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Recent progress and challenges of adsorptive membranes for the removal of pollutants from wastewater. Part II: Environmental applications

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ABSTRACT

Adsorptive membranes have attracted attention recently and have been employed to remove variety of pollutants from wastewater. Part I of this work was devoted to provide an overview on the latest progress in their fabrication techniques. This part is devoted to review the studies performed towards environmental applications. Adsorptive membranes were used to remove pollutants such as dyes, heavy metals, and pharmaceuticals. The major findings of this review include presenting the various benefits associated with the use of adsorptive membranes in micropollutants removal from water samples and discussing the potential utilization of bio-adsorbents such as chitosan. While adsorptive membranes proved their effectiveness in removing several pollutants, they still however, suffer from various drawbacks and challenges on a large scale implementation. These drawbacks include the low adsorption capacity, the cost, reusability and fouling. Finally, the paper concludes that exploiting adsorptive membranes in the removal of emerging pharmaceutical compounds in particular have not yet been researched extensively in the literature and more efforts should be focused in this direction.

1. Introduction

Purifying water and removing pollutants to produce drinkable water is a major research topic in the scientific community. The emergence of hazardous materials in wastewater is a dangerous phenomenon that impacts the water's quality and safety. These materials include heavy metals, pharmaceutical materials, and dye materials. The emergence of such pollutants has risen over the years due to the growth in population globally, industrial activities, urbanization, and climate change which all contributed in water scarcity all around the world [1,2]. Mainly due to population growth, the amount of available water was reduced to 1250 cubic meters in 1995, and the trend is expected to decrease to 650 cubic meters worldwide by 2025. Therefore, this water shortage crisis drives wastewater treatment progressively in the near future [3]. The presence of contaminants in water continues to impose a threat on human lives and the environment. For example, heavy metals like cadmium (Cd^{2+}) and lead (Pb^{2+}) can accumulate in humans and animals' tissues causing serious health problems like kidney damage, cancer, and nervous system complications [4]. Moreover, pharmaceutical waste materials affect the quality of drinking water resources, e.g. spreading antibiotic resistance, and toxicity to aquatic organisms [5]. Therefore, many studies were devoted to finding efficient and cost-effective water treatment technologies for the removal of these contaminants from wastewater combined with many treatment technologies as reverse osmosis, ion exchange, and adsorption. However, these methods suffer from limitations and drawbacks which shifted the focus towards utilizing adsorptive membranes technology for this purpose.

Adsorptive membranes possess the dual function of both adsorption and membrane separation and encompass the advantages of the previous technologies. In addition, their diverse classifications, configurations, and filler types favors adsorptive membranes technology as they can be utilized to remove numerous contaminants from wastewater. Moreover, the large surface area and redundancy of adsorption sites are important factors for the efficiency of adsorption and removal of pollutants from wastewater [6]. Adsorptive membranes are mainly applied to remove the soluble micropollutants that cannot be easily removed via known and commonly used water treatment methods and escape the treatment to reach the environment. Over the years, this technique has proven to enhance many treatment aspects such as removal rates, rejection, selectivity, permeability, water flux, and efficiency compared to other treatment techniques.

Adsorptive membranes classifications and synthesis techniques were discussed in part I of this work [1]. This part is devoted to discuss the environmental applications of adsorptive membranes in removing pollutants such as heavy metals, dyes, and pharmaceuticals. It also reviews

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List of abbreviations

MF	Micro-filtration
UF	Ultra-filtration
PAN	Polyacrylonitrile,
PES	Polyethersulfone
AMs	Adsorptive Membranes
PVDF	polyvinylidene flouride
MWCNTs	Multi-walled carbon nanotubes
FMWCNTs/CS	Fibrous Multi-walled Carbon nanotubes/chitosan
PVT	Polyvinyl tetrazole
PVA	Poly (vinyl alcohol)
PPCP	Pharmaceuticals and personal care products
SEM	Scanning electron microscopy
DFUF	Dual-functional ultrafiltration
EDTA	Ethylenediaminetetraacetic acid

the application of bio-adsorbents like chitosan, and presents some recommendations for future research and directions. This paper is a comprehensive discussion on environmental perspectives that focus on the utilization of bio-adsorbents like chitosan for the removal of different types of pollutants as well as their challenges. Additionally, this paper provides an overview for the alternative and sustainable bio adsorbents/membrane materials that can be used. All the preceding points, as far as the authors are aware, were not reported previously in the literature.

2. Environmental applications of adsorptive membranes

2.1. Removal of dye materials

Dyes are chemical compounds that can get attached to surfaces or fabrics to impart colour. They are mostly complex organic molecules that resist detergents. Many types of synthetic dyes and pigments have extensive applications in several industries like textile, leather, plastic, paint, paper, printing, food processing, and cosmetic industries [7]. The most common synthetic dyes are triarylmethane, azo, and carotenoid. There are more than 100,000 distinct types of dyes produced with a rate of 9×10^6 tons/year which highlight the impact of the effluents discharged in water streams by this industry [8]. Dyes have two general categories:

1) Anionic dyes, which are used for silk, nylon, and wool. Examples include anthraquinone, azine, triphenylmethane, and xanthene.
2) Cationic dyes, which can be used for paper, modified nylons, polyesters, polyacrylonitrile, and in medicines. Examples include crystal violet and methylene blue [9].

However, dyes can be classified to other categories based on different factors such as: their chemical composition or their applications [8]. They come in different chemical structures mainly based on substituted aromatic and heterocyclic groups, and many dyes are azo compounds linked by an azo bridge [10].

Pollutants like dyes are continuously discharged to the environment like methyl orange (MO) for example, as annually more than 10% of the produced 700 kt dyes are effluents coming from different industries [11]. Therefore, the wastewater containing dyes needs to be treated and purified for safety and environmental purposes [12]. As most of these dye wastes are toxic, carcinogenic, and poses a serious hazardous threat to aquatic living organisms and to humans. In addition, the exposure to these dyes can pose acute and chronic effects on the organisms; some dyes consume the dissolved oxygen in water which threats the aquatic life. The strength of these effects depends on the time of exposure and the concentration [9]. Moreover, even low levels of dyes in water, less than 1 ppm, is dangerous, undesirable, and very challenging to remove [13]. Table 1 presents the main dyes found in wastewater with their concentration ranges being discharged in aqueous effluents from dyes manufacturing and textile industries.

