



Review on wastewater treatment for man-made cellulosic fibre industries: recent advancement and future prospects

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Received: 15 April 2023 / Accepted: 30 October 2023 / Published online: 28 November 2023
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Abstract Man-made cellulosic fibres (MMCF) are gaining attention due to their sustainability compared to other fibres. The demand for MMCF has increased due to improved standard of living leading to their accelerated production. Substantial amount of water and chemicals are required for the production of MMCF generating a significant volume of wastewater. This review highlights the type of effluents generated in MMCF industries, their characterization, along with global discharge standards. Chemical oxygen demand, dissolved salts, and heavy metals

are found as the major source of impurities. Current effluent treatment methods are critically reviewed to identify the challenges and scope for technological advancement focusing on impurities reduction, value recovery, and water recycling to meet the stringent disposal norms. Additionally, the criteria for the selection of a techno-economically feasible treatment solution are also discussed. Further, the areas for improvement in conventional zero liquid discharge system for MMCF industries have been highlighted.

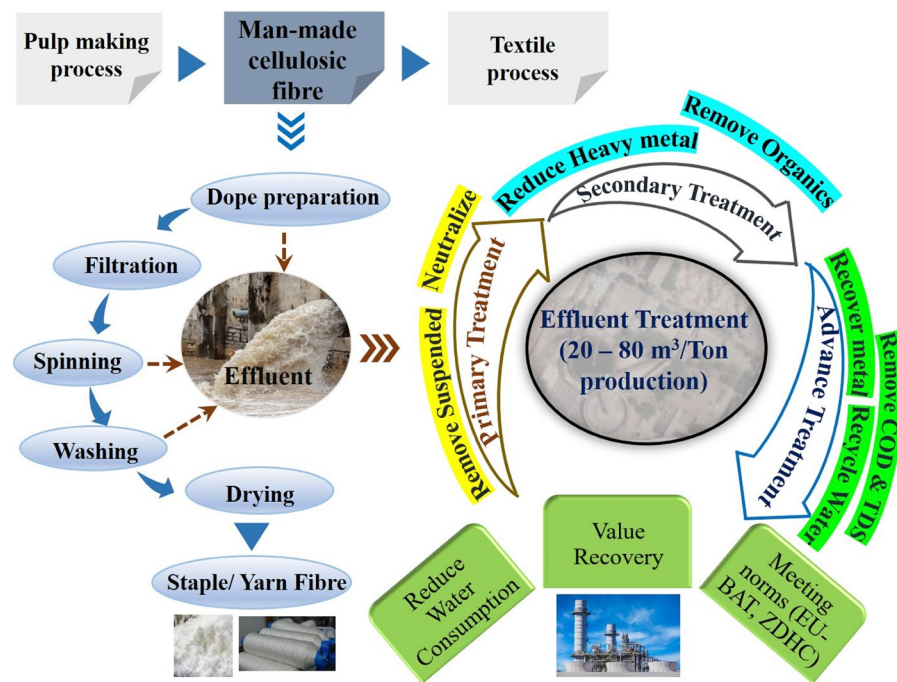
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Graphical abstract



Keywords Man-made cellulosic fibre · Disposal standards · Wastewater treatment · Advanced technologies · Techno-economically feasible · Zero liquid discharge

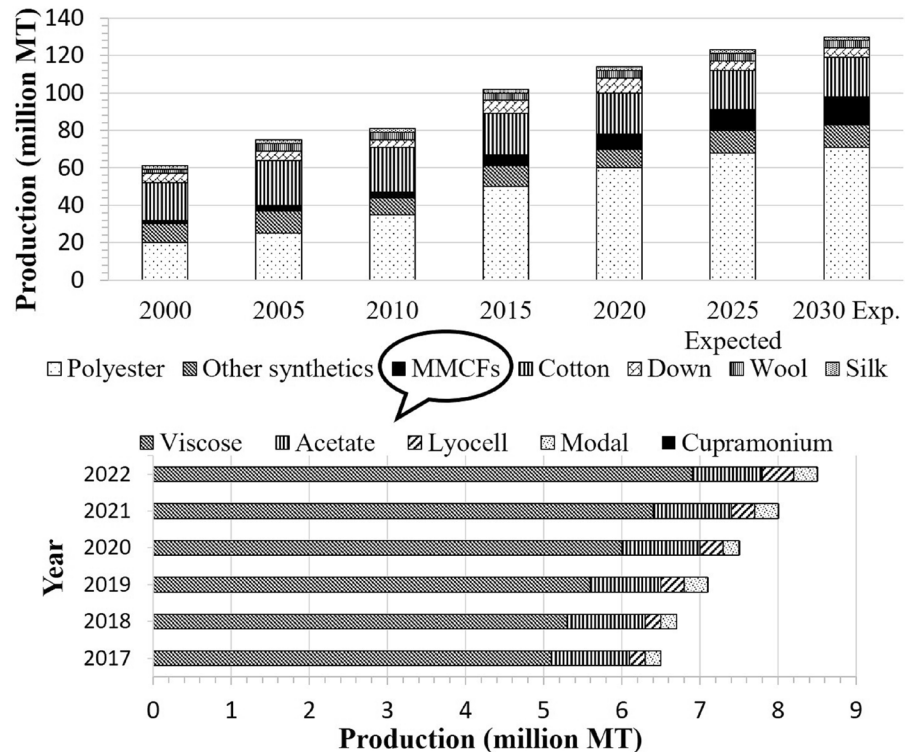
Introduction

The fibre production industry has undergone dramatic changes in the last century. Natural fibres, like, cotton, wool, silk, etc., were used widely until the industrial revolution in the nineteenth century (Seisl and Hengstmann 2021). Man-made fibres were developed in the twentieth century and had widespread applications in the textile industry (Mondal et al. 2022). A recent market survey published by the European Man-made Fibres Association indicated that the global production of these fibres was increased steadily over the past few decades compared to the natural fibres (CIRFS 2017). These fibres have high production

rate, low cost and can be modified to suit variety of applications.

Man-made fibres can be classified further into synthetic and cellulosic. Synthetic fibres such as polyester, polyamides (Nylon 6, Nylon 66), and acrylic are produced from high molecular weight synthetic polymers (Shirvanimoghaddam et al. 2020). However, they are non-biodegradable and their production can cause environmental hazards (Stone et al. 2020), whereas, cellulosic fibres are attractive in this regard. Man-made cellulosic fibres (MMCF) are produced from natural plant sources, like, wood pulp, cotton linter, etc. (Shen et al. 2010) that are abundant, and the fibres are biodegradable and biocompatible making them a suitable substitute for the synthetic fibres. Due to their desirable qualities, like, comfort, breathability and softness, the demand for cellulosic fibres is increasing globally, especially in the textile industry (Mondal et al. 2019). With an annual production of more than 8 million metric tons (refer Fig. 1), MMCF have market share of around 6.7% (Opperskalski et al.

Fig. 1 Global production of fibres over the years and growth of MMCF production (CIRFS 2017; Opperskalski et al. 2020)



2020) and by 2030 viscose fibre (type of MMCF) is projected to account for 8.5% of the total fibre market (Changing Market Foundation 2018).

Wood pulp is used as raw materials in cellulosic fibre production industries that are further sent to the textile mills to be drawn into the cloth and other uses (Vallejos et al. 2022). These fibres are not the end products but are important intermediates which are converted to variety of textile products. The leading manufacturing companies in cellulosic fibre are Grasim Industries, Lenzing, Sateri, Tangshan Sanyou, Aoyang, Shandong Helon, etc.

The amount of wastewater generated depends on the type of fibre. For example, the processing of cotton fibre and MMCF industries generate around 80–200 m³ and 20–80 m³ of wastewater per ton of production, respectively (Jiang et al. 2020; Kozłowski et al. 2012). MMCF are more sustainable as they consume less water compared to the natural fibres. Generally, untreated MMCF wastewater consists of suspended and dissolved solids, organic and inorganic impurities, and foul odour affecting the environment adversely (Riquelme et al. 2022). Thus, the wastewater treatment of the fibre industry is an area

of considerable interest to make the industry more sustainable. Currently, MMCF industries are adopting various treatment technologies to meet the increasingly strict discharge norms (Uddin 2021).

There are review articles about the effluent of textile industries (Hussain and Wahab 2018), the manufacturing process of MMCF (Sayyed et al. 2019), and the environmental impact (in terms of energy, water, and land footprint) of MMCF industries (Shen et al. 2010). However, the review of the effluent from MMCF industries is not available. The aim of this review paper is to highlight the hazardous pollutants in the wastewater produced by MMCF industries and evaluate of the current treatment technologies to identify the scope for future advancement to meet the stringent disposal standard (like EU-BAT: European union through best available techniques, ZDHC: zero discharge of hazardous chemicals). Also, this article offers insight to the advanced technologies relevant to the treatment of MMCF wastewater for future research.

