Dairy Wastewater Treatment by Membrane Separation Technology

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Contributor: Aws N. Al-Tayawi, Elias Jigar Sisay, Sándor Beszédes, Szabolcs Kertész

Water pollution caused by population growth and human activities is a critical problem exacerbated by limited freshwater resources and increasing water demands. Various sectors contribute to water pollution, with the dairy industry being a significant contributor due to the high concentrations of harmful contaminants in dairy wastewater. Traditional treatment methods have been employed, but they have limitations in terms of effectiveness, cost, and environmental impact. Membrane separation technology (*MST*) has emerged as a promising alternative for treating dairy wastewater. Membrane processes offer efficient separation, concentration, and purification of dairy wastewater, with benefits such as reduced process steps, minimal impact on product quality, operational flexibility, and lower energy consumption. However, membrane fouling and concentration polarization present major challenges associated with this technique. Therefore, strategies have been implemented to mitigate these phenomena, including pre-treatment prior to MST, coagulation, and adsorption. 3D printing technology has gained prominence as one of the latest and most notable advancements for addressing these issues.

Keywords: dairy wastewater ; conventional treatment ; membrane filtration methods

1. Introduction

In the early 1960s, the first defect-free, high-flux anisotropic reverse osmosis (*RO*) membrane was created at the University of California, Los Angeles (*UCLA*) due to growing worries about the drinking water supply. Two *UCLA* graduate students, Sidney Loeb and Srinivas Sourirajan, found an efficient method for producing *RO* membranes [1][2]. Their lab-scale desalination equipment, the so-called “big dripper”, produced tiny volumes of fresh water, but it spawned a global business worth billions of dollars. The discovery of asymmetric membranes by Loeb and Sourirajan is typically considered the beginning of contemporary membrane research. In addition, it is considered the basis of industrial membrane processing [3].

In the food and beverage industry, applying membrane processes as an alternative to classical separation, purification, and concentrated product methods for “sustainable production” and a “zero waste approach” is a popular and rising topic. Depending on the variety of applications, the reasons for the widespread use of suitable membrane processes in the food and beverage industry are as follows: (i) reducing the number of process steps in comparison to traditional methods; (ii) relying on minimized changes in the loss of aroma and nutritional components of food and beverages due to the use of high temperatures in traditional methods and improving end product quality; and (iii) high process selectivity [4]. Also, membrane processes have built-in advantages when making a process more efficient, mainly because they reduce the amount of equipment needed, offer much operational flexibility, and use less energy [5].

2. Membrane Filtration Methods

2.1. Microfiltration

Microfiltration (*MF*), like all other membrane separation procedures, is a technology that permits the differential concentration in the liquid retained by the membrane, known as *MF* retentate, of the components having a pore width larger than the average pore size of the membrane [6]. Membranes with a diameter ranging from 0.1 to 10 μm are used in microfiltration (*MF*). Thus, particles larger than 0.1 μm are included in the retentate, and the pore size can vary depending on the application [7]. The typical TMP ranges between 0.03 and 0.20 MPa [8]. As one of the dairy applications of this process is the retention of bacteria and spores, it is necessary to control the size of the membrane pores, which should be small enough to retain microorganisms without compromising the composition of the permeated milk [9][10][11]. Introduced were commercial ceramic membranes and the idea of uniform transmembrane pressure (*UTP*) for regulating hydrodynamics and fouling during membrane filtration (*MF*) of dairy fluids. This breakthrough led to the resolution of technical issues, including late emmental cheese expansion, spore removal from whey, effective defatting of milk and whey, and casein micelle separation from milk [1].

2.2. Ultrafiltration

Ultrafiltration (*UF*) can prevent the passage of molecules larger than 0.001 μm due to membranes with pores ranging from 0.01 to 0.001 μm [7]. Typically, ultrafiltration (*UF*) employs membranes with a cutoff of 1–800 kDa and a *TMP* range of 0.1–1 MPa [8]. *UF* can retain proteins and fat while allowing vitamins, minerals, and lactose to pass through. The use of *UF* in dairy product development improves yield, nutritional functionality, and sensory characteristics [12]. This process is helpful for protein concentration and purification, and it distinguishes itself in cheese production by providing higher protein concentration and better nutritional characteristics to the product. Another common use for *UF* is manufacturing milk protein concentrate (*MPC*) [13][14]. Ultrafiltration (*UF*) was suggested as a potential technology for concentrating milk solids, mainly proteins [1].