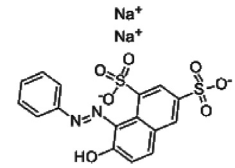
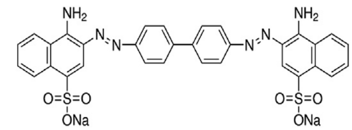
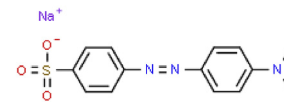
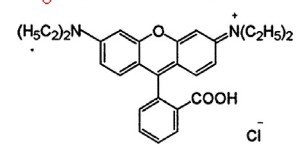
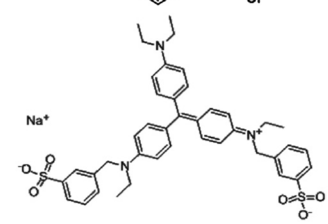
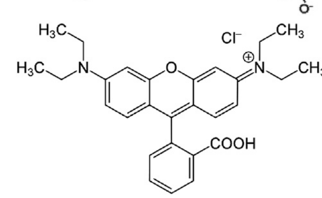
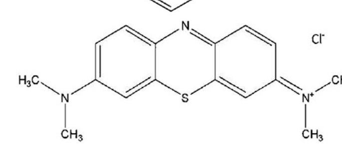
Governments have set regulations and environmental restrictions to control the quality of water regarding the presence of dye materials. The treatment of wastewater containing dye effluents has been a major concern for many industries. The conventional treatment methods typically in practice are physico-chemical processes that employ adsorption, oxidation, and chemical precipitation [15]. Coagulation, biological oxidation, and membrane filtration are also used but each method has its limitations and drawbacks in application [12]. The most common method of dyes removal from water is adsorption [9] due to its relatively low energy requirement, efficiency and insensitivity to toxic pollutants. Natural materials have been used for this purpose, such as pine wood dust and clay minerals [9]. One of the most commonly used adsorbents for dyes removal is activated carbon due to its high adsorption capacity, yet it is expensive, and regeneration is an issue. Therefore, several studies shifted towards the investigation of biosorbent materials [10]. However, the drawbacks due to adsorption can be avoided by the use of adsorptive membranes.

The use of adsorptive membranes for dyes removal from wastewater is an attractive practice. The attention is directed on using biosorbent materials due to their low cost and being environmentally friendly. chitosan-based membranes are being extensively investigated, since it is more chemically flexible than cellulose and chitin because of the presence of free amino groups. And it is widely used due to its hydrophilicity, biodegradability, and high affinity to dyes and some metal ions. chitosan was previously used to adsorb acid dyes [16]. The chemical structure of chitosan is presented in Fig. 1.

Cellulose is also among the most available biomaterials on earth which makes it environmentally friendly with a low cost. Cellulose was studied as nano reinforcements in polymer matrix to produce an effective membrane. The adsorptive membrane developed in a study contained ultrafine cellulose nanocrystals impregnated in an electrospun polyacrylonitrile nanofibrous scaffold endorsed by polyethylene terephthalate [17]. As these cellulose nanocrystals possess a negatively charged surface density attributed to the negative functional groups SO^{3-} and/or COO^{-} that lead to high adsorption capacity to remove the positively charged compounds like crystal violet dyes for example [17]. The main attribute of the adsorption depends on surface interactions, thus the functional groups on the adsorbent surface play significant part in adsorption phenomenon. Overall, functional groups determine the effectiveness, selectivity, and reusability of the membrane produced [17]. Larger surface area and adsorption sites enhances the removal of contaminants from wastewater. The cellulose nanocrystals functional groups are responsible for dyes' removal by electrostatic interaction making them potential candidates for fabricating composite membranes [18]. While chitosan can be used as a matrix, it is mostly used for the removal of acidic dyes that are negatively charged because of its functional group. When the positively charged dyes (Victoria Blue 2B, Methyl Violet 2B and Rhodamine 6G) were present in the water to be treated, electrostatic interaction occurred between these pollutants and the high negative charges on the membrane's surface. This is attributed to the nanocrystals where stable ionic bonds were formed between the dyes' molecules and membrane surface that prevented them from passing through the membrane. In this adsorptive membrane technology, the freeze-drying process was used in loosely bounding the cellulose nanocrystals (CNCs) with the chitosan polymer chains in a 3D network by cross-linking [19]. This was a useful study as these dyes which were removed by 98% are categorized under the "very hazardous" contaminants that threat humans and the aquatic life [19]. This study also proved that cross-linking of nano-composite enhanced mechanical stability and showed decrease in surface area and pore size [19].

Similarly, another study used a fabricated nano-fibrous membrane

Table 1
Dyes found in textile wastewater with their concentration ranges [14].

Main dyes found in WW	Classification of the Dye	Pollutant concentration range	Chemical Structure
Acid orange 10 (C.I. 16230)	Anionic	14 mg/L	
Direct red 28 (Congo red)	Anionic	10–120 mg/L	
Acid orange 52 (methyl orange)	Anionic	10–120 mg/L	
Basic violet 10	Cationic	10–120 mg/L	
Acid violet (C.I. 42650)	Anionic	10–40 mg/L	
Rhodamine B	Cationic	10–40 mg/L	
Basic blue 9 (methylene blue)	Cationic	–	

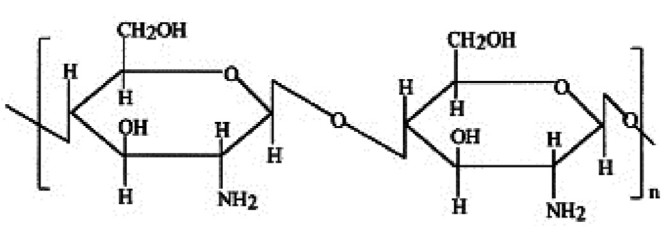


Fig. 1. Chitosan Structure [12].

with micro–nano structured poly(ethersulfones)/poly(ethyleneimine) to remove three anionic dyes (Sunset Yellow, Fast Green and Amaranth) in the same manner related to charges interactions [20]. chitosan membranes have attracted a special attention in the removal of dyes as they showed positive outcomes and advantages including low cost fabrication, high adsorption capability, and selectivity in removing dye materials [21]. In one experiment, a fabricated thin film composite membrane by a mixed matrix of nano-clay (Cloisite 15A and 30B)/chitosan nano-composite coated on commercial polyvinylidene fluoride microfiltration membrane was applied for methylene blue removal. This fabrication was done without a cross-linker agent [22]. Their study showed that methylene blue removal improved with adding organoclay particles in the chitosan matrix. Cloisite 15A mixed TFC membranes proved to be effective for methylene blue removal from water, while acidic dye (acid orange 7) was efficiently eliminated by Cloisite 30B/chitosan coated membrane at acidic pH values [22]. This was explained by the electrostatic interactions of clay and chitosan in the presence of high concentration of proton. These two dyes are excessively used in textile industry and widely presented in wastewater with threatening effects to human health due to being bio-toxic and carcinogenic [23]. Therefore, the effective removal shows the promising results of nanoclay insertion in nanocomposite membranes.