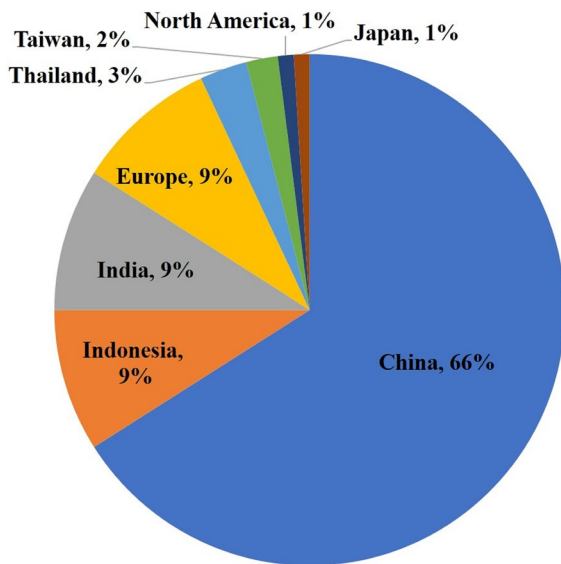


Fig. 2 World producers of viscose fibres (Freitas et al. 2017)

Overview of MMCF industries

Man-made cellulosic fibres can be classified broadly based on their production process. Globally, five types of MMCF are produced: viscose, lyocell, modal, acetate, and cuprammonium. These are also known as regenerated fibres, as dissolved wood pulp needs to be regenerated to produce the fibre.

Viscose fibre

The most important MMCF is viscose fibre, as it captures around 80% of the global market share with an annual growth rate of 6–7% in the last five years (Seisl and Hengstmann 2021). The viscose process comprises of various steps that must be controlled carefully to produce the desired quality of the product. By modifying the process parameters in one or more steps, it is possible to generate a variety of fibres. The first step in the manufacturing process is the dissolution of wood pulp, where the cellulose is allowed to react with sodium hydroxide to form alkali cellulose followed by ageing for depolymerisation. Subsequently, carbon disulphide (CS_2) is used to perform xanthation reaction forming orange crumb known as cellulose xanthate. Further, cellulose xanthate is dissolved in caustic soda to get a viscose dope. This solution is extruded through a spinneret having numerous holes into a dilute sulphuric acid bath where the cellulose is regenerated to form the filaments of fibre. The process parameters, such as spinning temperature, spinneret type, chemical concentration of regeneration bath, etc., are the major factors controlling the quality of the fibres. Viscose fibres are used in many applications, such as, clothes, upholstery, bedding materials, etc. The world's largest viscose producing regions are depicted in Fig. 2 (Freitas et al. 2017).

Table 1 Comparison of MMCF processes based on sustainability

Fibre	Process advantages	Challenges
Viscose fibre	Low cost Good quality of sodium sulphate salt as a by-product High elongation and low breaking strength of fibre	CS_2 recovery > 90% Effluent with zinc and sulphate impurities
Modal	Low-cost fibre, process is similar to viscose Good water absorption with soft and smooth fibre	Prone to stretching and pilling Effluent with zinc and sulphate impurities
Lyocell	Less process steps due to direct dissolution Biodegradable solvent (NMMO) with ~99% of solvent recovery High tensile strength of biodegradable fibre	Not as economical as other fibres Fibrillation of fibre
Acetate	Use of less hazardous chemicals, like, acetic acid Fibres with superb comfort and good absorbability of water	Poor fibre strength with retained static electricity Acetone pollution due to evaporation
Cuprammonium fibre	Fibres with high tensile strength with softness than viscose Use of pulp with a lower degree of polymerization	Expensive due to consumption of cuprammonium Environmental issues due to copper and ammonia

Modal fibre

Modal fibres are the second generation of viscose fibres with 2.8% share of the MMCF market (Opperskalski et al. 2020). They are made by modified viscose process with higher degree of polymerisation using altered precipitating bath solution. As a result, these fibres have improved properties, such as, better wear, higher dry and wet strength, and better dimensional stability.

Lyocell fibre

Lyocell is the third most produced MMCF as it has a market share of around 4.3% and it is considered the future of MMCF. The estimated compound annual growth rate from 2017 to 2022 is around 15% (Opperskalski et al. 2020). Fewer process steps are required to manufacture lyocell fibre compared to viscose fibre. Lyocell fibres are produced through the solvent spinning process (Sharma et al. 2019). In this case, the cellulose is directly dissolved in the solvent N-methyl morpholine N-oxide (NMMO) and the solution is then filtered and spun through the spinnerets to make the filaments, which are converted into fibres. The NMMO is recovered (~99%) from this aqueous solution and can be reused in the production process (Woodings 1995).

Acetate fibre

The market share of the acetate fibre is around 14% and it is used mainly in non-textile applications. Wood cellulose is swollen by acetic acid, converted to cellulose acetate using acetic anhydride and dissolved in acetone (Win Win Textiles 2020). Derivatized cellulose is used in this process for cellulose regeneration, like viscose/modal. The resulting viscous solution is extruded through the spinnerets into warm air to form filaments and the evaporated solvent acetone is recovered. The filaments are then wound as filament yarns or collected as tow.

Cuprammonium fibre

It is the least produced fibre under the MMCF with a market share of less than 1% (Opperskalski et al. 2020). Cotton linters are the source of cellulose

and these are used to produce cuprammonium fibres. When a solution of cellulose in cuprammonium hydroxide is diluted with water or treated with dilute sulphuric acid, the cellulose is regenerated. Like lyocell, direct dissolution step is used for cuprammonium fibre (Sayyed et al. 2019). The advantages and challenges of the various MMCF production processes are summarized in Table 1.

Source of wastewater

In the MMCF industries, 5–10% of the process water is used to manufacture the cellulose fibres (Shen et al. 2010). In the viscose and modal fibre manufacturing process, wastewater is generated during different steps of fibre making (Indian Standards 2003) (refer Fig. 3a). The wastewater streams can be classified broadly into two types: alkaline and acidic (refer Table 2). Acidic waste is characterized by foul odour, high temperature, and high concentration of zinc. It also contains sodium sulphate, zinc sulphate, sulfuric acid, oils, and surfactant with pH of 1 to 2 (Lun and Zhang 2014). It is generated mainly from the spinning processes (like, spinbath, makeup tank, etc.), the evaporation stage, and the fibre-washing stage. Calcium and heavy metals, like zinc, is released during spinning of filaments and wash-water from the equipment. Alkaline wastewater is characterized by high content of caustic soda and cellulose (Ding et al. 2017). The source of the alkaline waste stream is mostly from the dope preparation (specifically from the filter press, ripening, and deaeration vessels) and the fibre-washing stage due to the desulphurisation of fibre. The presence of lignin, hemicellulose, resin, etc., increases the organic concentration in the alkaline wastewater. The dissolved salts are due to trithiocarbonate and sodium carbonate that are formed during the xanthation stage and in the presence of acids (like, acetic) which are added to the dope preparation stage. Apart from acidic and alkaline streams, there are cooling and condensate water streams, which are mostly present in the carbon disulphide and the sulphuric acid plant. Due to the presence of less impurities, the condensate water can be reused in the process reducing the overall water consumption. Similarly, many plants reuse the miscellaneous wastewater in the process generated from floor washing, water treatment plant, and sanitary waste. Apart from the

Fig. 3 Source of wastewater stream at **a** viscose and modal fibre, **b** lyocell fibre, **c** acetate fibre, and **d** cuprammonium fibre making process

