volatilization (evaporation would). Also, freeze concentration can be a cost-efficient technology in terms of energy consumption with no requirement for additional chemicals. Freeze concentration can be a major contributor to the biorefinery concept behind the next generation of integrated processes to obtain bio-based fertilizers from waste, regardless of the undervalued nature of the raw material. The wide spectrum of opportunities that freeze concentration offers is beginning to emerge, driving development efforts towards extrapolation of freeze concentration processes creating innovative applications and technologies for full-scale deployment. Obviously a corresponding economic assessment of both capital and operational cost is

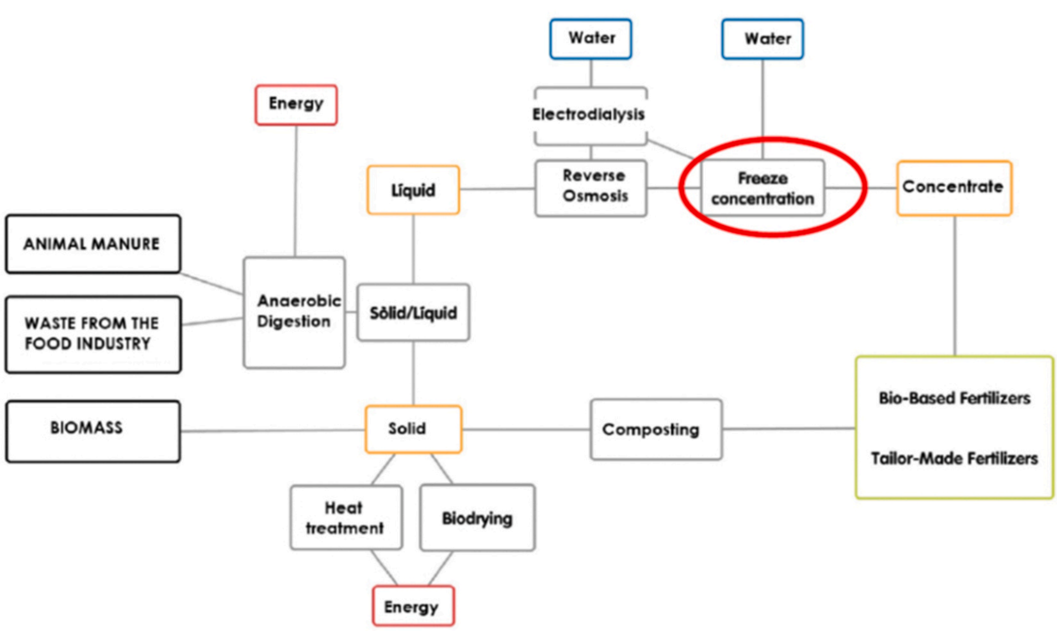

Fig. 10. A suggestion for the integration of freeze concentration with other technologies to produce a rural biorefinery.

necessary and comparison to other technologies is paramount. Finally, freeze concentration has great potential for the recovery of nutrients from animal manure. However, industry, law makers and the scientific community need to work in unison to fully realize the potential of this novel technology.

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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