A natural filler to improve the filtration of chitosan membrane was used with montmorillonite (MMT) which is a clay mineral characterized by good biocompatibility, biodegradability, and excellent mechanical properties [24]. The advantages of clay and chitosan were combined while reducing any drawbacks. Different ratios of montmorillonite embedded in the membrane that ranged from 10 to 50% for the removal of Bezactiv Orange V-3R dye of different concentrations were studied. The results showed that the adsorption increased with increasing the content of MMT in the membranes. This adsorption material resulted in enhancing the performance [10]. Similarly, another study reported nanocomposite adsorptive membrane based on chitosan–montmorillonite nanosheets added to the polyethersulfone membrane matrix by mixing with dope solution. It was a “loose” novel hybrid type adsorptive membrane with a finger-like structure that showed excellent performance, excellent hydrophilic nature, and competent mechanical strength in a range of pH values. This was proven by the increase in water flux from 32 $L\ m^{-2}\ h^{-1}$ to 68.82 $L\ m^{-2}\ h^{-1}$ with increasing the CS– MMT content from 0 to 1.0 wt% [25]. This AM was utilized for Reactive Black 5 and Reactive Red 49 dyes’ removal which are organic dyes that can react with a fibre to form a covalent link. This membrane was fabricated by blending chitosan–Montmorillonite nanosheets via phase inversion and resulted in better antifouling nature. Therefore, use of nanosheets is a promising technology since the nanofiltration membrane has a relative “loose” selective layer capable of separating dyes and multivalent salts with high flux recovery ratio of 92% [25].

Polyethersulfone nanofiltration membranes were synthesized by blending of O-carboxymethyl chitosan/Fe_3O_4 nanoparticles by phase inversion for the removal of Direct 16 dye [26]. These modified mixed matrix adsorptive membranes, which characterized by flat sheet form, showed greater pure water flux and permeation in comparison with the unfilled membrane. The dye removal was motivated by the negative charge of the membrane surface created from the addition of O-carboxymethyl chitosan coated Fe_3O_4 nanoparticles in the matrix [26].

A hybrid chitosan membrane containing oxidized starch and silica was used for the removal of direct dyes Blue 71 and Red 31. These direct dyes containing one or more azo groups (-N=N-) are commonly used for dyeing and printing of cellulosic fibers and their blends [27]. They are harmful and any traces in water even in the nano ranges can reduce the penetration of sunlight, thus affect the photosynthesis of plants. In addition, they are carcinogenic, mutagenic, toxic, and allergenic [28]. This hybrid membrane type has shown good thermal stability and swelling properties, and an increased adsorption with pH increasing [27]. Additionally, a combined technique of electrospinning and spraying was used for fabricating nano-fibrous chitosan membrane enhanced by carboxylated MWCNTs as an innovative technique instead of blending. This adsorptive membrane was investigated in removing methylene blue and methyl orange from water [29]. As MWCNTs have attracted attention in the membranes filed due to their better selectivity without compensating the flux, in addition to good electrical, thermal, mechanical, and water transport properties. As a result, the bright outcomes regarding the rejection percentages obtained which reached 86.2% and 83.6% show the promising technology of combining electrospinning and spraying techniques [29]. Scanning electron microscope (SEM) image is shown in Fig. 2 when the two approaches are combined in this study.

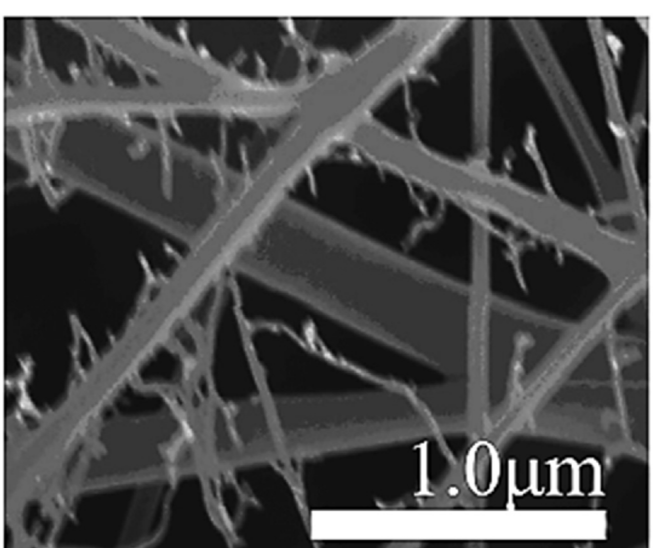


Fig. 2. SEM image of f-MWCNTs/CS [89].

Additives were incorporated to improve chitosan’s performance for dyes removal from water by changing membrane’s surface charge. This is due to enhancement of the electrostatic and chemical bonding adsorption of dyes. For instance, TiO_2 nanoparticles were added as a surface acting agent to polysulfone membrane that increased the flux and dye elimination [30]. Nanoparticles sizes and quantum effects allow the enhancement of the pore structure of the membranes, the improvement of their hydrophilicity, anti-fouling property, and flux [31].

Despite the improvements observed upon the usage of Chitosan for the removal of dyes from wastewater, it showed several disadvantages as well. The efficacy strongly depends on the pH, as it works at acidic pH due to the chemical nature of the dye. Therefore, the pH of the wastewater need to be modified to more acidity before the purification process [10]. Also, chitosan gets swollen in water and thus might lose its physical structure, in addition to its low mechanical strength. To overcome these issues, chitosan usually reacts with a cross-linking agent to link chitosan chains via covalent bonding where its function react with amino groups [27].

The summary of these studies’ removal efficiencies and experimental operation conditions is presented in Table 2 based on the available information presented by the authors.:

2.2. Removal of heavy metals

The rapid development of industrialization is beneficial to the life of mankind; however, it has increased the heavy metal-containing effluent discharge which possess threats to the public health and wellbeing. Heavy metals as anions or cations are among the most hazardous pollutants in water coming from human activities like mining and manufacturing. These metals include lead (Pb), arsenic (As), chromium (Cr), mercury (Hg), nickel (Ni), and copper (Cu) which are toxic and dangerous to humans and animals even at very low concentrations [33].

