Removal of organic matter and heavy metals of low concentration from wastewater via micellar-enhanced ultrafiltration: an overview

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Abstract. As a new and effective means of wastewater treatment, the micellar-enhanced ultrafiltration (MEUF) has been extensively studied. In this paper, MEUF was introduced from the aspects of theory basis, ultrafiltration membranes, and surfactants. Additionally, the latest research achievements in removing organic matter and heavy ions, its application in actual wastewater, and the characterization parameters of MEUF were introduced and summarized. Then, influences and mechanisms of the primary operation parameters, including surfactant concentration, pH, electrolytes, and transmembrane pressure on the performance of the MEUF process were analyzed. Finally, existing problems in the MEUF process were identified and developmental trends were predicted.

1. Introduction
Micellar-enhanced ultrafiltration (MEUF), which was first developed by Dunn et al. in 1985 for the treatment of wastewater, combines the use of surfactants and ultrafiltration membranes for the removal and recycle of organic matter and heavy metal ions of low concentration in wastewater [1]. Its beneficial characteristics include strong separation capability, easy extension, and low energy consumption [2]. In virtue of efforts over the past two decades, MEUF has achieved great progresses and its industrial applications have been observed.

1.1. Principles of MEUF
In cases the surfactant concentration is higher than the critical micelle concentration (CMC), the surfactant forms an aggregate with a hydrophobic domain inside and hydrophilic region outside, i.e. micelle. Based on the like dissolve like theory, organics have increased solubility inside the micelle while charged groups on the surface attract counter ions in the solution through electrostatic interaction. Therefore, aggregates of organics-micelle or metal ions-micelle in the solution can be blocked by an ultrafiltration membrane with a pore size smaller than the diameter of the aggregates.

With a deeper understanding of MEUF, studies in recent years have focused on reverse micelles [3]. Compared to normal micelles, reverse micelles are surfactant aggregates in apolar solvents with hydrophilic groups inside and hydrophobic groups pointing toward an apolar phase. The hydrophilic inner-core of reverse micelles can dissolve hydrophilic materials inside and achieve successful removal or recycle through ultrafiltration.
1.2. Ultrafiltration membranes

Defined as the maximum molecular weight of substances of which 90% are retained, the molecular weight cutoff (MWCO) of a membrane is an important factor that influences the treatment efficiency of MEUF. A membrane with a MWCO larger than the micelle molecular weight is often adopted in the MEUF process. Usually, the permeation flux is higher for a membrane with a larger MWCO, although the removal efficiency for contaminants is lower. The hydrophobicity of a membrane also has a certain impact on MEUF. If a hydrophilic membrane is used to remove hydrophobic organics, hydrogen bonds between the membrane surface and water molecules attract water molecules, thus reducing attraction and contamination to the membrane. Commonly used hydrophilic ultrafiltration membranes include polyacrylonitrile hollow fiber membranes, cellulose acetate membranes, polyamide membranes, and polyethersulfone membranes; hydrophobic ultrafiltration membranes include polysulfonemembranes, PESF membranes, polyphenylene ether membranes, and ceramic membranes. Advances on ultrafiltration membranes aim to reduce membrane contamination and improve removal efficiency. Ngang et al. improved hydrophilicity using ultrafiltration membranes doped with TiO₂ nanoparticles, which increased the permeation flux and reduced contamination of the membrane [2]. Furthermore, an external magnetic field was applied to the membrane to reduce the concentration polarization phenomenon during removal of charged ions with MEUF, hence reducing membrane contamination, increasing permeation flux, and improving removal efficiency [4, 5].

1.3. Surfactants

Surfactants can be classified into anionic, cationic, and nonionic surfactants. Common anionic surfactants include sodium dodecyl sulfate (SDS), sodium dodecylbenzenesulfonate (SDBS), and linear alkylbenzenesulfonate (LAS) [6, 7]; common cationic surfactants include cetyltrimethylammonium bromide (CTAB), n-octyltrimethylammonium bromide (OTAB), and cetlypyridinium chloride (CPC) [8]; nonionic surfactants include polyoxyethyleneoctyl phenyl ether (TX-100), oxyethylated coconut fatty acid methyl esters (OMC), and polyoxyethylene glycol dodecyl ether (Brij-35) [9, 10]. In recent years, research trends have included the pursuit of surfactants to remove certain contaminants. EI-Abbassi et al. discovered that SDS efficiently removes polyphenols in MEUF [11]. Fang et al. reported that CPC is superior to OTAB and CTAB in removing phenols [8]. Usually, anionic surfactants are chosen for the removal of metal ions, while cationic surfactants are preferred for anions and organics [12].