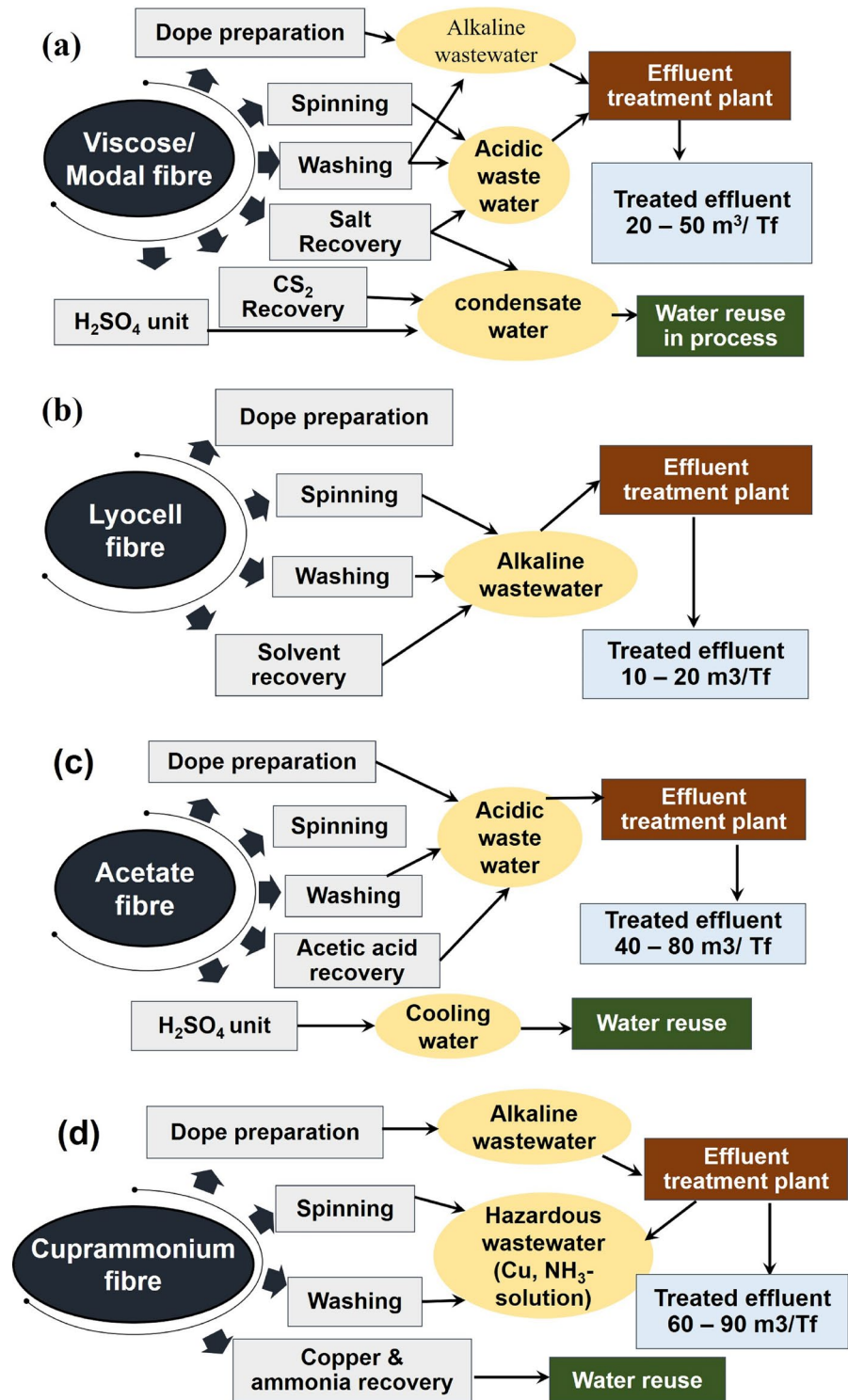


Table 2 Acidic and alkaline process wastewater generated in viscose and modal fibre process

Parameters	Acidic wastewater	Alkaline wastewater
pH value	1–2	9–11
Acidity, mg/L	2200–7700	–
Alkalinity (as CaCO ₃), mg/L	NA	310–790
Total solids, mg/L	8900–32,900	1480–2940
Total dissolved solids, mg/L	8892–31,950	960–2260
Suspended solids, mg/L	70–2210	236–680
COD, mg/L	390–790	154–1160
Zinc (as Zn), mg/L	181–315	–
Sulphate (as SO ₄), mg/L	8400–32,188	267–1315
Chloride (as Cl), mg/L	–	148–242

organic impurities, zinc as a heavy metal and sodium sulphate as total dissolved salts (TDS) contribute significantly to viscose and modal process wastewater.

In lyocell process, less wastewater is generated compared to other MMCF due to few numbers of process steps. Most of the wastewater generated in lyocell process is from the solvent recovery and washing stages that is alkaline in nature (refer Fig. 3b) as NMMO and caustic soda are used in the process to dissolve the pulp and to wash the recovery columns (Schuster et al. 2004). NMMO is biodegradable but contributes significantly to the chemical oxygen demand (COD) in wastewater. Apart from the COD load, the lyocell effluent also has small amount of sodium chloride as TDS, coming from the solvent recovery stage (Meister and Wechsler 1998).

Acetate fibres have the second-largest market share among the MMCF. The generated wastewater is mostly acidic containing acetic acid, acetate, fines of cellulose acetate, and sulphuric acid (Sayyed et al. 2019). It is generated during the dope preparation and fibre washing stage (refer Fig. 3c). Excess water is required to hydrolyse cellulose triacetate to release acetic acid during fibre washing, producing large volume of effluent. During the fibre-spinning stage, acetone is used in the regeneration bath and is recovered in the subsequent stages and therefore, does not contribute much to the wastewater. The overall acetate fibre effluent has an appreciable amount of sulphate impurities in the form of TDS, along with the organic impurities.

The manufacturing of cuprammonium fibre is limited as hazardous chemicals, like, ammonia, copper, and sulphuric acid are used in this process. Consequently, hazardous waste streams are generated (refer Fig. 3d) during the spinning and washing stages.

Alkaline waste streams are obtained in the dope preparation and washing stage because of the alkaline wash (Win Win Textiles 2020). The cuprammonium process also generates a large volume of wastewater with high COD and sulphate load.

In general, MMCF production is a chemical intensive process involving several non-biodegradable, biodegradable, and hazardous chemicals. Additionally, unused reagents remain after the final stage along with water. The wastewater of MMCF industries typically has large amount of total suspended solids (TSS), high COD, biological oxygen demand (BOD), metallic ions (mostly zinc, calcium, etc.) and TDS mostly in the form of sodium sulphate. Although the composition of the effluent depends on the type of fibre being manufactured, it should be noted that conscious efforts are exercised to maintain the standard effluent discharge norms.

Regulatory standards for effluent disposal

Several governments and environmental protection agencies have enforced rules and regulations to limit the discharge of harmful pollutants in water bodies (Ghumra et al. 2021). As mentioned earlier, among the different MMCF, viscose and modal have the dominant share in the global market. Along with the growing trend, industries need to adopt a responsible manufacturing process to meet the EU-BAT norms, apart from the country-specific regulations (Sah 2020). Furthermore, guidelines for the effluent disposal for MMCF have become more stringent over the years focusing on aspirational standards set by ZDHC (ZDHC 2023). This can be achieved by aligning the

Table 3 Disposal standards defined by countries, EU-BAT, and ZDHC relevant for MMCF industries (Asian Development Bank 2017; Government of Canada 2012; Qu and Fan 2010; Tuddao 2019; U.S.A Environmental Protection Agency 1972)

Parameters	India	Canada	China	United States	Thailand	Philippines	Indonesia	Bangladesh	Sri Lanka	EU-BAT	ZDHC
pH	5.5–9	6.5–8.5	6–8.5	6–9	5–9	6–9	5–12	6.5–9	6–8.5	–	6–9
Temperature, °C	50	30	–	40	–	40	40	40–45	40	–	Δ5
TDS, mg/L	2100	2000	–	2000	2000–5000	1200	–	2100	2100	200,000–300,000*	–
TSS, mg/L	100	40	35	30	30–150	90	60	100	500	–	30
Sulphide, µg/l	2000	200	1000	200	–	–	1000	1000	2000	0–300	500
COD, mg/L	250	80	125	80	125–400	200–300	250	200	600	3000–5000*	–
BOD ₅ , mg/L	30	50	25	30	20–60	30–200	85	150	200	–	5
Zinc, µg/l	5000	30	5000	<10,000	–	5000–10000	10,000	5000	10,000	10–50*	500
Calcium, µg/l	–	–	–	200,000	–	200,000	–	–	240,000	–	–
Copper, µg/l	3000	<1000	1000	<1000	1000	1000	2000	500	3000	–	250
AOX, µg/l	1000	500	–	500	–	–	1000	2000	1000	500	200

* numbers define in g/Tf where, Tf—Ton of fibre production

manufacturing units with sustainable treatment methods to generate cleaner outputs. Some of the regulations in various countries with EU-BAT and ZDHC (aspirational) norms relevant to MMCF industries are presented in Table 3.

Conventional effluent treatment process

MMCF industries generate large quantities of effluent and they are treated mostly in primary, and secondary methods to make them suitable to be released (refer Fig. 4). The different stages of wastewater treatment in the fibre industry have been discussed in the subsequent sections.

Primary treatment

Generally, the purpose of primary treatment is to remove coarser particles with suspended solids. The common primary treatment steps used in the industries are presented below.