Heavy metals are dispersed in the environment by natural processes like volcanic eruptions, spring waters, and erosion, or by anthropogenic practices such as fossil fuel combustion, agricultural activities, and industrial activities. The heavy metals present in water can have many adverse health effects on humans [34].

Heavy metals existence in water is a threat to humans’ lives and the environment. Hence, their removal is crucial. During the past decades, heavy metals removal was performed using conventional treatment techniques like ion exchange, coagulation-flocculation, adsorption, and membranes to meet the required standards set by countries or regions. While recently, the use of adsorptive membranes has also become an efficient technique that combines many advantages such as low energy consumption, and improved permeate flux [33].

2.2.1. Mechanism of heavy metals removal via adsorptive membranes

Adsorptive membranes can be modified to contain reactive functional groups like $-NH_2$, $-SO_3H$, and -COOH groups, via ion exchange or surface complexation. These functional groups are essential for metal ions attachment, thus, removed when contacting the membrane surface regardless of the pore sizes that are larger than the metal ion size [33]. To better describe the mechanism of adsorptive membranes removal of heavy metals, it is crucial to understand that their surfaces are charged by adsorption and ionization, as the adsorbent may contain charged ionic functional groups. These surfaces can be split to Stern layer and diffuse layer [6] which are shown in Fig. 3. The region A in the figure shows charged functional groups that attract ions in the feed solution, bring them to diffuse, thus, leads to cation exchange process. The passage of the feed solution in the membrane pore can be convective mass transfer or diffusion mass transfer as seen in region B. Region C represents the case when there is no net charge on the membrane surface. For example, chitosan based adsorptive membranes are used for copper ions removal,

Table 2
Summary of dye removal studies operation conditions.

Dyes to be removed	Adsorptive Membrane	Removal efficiency	Operation Conditions	Reference
Victoria Blue 2B, Methyl Violet 2B and Rhodamine 6G	Cellulose nanocrystals (CNCs) in chitosan matrix composite membrane.	98%, 84% and 70% respectively	Initial Concentrations 1 mg/L with optimum pH 5.01 for model wastewater (synthetic WW).	[19]
Methylene blue	Organoclay/chitosan nanocomposite coated on the commercial polyvinylidene fluoride (PVDF) microfiltration membrane.	–	aqueous dye solution with distilled water and initial concentration of 1 mg/L at neutral pHs.	[22]
Reactive Black 5 and Reactive Red 49	Nanofiltration membrane by blending with chitosan –Mont- morillonite (CS–MMT) nanosheets.	92%	Dye solution with prepared with deionized water and a pH 5.	[25]
Direct Red 16 dye	Polyethersulfone (PES) nanofiltration membranes with O- carboxymethyl chitosan/ Fe3O4 nanoparticles.	99%	Dye solution in distilled water studied in a dead-end stirred under operating pressure of 4 bar and pH= 6.	[26]
Acid Red 249 and Reactive Black 5	Composite nanofiltration (NF) membrane using polysulfone ultra- filtration membrane as support layer and TiO2 nanoparticles.	99% and 93% respectively.	At the optimal preparation conditions of 3 wt% PEI, 0.3 wt% HACC, 0.9 wt% TiO2, 1.5 wt% TMC, reaction temperature of 20 °C and reaction time of 60 s. The solution to be treated was prepared using deionized water.	[30]
Direct Red 75, 80 and 81, and Direct Yellow 8 and 27	Nanofiltration (NF) polyamide (PA) composite membranes	Almost 100% for all of the dyes producing colorless water.	Aqueous solution with initial concentration of dyes of 1000 ppm prepared at room temperature. Artificial dyeing wastewater containing Direct Red 75, PVA, NaCl and Na_2SO_4 as components of the wastewater was used.	[32]

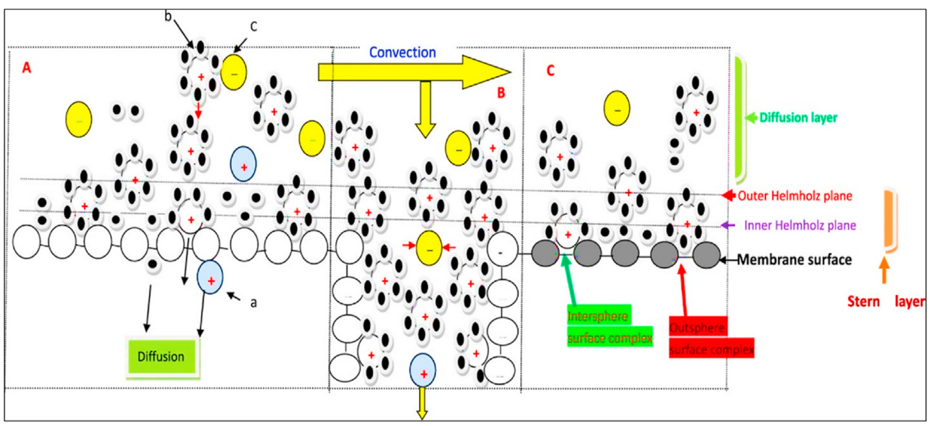


Fig. 3. A schematic representation of electrical double layer in which ion exchange occurs (A and B) or surface complexes are formed on the membrane's surface (C) [6].

since they have high content of amine ($-NH_2$) functional group which form surface complexes in aqueous solutions with Copper. As a pair of lone electrons from the N atoms get shared with the copper ions, that results in increasing the oxidation states and changing the binding energies of the nitrogen atoms [35].

Adsorptive membranes are porous membranes that carry functional groups on their external and internal surfaces. The functional groups can bind with heavy metal ions by surface complexation or ion exchange mechanism [36]. This ion exchange mechanism occurs when the adsorbent have the active sites with free electrons or an electric charge after which an electrostatic interaction between the adsorbent and the substance occurs [33]. Hence, heavy metal ions are eliminated from the passing wastewater when they touch the membrane surface even if their size is much smaller than the membrane's pore size. This is a preferred technology over traditional porous membranes which remove particles by size exclusion only depending on the pores size. Therefore, the use of the adsorptive membrane appears as an innovative hybrid technique that removes small pollutants as heavy metal ions [36].

Adsorption is mainly controlled by the functional groups on the surface, as they control the selectivity and the mechanism. Carboxyl, sulfonic and phosphonic groups adsorb pollutants by ion exchange, whereas groups with nitrogen like amine and thioamide groups chelate cations and anionic adsorb via electrostatic interactions. It has been demonstrated that amine groups are the most effective in pollutant removal from aqueous solutions [37].