Mixed surfactant systems are attracting increasing attention and have been extensively studied in recent years. The advantages of using mixed surfactant systems include greatly reduced amount of surfactant being used, cost, surfactant concentrations of permeated solution, and secondary pollution. Nonionic surfactants and ionic surfactants are often combined. This principle is based on the concept that nonionic surfactants act as a grid on the anionic surfactants, reducing the repulsion between anionic surfactants and thus promoting the formation of micelles. Beislak et al. adopted a mixture of SDS and OMC-10 for the removal of methylene blue (MB) [9]. The CMC of the surfactant decreased from 8.3×10⁻³ M to 2.0×10⁻³ M when the ratio of SDS to OMC-10 was 4:1, and using a mixed surfactant showed no impact on the removal of MB. Paiboon et al. mixed SDS with nonionic surfactant of TX-100 and NP12 in MEUF [13]. This method improved the removal efficiency of Pb²⁺ and the surfactant, which solved the problems of low SDS removal efficiency when using only SDS, and low Pb²⁺ removal efficiency when using only anionic surfactants. Huang et al. used a mixture of SDS and TX-100or Brij-35 in MEUF for the removal of Cd²⁺ ions and discovered that although the CMC was effectively reduced by adding nonionic surfactants to the solution of anionic surfactants, excess nonionic surfactants reduced the micelle adsorption of Cd²⁺ ions [10]. This was because the charge density was reduced with the addition of nonionic surfactants, which in turn reduced the electrostatic adsorption. Consequently, there is an optimum ratio when using multiple surfactants.

The Gemini surfactant is a new and effective surfactant that is different from traditional surfactants in structure and property in that it contains two hydrophobic chains and two hydrophilic head groups. The CMC of the Gemini surfactant is quite low and its adsorption of charges of different polarity is
stronger due to its stronger charge compared to traditional surfactants. Zhang et al. studied cationic Gemini surfactants in MEUF for the removal of phenols and obtained excellent removal efficiency, which showed great application potential [12].

Along with the development of MEUF, requirements for surfactants are also advancing. Using biodegradable surfactants for reducing secondary pollution to the environment and the subsequent treatment expenditures are trends in MEUF development. Monem et al. adopted arhamnolipid bio-surfactant for the removal of heavy metal ions, copper, zinc, nickel, lead, and cadmium, and demonstrated the ability to lower the concentrations to below federal Canadian regulations [14].

2. Application of MEUF

2.1. Removal of organics with MEUF

Great efforts have been made to investigate the mechanism of organic removal by MEUF. The removals of methylene blue and various reactive dyes such as Reactive Black 5 and Reactive Orange 16 have been demonstrated (>99%) [15]. Xu et al. studied the interaction between phenols and CPC and found that phenols were adsorbed at the interface between water and micelle due to the electrostatic interaction between $C_6H_4O$ and $C_5H_5N^+$ or increased solubility in hydrophilic domain of CPC micelles at a low concentration of phenols while at a higher concentration of phenols, phenols at the surface of micelles and in the hydrophilic domains saturated and entered the palisade layer of micelles but were still closed to the hydrophilic regions of CPC [16]. Deriszadeh et al. removed naphthenic acids by MEUF and discovered that the amphiphilic nature of the acids reduced the CMC value of the CPC surfactants, hence reducing membrane contamination and CPC concentration in the permeated solution and improving the removal efficiency [17]. To date, the application of MEUF in organics-containing wastewater treatment has not gained sufficient significance. Vinder et al. applied MEUF in the treatment of adsorbable organic halogens (AOX) in industrial wastewater and found that the removal efficiency was mainly determined by the characteristic of the surfactant and electrolyte concentration. In the presence of nonionic surfactants, when the solution conductivity is 4.0 mS/cm, the removal efficiency can exceed 90%, and the concentrations of anionic surfactants, SDS, and SDBS are lower than their CMC. El-Abbassi et al. treated wastewater from an olive mill, which contained polyphenols using MEUF [11]. The removal efficiency of polyphenols was 74%, and a faintly-colored permeate (>87%, less dark) was achieved at the transmembrane pressure of 4 bars and SDS concentration of 10CMC under pH 2. Furthermore, MEUF has been tested in the removal of sulfonamide antibiotics of low concentrations in pharmaceutical wastewater with a removal efficiency that exceeded 70% [18]. In addition to the removal of contaminants, MEUF applications for recycling of valuable low-concentration organics in solutions have also been explored. For example, Schwarzze et al. reported recycling an expensive catalyst in solutions using MEUF [19]. Geantaet al. reported recycling lactic and citrate acids in solutions using MEUF [20].

