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Review and assessment of the separation and recovery of zinc from the aqueous stream

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ABSTRACT

Effluents from industries include high levels of toxic heavy metals and are considered to be one of the serious sources of water pollution. Galvanizing, electroplating, tannery, textile, and dye industries are the major sources of heavy metal contaminants. To protect the environment from the toxic effects of the effluents, it is highly necessary to treat this industrial effluent before discharging it into the water bodies. This paper focuses on the critical analysis of conventional separation processes such as adsorption, chemical precipitation, coagulation and flocculation, and membrane separation processes for the separation and recovery of zinc from wastewater. This paper deals with the analysis of effective separation technologies for the removal of zinc from industrial effluent. The advantages and limitations of the technologies for the separation and recovery of zinc have been compared and critically analyzed. This paper also provides the outline of hybrid membrane technology in the ultrafiltration process known as complexation-enhanced ultrafiltration for the separation and recovery of zinc from the aqueous stream.

Keywords: Zinc; Heavy metals; Adsorption; Chemical precipitation; Membrane processes

1. Introduction

water is the source of life and vitality for millions of people around the world. One of the persistent issues we have today is water pollution. The rapid growth of industrialization and population has created a polluted environment. One of the major concerns related to rapid industrialization is the generation of a huge volume of wastewater contaminated with heavy metals which is directly discharged into the water bodies without proper treatment [1]. Electroplating, pigment, textile and leather industries, paper and pulp industries, galvanizing sectors, petrochemical, and refineries are the main sources of wastewater. Metal finishing industry waste, medical trash, electronic scraps, battery waste, and other industrial wastes are the few sources of wastewater. Heavy metal pollutants, unlike organic contaminants, are not biodegradable, hazardous, and tend to accumulate in living organisms. Due to their poisonous or carcinogenic effects and accumulation through the food chain, heavy metal contamination of aquatic organisms is a global environmental concern [2–5]. These heavy metal ions, however, can be transformed into less hazardous compounds. The proper separation and recovery of heavy metals could produce value added compounds. Toxic metals such as mercury, nickel, zinc, copper, lead, chromium, iron, etc., can persist in either chemical or mixed forms, making their removal from wastewater a challenging task. However, at the trace level, these heavy metals are required by living beings for metabolic activities. But when present in higher concentrations, these metals can affect human health irreversibly. Heavy metals in open water kill aquatic life, deplete oxygen, and produce algae blooms. When heavy metals

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are dumped into rivers or any water bodies, they become hydrated ions, which are significantly more dangerous than metal atoms. These hydrated ions disturb the enzyme process, causing absorption to speed up [6,7]. The World Health Organization (WHO) has established the standards for the maximum allowable limits of certain heavy metals in potable water and industrial wastewater to be discharged into water resources, as well as the repercussions for human health if these limits are exceeded. The tolerance limit of the toxic heavy metals is shown in Table 1.

Zinc is one among the heavy metals, and it reacts moderately with oxygen and a few non-metallic compounds to produce hydrogen, and dilute acids. But presently, the concentrations of zinc are rising at an abnormal rate as a result of human-induced addition. Huge quantities of zinc are being consumed during industrial processes such as mining, burning of coal, fossil fuel combustion, burning of waste, fabrication of steel, steel galvanization to the production of the negative plate in electrical batteries, alloy preparation, smelting, etc. It is used in the galvanizing industry, electroplating industry, as a pigment in the plastic industry, etc. It is also employed in rubber manufacturing as a catalyst and also as a heat disperser in the finished product [8]. Hence, the effluents from these processes contain an enormous volume of zinc. Since it possesses excellent antioxidant properties, which can protect the human body from premature aging, almost every single tablet contains it. It has a significant impact on human health when it is consumed at a trace level. High concentrations of zinc can cause health hazards such as gastrointestinal diseases, lung disorders, risk of cancer, irritation stomach cramps, and so on [9]. The issue with the pollution related to heavy metals in water and aquatic organisms, especially fish, requires continuous monitoring and surveillance because these elements do not disintegrate and tend to biomagnify human beings through the food chain. So, it is very important to remove heavy metals from the industrial effluent, before discharging the effluents into the water bodies. In this context, in recent years research and development are being focused on industry-specific methods and technologies for separation as well as recovery of zinc from wastewater.

This review paper aims to provide an overview of the different technologies adopted for the separation and recovery of zinc from industrial effluent. This study critically analyses the existing technologies, and recent practices as well as research work that are involved in the removal and recovery of zinc. Separation or removal efficiency is measured in terms of percentage removal, rejection, or retention.

Various separation technologies have been utilized for the separation of zinc from industrial wastewater. Chemical based separation processes such as precipitation, coagulation, flotation, adsorption, ion exchange process, electrochemical process, and physicochemical processes such as membrane separation process, electrodialysis, etc., have been reported in the literature for the separation of Zn(II) from the effluent. In recent years, carbon nanomaterials are gaining importance for the separation of heavy metal ions from the effluent. There is tremendous progress in the synthesis of innovative carbon nanotubes for effluent treatment. The emerging technologies utilize zeolite-based composite materials for effluent treatment due to their successful employability as an adsorbent as well as an excellent photocatalyst [10]. This paper highlights the emerging technologies with their feasibility and also drawbacks for the removal of Zn(II) from the effluent.