Utilizing adsorptive membranes for the removal of heavy metal ions have been researched extensively, and many types of AMs were investigated for this purpose. A recent work discussed the applications of various adsorptive membranes such as polymeric membranes (PMs), polymer-ceramic membranes (PCMs), electrospinning nanofiber membranes (ENMs), and nano-enhanced membranes (NEMs) [38]. As polymeric membranes contain functional groups such as amine, carboxyl, and sulfonic acid in biopolymers or synthesized polymers which are advantageous due to their adsorption capacities for heavy metals' removal. While polymer ceramic membranes with natural clay materials are less common due to fouling and their thermal stability limits. Electrospinning is a common fabrication technique to produce fibres with nanometer to micron diameters into long polymeric fibres to achieve nanofiber membranes (nanostructure membranes) with many advantages like high porosity membranes (>90.0%) and practicability. Moreover, nano-enhanced membranes possess unique characteristics due to the structure and surface properties of nanomaterials (carbonaceous, nanometal oxides) such as the larger surface contact and higher reactivity) [38].

Natural polymeric adsorptive membranes are widely used for heavy metals' removal. chitosan based membranes has showed excellent performance in this purification process. The chitosan biopolymer is simply extracted from the shells of crustaceans like shrimps, crabs and lobsters.

Removal of heavy metals by chitosan is attributed to having amine and hydroxyl groups present on chitosan [36]. The removal of Cd(II), Cu(II), and Ni(II) metal ions was studied using chitosan adsorptive membranes with polysulfone polymer and applying phase-inversion to achieve ultra-filtration. Synthesized *N*-phthaloyl chitosan was used in blending modification of poly(ether imide) ultrafiltration membrane for the removal of trivalent ions. Increasing the content of *N*-phthaloyl chitosan particles showed effective results in reducing the size of the membrane pores, increased hydrophilicity and permeate flux [39].

Synthetic polymers are also used for removing heavy metal ions from water. For example, the novel adsorptive ultrafiltration membranes made from polyvinyltetrazole-*co*-polyacrylonitrile was used for Cu (II) ions. As the PVT parts were the main binding sites for the adsorption of copper ions, and the membranes were hydrophilic so that water was adsorbed by hydrogen bonding interactions. This study demonstrated that the membrane adsorbents were greatly selective for adsorbing Cu (II) ions over Pb (II) ions under the same experimental conditions, in addition to that it showed higher adsorption capacity than other types of membranes discussed in literature. The regeneration of the membrane occurred with using 0.25 mM ethylenediaminetetraacetic acid (EDTA) solution that can be furtherly used for heavy metal removal. Nonetheless, the permeability of the hollow fiber membrane was lower than flat sheet membranes [40].

A mixed matrix polymer membrane type was studied where a thiol functionalized mesoporous poly (vinyl alcohol)/SiO_2 composite nanofiber membrane was used. It was fabricated by electrospinning and utilized for Cu (II) removal with an enhanced adsorption capacity in comparison with the pure membrane. The highest adsorption capacity obtained was 489.12 mg/g at 303 K. That is a result of the modification with $–NH_2$, –SH, or $–HSO_3$ functional groups that are reactive with metal ions. For instance, the sulfur atom of the –SH group can form chelates with them which is a type of bonding [41]. Moreover, a novel polyvinyltetrazole-grafted resin showed maximum adsorption capacity for Cr (III) (3.36 mmol/g), then Cu (II) (2.65 mmol/g) and Pb (II) (1.52 mmol/g) at pH 5.0 with high selectivity for these ions over other ions. This adsorptive membrane showed excellent desorption rate, reusability, and chemical stability under acidic and alkaline conditions [37].

A multifunctional porphyrin membrane was used for the selective Cd ions removal [42]. Polymer brushes were grafted on a porous chitosan/cellulose acetate blend base membrane surface by surface-initiated atom transfer radical polymerization *(ATRP)* method. It was prepared with the ability of displaying different colours changes as a response to the occurrence of cadmium ions in the aqueous solutions, as it turns from yellow to green even at low concentrations. This is called a multifunctional membrane since it simultaneously detects and removes the ions in wastewater through one process. An improved adsorption for cadmium in a large range of pH and concentration was achieved using such developed adsorptive membrane [36]. A synthetic membrane with poly(4-vinylpyridine) as a complexing polymer in a poly(vinyl alcohol) matrix also showed great affinity for Hg(II) ions [43].

Heavy metals can be removed by nonspecific adsorption on solid matrices such as metal oxide, and activated carbon, or by specific adsorption on appropriate sorbents having complexing or chelating agent that interacts with the metal ion. In addition to a support matrix that could be either inorganic (e.g, aluminum oxide, silica or glass) or organic (e.g poly(methyl methacrylate), polystyrene, cellulose). The difference between the specific and nonspecific adsorption is that the specific sorbent consists of a ligand that interacts with a certain metal ion unlike the nonselective adsorption [43].

Furthermore, polyethylene terephthalate (PET) is a semicrystalline thermoplastic polymer which is utilized to produce different products like bottles, fibers, molded parts, and films. This polymer proved to be a proper material for adsorptive membranes' support. Using PET is applied in removing chromium Cr(VI) from water due to its excellent mechanical properties and simple processing [44]. In this study, electrospinning of PET bottle waste in the form of nanofibers was used to prepare the membrane support. Then, the surface was activated by cold plasma and functionalized with chitosan [45]. The plasma treatment showed significant functionalization with chitosan. Furthermore, sunlight active Polysulfone with TiO_2 hydrophilic nanoparticles hybrid membrane was used to remove chromium from water, as it reduces poisonous Cr (VI) to less toxic Cr (III) by photoreduction and reject it from polluted water. This mixed matrix hybrid membrane demonstrated superior hydrophilicity and permeability with enhanced metal ion reduction and rejection after the uniform dispersion of the appropriate photocatalyst [46]. However, this photoreduction reaction needs the presence of an acid to prevent any accumulation of holes and hydroxyl radicals in the reaction medium, in addition to the need of a filtration step and the formation of agglomerates in the membrane matrix which are considered drawback for future research focus [47].

The use of adsorptive membranes is a promising solution to many technical problems that could be encountered using the conventional methods for heavy metals removal from water. For instance, infusing nanoadsorbents into polymeric membranes; since using these nanomaterials in fixed column bed or any other flow systems is not the best approach due to inadequate mechanical strength, high pressure drop, and difficulty in separation from the aqueous solution [48]. Also, using the high pressure dependant membranes like nanofiltration (NF) and reverse osmosis (RO) [49] which use size exclusion and Donnan charge repulsion mechanisms to remove heavy metal ions have drawbacks such as requiring high pressure, producing low permeate, and forming very high concentrated retentate solution. Therefore, having nano-adsorbents immobilized on the membranes can effectively remove heavy metal ions when the polluted water pass through the cross-section of the membrane to produce purified permeate with better selectivity and permeability and overcome any problems faced when applying each technology on its own [50]. It should be noted that surface area of the nanoparticles can be lost because of the interactions between nanoparticles and the polymers, hence, optimum loadings are used.