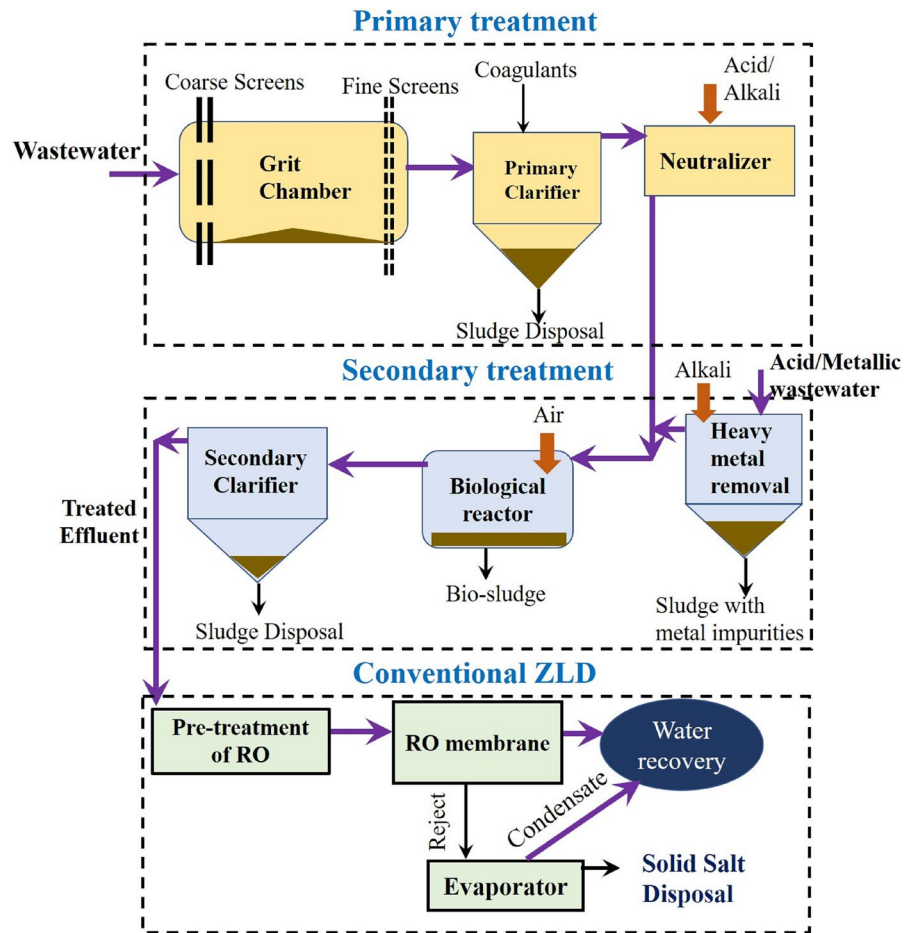
Removal of solid lumps

Wastewater collected from different processes is passed through a coarse screen or a strainer. Screening is done to remove large suspended materials, such as, fibres, gritty material and solid impurities (Kumar and Saravanan 2018). These screens typically have openings of 6 mm (0.25 in) or higher. The equipment, like, bar screens, strainers etc. (Singh and Murthy 2017), are used to filter the large solids allowing the water to pass through. This prevents pipe blockage, accumulation of unwanted materials in the treatment plant, and damage to other equipment.

Removal of suspended solids

Wastewater is then passed into a grit chamber or pond to remove the suspended solids through gravitational settling. This process works on the difference in density between the bulk liquid and the solid particles. The grit chambers have high residence time of 8–16 h and the easily settleable materials are removed. There are usually two types of grit chambers: horizontal and aerated chambers (Kumar and Saravanan 2018). The

Fig. 4 Schematic for wastewater treatment used typically in MMCF industries



exit of the grit chamber is equipped with fine screens to remove the fine solids/ lumps. The typical opening size for fine screens is around 1.5–6 mm.

The wastewater is then sent to the primary clarifier. Mostly, coagulants and flocculants, such as polyelectrolytes, aluminium sulphate (alum), ferrous sulphate, ferric chloride, and ferric chloro-sulphate are added to aid in the sedimentation (Sher et al. 2013). Coagulation works by adding chemicals with an opposite charge to those of the suspended solids in the water. These chemicals are attracted to the solids, neutralize them, and help them to sediment. The solids floating on the surface are removed by the skimmers. The settled particles are collected as sludge and sent to thickeners for dewatering. The most commonly used thickening processes are rotary drum, gravity thickening, and centrifuge thickening (Turovskiy and Mathai 2006). After sludge dewatering, the mother liquor is sent back to the primary clarifier and the thick

sludge is sent for solid waste disposal. A high degree of thickening is desirable so that the cost of sludge treatment can be minimized. Lamella clarifier is used commonly instead of conventional clarifier due to its lesser footprint along with better carbon capture from the wastewater (Gulhan et al. 2022).

Neutralization

Fibre manufacturing plants generate both acidic and alkaline waste streams. These are either combined to neutralize them, or chemicals such as sulphuric acid and lime are added so that the pH of water is neutral (Singh et al. 2008). Neutralization is necessary to ensure suitable conditions for consequent biological treatments.

Secondary treatment

The main objective of the secondary treatment is the reduction of the heavy metals, COD, and BOD of the effluent. The technologies used in this stage of wastewater treatment are as follows:

Reduction of heavy metal, Zinc

Some of the streams generated during the fibre manufacturing process, like, acidic process waste has high zinc load (refer Table 2). It is important to reduce the heavy metal load to meet the effluent discharge norms, as this stream makes up about 80–90% of the overall effluent. Additionally, it is important to reduce the load of heavy metals before biological treatment to facilitate the growth of the bacteria (Shrestha et al. 2021). The streams containing high zinc concentration are treated upfront before they are mixed with other effluent streams, as the removal of zinc from dilute streams is not cost-effective.

Conventionally, industries carry out chemical precipitation to recover the zinc /metal as zinc hydroxide ($\text{Zn}(\text{OH})_2$) or metal hydroxide in a clarifier (Chen et al. 2018). Even the solubility of different metal hydroxides varies with pH (Marchioretto et al. 2005). Generally, lime is added to the acidic wastewater stream generated from MMCF industries to raise the pH in the range of 8 to 10 to get rid of the acidity and metallic impurities and precipitate inorganic hydroxide (Jha et al. 2004). However, this process has the disadvantage of generating gypsum and metal hydroxide as precipitates that are not easy to separate. Separation is important from an economical perspective as zinc can be reused as a raw material. Some industries have explored using stepwise chemical precipitation for zinc recovery (American Enka Company 1971), where chemical precipitation is used for acid removal and metal precipitation separately. Though, the recovered metal may contain certain impurities and further treatment may be required to obtain pure zinc.

Removal of organics

The organic load of the effluent is reduced by the biological treatment. The working principle is that the organics in wastewater serve as nutrients for bacteria, that digest them helping in their separation from

water. The conditions of the influent stream to the biological treatment tank, such as pH, BOD/ COD ratio, sulphide, chloride, etc., are required to be maintained to ensure the optimal growth of the bacteria.

The organic components of water are oxidized and degraded during aerobic and anaerobic biological treatment. Aerobic bacteria are used in the aerobic biological treatment to convert the organic matter into carbon dioxide, water, and microbial sludge. The process includes an aeration tank to ensure the dissolved oxygen concentration and a settling tank to separate the sludge from water (Revilla et al. 2014). Aeration can be done with the help of the devices, such as diffused aerators, surface aerators, etc. On the other hand, anaerobic treatment is carried out in absence of oxygen and the presence of the anaerobic bacteria, like, sulphate reducing bacteria (SRB). It is generally carried out for wastewater having high COD load of 8000–15000 ppm, BOD concentration of 4000–5000 ppm, and BOD/COD ratio < 0.3 (Costa et al. 2017; Enitan et al. 2017). This process generates methane as a by-product that can be used further as fuel. Shoukat et al. (2019) proposed a hybrid aerobic-anaerobic system that was able to remove 99.5% COD from the textile wastewater. The biologically treated effluent is then sent to a secondary clarifier that removes dead bacteria, etc., from the effluent. The clarifier usually has a residence time of around 6 to 7 h which allows the sludge to either sediment or float on the surface based on density that can be separated further.

Use of activated sludge is an alternative treatment for organic degradation, like, NMMO in lyocell wastewater is biodegraded in conventional wastewater treatment plants using activated sludge (Meister and Wechsler 1998). Further emerging technology to treat fibre industry wastewater is the use of a membrane bioreactor (MBR) (Hu et al. 2018; Kong et al. 2023). This technology has several advantages: (i) it eliminates the need for secondary clarification of the biologically treated water; (ii) it has a smaller footprint; (iii) it requires lower maintenance and achieves better removal of organics compared to the activated sludge process (Jallouli et al. 2023; Jegatheesan et al. 2016). A stepwise chemical treatment process, consisting of a moving bed biofilm reactor (MBBR) followed by a MBR was reported to reduce the COD of the leachate from a viscose rayon plant by 85% (Camper and Bott 2014). The major challenge in using direct membrane