2. Separation processes

2.1. Chemical precipitation

Chemical precipitation is a simple and easily mechanized treatment process. This is one of the commonly used techniques for the removal of Zn(II) from industrial

Table 1
Toxic heavy metals tolerance limits and their impact on human health [1,74]

Heavy metal ions	Permissible limit for drinking water (mg/L)	Permissible limit for wastewater (mg/L)	Major sources	Health hazards
Zn	3	2–5	Metal plating, refineries, brass manufacture	Skin irritation, anemia, nausea, vomiting, damage to the nervous system, dermatitis
Hg	0.006	0.05	Pesticides, batteries, paper industry	Oral ingestion, issues in the gastrointestinal, damage to the nervous system, and kidney, and increases the incidence of some benign tumors
Cu	2	1	Metallurgical industry, electroplating industry	Liver and kidney damage, anemia, gastrointestinal effects, irritation in the stomach
Cr	0.05	0.05	Mine, mineral sources	Causes cancer, mainly lung cancer
Pb	0.01	0.01	Automobile emissions, coal burning, pesticides, paints	Fatal infant encephalopathy, chronic damage to the nervous system, liver, and kidney.
Ni	0.02–0.07	0.02	Metal smelters, pesticides, fungicides.	Difficulty in sleeping, irritability, nausea, vomiting
Cd	0.003–0.005	0.003	Welding, fertilizer, electroplating, pesticides.	bone marrow, lung disease, bone defects, lung cancer

wastewater. This process requires a huge quantity of chemicals to reduce the concentration of Zn(II), which is one of its drawbacks. This also leads to the generation of a huge volume of secondary pollutants such as sludge. In the chemical precipitation process, the precipitant added with the effluent reacts with Zn(II) and converts it into an insoluble precipitate. To separate these insoluble precipitates, the effluent has to undergo additional separation processes like sedimentation, filtration, or/and membrane separation processes [11,12]. The process flow diagram is shown in Fig. 1.

In chemical precipitation processes, impurities from a solution are separated as sediment, which can subsequently be filtered, and centrifuged. The precipitate is a complex compound that forms between the precipitation agent and heavy metal ions, which lowers the metals bioavailability. pH is one of the most essential factors in chemical precipitation. The type and concentration of metal ions, the precipitation reagent utilized, the presence of additional chemicals, and reaction circumstances are all factors that can affect the effectiveness of chemical precipitation processes [13]. The conventional methods of chemical precipitation for the separation of heavy metals involve metal hydroxide precipitation and metal sulfide precipitation [13]. Various precipitating agents such as calcium hydroxide ($Ca(OH)_2$ [14], calcium carbonate ($CaCO_3$) [13], soda ash (Na_2CO_3) [15], sodium sulfide (Na_2S), calcium oxide (CaO) [14,16], sodium hydroxide (NaOH) [17], potassium hydroxide [17], hydrogen sulfide [18], oxalic acid [19], ethylene diamine tetraacetate [20] have been reported in the literature for the separation of Zn(II).

The mechanism involved in the metal hydroxide precipitation is as follows:

$$M^{2+} + 2(OH)^{-} \leftrightarrow M(OH)_2 \quad (1)$$

The dissolved metal ion (M^{2+}) in the effluent stream reacts with the precipitant (OH^-) and produces insoluble metal hydroxide precipitate M(OH) [13]. In the case of metal sulfide precipitation, sulfide is added to the effluent to produce the insoluble metal sulfide precipitate.

Ray et **al**. [21] investigated a two-step treatment method that involves electro-Fenton treatment for chemical oxygen demand reduction and then zinc removal through chemical precipitation. Zn^{2+} was precipitated in $Zn(OH)_2$ form from treated wastewater after the electro-Fenton experiment by adding calcium oxide (CaO). Separation efficiency was found to be 99.3% at pH 10. The formation of the main negative soluble species was reduced when the pH was increased beyond 10.

Chen et **al**. [15] studied the efficiency of precipitating agents such as $Ca(OH)_2$, Na_2S, and Na_2CO_3 for the removal of metal ions Zn, Cu, and Pb. Particle size variations and chemical phase conversion of precipitates were given specific attention. Copper and zinc were successfully removed from the aqueous solute ions with the three precipitants, while lead was more efficiently separated by sodium sulfide with a removal efficiency of 99.75%.

Oncel et **al**. [22] examined the separation of Cu(II) and Zn(II) using precipitating agents such as NaOH, $Ca(OH)_2$, and Na_2CO_3. The separation was found to be more than 90%. Zinc and copper were precipitated as amorphous hydroxides in the sludge product, including $Zn(OH)_2$, and $Cu(OH)_2$. The volume of sludge generated was found to be quite low. As a result, drying processes could be economical for sludge treatment. Soda ash could be used as a cost-effective precipitating agent for Cu(II) and Zn(II) from industrial wastewater. Shah et **al**. [23] studied the selective separation of Cu(II) and Zn(II) followed by the recovery of zinc as zinc oxide using precipitation stripping. The recovery of zinc was reported to be more than 90%.

However, due to certain limitations such as huge volume of chemical consumption, sludge handling and disposal problems and limited possibility of recovering the metal ions and precipitating agent, chemical precipitation lacks in the full fledge application for the separation of heavy metal ions.

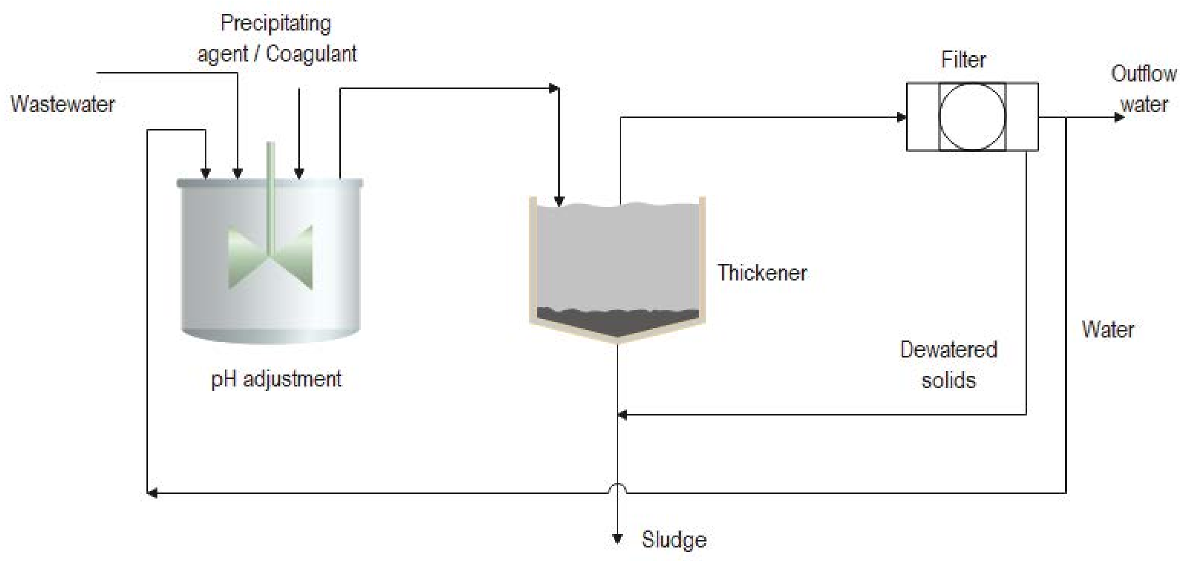

Fig. 1. Process flow diagram of conventional heavy metal precipitation.