2.2. Removal of metal ions by MEUF

Many studies have been conducted on using MEUF for the removal of low-concentration heavy metal ions including arsenic, zinc, cadmium, copper, nickel, and lead [13-14]. In recent years, the removal of heavy metal ions has advanced from removing only one kind of heavy metal ion to removing various heavy metal ions simultaneously. Fang et al. found that in the systems of one metal ion and mixed metal ions under the same experimental conditions, the removal efficiency of $\text{Cd}^{2+}$, $\text{Cu}^{2+}$, and $\text{Ni}^{2+}$ ions was similar (all higher than 98%), indicating that the removal mechanism of MEUF was based on the electrostatic adsorption of heavy metals onto the micelles, and was dependent only on the valence number of heavy metals and independent of the kind of heavy metals [21]. Using MEUF for removing heavy metal ions is progressing towards treating actual wastewater. Aguirre et al. removed heavy metals from phosphorous-rich drainage waters (pH of 3.2 and SDS concentration of 75.6 mM) of a fertilizer company using MEUF; the removal efficiency of cadmium and copper was 84.3% and 75.0%, respectively [22]. At the same time, MEUF is making its way into the recycling of low-concentration
heavy metals in solutions. Kedari et al. utilized MEUF for the recovery of low-concentration americium (III), and achieved a recovery efficiency of 100% with SDS as the surfactant and a solution of pH level of ≥ 3 [23].

2.3. Simultaneous removal of organics and heavy metals using MEUF

With environmental concerns growing and contaminant compositions in the environment becoming more complex, the simultaneous removal of organics and heavy metals is gradually becoming a necessity; this need has attracted growing attention and extensive investigations. Tung et al. used a mixture of SDS and Triton X-100 to simultaneously remove Cu²⁺ and phenols, and discovered that the removal efficiency of the Cu²⁺ was higher than that of Cu²⁺ alone, while the removal efficiency of phenols was lower than that of phenolsalone [24]. Witek et al. proposed that the presence of Cr³⁺ did not influence the removal of phenols with a surfactant system of SDS or CTAB [25]. Misra et al. demonstrated experimentally that in a SDS surfactant system, the presence of UO₂²⁺ did not affect the removal of di-butyl phosphate (DBP) [26]. Zeng et al. found during the simultaneous removal of Cd²⁺ and MB that the presence of MB increased the removal efficiency of Cd²⁺ when the SDS concentration was lower than 1 CMC [27]. The removal efficiency of Cd²⁺ reached 98.8% when the concentration of MB and SDS was 4 mg/L and 1 CMC, respectively. However, the MB removal efficiency reached 96.5% in the presence of Cd²⁺, which was a decrease of 3.4% compared to that of MB alone.

2.4. Technical parameters of MEUF

Common technical parameters of MEUF include removal efficiency and permeation flux. Removal efficiency is defined as the ability to retain or reject contaminants with the use of a membrane under certain conditions, and is often expressed as follows:

\[
R = \frac{C_R}{C_P} - 1 \cdot \frac{C_R}{C_P}
\]  

where \(R\) is the rejection rate or removal efficiency of the contaminant; \(C\) is the concentration of the contaminant; subscripts \(R\) and \(P\) denote concentrated solution and permeated solution, respectively.

Permeation flux is defined as the volume of permeated solution passing through the membrane per unit time and per unit area. It is often used to indicate wastewater treatment efficiency of the membrane and the contamination level. Permeation flux is usually expressed by the following equation:

\[
J = \frac{Q}{A}
\]

where \(J\) (L·m⁻²·h⁻¹) is the permeation flux of the membrane; \(Q\) (L·h⁻¹) is the feed solution flow passing through the membrane; and \(A\) (m²) is the effective area of the membrane.