Table 4 Comparison of advanced treatment methodologies for COD reduction

Treatment	Method	COD load (ppm)	COD reduction	Mechanism	References
Chemical	Chlorine, Hypochlorite	Not available	10–20%	$Cl^- / OCl^- + H_2O \rightarrow \dot{OH} + HCl / HOCl / (Hydroxyl\ radical)$	Quader (2010)
AOP	Ozone, Hydrogen Peroxide	–	15–20%	$\dot{OH} + RH \rightarrow \dot{R} + H_2O$ (Organic degradation) $O_3 + H_2O_2 \rightarrow 2\dot{OH} + 3O_2$ (Hydroxyl radical) $\dot{OH} + RH \rightarrow \dot{R} + H_2O$ (organic degradation)	Mirza et al. (2020)
	Electro-Fenton process	COD: 2000–3000	80–90%	$Fe \rightarrow Fe^{2+} + 2e^-$ (Electrode) $H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + \dot{OH} + OH^-$ (Hydroxyl radical) $\dot{OH} + RH \rightarrow \dot{R} + H_2O$ (Organic degradation)	Ghosh et al. (2011)
	HC + Fenton	COD: 2000–3000	70–80%	$HC + H_2O \rightarrow \dot{H} + \dot{OH}$ (Hydroxyl radical) $H_2O_2 + Fe^{2+} \rightarrow Fe^{3+} + \dot{OH} + OH^-$ (Hydroxyl radical) $\dot{OH} + RH \rightarrow \dot{R} + H_2O + \dot{R}$ (Organic degradation)	Gujjar et al. (2021)
Electrochemical method	Electrocoagulation (iron electrode)	–	75–85%	$M \rightarrow M^{n+} + ne^-$ (Anode) $H_2O + e^- \rightarrow OH^- + H_2$ (Cathode)	Sahu (2019)
	Electrocoagulation (aluminum electrode)	COD: 280–340	20–70%	$M^{n+} + H_2O \rightarrow M(OH)_n + nH^+$ (overall) $OH^- + RH \rightarrow H_2O + R^+$ (Organic degradation)	Amri et al. (2021), Bener et al. (2019)
	Electrocoagulation + advanced oxidation	COD: 3000–8500	88–100%	$O_3 + H_2O \rightarrow 2\dot{OH} + O_2$ (Hydroxyl radical) $Fe^{2+} + O_3 \rightarrow Fe^{3+} + \dot{OH} + OH^-$ (excess radicle)	Aziz et al. (2016)
	Electrochemical oxidation (Titanium anode)	COD: 250	64%	$Cl^- \rightarrow Cl_2 + e^-$ (Impact of electrolyte, Anode) $H_2O + e^- \rightarrow OH^- + H_2$ (Cathode) $Cl_2 + 2OH^- \rightarrow H_2O + OCl^- + Cl^-$ (Overall) $OCl^- + RH \rightarrow CO_2 + H_2O + R^+$ (Organic degradation)	Ramesh et al. (2021)
Membrane	Polymeric—Ultrafiltration	Hemicellulose: 20,000–30000	55–65%	Commercial Alkali assisted ultrafiltration membrane with nominal MWCO 3 kDa shows the highest rejection	Singh and Murthy (2017)
	Polymeric—Nanofiltration	Hemicellulose: 17,000	65–90%	Polyethersulfone type commercial membrane with MWCO 200–1050 g/mol shows the highest retention	Schlesinger et al. (2006)
	Ceramic—Ultrafiltration	Hemicellulose: 20,000–40000	60–70%	Used ceramic membrane from TAMI Industries having MWCO 3–5 kDa for effluent study	Singh et al. (2018)

filtration as an alternative to the biological wastewater treatment process is the fouling of the membrane lowering the process throughput and reducing the membrane life (Tuluk et al. 2022).

Advanced methodologies to meet stringent norms

The fibre industry wastewater is conventionally treated upto the secondary treatment stage before its release into waterbodies. The typical composition of the effluent after secondary treatment is: COD: 100–250 ppm, BOD: 30–80 ppm, TDS: 2000–6000 ppm, TSS: <10 ppm, inorganics: 5–10 ppm, hardness: 800–1500 ppm, and the pH of water is around 7.2. However, since 2020, increasing number of industries are trying to achieve the standards set by the EU-BAT norms for wastewater disposal, and advanced treatment is required to match these stringent standards as discussed in the subsequent sections.

Reduction of COD

The biological treatment step leads to bottlenecks while treating large volume of effluent generated for increased fibre production due to the enhanced organic load. As a result, advanced COD reduction processes are becoming important (refer Table 4). COD reduction of fibre industry wastewater can be carried out using strong oxidising agents, like, hypochlorite, dissolved chlorine, etc. They are able to break the COD chain and reduce COD by 10–20% (Wei et al. 2017). Similarly, advanced oxidation processes (AOP) are also viable solutions (Chaturvedi et al. 2021; Oturan and Aaron 2014) as Fenton's reagent, hydrogen peroxide, and ozone are used to react with the organic pollutants. In lyocell process, NMMO in the effluent can be oxidised by ozone, and the oxidation products are biodegradable (Stockinger et al. 1996). Fenton's reagent is a combination of ferrous sulphate and hydrogen peroxide, where the Fe^{2+} ion generates strong oxidizing radicals that can attack the organic compounds (Malik et al. 2020). The drawback of the Fenton process is the increase in the iron (Fe) load which requires a separate unit for treatment. While reagents, like, hypochlorite and chlorine can oxidize the organic compounds. However,

handling and storage of such hazardous materials are challenging. Even the process is expensive as it would cost around 0.6–1 USD/m³ of water to remove around 97% COD (Gujar et al. 2021). Apart from the oxidizing chemicals, hybrid treatments also work well in removing the COD load in fibre generated wastewater. For example, a stepwise combination of the electro-Fenton process, involving the electrochemical dissolution of iron (Fe), and chemical precipitation can be adopted to treat the rayon industry wastewater for lowering COD and zinc (Zn) concentration (Ghosh et al. 2011). Even biological oxidation in a sequential batch reactor (SBR) by combining Fenton's reaction with biological process results 88%–98% removal of COD (Rodrigues et al. 2014).

Hydrodynamic cavitation (HC) is another promising technology that works on the principle of the passage of liquid through a constriction, like, orifice plate, venturi, or throttling valve (Carpenter et al. 2017). This causes the formation of cavities, that consequently implode once the pressure is recovered downstream of the constriction (Panda et al. 2020). As a result, highly reactive radicals, such as OH , $\dot{\text{H}}$, HO_2 etc., are generated that can degrade organic compounds (Tao et al. 2016). This process has several advantages, such as chemical free operation, large potential to degrade a wide range of organic molecules, cost-effectiveness, and less energy intensive. It is demonstrated that cavitation coupled with the Fenton oxidation resulted in maximum COD reduction of 86% and a TDS reduction of 47% even at fairly low cost ~0.2 USD/m³ of water (Gujar et al. 2021). Similarly, Badmus et al. (2020) treated textile wastewater using combination of Fenton process with HC and achieved 74% reduction in total organic carbon content. Several companies, such as, Vivira Process Technologies are working on the commercial applications of HC for wastewater treatment (Ranade et al. 2021).

Among all the new and innovative technologies, electrochemical treatments, like, electrocoagulation and electrooxidation are applied globally for remediating effluent impurities (Garcia-Rodriguez et al. 2020). Electrochemical treatments are advantageous due to compact design and energy efficiency. The selection of the electrode is crucial to decide the COD rejection. For example, iron electrodes in electrocoagulation process can increase COD rejection up to 84% (Sahu 2019). Further

rejection can be achieved by using hybrid system, like, combination of electrocoagulation and AOP (GilPavas et al. 2019). The combined effect of electrocoagulation and AOP enhances COD and colour rejection, whereas lower energy consumption is observed (5.8 kWh/m^3) using an ozone-electrocoagulation process (Aziz et al. 2016). Further reduction of energy consumption ($\sim 3.0 \text{ kWh/m}^3$) with better COD rejection is possible using electrochemical oxidation (Rakhmania et al. 2022). However, the major challenge in electrochemical treatments is their scalability.

Alternatively, membrane filtration is an emerging technique to reduce COD in cellulosic wastewater due to higher selectivity. Membranes work on the principle of the selective permeation based on their pore size and they can separate a wide range of pollutants. Several studies have reported the potential of membranes in reducing COD from wastewater. Persson and Jönsson (2010) reported high hemicellulose retention (above 90%) from pulp mill process water using ultrafiltration polysulphone membrane. Further reduction is possible using nanofiltration or reverse osmosis membrane but at the cost of lower flux. Likewise, Astegger et al. (1992) subjected the lyocell wastewater containing NMMO to reverse osmosis for the separation of water from dilute aqueous solution of NMMO, N-methylmorpholine or morpholine or mixtures that contributed to COD. For COD removal, ceramic membranes made from inorganic materials, like, alumina, zirconia, etc., are also gaining attention due to their advantage of high chemical and thermal stability (Amin et al. 2016). Singh et al. (2018) studied the performance of ultrafiltration ceramic membranes and found that they were able to separate hemicellulose effectively from a highly alkaline stream of viscose process containing 17–18% sodium hydroxide. Krawczyk et al. (2011) studied the performance of a tubular ceramic membrane for the removal of hemicellulose from a viscous solution originating from wheat bran, and they reported high flux ($62 \text{ L/m}^2\text{h}$) and high hemicellulose retention of 96%. Several companies, such as Tami industries, Pall Corporation, etc., have developed commercial ceramic membranes for industrial applications (Barredo-Damas et al. 2012). However, despite their advantages, membranes suffer from fouling, high energy consumption, high capital cost, and the generation of a secondary concentrated wastewater stream

(Horovitz et al. 2020) which can be a part of future research.