The separation of Zn(II) by metal hydroxide precipitation is a simple technique and depends on the pH of the solution. However, this process generates a huge volume of sludge which is difficult to dewater [13]. Metal sulfide precipitation lacks due to the low solubility, colloidal precipitate formation, and possibility of toxic hydrogen sulfide generation. In recent years, to overcome these limitations, the research has been focused to employ alternate precipitation methods such as precipitation using chelating agents, dithiocarbamate compounds, etc., for the separation of Zn(II) from the effluent [13].

2.2. Coagulation and flocculation

Coagulation is a process that involves the formation of large aggregates from insoluble particles. The coagulants such as aluminium sulfate [24], magnesium chloride [24], poly aluminum chloride [24], and ferric chloride – polyacrylamide [25] were reported in the literature for the separation of Zn(II). In the flocculation process, flocculants bind with the target metal and form huge agglomerates which can be separated by sedimentation, filtration, etc. Natural and synthetic polymers which are water soluble have been reported in the literature. Synthetic water polymers have attracted attention due to their ability to create stable and large agglomerates. However, they lack in their implementation due to their non-biodegradability. In recent years, functionalized bioflocculants have been investigated for the separation of Zn(II). The functionalized bio flocculants such as starch-graft-P[acrylamide-acrylicacid], starch-graft-P[acrylamide-2-acacrylamido-2-methyl-1-propanesulfonicacid] [26], were utilized for the separation of Zn(II).

Amuda et **al**. [25] have investigated the separation of Zn(II) from beverage industry effluent using ferric chloride in combination with polyacrylamide as a coagulant. This work was focused on the separation efficiency using ferric chloride and polyacrylamide independently, and the combination of ferric chloride-polyacrylamide. It was observed from the experimental findings that the combination of ferric chloride-polyacrylamide could yield better separation when compared to the ferric chloride and polyacrylamide combination.

Hankins et **al**. [27] studied the separation of Zn(II) which involves the binding of Zn(II) with humic acid, followed by coagulation and flocculation using poly diallyl dimethyl ammonium chloride. The effect of parameters such as polyelectrolyte dose, pH, the concentration of humic acid, and Zn(II) was evaluated. To isolate the free metal ions from the solution ultrafiltration was carried out. The percentage separation Zn(II) was found to increase with coagulation–flocculation of humic acid by poly(diallyldimethylammonium chloride).

In spite of the highlights of the coagulation–flocculation process such as simple and easy to adopt, there are certain limitations such as consumption of chemicals, increased sludge volume, handling, and treatment of sludge, need for secondary treatment processes. The recently carried out works in this area attempt to overcome all these limitations and recover Zn(II) as a value-added component to successfully implement the process on a large scale. The various precipitating agents, coagulants, and flocculants employed for the removal of Zn(II) are shown in Table 2.

2.3. Adsorption

Adsorption is a widely employed technology for the removal of heavy metal contaminants from wastewater. Ease of operation, low operational cost, high separation efficiency, and feasibility of regeneration of adsorbents were all the noted benefits of this process. When it comes to heavy metal removal from the effluent, adsorption by activated carbon is the widely adopted technology because of its higher surface area and affinity towards heavy metals. This review elaborates on the removal of Zn(II) from industrial wastewater through the adsorption process. A variety of adsorbents have been developed for the removal of Zn(II) with promising results. Although activated carbon, is a popularly used absorbent for heavy metal removal, its use is restricted due to its high cost and limited regeneration potential. In the past few decades, researchers have been working towards the synthesis of low-cost novel adsorbents as a potential alternative to activated carbon to achieve equivalent efficiency [7,28].

2.3.1. Adsorbents

Carbon-based adsorbents were employed widely for the separation of Zn(II). The functional groups of adsorbents such as phenyl, and carboxyl can boost the surface charges to facilitate the separation process. But these carbon-based adsorbents are very expensive due to their complexity in

Table 2
Review of different precipitant/coagulant/flocculent for the removal of Zn(II)

Separation method	Precipitant/coagulant/flocculent	pH	Removal percentage	References
Chemical precipitation	CaO	9–10	99.3	[21]
	$Ca(OH)_2$	11	99.65	[15]
Chemical precipitation	Na_2S	10	98.9	
	Na_2CO_3	9	99.69	
Chemical precipitation	NaOH	10	99.89	[75]
Coagulation–flocculation	$FeCl_3$	7	36	[76]
	Flocculant	7	42	
Coagulation–flocculation	PAX-215	–	33	[77]
	Chitosan	–	51	

synthesis, which limits their implementation in industrial applications [29,30].

In the past few years, chitosan has attracted attention as a feasible adsorbent for the separation of heavy metal ions from aqueous effluent. It is a natural biopolymer that has an affinity for the metal ions in wastewater. This affinity towards the metal ions is mainly due to the amino and hydroxyl groups present in it. Despite its distinctive characteristics, it has minimal mechanical durability and low regeneration capability. Due to the lower surface area, mass transfer resistance, high crystallinity, and low porosity, it is very difficult to apply chitosan in powder or flake [31,32].

Several adsorbents synthesized from natural minerals such as zeolite and clay have been employed for the separation of Zn(II) from the wastewater due to excellent stability, selectivity, adsorption capacity, surface charge, and hydrophilicity, and high swelling capacity [33,34]. Furthermore, acid washing, pillar bearing, and thermal treatment can enhance the size of the pores, their volume, and specific surface area, which results in a significant boost in the effectiveness of adsorption. Natural minerals may be more cost-effective. However, after a few cycles, the removal efficiency may drop. As a result, various modification procedures, such as calcination and impregnation, have been proposed to improve the adsorption capacity [35,36].