2.3. Nanofiltration

Membranes with pores ranging from 0.001 to 0.0001 μm are used in nanofiltration (*NF*) processes [7]. *NF* can concentrate small molecules with molecular weights equal to or greater than 100 kDa. Where sugars, amino acids, dyes, and salts can be retained by *NF* membranes [15]. It can also concentrate whey proteins in milk to produce derivatives. Because of the interaction between the membrane, the solution to be filtered, and electrostatic repulsion, the *NF* process is capable of high retention of organic compounds [16]. Nanofiltration (*NF*) employs membranes with a typical cut-off of 150–700 kDa for the concentration and partial demineralization of whey or milk streams, thus removing dissolved mineral salts in inverse proportion to their valence. In ratio to their concentration in the retentate, the demineralization capability is counterbalanced by the partial penetration of low molecular weight components such as lactose. Typical operating pressures for this process are 1–3 MPa [8].

2.4. Reverse Osmosis

Reverse osmosis (*RO*) is a process in which membranes with pores smaller than 0.0001 μm are used [17]. Only water can pass through at pressures between 3.5 and 10 MPa [18]. These membranes can retain larger ions and compounds while releasing water into the permeate and can be used for milk preconcentration; this process has increased osmotic pressure and feed stream viscosity, resulting in severe fouling and permeate flow reduction problems. Several studies to promote optimization have focused on the disadvantages of this process [8].

3. Challenges and Future Perspectives

3.1. Fouling Phenomena

Fouling phenomena refer to the limitations encountered in membrane filtration processes, primarily attributed to membrane fouling and concentration polarization [19][20]. These phenomena lead to a decline in flux, resulting in decreased productivity over time [21]. Concentration polarization occurs due to the preferential passage of certain species across the membrane, accompanied by the accumulation of other species at the membrane surface, which results in a reduction in permeate flux [22]. While concentration polarization is typically reversible by adjusting operational parameters like increasing cross-flow velocity [23], it may also involve the formation of a gel layer at high species concentrations, which cannot be rectified solely through operating condition modifications [24][25]. The development of a gel layer necessitates washing to restore the membrane’s characteristics [26].

Fouling remains a significant obstacle in membrane processes [27]. It generally arises through two main routes: foulant adhesion/deposition and the foulant layer filtering process [5]. Fouling occurs due to the interaction between foulants present in the separation solutions, which can include particulate matter, colloidal particles, biomacromolecules, and the membrane surface [28][29]. The foulants physically and chemically interact with the membrane surface, leading to chemical degradation of the membrane material [30][31]. Microorganisms and biomacromolecules non-specifically adhere to the membrane surface, blocking or significantly reducing the membrane pores, thereby causing a notable decline in permeation flux and separation efficiency [32]. The fouling phenomenon can be characterized by different mechanisms, including the complete blocking model, intermediate blocking model, standard blocking model, and cake layer model [33].

Membrane fouling has been examined extensively. Recent trends include in situ real-time monitoring approaches for membrane fouling, sophisticated characterization techniques such as *HPLC* coupled mass spectrometry and advanced simulation methodologies such as molecular simulation [26].

Numerous approaches have been employed in addressing membrane contamination, encompassing chemical and physical treatment modalities. Presently, environmental scientists are actively exploring contemporary and sustainable methodologies that involve the utilization of environmentally benign or recycled materials for the remediation of membrane pollution. The following elucidation highlights several methodologies employed in the treatment of membrane pollution.

3.2. Pretreatment before MST

Various pretreatment methods are utilized in membrane filtration. In order to enhance membrane performance, it is crucial to initially identify the primary causes of membrane fouling [34]. The effectiveness of pretreatment in reducing membrane fouling relies heavily on several important factors, which include the type of pretreatment agent employed (such as coagulant, adsorbent, oxidant, or bio-filter), the dosage and mode of dosing (continuous or intermittent), the mixing technique, the temperature, the properties of natural organic matter (NOM) (such as charge density, hydrophobicity, molecular size, and molecular weight), the solution environment (pH and ion strength), and the characteristics of the membrane itself (such as hydrophobicity, membrane charge, and surface morphology) [35].

3.3. Coagulation

Coagulation is used as a pretreatment process to increase the rate of particle aggregation. It is the most common and effective pretreatment process due to its low cost and relatively simple operation [36]. It is still a promising method for reducing membrane fouling while improving turbidity, dissolved organic carbon (*DOC*), and microorganism removal [37][38]. It is critical to optimize the coagulation process [39]. To begin, the type of coagulant used can significantly impact the performance of membranes, and under-dosed coagulation could harm membrane performance. An adequate coagulant dose significantly reduced fouling and improved membrane performance, resulting in high removal rates of microorganisms and other waterborne impurities under optimal coagulation conditions. Optimizing operating conditions, such as raw water pH, improves coagulant performance, resulting in less fouling and improved membrane performance. Other coagulants, such as alum or ferric chloride ($FeCl_3$), may necessitate pH adjustments for optimal performance. Coagulant performance may also be affected by the mode of coagulation. Coagulants can be used in-line or in standard mode. In-line coagulation occurs without sedimentation or pre-filtration, whereas standard coagulation does [40].