In addition to removal efficiency and permeation flux, new parameters have been deduced in succession and adopted in current studies; examples include the distribution coefficient (D), adsorption amount of micelle per unit (L), and relative adhesion coefficient of contaminant (K). D represents the distribution of contaminants between the concentrated and permeated solutions. A higher D value indicates a higher degree of enrichment of contaminants and better removal efficiency. It can be expressed by the following equation [28, 29]:

\[
D = \frac{C_R}{C_P}
\]

Adsorption amount of micelle per unit (L) characterizes the amount of contaminant adsorbed by a micelle unit, and can be expressed by the following equation [27, 28]:

\[
L = \frac{C_R}{S_R} \cdot \frac{C_P}{S_P}
\]

where \(S\) is the concentration of surfactant. In the case of a mixed surfactant system, \(SR\) is the sum of the concentrations of all surfactants in a solution.
The relative adhesion coefficient of contaminant (K) represents the relative adsorption amount of contaminant on the micelle, and can be expressed by the following equation [28, 29]:

$$K = \frac{C_M}{C_W} \frac{S}{S}$$

(5)

where subscripts M and W denote micelle and water phases, respectively; and S is the concentration of surfactant in the micelle phase.

With the introduction of new parameters, the principle of the enhanced ultrafiltration of the micelle is understood more profoundly. Paiboonet et al. used distribution coefficient (D) and relative adhesion coefficient of contaminant (K) to describe the removal of lead ions in wastewater using MEUF based on mixed surfactants, and demonstrated that an increased surfactant concentration simultaneously improved the removal efficiencies of lead ions and surfactants as more surfactant monomers formed micelles, which strengthened the micelles’ adsorption of lead ions [29]. In a study of the simultaneous removal of nickel and cobalt using MEUF, Karate et al. manifested the highest efficiency of heavy metals removal at the early stage of ultrafiltration through a comparison of the changes of removal efficiency and distribution coefficient under different ultrafiltration volumes [28]. The results were also verified by the adsorption amount of micelle per unit (L) and relative adhesion coefficient of contaminant (K).

3. Factors that influence the treatment efficiency of MEUF

3.1. Effects of surfactant concentrations

Micelles are only observed when the surfactant concentration in the solution is no less than 1 CMC; however, the concentration polarization effect resulting from the membrane filtration in MEUF causes abundant surfactants to aggregate near the membrane surface, which leads to the formation of micelles and a much higher concentration of CMC. Therefore, with MEUF, contaminants can be removed even if the surfactant concentration is lower than 1 CMC [6]. Samper et al. used SDS and low-concentration LAS to remove heavy metal ions in solutions; more than 90% of the heavy metal ions were removed when the SDS concentration was lower than 1 CMC, and almost 100% were removed when the LAS concentration was lower than 1 CMC. In a certain range, the removal efficiency of contaminants increases with an increasing surfactant concentration with MEUF. However, excess surfactants can result in compact micelles and a reduced hydraulic radius, which causes the solubilization of organics by the micelles to be reduced. This results in a simultaneous change in the morphology of micelles from ball-shape to rod-shape. In such a case, both the solubilization of organics and adsorption of heavy metals by micelles are significantly reduced, and they penetrate the membrane into the permeated solution easily, which leads to a reduced treatment efficiency [30, 31]. Huang et al. increased SDS concentration from 8 mM to 80 mM in MEUF and the resultant MB removal efficiency decreased from 99.34% to 87.14% [31]. Moreover, it was discovered that excess micelles readily aggregated on the membrane surface and formed a gel layer, which lead to a reduced permeation flux.

3.2. Effects of pH value

The heavy metal ions removal efficiency of MEUF increases gradually with increasing pH levels. This can be attributed to the fact that H+ ions in the solution are adsorbed by charges of different polarity on the micelle surface and compete with metal ions, which reduces the available adsorption sites of the micelle surface for the metal ions [23]. Therefore, it is not suitable to use MEUF for heavy metal removal under acidic conditions. Aguirre et al. found that the optimum pH was 3.2 for the removal of copper and cadmium from phosphorous-rich drainage waters of a fertilizer company using MEUF[22].