Another category of adsorbents includes magnetic adsorbents, mainly magnetic nanoparticles like Fe_3O_4. Carbon, chitosan, starch, polymers, or biomass could be the base materials. Magnetic field, redox activity, and surface charge all influence the adsorption process. Iron oxides such as hematite α-Fe_2O_3, maghemite γ-Fe_2O_3, magnetite Fe_3O_4, zero-valent iron nanoparticles, and spinel ferrites are some magnetic adsorbents proposed in the literature [37]. The adsorbents utilized for the separation of Zn(II) from the aqueous stream are shown in Table 3.

Mo et **al**. [38] reported the review on the functionalized zeolite for the separation of Zn(II). Their review work highlighted the impact of the functionalization of zeolite, the potential application of the adsorbent for the separation of Zn(II) and other heavy metals, mechanism of separation. Zhang et **al**. [34] investigated the adsorption study on the separation of Zn(II) from sewage using NaP zeolite developed from coal fly ash as an adsorbent. The concentration of Zn(II) in the feed solution was 100 mg/L, the optimum pH was found to be 5 and the maximum adsorption capacity was found to be 40 mg/g. The separation mechanism was described by pseudo-second-order kinetics and Langmuir adsorption isotherm.

Zeolite-based polymer composite materials are also emerging as novel adsorbents for the separation of heavy metal contaminants from aqueous streams due to their mechanical and chemical stability, surface area, flexibility, etc [10]. The blend of organic and inorganic composite materials could very effectively separate Zn(II) from the aqueous stream as compared to either organic or inorganic compounds as adsorbents. Zeolite-based nanocomposite materials are synthesized by incorporating metal oxide nanoparticles, and carbon-based compounds [10]. It could be employed for the removal of both cationic and anionic pollutants from the aqueous stream owing to their pore size, photoactivity, physiochemical stability, and ease of operation [10,39,40]. The functional modification of zeolite could be done by acid/base treatment, ultrasonic and thermal modification to increase the pore volume and diameter [10]. Panek et **al**. [41] investigated the simultaneous separation of Zn(II) and Pb(II) using zeolite as well as zeolite-carbon composite as an adsorbent. The results exhibited the higher separation efficiency of natural zeolite with Zn(II) due to their ion exchange capability. Due to the smaller ionic radius, the movement of Zn(II) towards the actives sites of the adsorbent was much faster than Pb(II). The ionic interaction was explained by the following equation:

$$Zn^{2+}_{(solution)} + 2Na^{+}_{(zeolite)} \rightarrow Zn_{(zeolite)} + 2Na^{+}_{(solution)} \quad (2)$$

Table 3
Brief review of the adsorbents employed for the separation of Zn(II) from wastewater

Adsorbents	pH	Percentage adsorption/Adsorption capacity	References
Cork powder	6.5	92	[78]
Natural clinoptilolites	8	97	[79]
Conditioned clinoptilolites CC_1	4–8	99	
CC_2	4–8	99.7	
CC_3	4–8	97	
Powdered fish bones	5	98	[80]
Activated carbon derived from oil palm empty fruit bunches	–	98	[81]
Bagasse fly ash	4	100	[82]
Nanostructured cedar leaf ash	–	94	[83]
Bentonite of Mostaganem	9	86.3	[84]
Kaolin		81.4	
Egyptian oil shale	–	99	[85]
Ulva fasciata sp.	5	76.42	[86]
Natural zeolite	–	656 mg/g	[41]
Zeolite-carbon composite	–	600 mg/g	
Fe_3O_4-SiO_2	–	119 mg/g	[87]

Substantial percentage desorption was observed with hydrochloric acid treatment of the adsorbent [41]. However, a great challenge lies in the reusability of the adsorbent.

The adsorption process has proven to be a promising methodology for the separation of heavy metal ions. However, due to the cost of adsorbents, and limitations in the recoverability of adsorbents, this process lacks applications in large-scale operations. In recent years, low-cost adsorbents and nanocomposite adsorbents have been formulated and applied successfully for separation studies on a lab scale and pilot scale.

2.4. Membrane separation process

During the 1980s, membrane separation processes advanced at a breakneck speed. When compared to the other conventional processes, membrane separation technologies have merits such as (i) better separation, (ii) less energy requirements in the case of low-pressure membrane separation processes, (iii) less chemical usage, (iv) easy to operate and compact, (v) ambient operational conditions, and (v) can be combined with other conventional methods [12]. Because of these applications, membrane processes find applications in the food and beverage industry, biotechnology, wastewater treatment, and eventually for the treatment of heavy metals containing wastewater. The performance of the membrane can be influenced by a variety of factors. Membrane material, pore size, and contact angle are the essential characteristics. Materials should be selected in such a way that they aid in membrane fabrication with excellent chemical resistance and less fouling. Membrane fabrication involves the use of a variety of materials, including metallic, ceramic, and composite materials. Other innovative fabrication of membranes involves incorporating catalytic nanoparticles in the membrane structure. The characteristics of pressure driven membrane processes are shown in Table 4.

Polymers are a popular choice in membrane fabrication due to their low cost and porous nature. Some of the most widely employed polymeric membrane materials are cellulose acetate, polyvinylidene fluoride (PVDF), polyacrylonitrile (PAN), polypropylene (PP), polyethersulfone (PES), and polysulfone (PSU). In some circumstances, ceramic materials outperform polymeric materials, due to their small pore size distribution, and great thermal, mechanical, and chemical resilience. Ceramic membranes are made using alumina, silica, titania, zirconia, oxide mixes, sintered metals, etc [11].

2.4.1. Microfiltration

Microfiltration (MF) is considered to be one of the oldest commercially adopted pressure-driven membrane separation processes. MF can retain suspended particles, proteins, yeast cells, and significant pathogens. MF is a versatile membrane process since it can reject a wide spectrum of large-scale pollutants [42]. Because of the vast range of pore sizes, this pressure-driven membrane process can be used in a variety of fields such as pharmaceuticals, food [43], desalination [44], biotechnology, and wastewater treatment [45]. Because of its low efficiency, the application of MF for the separation of heavy metal has received only trivial consideration. However, by modifying the membrane materials and also through pre-treatment of the effluent MF could be adopted significantly for the separation of heavy metals [28]. In recent years, complexation followed by MF has been widely adopted as a promising technology for the separation of heavy metal ions from the aqueous phase. This process involves the formation of metal complexes with polymer ligands followed by the separation using the MF membrane. A variety of synthetic polymers such as polyvinyl alcohol, poly acrylic acid, polyethylenimine, and biopolymers such as chitosan and pectin were employed as complexing species in this process [46]. The schematic representation of complexation followed by the MF process is shown in Fig. 2.