3.4. Activated Carbon Adsorption

The process of foulant adhesion to an adsorbent surface, known as “adsorption”, is commonly used as a pretreatment method. Adsorbents possess a high porosity and a large specific surface area, allowing them to absorb or accumulate impurities effectively [33]. Among the various adsorbents, powdered activated carbon (PAC) is widely utilized in membrane filtration applications [41]. Adsorption can be combined with membrane filtration in two configurations, similar to pre-coagulation: a unified membrane reactor or a separate reactor following a PAC reactor. Two dosing methods are employed: step input, which introduces PAC into the reactor at a constant rate, and pulse input, which adds all the PAC at the beginning of the filtration cycle. Optimal PAC dosage should be determined through preliminary tests before implementation. Additionally, PAC size must be optimized, considering the potential impact on membrane integrity due to abrasion and the specific material, PAC type, and configuration used. To address the challenges associated with carbon fiber felt (CFF) [41], a separation step has been proposed to prevent direct contact between the PAC and membrane surface. While PAC adsorption is cost-effective [42], its suitability as a pretreatment method in developing countries needs to be evaluated to determine if PAC particles can enter membrane pores and cause fouling. The possibility of some impurities not being absorbed by PAC but readily entering membrane pores may restrict the widespread adoption of PAC [34].

Several authors have explored the theoretical advantages of specific pretreatment methods and integrated multiple approaches to compensate for limitations. Integrated systems often come with high capital costs, which can be challenging for developing countries. However, if such systems effectively control fouling and improve membrane performance, operational costs may be reduced. In situations where poor-quality source water needs to be transformed into high-quality effluent, even if the total costs are high, integrated systems may be the only viable option. However, it is important to note that no single known technique can effectively control fouling [33]. Furthermore, some integrated systems might even exacerbate membrane fouling. One possible explanation for the conflicting performance of integrated pretreatment systems is the formation of precipitates resulting from the combination of certain pretreatment procedures, which can be detrimental to membrane fouling. Therefore, it is crucial to thoroughly evaluate any adverse consequences when implementing integrated systems. Although the capital costs of filtration systems may increase with integrated pretreatment, current research efforts should focus on optimizing specific pretreatment methods to enhance membrane permeability [34].

3.5. Mitigate Membrane Fouling Using 3D-Printed Promoters

As mentioned above, fouling removal from membranes continues to be a formidable barrier to their widespread adoption, as cleaning is expensive and generates significant waste [43]. As a result, there is much interest in new membrane materials and/or structures that can reduce fouling and the use of cleaning agents. The main goal in all cases is to reduce interactions between the foulants and the membrane surface [44], either by changing the wetting behavior of the membrane [45] or by promoting fluid turbulence at the membrane surface via surface structuring [46]. As a result, the latter approach is preferred because it applies to commercial membrane materials. Turbulence is created primarily by generating vortices near the membrane surface due to regular or irregularly patterned structures such as pillars, lines, or indents [6]. These patterns are created through various techniques; one of the latest technologies is 3D printing, which is a new membrane fabrication technology that allows the creation of more complex and irregular membrane shapes and structures that are impossible with current methods [6][47].

Ref. [48] state that fouling is frequently controlled by turbulent flow, which requires more energy. In the flow channel of tubular membranes, turbulence promoters or static mixers can be inserted. They deflect the fluid, induce vortices, improve particle back-transport, and increase the shear rate at the membrane surface, all of which help to prevent fouling. However, more is needed to know how the geometry of such turbulence promoters affects fouling reduction.

Ref. [49] explain that changing the hydrodynamic conditions in the membrane module can result in improved mixing efficiency and flow conditions, incorporating three-dimensional (3D)-printed spacers into the module can improve mixing efficiency and flow conditions. Three-dimensional-printed spacers in the module can improve mass transfer through the *UF* membrane by reducing concentration polarization and fouling. Three-dimensional printing has the potential to enable a promising new class of efficient laboratory filtration devices. On the other hand, higher mechanical stirring into the module can reduce membrane fouling by increasing the shear rate on the membrane's surface.

Researchers have taken an interest in adapting variants of 3D printing techniques to membrane manufacturing as their resolution has improved to the micrometer or even nanometer level. Ref. [50] indicate in their research that according to Scopus database statistics, there has been an increase in membrane papers related to 3D printing over the last decade, mirroring the increase in papers on 3D printing overall. Customizing spacers for membrane processes such as *UF*, *RO*, forward osmosis (*FO*), and membrane distillation (*MD*) was the focus of the early work on membrane-related printing technology.

Turbulence promoters are a promising alternative for improving hydrodynamic conditions in membrane separation processes [51]. These devices reduce particle deposition by increasing the shear rate on the membrane surface [52]. Turbulence promoter geometry is also essential in their effectiveness in membrane filtration processes. Devices based on 3D printing make significant progress in the design of turbulence promoters because 3D printing technology allows the creation of several complex geometric shapes from various materials [5].

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