Adsorptive nanocomposite membrane is fabricated from the dispersion of nanosized adsorbents in the continuous polymeric matrix. This is done by casting followed by evaporating the solvent under controlled conditions [51]. Many studies investigated the applications of nanoparticles as membranes fillers in the adsorption of heavy metal ions from aqueous solutions, and Table 3 presents their special characteristics based on the available information presented by the authors.

Many factors need to be considered to decide whether the adsorptive membrane is suitable for this kind of operation or not; these include the mechanical strength, water permeability, adsorption capacity, surface charge alteration, water flux, and ions selectivity. For instance, adding nano-sized adsorbents in adsorptive nanocomposite membranes showed enhancements in adsorption capacity and selectivity, and affinity for targeted metal species. In addition, adding the optimum amount of nanoadsorbents increase the mechanical strength of the adsorptive membrane due to the interaction between them and the polymer matrix that results in forming thicker skin layer and suppressing macrovoids; this would increase the rigidity and elasticity of the structure [50]. Mukherjee [56] showed that as the loading of graphene oxide nanoparticles increased from 0 to 0.1 wt% in PSf membrane, the mechanical strength of the nanocomposite membrane increased significantly. However, when it exceeded 0.5 wt% it decreased due to increasing pore density. Therefore, an optimum loading should be applied to enhance the strength without overdoing to avoid an opposite response. This mixed matrix membrane showed high rejection and adsorption capacity for Pb, Cu, Cd and Cr ions [56].

Moreover, fabricating rougher membrane surface to increase water permeability can be achieved by adding hydrophilic nanoparticles. Abdullah et al. [57] studied polysulfone/hydrous ferric oxide nanoparticles ultrafiltration mixed matrix membranes that were fabricated for removal of lead (Pb^{2+}). The authors showed that by increasing the loading of the nanoparticle, many characteristics were enhanced. As the amount of hydroxyl group interacted with PSf polymer increased, hence membrane became more hydrophilic to attract more water molecules to

Table 3
Nano-adsorbent characteristics.

Nano-adsorbents	Special Characteristics	Adsorbent Capacities	Operational Conditions	Reference
Metal oxides and metal-organic frameworks (MOFs)	Excellent performance in removing Pb(II), Hg(II), and As(III)1.	8.40–313 mg Pb(II) g^{-1}, 0.65–2173 mg Hg(II) g^{-1}, 49.5–123 mg As(III) g^{-1}.	Aqueous solution with deionized water and pH < 6.	[52]
Carbon Nanotubes	High adsorption capacity.	It reached 181.8 mg Hg(II) g^{-1} when using Ox-MWCNTs impregnated chitosan beads.	Wastewater of some industrial cities in Egypt with initial concentration of 1000 mg/L and pH 4.	[53]
Graphine Oxide	Unique physicochemical characteristics.	250 mg Pb(II) g^{-1}, and 72.6 mg Cu(II) g^{-1}.	Aqueous solution with Pb(II) at with initial concentration of 25 mg/L, pH 6 and 298 K. Aqueous solution with Cu(II) of pH 5.7 and temperature of 303 K.	[54]
Zeolites	High surface area and hydrophilic nature.	838.7 mg Cd(II) g^{-1} using polyvinylacetate polymer/NaX nanocomposite nanofibers.	Aqueous solution with distilled water containing Cd(II) with initial concentration of 5 mg/L at pH 5.0.	[55]

the surface and thus increase the permeability of the adsorptive membrane [57].

A new research direction is focusing using conductive polymeric-based materials [58] such as adsorbents, photocatalysts, membranes, and supporting beds for the purpose of heavy metal ions removal from wastewater. This is due to their outstanding properties that outweighs the traditional materials and semiconductors usually used such as their mechanical stability, ease of functionalization, chemical versatility, ease of preparation, environmental sustainability, and high electrical conductivity. Conductive polymers include polyaniline, polypyrrole, and polythiophene that can be combined with other polymers, nanoparticles, and bio-based materials for wastewater purification [58]. These materials can be investigated thoroughly in future research.

2.3. Removal of pharmaceutical compounds

One of the current concerns that poses threat to humans and the ecosystem is what's called 'emerging organic contaminants' (EOCs). This term covers the newly discovered compounds in the environment like pharmaceuticals and personal care products (PPCPs) [59]. Pharmaceuticals, as medicines or drugs, are chemical compounds used for many purposes like medical diagnosis, cure, treatment, or prevention of diseases for humans and animals [5]. These substances are an essential category of emerging environmental contaminants, which recently led to increased concerns as huge amounts are being discharged and ending up in surface waters and wastewaters.

These chemicals are excessively consumed by modern societies these days, such that they find their way to the environment through sewage treatment plants or direct discharge from the skin while swimming or bathing [60]. Generally, pharmaceuticals industry is a thriving sector with continuous increase in consumption driven by the need to treat ageing-related and chronic diseases. Moreover, the current lifestyle, nutrition, and modernity have resulted in a tremendous increase in many diseases like diabetes, high blood pressure, cholesterol, cancer, and psychological illnesses. For example, the COVID-19 pandemic has enlarged the general worldwide occurrence of depression and anxiety which increased the consumption of antidepressants, thus their presence in wastewater [61,62]. Many pharmaceuticals do not completely degrade after application; therefore, their metabolites and unchanged forms are excreted thus entering the ecosystem. Pharmaceutical residues are continuously introduced to the aquatic environment as traces of low concentrations in the ng L^{-1} or μg L^{-1} range [63], as a study with targeted analysis of grab water samples from a river in the UK discovered a total of 33 pharmaceuticals, illicit drugs, drug metabolites, personal care products and pesticides with mean concentrations at 40 ± 37 ng L^{-1} [64]. This was facilitated by the use of different technologies that allowed the detection of these compounds at such low levels. For instance, liquid chromatography-tandem mass spectrometry (LC–MS/MS) is used for pharmaceuticals and illicit drugs detection in wastewaters due to its sensitivity, quantitative precision and selectivity via multiple reaction monitoring [65].