Trivunac et **al**. [46] studied the efficiency of sodium carboxymethyl cellulose (Na-CMC) as a complexing agent for the removal of zinc ions from water through MF complexation processes. The studies were carried out in a stirred dead-end cell. The generated polymer-metal combination was separated using versapor membranes. The study showed that the usage of Na-CMC for the removal of zinc is quite promising, with higher rejection coefficients of 99%. The high rejection of metal ion was observed at pH 7 and 8 owing to the higher ionic strength. Sekulić et **al**. [47] studied the complexation microfiltration process for the removal of Pb, Cd, and Zn using two water-soluble derivates of cellulose. In this paper, the artificial neural network (ANN) tool was employed to predict the rejection efficiency by MF. The results showed that ANN could be employed successfully for predicting the percentage rejection of heavy metals. Mavrov et **al**. [48] selected synthetic zeolite P as a bonding agent for the removal of Zn, Cu, and Ni through a hybrid process combining flotation and submerged microfiltration.

2.4.2. Hybrid ultrafiltration processes

Ultrafiltration, one of the widely employed pressure-driven membrane processes, employs porous membranes with a molecular weight cut-off (MWCO) of 1–300 kDa with a pore diameter of 2–100 nm. Ultrafiltration (UF) is applied for concentrating macromolecule solutions by allowing only solvent molecules and low MW solutes to pass across the membrane [49]. This paper mainly focuses on the removal of zinc. One of the drawbacks of applying the ultrafiltration process for heavy metal removal is that the size of the heavy metals is relatively smaller than the pores of the UF membrane, such that these heavy metal ions could easily pass through the UF membrane which led to the idea of hybrid ultrafiltration processes such as complexation enhanced ultrafiltration (CEUF), micellar-enhanced ultrafiltration (MEUF), etc. In these methods, the size of the target molecules (heavy metals) has been enhanced through complexing species such as N-N-N-triethylammonium chitosan (TEAC) [50], carboxymethyl chitosan (CMCh) [6], polyethyleneimine (PEI) [51], poly(N,N-dimethylaminoethyl methacrylate) (PDMAEMA) [52], carboxymethyl-β-cyclodextrin (CM-β-CD) [53], unmodified starch [54], acacia gum (AG) [55], polyvinylamine (PVAm) [56]. In MEUF, the addition of enhancing agent results in the formation of precipitates that could bind with metal ions to form a macromolecular structure of metal and surfactant complex. During the separation process, the UF membrane retains the macromolecular

Table 4
Characteristics and solutes retained by MF, UF, RO, and NF [88]

Pressure driven membrane processes	Molecular weight cut-off (kDa)	Membrane type	Solutes retained
Microfiltration	100–500	Porous, asymmetric, or symmetric	Suspended solids, bacteria
Ultrafiltration	20–150	Asymmetric, microporous	Suspended solids, virus, bacteria
Nanofiltration	2–20	Thin film composite, asymmetric, tight porous	Multivalent ions, suspended solids, virus, bacteria
Reverse osmosis	0.2–2	Semi-porous, thin film composite, asymmetric	All the contaminants including monovalent ions

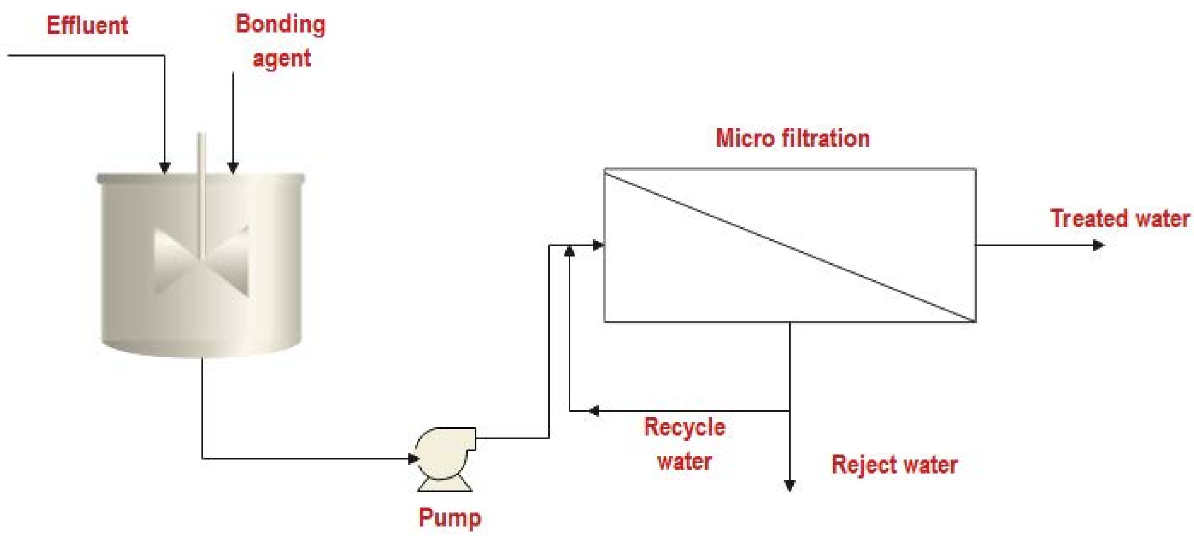

Fig. 2. Schematic representation of complexation followed by microfiltration process.

structure and non-complexed species only will flow through the UF membrane. High retention of the metal-surfactant complex can be achieved when a cationic surfactant is employed for the removal of anionic metal ions and an anionic surfactant is employed for the removal of cationic metal ion.