Recovery of heavy metal

Majority of the heavy metals is removed during the secondary treatment process. However, small concentration of heavy metal remains in the treated effluent. Advanced treatment processes remediate remaining heavy metal impurities and give an assurance to meet the stringent norms and recover the metals (refer Table 5). Ion-exchange resins have emerged as a viable option for heavy metal recovery (Batra et al. 2022). Ion exchange is a treatment method involving the passage of wastewater through resin beds. The cationic resins replace the cations in water with hydrogen ions and the anions with hydroxyl ions. The resin bed can then be regenerated for further use. The advantage of this method is that no sludge is produced, and the metal can be recovered selectively using a preferable resin bed. Bench scale studies have shown that the ion-exchange process using a chelating ion-exchange resin is technically viable to recover zinc and calcium from viscose rayon effluent (Jha et al. 2008). Several studies have shown that the resins from companies, like, Lanxess and Puro-lite can be used effectively in heavy metal removal from industrial wastewater (Abdelwahab et al. 2013). This method is effective for zinc concentration of 50–100 ppm. However, the associated problems are: (i) the contamination of the resin and (ii) the regeneration of resin is chemically intensive.

Adsorption using nano-adsorbents and nanoparticles is another promising way of removing heavy metals (zinc, copper, etc.). Generally, carbonaceous nanomaterials are effective because of higher adsorption capacity (Chai et al. 2021). Even, chemically modified lignocellulose (using alkali, peroxide, etc.) has also been proved as an effective nano-adsorbent for the removal of heavy metals from aqueous streams due to higher adsorption and desorption capacity (Gao et al. 2018; Zhang et al. 2020). Among all, metal nanoparticles synthesized from metal oxides are promising in this context as source is abundant, low-cost, and high specific surface area of the adsorbent (Kaushal and Singh 2017). However, maintaining rejection after few regeneration cycles is a challenge which can be resolved with detailed regeneration strategy (Kim et al. 2013). Nevertheless,

Table 5 Advanced technologies for heavy metal removal and recovery

Technologies	Input parameters	Efficiency	Optimize condition	References
Ion-Exchange Resins	50–100 ppm	99.4% recovery of Zn	Extraction of Zn enhances at lower equilibrium pH and increases with resin dose (25 g/L water)	Jha et al. (2008)
Thiol-lignocellulose sodium bentonite (TLNB) nano-adsorbent	20–1500 ppm	> 90% remove heavy metals	Maximum adsorption of Zn from effluent observed at pH 4.5 at 43°C with 100 min contact time. The ability of metal binding in order Cd < Zn < Hg	Zhang et al. (2020)
Fe ₃ O ₄ / MnO ₂ nanocomposite	10 ppm	95% recovery of heavy metal (Zn, Cu, etc.)	90% sorption of Cu and 80% in Zn observed at 6.3 pH with 30 min contact time	Kim et al. (2013)
Electrocoagulation	50–250 ppm	> 95% remove heavy metals (Zn, Ni, Cu)	$Al_{(s)} \rightarrow Al^{3+} + 3e^{-}$ (Anode) $H_2O + 2e^{-} \rightarrow 2OH^{-} + H_2$ (Cathode) $M^{+} + OH^{-} \rightarrow MOH \downarrow$ (Coprecipitation) Current density 3.3–98 A/m ² for 30 min at pH10	Heidmann and Calmano (2008)
Emulsion Liquid Membranes	0.3–200 ppm	90–99% Zn recovery	$M^{2+} + 2RH(\text{extracts}) \leftrightarrow MR_2 + 2H^{+}$ 97% extraction of Zn and Cu is possible using D2EHPA and LIX860 respectively in 10 min	Tandlich (2010)
Nanofiltration Membranes	< 10 ppm	> 90% rejection of heavy metal	Used commercial polyamide film NF membrane. Maximum rejection obtained at 3 pH, 10 bar pressure with flux rate of 54.5 L/m ² h	Kočanová et al. (2017)

the major limitations of the nanosized adsorbents are: (i) they cannot be used in a continuous column due to high pressure drop and (ii) the nanoparticles leach out with the treated stream and require nanofiltration for the recovery (Baskar et al. 2022). Thus, the nano-adsorbents need to be immobilized in an appropriate media but that reduces the adsorption capacity due to the loss of surface area.

Another advanced technology is the electrochemical method for heavy metal removal and recovery (Jain et al. 2022). Heidmann and Calmano (2008) studied the impact of aluminium electrode in electrocoagulation with the addition of NaNO₃ salt in solution and obtained significant reduction of heavy metals (zinc, nickel, and cooper) in the form of metal hydroxide precipitate. Additionally, Ferreira et al. (2013) identified that the presence of an aluminium electrode can remove the heavy metals using lower energy (0.6 kWh/ m³) without adding any salt. Moreover, it also reduces the hardness of the stream due to the precipitation of calcium carbonate in the reactor. Metal recovery using this technology can be effective if the concentration of the competitive ions is less,

even if the presence of the organic impurities influences the electric field (Al-Qodah and Al-Shannag 2017).

Several studies have also reported the successful removal of zinc using emulsion liquid membranes (ELM). Liquid membrane-based separation processes are usually based on the liquid–liquid extraction. Emulsion liquid membranes are double emulsions stabilized by the surfactants, although poor recovery of solvent increases the operating cost (Kumar et al. 2019). The use of ELM for zinc removal and recovery from viscose effluent was reported at a fibre plant in Lenzing, where the process can treat up to 75 m³/h of zinc bearing wastewater with the zinc concentration ranging from 0.3 to 200 mg/L with 99.5% extraction efficiency (Raghuraman et al. 1994; Tandlich 2010).

There are many state-of-the-art technologies that are developed by research groups and companies for heavy metal removal and recovery from industrial wastewater. Like effluent with low zinc loads (around 10 ppm) can be treated using low molecular weight cut-off nanofiltration membranes with higher rejection efficiency ~ 90% (Xiang et al.

2022). On the other side, Rajivgandhi et al. (2022) found a novel bioactive catalyst assisted hybrid approach for the removal of heavy metals (50–85%) from textile effluents. Long-run trial using these state-of-art technologies is required before going for commercialization.

Reduction of TDS

A major component (~90%) of the total dissolved solids in MMCF wastewater is divalent salts, mainly, sodium sulphate. Sulphate is non-toxic, but it has a high scaling potential. After secondary treatment, the concentration of sulphate (SO_4^{2-}) in the stream is more than 1000 ppm. In order to match the stringent discharge norms and for water recycling, further treatment is required for sulphate remediation (refer Table 6). The most conventional method for sulphate reduction is precipitation in the form of ettringite ($\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot 26\text{H}_2\text{O}$) and sodium-jarosite ($\text{Na}[\text{Fe}_3(\text{SO}_4)_2(\text{OH})_6]$). In ettringite precipitation, lime is used to attain a pH up to 12 and aluminium salt is added to promote precipitation (Runtti et al. 2018). However, the challenges are: (i) higher solubility of magnesium sulphate in water that hinders precipitation, and (ii) the process is chemical intensive as the treated stream needs neutralization before disposal (Dou et al. 2017). Sodium-jarosite precipitation requires a low pH (2–3), high pressure, and high temperature with 1–3 h contact time, where sodium is replaced by potassium or ammonium ions. The increased pH also hinders the sulphate precipitation (Casas et al. 2007).

Apart from chemical-based separation, adsorption is another attractive option for the separation of sulphates from the effluent stream. Silva et al. (2012) used limestone for the adsorption of sulphate from mine wastewater and the water was used for regeneration, although the required contact time for adsorption was higher due to less (23.7 mmol/g) adsorption capacity. Alternatively, ion-exchange is another option for removing impurity ions using selective ion exchange media. Guimarães and Leão (2014) identified a weak base polystyrene resin (Amberlyst A21) for the selective reduction of sulphate from varying concentration of sodium sulphate effluent and reported a maximum adoption capacity of 11.6 mg/mL of resin for both batch and fixed-bed column.

After resin column, saturated caustic should be used for column regeneration.

The removal of microbial sulphate from industrial effluent using SRB is studied since long (Hansen 1994). In an anaerobic condition, the microbe uses sulphate to oxidize organic species or hydrogen, and simultaneously forms sulphide which can precipitate along with metals or converts in the form of gaseous hydrogen sulphide (Zhang et al. 2022). Typically, up-flow anaerobic bioreactors are preferred for sulphate reduction at pH 2–8. Furthermore, acidophilic and acid-tolerant microbes are used to survive in lower pH (Santos and Johnson 2017). In order to optimize the hydrogen sulphide formation, sulfidogenic bioreactors are also constructed (Kousi et al. 2015). Overall, the process requires longer hydraulic retention time, where a high sulphide concentration (~3500 mg/L) takes longer for sulphide formation than a lower concentration (1000 mg/L) (Bernardez et al. 2012). A novel method was invented by Lee et al. (2014) by using SRB and sulphide-oxidize biofilms as a microbial fuel cell anode to convert sulphide to elemental sulphur.