The influence of pH on organics removal using MEUF is less obvious than that on heavy metals, which relates to the property of organics to some extent. Studies have shown that pH change does not influence the removal of dyes, e.g. MB and Safranin T; the removal efficiency of both is above 99% [31, 32]. Based on a study of phenols removal using CTAB as the surfactant in MEUF, Sabatino et al. found that the removal mechanisms were affected by the pH value, although the pH changes did not
significantly affect the removal efficiency [33]. Phenols are adsorbed on the micelles surface by ionic adsorption under neutral conditions (pH=6.7) while under basic conditions (pH=9.94), phenols are removed due to increased solubility in the palisade layer of micelles. Venkataganesh et al. studied the removal of naphthenic acid (NA) using MEUF and found that under acidic conditions, carboxylic groups of NA were ionized to neutral and NA molecules became nonionic, which resulted in its increased solubility in micelles; under basic conditions, the NA decomposed and its water solubility increased [4]. At the same time, the CMC of SDS micelles decreased at low pH levels while the CMC remained unchanged at higher pH levels (>5). The NA removal efficiency decreased from 82% to 68% when the pH level increased from 3 to 9.

3.3. Effects of electrolytes
Due to the presence of various electrolytes in wastewater, it is necessary to understand the influence of electrolytes in the MEUF process. Electrolytes reduce the CMC of surfactants. This is primarily realized by the salting-in and salting-out effects from hydrophobic groups of surfactants for nonionic surfactants, by the neutralization of charges in micelles, and the reduction of electrostatic adsorption for ionic surfactants. The addition of concentrated electrolytes significantly reduces the metal ions removal efficiency because cations in the electrolytes compete with metal ions in the solution for the adsorption sites on the micelles [21]. Since the number of micelles increases and micelle volume decreases after the addition of electrolytes, hydrocarbons soluble in the micelle core increase while polar molecules soluble in the palisade layer of micelle decrease. Therefore, organics removal effects of electrolytes are related to the property of organics. Organics, like dyes, usually have increased solubility in the micelle core and palisade layer; thus, electrolytes have no obvious effect on organics removal efficiency [34, 35].

Previous studies showed that the permeation flux decreases with the addition of electrolytes because the repulsion between surfactants in the solution decreases, the number of micelles increases, and the concentration polarization phenomenon becomes severe, resulting in increasing deposition of micelles on the membrane surface [31]. Simultaneously, electrostatic adsorption causes the electrolyte ions to be adsorbed onto the charged micelle surface, which reduces the repulsion between micelles, densifies the gel layer on the membrane surface, and increases the resistance on the membrane surface [4]. Furthermore, viscosity of the solution and hydraulic resistance increase with the addition of electrolytes.

3.4. Effects of trans-membrane pressure
The effects of trans-membrane pressure on MEUF can be discussed based on contaminant removal efficiency, permeation flux, and flux reduction volume. Usually the change in trans-membrane pressure does not influence the removal of organics and heavy metals as contaminant removal is largely achieved by its combination with micelles [35, 36]. However, micelles are squeezed when the trans-membrane pressure is over-high, resulting in reduce dorganics solubility. At the same time, overlarge pressure increases the possibility of contaminant permeation through the membrane [31]. A change in trans-membrane pressure significantly affects permeation flux. Increasing the trans-membrane pressure overcomes permeation pressure and membrane resistance, and increases permeation flux [31]. However, higher trans-membrane pressure accelerates the growth of the gel layer, which increases the thickness and flux reduction amount [4].

4. Conclusions and future research needs
Traditional ultrafiltration membranes are limited by contamination issues, flux reduction and rapid membrane consumption. Hence, a novel anti-pollution ultrafiltration membrane and efficient membrane cleaning method are needed. Since a surfactant of about 1 CMC remains in a permeated solution, it is necessary to have a biodegradable surfactant with a low CMC. The permeated solution can be simultaneously treated again at a low cost, and currently used methods include foam fractionation and activated carbon adsorption [37, 38]. There are usually various organics and heavy
metal ions in actual wastewater; therefore, in order to realize industrialization and based on the treatment of uncomplicated wastewater, studies regarding MEUF should focus on the treatment of wastewater with multi-components. Besides, there are concentrated surfactants, organics, and heavy metals in concentrated solutions, and it is an inevitable trend of MEUF development to efficiently recycle surfactants, valuable organics, and heavy metals from such solutions.

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