Innocenzi et **al**. [57] performed the MEUF process for the separation of Zn(II) and yttrium from simulated wastewater. They employed sodium dodecyl sulphate as a surfactant and performed the separation studies in a mono-tubular ceramic UF membrane with molecular weight cut-off of 210 and 1 kDa. The percentage retention of the metal ions (99%) was excellent with a UF membrane of 1 kDa MWCO. The chief limitation of MEUF is that it results in the generation of secondary pollutants if the metal-surfactant is not properly disposed of.

CEUF is a form of water-soluble polymeric agent-based ultrafiltration purification technique. The polymeric agents bind to metal ions to form macromolecule, which gets retained by the membranes. Since this process greatly depends on the solution pH, the metal ions and polymers could be recovered by altering the solution pH and can be reused [58]. There are only a few papers reported in the literature on the separation of zinc ions through CEUF. The CEUF process has advantages like high selectivity, because of the selective bonding between the metal ion and polymer ligands, minimal energy requirements, and no limitation of osmotic pressure. Also, the fouling problem is minimum due to the possibility of backwashing of the membrane. The pressure-driven membrane separation processes such as reverse osmosis and nanofiltration suffer the problem of limitation of osmotic pressure which restricts the absolute separation of metal ions by these processes. The schematic representation of the CEUF process is shown in Fig. 3. A brief literature review of the different complexing species employed for the removal of Zn(II) from wastewater is shown in Table 5. The mechanism of complexation-enhanced ultrafiltration is shown in Fig. 4. The separation is by ionic interaction of metal ion with polymer ligands as well as chelation. The separation mechanism is represented in Fig. 5.

Kavitha et **al**. studied the separation and recovery of heavy metal ions by CEUF, also known as size-enhanced ultrafiltration. The percentage retention of metal ions Cu(II), Ni(II), Pb(II), and Zn(II) was observed to be more than 95% and the recovery of metal ions was observed to be more than 75%. The results revealed that the complexation was more pronounced at basic pH due to the high affinity of OH^- present in the polymer ligands to complex with the metal ions. It was also observed from the results that the percentage retention was quite high even at neutral pH. Hence,

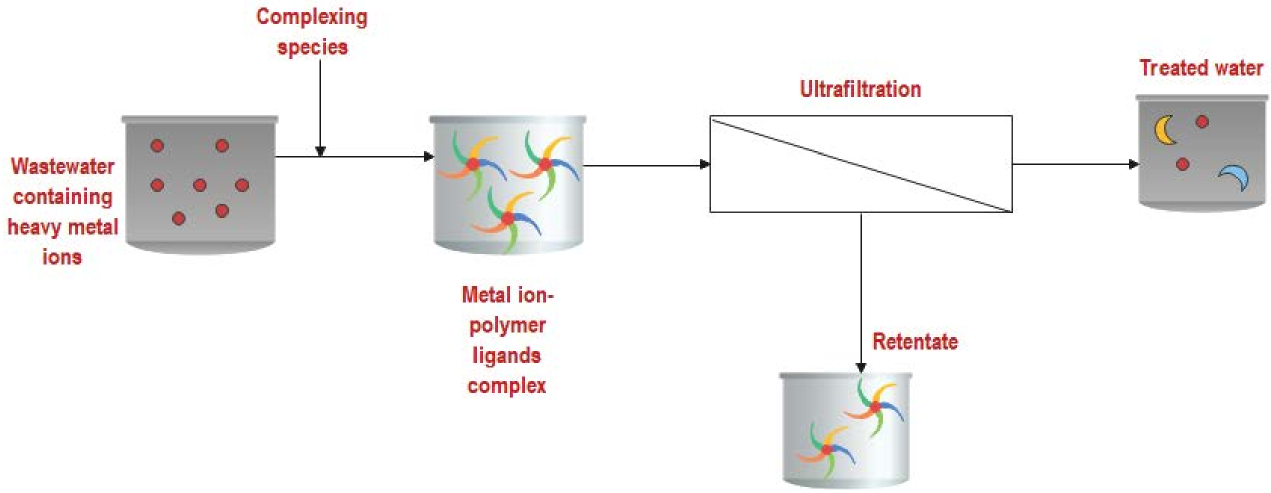

Fig. 3. Complexation-enhanced ultrafiltration process.

Table 5
Brief review on different complexing species for the removal of Zn(II)

Metal ions	Complexing species	Membrane material	Molecular weight cut-off (kDa)	Rejection percentage (%)	References
Zn^{2+} Ni^{2+}	Polyacrylic acid (PAA)	Polyethersulfone	10	99.1 93	[89]
Zn^{2+} Cd^{2+} Pb^{2+}	Sodium dodecyl sulfate (SDS)	Polyethersulfone	–	99 97 99	[90]
Zn^{2+} Cu^{2+} Ni^{2+} Pb^{2+}	CMCh	Polyethersulfone	50	87 100 100 90	[6]
Zn^{2+}	SDS	PAN-MEOF	30	84.67	[91]
Zn^{2+} Pb^{2+} Cr^{3+} Cr^{4+}	Unmodified starch	Polysulfone hollow fibre	10	87.82 83.86 87.74 92.06	[92]
Zn^{2+}	PAAS	Polyethersulfone	30	95.3	[93]
Zn^{2+} Pb^{2+}	Unmodified starch	Polyethersulfone	10	96 66	[94]

without much altering the pH of the effluent the separation could be achieved by CEUF.

Mehenktaş and Arar performed CEUF using sodium lignosulfonate as a complexing species for the separation of Zn(II) from both synthetic and electro-plating effluent. The maximum percentage retention of 90% was observed at pH in the range of 3–7. There was no impact of monovalent ions present in the solution for the separation of Zn(II) and observed a significant impact of divalent ions. With the electroplating effluent, the percentage retention of Zn(II) was observed to be 80%. Substantial separation of Zn(II) from the effluent is feasible without much altering the initial pH and with a minimum quantity of complexing species. Hence, CEUF is a promising methodology for the separation as well as recovery of Zn(II) from the aqueous stream.