The continuous input or inadequate removal at treatment plants shifted the focus towards this type of contaminants [60]. Anti-inflammatories and analgesics like paracetamol, ibuprofen, and diclofenac, Antidepressants such as benzodiazepines, and Antiepileptics like carbamazepine. In addition to Lipid-lowering drugs as fibrates, β-blockers, Antiulcer drugs, and antihistamines which includes ranitidine. Other types are Antibiotics such as tetracyclines, macrolides, β-lactams, penicillins, quinolones, and imidazole derivatives [66].

The pharmaceutical industries uses the conventional term "active pharmaceutical ingredients" to define substances that are pharmacologically active, resilient to degradation, capable of causing serious effects on water organisms, and have an adverse impact on human's health [66]. For example, it can cause fish's nephridial tissue necrosis, influence the growth of alga and duckweed, and enhance the microbial resistance to antibiotics [67]. The U.S. Environmental Protection Agency and European Union added pharmaceuticals to their watch list for water quality [5].

They have special characteristics that differentiate them from other contaminants such as having molecular masses less than 500 Da mostly, and they can be produced by large and complex molecules that have different molecular weights, functionality, structure, and shape. Moreover, they are polar molecules having more than one ionizable group. This degree of ionization and its characteristics depend on the pH of the medium. Also, they can be lipophilic, modestly soluble in water or highly persistent in water depending on the type of pharmaceutical, and most of them are photoactive as they absorb luminous radiations [66]. This proves the diversity of pharmaceutical compounds' types and characteristics.

In wastewater treatment plants, mixtures of pharmaceuticals with diverse chemical structures exist simultaneously. Pharmaceutical contaminants used to be removed from water using physico-chemical treatments with a secondary system in wastewater treatments plants that contains a biological reactor formed with activated sludge [68]. This process can remove paracetamol, acetylsalicylic acid, and ibuprofen. However, these processes have a limited capacity as most of the compounds do not get metabolized by microorganisms as a source of carbon and can hinder the activity of these microorganisms. Other treatment methods include adsorption/bioadsorption on activated carbon, ozonation, photooxidation, radiolysis and electrooxidation without and with active chlorine generation. Examples of such adsorbents studied include activated carbon from lotus stalks, olive-waste cake, coal, wood, plastic waste, cork powder waste, peach stones, coconut shell, and rice husk [66]. But, these have shown drawbacks like the uncertainties in the interactions, mechanisms, and kinetics which impedes such application on the industrial scale [66]. Other adsorptive materials include: CNTs especially multi-walled carbon nanotubes (MWCNTs), natural clay materials such as bentonite, and ion exchange materials that were also reviewed for antibiotic removal [69].

Rsearchers also studied the different technologies such as coagulation and flocculation, adsorption with their oxides as nanoparticles, and chemical oxidation or reduction [5]. However, these processes were also challenged by some limitations such as reduced removal efficiency, limited treatment conditions, and toxic by-products [5].

The use of adsorptive membranes appears as a promising technology for the removal of pharmaceuticals from wastewater. As this technique, encompasses the advantages of using adsorption and membrane technologies with eliminating their drawbacks. Some studies have explored this technology, which this section will be reviewing.

For instance, cellulose acetate/Mg-Al layered double hydroxide nanocomposite adsorptive membranes were used to eliminate pharmaceutical compounds from wastewater [70]. It was applied to remove Diclofenac sodium and tetracycline. This showed excellent adsorption capacity and better permeability than using the polymer membrane alone. Tetracycline is an antibiotic and diclofenac sodium (DS) is an anti-inflammatory drug. The use of cellulose acetate porous membrane alone showed such drawbacks as low water permeability, inadequate mechanical strength, and weakness to chemical and microbial attacks [70]. Therefore, blending or adding nanofillers have helped in overcoming these issues, since layered double hydroxides are active adsorbents with large surface areas, extraordinary thermal stability, and porosity which was incorporated by phase inversion as Mg-Al LDH inserted with sodium dodecyl sulfate (SDS) within the polymer matrix [70]. These nanocomposite adsorptive membranes proved to be more hydrophilic according to contact angle measurements with ten times increase in adsorption capacity for diclofenac sodium with respect to the original membrane. This improvement resulted from the electrostatic interactivity between the negatively charged drug molecule and the positive charged Mg-Al LDH layers, yet it was different for tetracycline as the increase was smaller due to hydrogen bonding interactions [70].

Several adsorbent materials were studied such as biochar, clays, chitosan, agro-industrial wastes, and metal–organic frameworks (MOFs), however the removal effectiveness of pharmaceuticals depends on various factors like pH, temperature, and the affinity between adsorbent and pollutant [71]. Another research investigated the removal of xenobiotics which is a general classification that include numerous compound types employed in the chemical and materials industry like pharmaceuticals. A polysulfone membrane was used with polyvinylpyrrolidone additives and different organic acids that increased water flux and rejection [72].

Other studies included the technology of combined adsorption and ultrafiltration membranes [73]. Combined metal organic frameworks and ultrafiltration hybrid systems were used to treat pharmaceutically active substances such as ibuprofen and 17α-ethinyl estradiol, and natural organic matter [73]. Ibuprofen is a well-known pain killer, and 17 α-ethinyl estradiol (EE2) is a synthetic hormone. Metal organic frameworks have high tunable porosity, excellent capability for pollutants removal, and decreasing fouling in adsorbent ultrafiltration hybrid membranes. Although these pharmaceutically active compounds are found at low concentrations in ground, surface, and wastewater, they are very hazardous as they reach the aquatic environments and water supplies through the water cycle and pose threatening physiological effects. Moreover, this study compared the results obtained from their MOF-UF systems with having ultrafiltration membranes only, and with using powdered activated carbon as an adsorbent with UF membranes. It was noticed that better retention rate than UF only under pH of 3, 7, and 11 with no serious fouling because the MOFs adsorbed the selected pharmaceuticals effectively. Additionally, it showed superior results to using powdered activated carbon regarding water flux, retention, and anti-fouling performance as well [73].

Carbon nanotubes (CNT) have been widely researched previously as promising materials for water treatment due to their structure and excellent adsorption capacity [74]. The use of adsorptive membrane filtration for pharmaceuticals removal including Triclosan and ibuprofen have been studied [74]. However, many uncertainties were addressed concerning the toxic effects of ingested CNT when directly dosing it to the polluted water [74]. Consequently, they have to be accompanied with other practices to avoid contaminating the treated water. Membranes consisting a functional CNT layer on top of a substrate membrane was used. The substrate membrane which is a flat sheet polyvinylidene fluoride (PVDF) membrane acting as a mechanical support to the CNT layer, and as a barrier to avoid CNT leaking into clean water. This study indicated that using multi walled carbon nanotubes layers above a PVDF membrane substantially improved PPCP removal. This proved the promising potential of this technology for water treatment applications. Also, the process was affected by different PPCP–CNT interactions, as the efficiency was excellent with PPCP having aromatic rings like triclosan. Also, CNT that has a larger specific surface area favors PPCP molecules [74].