The most viable method to reduce dissolved salt concentration is the use of low molecular weight cut-off (MWCO) polymeric membranes such as nanofiltration (NF) and reverse osmosis (RO) (De et al. 2012; Mondal and De 2016). Membrane based processes are environmentally friendly as they do not require any external chemicals. NF and RO both are pressure driven membrane separation techniques and applicable to concentrate low molecular weight organic materials and salts while allowing water and solvents to the permeate. In case of RO, high pressure of about 35–100 bar is required to overcome the solution osmotic pressure across the membrane. Although NF membrane is separating dissolved components having a molecular weight cut-off of about 200–800 Da and molecular size of about 1 nm (Ahmad et al. 2022). It can also be used to separate inorganic salts with a much smaller size than the membrane pore size due to the electrostatic repulsion. The membrane retentate stream can be evaporated to recover the salts. To ensure the quality of recovered salts, prior treatment of the effluent is required to remove other impurities.

Membranes can be fabricated using different methods, such as phase inversion, grafting, interfacial polymerization, etc. Criteria of membrane selection are based on the salt rejection, pure water

Table 6 Available advanced treatments for sulphate reduction

Treatment	Methods	Sulphate load (ppm)	Rejection	Mechanism	Reference
Chemical treatment	Precipitate ettringite using hydrated lime and alumimum salt at pH 10.7–12	3000–6000 ppm SO_4^{2-}	80–90%	$6Ca(OH)_2 + 3H_2SO_4 + 2Al(OH)_3(s) + 20H_2O \rightarrow Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O \downarrow$	Germishuizen et al. (2018)
	Sulphate reduction by precipitating natron-jarosite at pH 2–4	~3000 ppm Na_2SO_4	–	$3Fe(SO_4)_3(aq) + Na_2SO_4(aq) + 12H_2O \rightarrow 2NaFe_3(SO_4)_2(OH)_6 \downarrow + 6H_2SO_4(aq)$	Casas et al. (2007)
Adsorption	Adsorption of sulphates using limestone bed	588–1100 ppm SO_4^{2-}	85%	Required calcite limestone of 25 g/L for 210 min	Silva et al. (2012)
	Selective separation of sulphate from weak base anionic resin	200–1800 ppm Na_2SO_4	96%	Preferable acidic condition with contact time ~ 150 min	Guimarães and Leão (2014)
Biological	Continuous 1.15 L up-flow bioreactor with chitinous substrate (pH 5–7)	1986 ppm SO_4^{2-}	60–80%	$8H^+ + 8e^- + SO_4^{2-} \rightarrow S^{2-} + 4H_2O$ (Sulphate reduction)	Aoyagi et al. (2017)
	Sulphidogenic bioreactor (2.3 L) with acid tolerant SRB (pH < 4)	2000–6700 ppm SO_4^{2-}	~98%	$8H_2 + H^+ + SO_4^{2-} \rightarrow HS^- + 4H_2O$ (Sulphide formation)	Ñancuqueo et al. (2016)
	Combination of SRB and sulphide-oxidizing bacteria biofilms on a microbial fuel cell anode (pH 7.5)	1150 ppm Na_2SO_4	84%	$CH_3COO^- + SO_4^{2-} \rightarrow HCO_3^- + HS^-$	Lee et al. (2014)

Table 6 (continued)

Treatment	Methods	Sulphate load (ppm)	Rejection	Mechanism	Reference
Membrane	Commercial cellulose acetate based NF membrane (NFCK)	790 ppm SO_4^{2-}	90%	Permeate flux ~4.5 L/m ² h at 3 bars in acid mine drainage (AMD)	Wadekar et al. (2017)
	Thin Film Composite (TFC) co-polyamide NF membrane modified with 3,5-diaminobenzoic acid	1000 ppm Na_2SO_4	96%	Operate at 4.5 bars and pure water flux ~50 L/m ² h in synthetic solution	Ahmad et al. (2004)
	Commercial NF270 (Dow Filmtec™), Flat sheet polyamide TFC membrane	1960–2768 ppm SO_4^{2-}	87%	UF membrane as a prefilter. NF270 operate at 10 bars and a flux rate of 40–100 L/m ² h in AMD	Aguiar et al. (2018)
	Poly(piperazine-amide)/silica thin film nanocomposite nanofiltration (TFNN) membrane	9600 ppm Na_2SO_4	84–92%		Lopez et al. (2018)
	Poly(piperazine-amide)/silica thin film nanocomposite nanofiltration (TFNN) membrane with aminated titanium dioxide	2000 ppm MgSO_4	94%	Silica nanosphere 0.05% (w/v) in organic phase. Obtained flux ~22 L/m ² h at 5 bars pressure using a synthetic solution	Li et al. (2015)
	Polyamide thin film nanocomposite (TFN) nanofiltration membrane with aminated titanium dioxide	1000 ppm Na_2SO_4	98%	0.03% TiO_2 - NH_2 in the aqueous phase. Obtain flux 50 L/m ² h at 5 bar in synthetic solution	Wei et al. (2020)
	Graphene oxide incorporated polyamide TFN nanofiltration membrane	1000 ppm Na_2SO_4	95%	0.3% GO with a base layer. Obtain flux ~18 L/m ² h at 8 bars in synthetic solution	Lai et al. (2016)
	Polysulfone (PSf) with dimethylacetamide (DMAc) membrane by Solvent Evaporation	1000 ppm Na_2SO_4	87%	10% PSf in DMAc 80μ casting thickness at 4 bars showed best in synthetic solution	Yuan et al. (2016)
	Cellulose acetate (CA) based reverse osmosis membrane	1% (wt/vol)	95%	Required pre-treatment before RO. Permeate flux 0.2–1 L/m ² h at pressure 12 bar in rayon effluent	Devmurari and Chandorikar (1983)

permeability, permeate flux, and fouling (Kheir-ieh et al. 2018). Polyamide composites or cellulose acetate are effective membrane materials to retain sodium sulphate or sulphate salts. Ahmad et al. (2004) modified the conventional polyamide thin film composite (TFC) membrane with a carboxyl group and found higher permeate flux as well as sodium sulphate rejection (96% for the feed concentration of 1000 ppm). TFC nanofiltration membranes have also been developed incorporating nanoparticles, like, silica, TiO₂, graphene oxide, etc., (Karimipour et al. 2021) to increase the surface roughness and permeability. Devmurari and Chandorikar (1983) designed a reverse osmosis plant to recover sodium sulphate from viscose rayon spent liquor and achieved a concentration hike of 15%. Additionally, several companies, such as FilmTech, Nitto Denko, Koch etc., are manufacturing commercial membranes with high (Lopez et al. 2018; Nyström et al. 1995) rejection of ~90% for feed sodium sulphate concentration < 10,000 ppm and that can be suitable for treating cellulosic fibre wastewater. However, further research is required to overcome the challenges, like, membrane fouling and the permeate flux decline, prohibitive cost of the membrane, limited chemical resistance, and scale-up issues.

Future scope in sustainable technologies for MMCF industries

The cellulosic fibre production industry is one of the largest industries with the potential of generating significant amount of wastewater (20–80 m³/ton of fibre production) and this is a big challenge for appropriate treatment before their disposal (Hugill et al. 2020). Focusing on sustainability and the increasingly strict disposal standards (like ZDHC), two approaches the MMCF industries should adopt: (i) reduction of impurities and recovery of useful components; (ii) recycling of the treated water.

Impurity reduction with value recovery

Most of the treatment technologies discussed in the previous sections have their own technical limitations and no technology is universally applicable to all kinds of wastewater streams. Therefore, the most suitable and economical solution needs to be selected

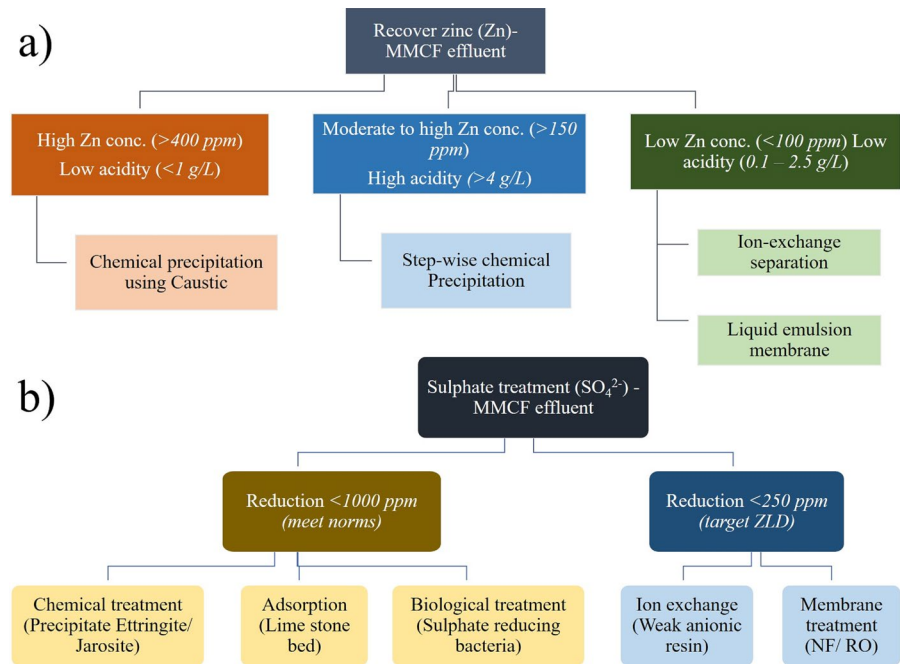
based on the effluent composition as well as the plant economics (Maryam and Büyükgüngör 2019).