2.4.3. *Nanofiltration*

Nanofiltration is a well-known pressure-driven membrane-based filtration technology. The pore diameter of nanofiltration membranes lies in the range of 1 to 10 nm which is lower than the pore diameter of ultrafiltration membranes. Nanofiltration (NF) activity represents a halfway point between ultrafiltration and reverse osmosis processes. NF is widely used in water and wastewater treatment, desalination, food, biotechnology, pharmaceutical, etc [60,61]. Several studies were performed for the removal of heavy metal ions through nanofiltration and obtained promising results. Kočanová et **al**. [62] have studied the effectiveness of the nanofiltration process for the removal of zinc from a real industrial sample as well as a synthetic binary aqueous

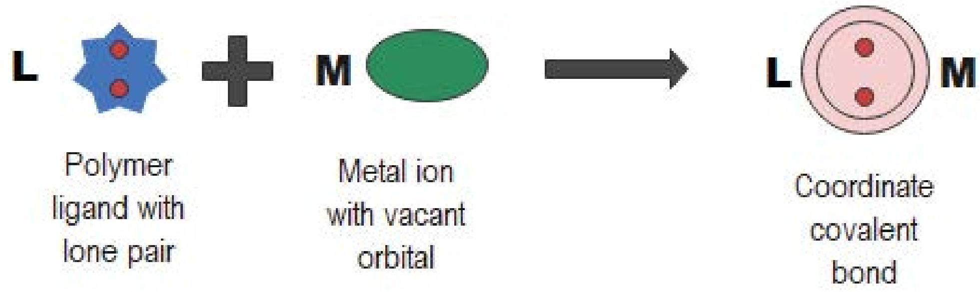

Fig. 4. Mechanism of complexation-enhanced ultrafiltration process.

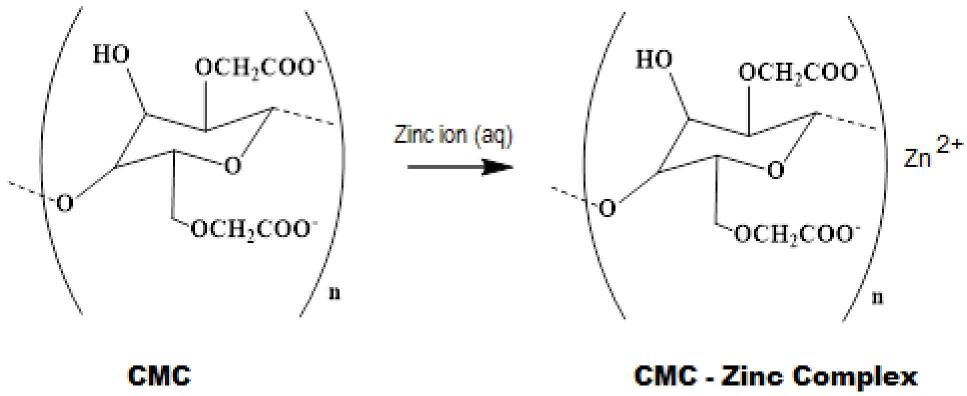

Fig. 5. Separation mechanism of Zn(II) using carboxymethyl cellulose by complexation-enhanced ultrafiltration process.

solution. AFC 40 nanofiltration membrane was employed for the separation process of Zn(II). The results of their studies were found to be promising as the AFC 40 membrane showed a high value of rejection (98%) of Zn(II) with real effluent as well as simulated effluent. Shukla et **al**. [63] studied the effectiveness of carboxylated-graphene oxide (GO)-incorporated polyphenyl sulfone (PPSU) nanofiltration membrane for the removal of Zn, Ar, Cr, Cd, and Pb. The incorporation of carboxylated-GO resulted in better performance of the membrane. This work exhibited that PPSU/carboxylated-GO nanocomposite membrane is highly economically favourable for heavy metal removal as it showed outstanding retention with acceptable volumetric flux. The rejection of metal ion was found to be governed by the Donnan and dielectric exclusion principle. The charge on the surface of the membrane was observed to be negative due to the presence of carboxylic groups, which could attract the cations and expel the anions. It was found to be negligible effect of transmembrane pressure on the metal ion rejection.

Moradi et **al**. [64] fabricated a novel antifouling nanofiltration membrane for the effective removal of metal ions. The membrane was prepared by introducing tetrathioterephthalate (TTTP) filler into the polyethersulfone (PES) matrix through phase inversion. This work exhibited promising results as the T-PES performed better when compared to PES. Also, T-PES embedded PES showed a high value of metal ion rejection for Zn, Cu, and Pb. One of the major limitations of NF membranes is severe fouling which leads to a drop in permeate flux. To overcome this issue, we need to do periodic cleaning of the membrane surface which is again involving operational costs. The work carried out by Moradi et **al**. [64] represented satisfactory permeate flux with T-PES NF membranes. Hence, NF with suitable composites of membrane material could enhance the feasibility of the separation of heavy metal ions such as Zn from an aqueous stream. In recent years many works have been carried out by researchers focusing on the suitable selection of material for the synthesis of composite NF membranes with promising results with the retention of heavy metal ions.

2.4.4. Reverse osmosis

The widely employed commercial pressure-driven membrane process, reverse osmosis (RO) has been acknowledged as an excellent desalination process. In recent years, a lot of improvements have been applied in RO technology, such as membrane material, processes, and also energy recovery which in turn increased the interest in its commercial applicability. RO process has been included in processes like separation, selective removal, and concentration [65]. Algureiri and Abdulmajeed [66] investigated the separation of heavy metal ions from industrial effluent by RO. The experimental findings showed more than 95% removal of heavy metal ions, and high productivity, involving medium-pressure application.

Ipek [67] investigated the removal of Zn^{2+} and Ni^{2+} from wastewater by a RO unit by modifying several parameters like initial solution pH and concentration of metal ions. The effects of EDTA as an aid to the membrane process were also investigated. Results shown were promising with a high rejection percentage of nearly 100% of Ni^{2+} and Zn^{2+}, respectively with an addition of EDTA at a concentration of 240 ppm.

Chung et **al**. [68] investigated a combined RO and ferrite process for a local metal plating wastewater treatment plant. Wastewater containing zinc and chromium ions was simultaneously purified and concentrated using disc-type RO modules. Zinc ions were recovered from RO concentrations via ferrite reaction. 99.7% of zinc ions were recovered after the ferrite reaction. The zinc plating waste stream containing 40 mg/L of Zn(II) was investigated for their studies and the concentration was reduced to 0.24 mg/L after treatment with RO. The detailed economic study was also conducted for the treatment of preplating rinse, zinc plating, and chrome plating wastewater. The combination of RO with ferrite process was proposed for the existing plant from the pilot scale study and economic analysis. As mentioned earlier, RO is a well-established technology for the separation of heavy metal ions. However, several works have been carried out for the past few decades, to enhance the membrane characteristics to minimize the fouling characteristics and also the economic operational cost related to the operational pressure.

2.4.5. Electrodialysis

Electrodialysis (ED) has been widely adopted for the treatment of brine and saline water. In recent years, ED has become an emerging technology for the separation of heavy metal contaminants. This process is based on the principle of transfer of anions and cations across anion exchange and cation exchange membranes. The ionic exchange membranes will allow the selected species (anion/cation) to permeate through it. This process involves the concentration and recovery of valuable metal ions. In the case of electroplating industries, a huge volume of wastewater containing valuable metal ions is generated, and by adopting the ED process it is feasible to recover those valuable species for reuse. Hence, ED could be an effective technique not only for separating heavy metals from the effluent stream but also for recovering valuable metal ions.

Choi and Jeoung [69] investigated the separation of Zn(II) from simulated wastewater and reported significant results. They employed 2 cells electro dialysis unit and observed that beyond the voltage of 10 V, there was no impact of voltage on the separation of Zn(II). They reported more than 97% removal of Zn(II). Boucher et **al**. [70] studied the separation of Zn(II) by employing commercial ED units. Buzzi et **al**. [71] performed the electrodialysis for the treatment of acid mine drainage containing the heavy metal ions Fe^{2+}, Fe^{3+}, Zn^{2+}, Mn^{2+}, etc. The studies were conducted with three different acid mine drainage samples and the current density in the range of 1–3 mA/cm^2 was applied. The results exhibited 100% separation of Zn^{2+} from the effluent.

For the selective separation of heavy metal ions, emerging technology known as the selective electrodialysis process has been employed. This mechanism utilizes pH dependency, ionic charge, and chelating species for selective separation. The schematic representation of heavy metal separation by ED is represented in Fig. 6. Reig et **al**. [72] investigated the separation of As(V), Cu(II), and Zn(II) in

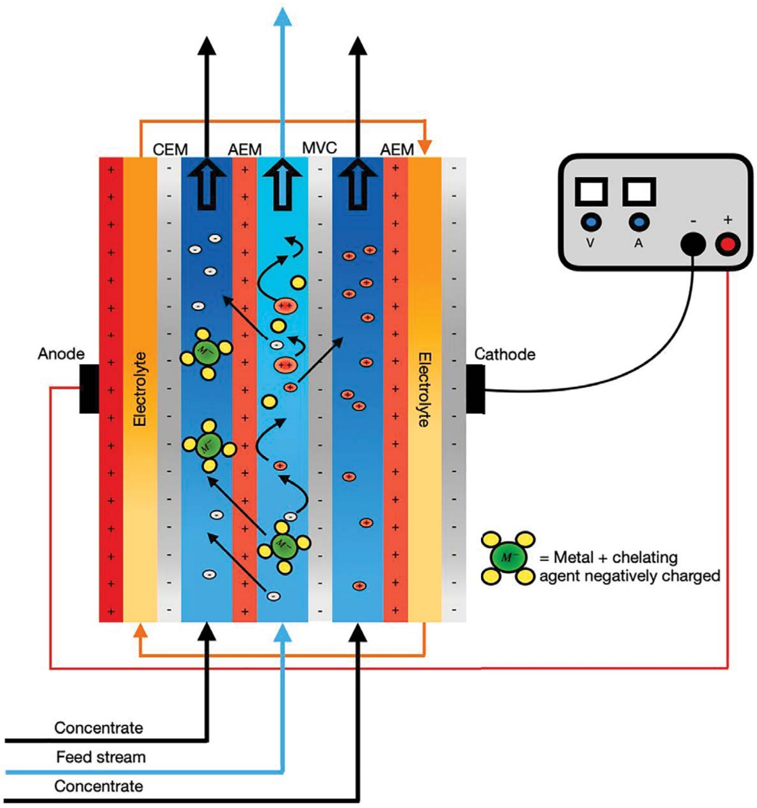

Fig. 6. Schematic representation of three compartment electrodialysis cell for the selective separation of metal ion.

a three compartment electrodialysis unit employing anion exchange, cation exchange, and monovalent ion exchange membranes. The monovalent ion exchange membranes are more energy efficient and their study reported 87% separation of Zn(II) with the energy consumption of 2.6 kWh/kg. By selective electrodialysis, they achieved 99.8% separation of Cu(II)-Zn(II) from As(V). Hence this technology has proven to be an effective methodology for the recovery of Zn(II) from mining effluents.

Membrane separation processes have excellent potential for the separation of heavy metal ions since it holds a diverse range of membrane characteristics and mechanisms. Despite their efficiency, still, most of the studies were at the research level and not implemented on large scale due to the stability of membrane, and membrane fouling [73]. Future studies should be focused on the methodology to overcome these limitations to implement the processes on large scale.

3. Conclusion

Environmental regulations have become more stringent during the past few decades, calling for treated effluent to meet the standard limits. Heavy metals containing wastewater have been treated using techniques such as adsorption, chemical precipitation, membrane filtration, biosorption, and coagulation/flocculation. It has been revealed from several studies that the adsorptive separation by biosorption and lost-cost adsorbent is effective and economical even for lower concentrations of heavy metals. As the contaminated species would need significant counter ions to overcome the limitation of solubility product, chemical precipitation directly may not be applicable. Membrane filtration processes for heavy metal removal have been widely studied and are considered to have excellent metal ion rejection capability. In recent years, one of the hybrid processes known as CEUF, an advanced process of ultrafiltration has been studied widely for the selective removal and recovery of zinc from industrial effluents with promising results and is considerably better than RO and NF, and also other conventional processes in terms of cost and metal ion retention capability. By overcoming the limitations and improving the membrane performance, there is an excellent chance for membrane separation processes as an adaptable and alternative technology for the separation and recovery of Zn(II) from the industrial effluent.

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