It will be better to treat the high COD stream upfront, although the challenge is in handling the harsh (acidic/ alkaline) process waste. In such scenario, ceramic membranes are emerging as a viable solution for COD removal and they can specifically be used for highly acidic waste stream due to their remarkable mechanical and chemical stability (Caltran et al. 2020; Xu et al. 2013). However, there is still further research required to upscale and improve the membrane life to handle huge quantities of effluent. For further reduction, a combination of HC with Fenton treatment can degrade the COD from 250 to 50 ppm (86% reduction) and can be economical for post-secondary treatment, while the capital cost with respect to the chemical storage and footprint requirement might be high.

Before the removal of heavy metals from the effluent, the metal containing stream (lean or rich) should be identified for economic circularity. In the case of viscose and modal production, the zinc consumption is around 2–10 kg/ton of fibres (Kishor et al. 2021). Some feasible options would be chemical treatment (Chen et al. 2018), ion-exchange separation (Jha et al. 2008) or the use of emulsion liquid membranes. The criteria for selecting the most techno-economically feasible solution is based on the concentration of zinc and acid in the effluent (refer Fig. 5a). In the case of low acid and the zinc concentrations, resin column and emulsion liquid membranes can be economical as tertiary treatment. Zinc recovery using caustic treatment can be economical where the acid concentration is less, and the zinc concentration is high. On the other hand, the stepwise chemical precipitation technique is advantageous in the case of high acid concentration with moderate to high zinc concentration (American Enka Company 1971).

The presence of TDS in the form of sodium sulphate is the most common problem for MMCF (except lyocell process) effluent. Based on the industrial need, an appropriate sulphate reduction technology needs to be selected (refer Fig. 5b). To meet stringent norms, ettringite precipitation using chemicals is an effective method to reduce sulphate from 3000 ppm. A treatment technology with a focus on water recycle or zero liquid discharge (ZLD) and further sulphate reduction can be achieved using membranes. The most conventional method to remove the

Fig. 5 Selection of techno-economic feasible technologies for **a** zinc recovery based on the acid-zinc concentration in effluent, and **b** sulphate reduction from effluent stream



sulphate salt is the use of reverse osmosis. Polyamide based nanofiltration can also be used to remove 90% of the divalent salts (like, Na₂SO₄) (Wei et al. 2020). It may be noted that NF is more cost effective in terms of both capital and operating costs than RO (Fornarelli et al. 2013). However, further research is required to increase the life of the membranes by reducing the fouling while maintaining high flux and high salt rejection (Lin et al. 2015).

Mostly in MMCF, the acidic wastewater is difficult to treat due to the high COD, TDS and inorganic impurities. The removal of COD and the recovery of chemicals (salts, acid) can be cost-effective and sustainable, while direct recycling of the process stream into the evaporators can lead to the build-up of impurities affecting the product quality and equipment life. Further, the recovery of chemicals from dilute streams through evaporation is not an economical option (Gawaad et al. 2011). It could be better if that stream is treated by sequential combination of chemical precipitation methods and ceramic membranes to recover metal hydroxide (Mondal et al. 2018; Samaei et al. 2018) and remove COD. However, further research is required to enhance the metal purity. Another alternative could be the use of anaerobic bioreactors for the simultaneous reduction of COD and TDS along with the metal recovery by adjusting the

pH beforehand (Ñancucheo et al. 2016). The use of a sulphidogenic bioreactor in the presence of acidophilic SRB and non-acid substrates (like glycerol) gives an added advantage in the presence of zinc, as it helps to buffer the pH along with the recovery of zinc in the form of zinc sulphide (Ñancucheo and Johnson 2011). Further, Parravicini et al. (2007) reported 50% reduction in the sulphate in viscose wastewater using anaerobic treatment with SRB. The challenges in anaerobic treatment would be handling high TDS, metabolic interaction between sulphates and other organics, etc. (Aryal 2021). In addition to nanofiltration, other options to recover and reuse the acidic process waste are ion exchange (Xu 2005), membrane distillation (Shirazi and Dumée 2022) and electro-dialysis (Gurreri et al. 2020). However, the most of these technologies are still in the preliminary stage of research.

Water recycle

ZLD systems are increasingly gaining importance as a strategy to reduce the liquid waste and maximize the water recycle. Additionally, salt recovery is also possible by using ZLD systems (Date et al. 2022). The Nagda plant of Grasim Industries became the world's first viscose unit to achieve ZLD in 2021 and

set a global benchmark in low water consumption (Birla Cellulose 2021). Generally, ZLD schemes are not cost-effective because of handling the large volume of wastewater and maintenance cost (Panagopoulos and Haralambous 2020). The last stage of the ZLD is usually reverse osmosis (refer Fig. 4) that has limitations in terms of concentration of TDS in feed, low efficiency, and membrane fouling (Elimelech and Phillip 2011). Instead, a combination of nanofiltration and reverse osmosis, or the use of forward osmosis (Shaffer et al. 2015) can be more effective. Alternatively, technologies, like, electrodialysis (McGovern et al. 2014) and membrane distillation (Subramani and Jacangelo 2014) can also be introduced to handle the concentrated salt streams. In the typical ZLD process, chemical based treatments and resins are generally used to remove inorganic impurities before passing through the membrane (Hube et al. 2020). The presence of organics even after secondary treatment is also an issue for the membrane fouling affecting the salt quality. This demands a pre-treatment for organic removal. Ramesh et al. (2021) suggested the electrochemical oxidation (EO) to remove 64% COD in the effluent before sending it for membrane separation that can be effective to reduce the membrane fouling. Additional improvement in the efficiency of the ZLD process is possible by optimization of chemical usage, reducing manual interventions, and strengthening the regulatory systems (Sueviriyapan et al. 2016).

The selection of the most suitable process depends on techno-economic feasibility. Long term successful running of a pilot unit can provide technical insights and data required to scale up these systems to the commercial level.

Conclusions

In the area of cellulosic fibres, the demand of the MMCF has increased with the rapid growth of the population. In the MMCF process, the generation of substantial amount of wastewater (20–80 m³/ton production) with high concentration of impurities, like, COD, TDS, and heavy metals is alarming due to environmental regulations and water scarcity. To ensure responsible and sustainable disposal of effluent streams, stringent measures such as EU-BAT and ZDHC are introduced.

Conventional treatment process with primary and secondary treatment for reduction of suspended, organic, and heavy metal impurities have limitations in complying with stringent regulations. Instead, either the amalgamation of new technologies with existing process or adopting sustainable treatment technologies are required to be evaluated and deployed focusing on reduction, recovery, and recycle. For example, MBR in combination with the conventional bioreactor can enhance the COD reduction from 80 to 99%; the use of hybrid oxidation system, like, HC + Fenton, ozone-electrocoagulation process after secondary treatment can meet the requirement. For heavy metal (zinc) recovery using ion-exchange resin or electrocoagulation processes can be effective among all available techniques. Also, chemical based treatment (ettringite) and biological treatment (anaerobic bioreactor with SRB) are promising for sulphate removal along with the membrane technology. For MMCF industries, NF can be a suitable alternative to RO as the most of the dissolved salt is divalent. The selection of suitable technology should be decided based on the effluent characteristics and economic feasibility. Keeping in mind the stringent disposal standards, a sustainable business approach needs to be developed focusing on impurities reduction with value recovery and water recycle. Incorporating digitalization can help in optimizing chemical usage. The final screening of the technology needs to be done based on rigorous pilot scale trials.

Acknowledgments This work is supported by Aditya Birla Science and Technology Company Private Limited (ABSTCPL) and Membrane Separation Laboratory, IIT Kharagpur. The authors are thankful to ABSTCPL leadership and all team members from the Fibre Textile Chemical department. Also thankful to all members of Membrane Separation laboratories for their valuable inputs and Knowledge Centre of ABSTCPL for their constant support in literature finding during this work. The author thanks Mrs. Sudha Kannan and Dr. Neeleshbharti Shukla for proofreading.

Author contributions SM: conceptualization, methodology, visualization, writing- original draft, summarizing, AAR: writing- original draft, data curation, methodology, investigation, SD: supervision, conceptualization, writing- review and editing, summarizing, PK: resources provision, Conceptualization, writing- review and editing.

Funding No funding was received for conducting this study.

Declarations

Competing interests No, the authors have no competing interests.

Ethics approval and consent to participate The authors followed all ethical responsibilities.

Consent to publication This manuscript is not submitted elsewhere except this journal.

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