Due to the impact of pharmaceutical compounds' presence in wastewater, more environmental risk assessment investigations are necessary for various pharmacologically active compounds and their metabolites [66]. However, there are limited studies on technologies that are capable of effectively removing pharmaceutical from contaminated water. Also, utilizing adsorptive membranes technology for this purpose has not been extensively studied which can be an idea for future research since there is potential. The summary of these studies' removal efficiencies and experimental operation conditions is presented in Table 4 based on the available information presented by the authors:

2.4. Common analytical techniques in adsorptive membranes

Some analytical techniques are crucially critical to understand and evaluate the behaviour of the adsorptive membrane. They are required as a preliminary step before assessing the performance of adsorptive membranes in environmental applications. Scanning electron microscopy (SEM) and Brunauer–Emmett–Teller (BET) techniques are widely used to investigate the membrane morphology and pore structure. SEM technique scans an electron beam over a surface to generate an image by electrons interaction with the sample to get surface topography and composition [75]. It is the most common approach to examine the structure of the membrane and evaluate the bulk and surface morphology to compare modified and unmodified membranes [22]. This analysis technique has many applications, for instance study the morphology before and after exposure to ultraviolet (UV) irradiation, measuring the composition for semi-quantitative results, identifying foreign materials in the membrane matrix of the polymer as well as their dispersion [76]. For example, such analyses showed clear images of the sponge-like and macrovoids-free porous structures of polyimide hollow fiber membranes [77]. BET analysis evaluates the specific surface area of materials and the pore area using nitrogen multilayer adsorption computed as a function of relative pressure. This is done by applying an automated analyser [78]. For example, it was used to determine the surface area of tyre pyrochar in an adsorption experiment to be $38.17 m^2/g$ [79]. Moreover, X-ray diffraction (XRD) is another method used to define the atomic and molecular structure of a material through irradiating it with incident X-rays, then determining the intensities and scattering angles of the X-rays scattered by the substance [80]. For example, it was used to study the locations of nanoclay particles in chitosan matrix of TFC membranes [22]. In addition, XRD was used to identify the functional group and magnetic properties of compounds. As an example, XRD analysis on Ferric oxide (Fe_3O_4) ferrofluids coated by oleic acid showed that the phase of the samples was Fe_3O_4 based on the diffraction peaks with the highest peak at 35.6° with the particle size of 8.8 nm [81]. Moreover, the dynamic contact angle measurement is another analysing test that was used in many experiments to study adsorptive membranes. This test is used to obtain information regarding hydrophilicity and hydrophobicity of the membrane by measuring the contact angle between the membrane's surface and water droplet using a contact angle goniometer, as membranes with lower water contact angle possess better hydrophilic properties [82]. Atomic force microscopy (AFM) can be used to characterize the interaction between the membrane surfaces and foulants to

Table 4
Summary of Pharmaceutical studies experimental conditions.

Pharmaceutical/compund to be removed	Adsorptive Membrane	Performance	Operation Conditions	Reference
Diclofenac Sodium and Tetracycline	cellulose acetate/Mg-Al layered double hydroxide (Mg-Al LDH) nanocomposite membranes	The membrane prepared with 4 wt% Mg-Al LDH loading exhibited the highest water flux (529 $L{\cdot}m^{-2}{\cdot}h^{-1}$)	Distilled Water solution with initial concentrations of 0.01 g/L DS (pH=7) and 0.01 g/L TC (pH=6.9) in the solution.	[70]
Xenobiotics p- nitrophenol (PNP)) (used in pharmaceutical industry)	Modified polysulfone nanofiltration membrane with organic acids	Highest rejection of PNP (85%) at pH 8	Aqueous media of double distilled water at room temperature (25 ± 2 °C) with initial concentration of 0.1 mM at different pH values.	[72]
Ibuprofen and 17α-ethinyl estradiol	Ultrafiltration hybrid systems combined with combined metal organic frameworks (MOF-UF)	The average retention rate of PhACs in MOF-UF (53.2%) was enhanced relative to the UF only (36.7%)	An aqueous water solution with deionized water and initial concentration of 10 μM with pH of 3, 7, and 11.	[73]
Triclosan, Acetaminophen and Ibuprofen.	Polyvinylidene fluoride membrane with MWCNT layers.	The removal ranged from 10 to 95% as it increased with increasing number of aromatic rings: (Triclosan > acetaminophen=ibuprofen).	Aqueous water solution using ultrapure water and Suwannee River fulvic acid to represent natural organic matter in natural water. pH values were varied from 4 to 10 which influenced PPCP removal by up to 70%; greater removals were observed with neutral PPCP molecules than with ions.	[74]

better understand the membrane fouling behaviour. This can be done via Nanosurf Mobile S microscope that shows three dimensional (3D) AFM images of prepared membranes including the roughness parameter [82, 83]. Furthermore, thermogravimetric analysis (TGA) can be used to investigate the loss of water from membrane material and degradation of membrane material during the heating process. TGA results are shown in plots that start at 100% mass and end at almost total mass loss of a membrane. Also, as the temperature increases, the thermal stability plots show the mass loss of a membrane [84]. In one study, it was used to monitor the thermal stability of the pristine as well as composite films [76]. Another study used TGA to plot the different weight loss stages of polyethersulfone/plant-waste-particles MMMs [85].

2.5. Future outlooks and challenges

Most research efforts discussed the use of polymeric based adsorptive membranes for the removal of heavy metals and dyes from wastewater. Most of these studies were conducted with aqueous synthetic solutions using distilled water rather than real wastewater samples. Hence, research efforts should be directed towards the evaluation of real water samples to have more representative results. In addition to the previous challenges and recommendations that were discussed in part I of the paper, the implementation of adsorptive membranes has other challenges that require more improvements. The efficient application of adsorptive membranes for the removal of emerging contaminants such as pharmaceuticals and personal care products remains a research problem. There is a lack in research studies that were performed on the preceding emerging pollutants unlike heavy metals and dyes that were heavily studied in the literature. Adsorptive membranes still suffer from several drawbacks such as the relatively low adsorption capacity because of the limited amount of the adsorbents that can be introduced into the membrane matrix. Moreover, detailed cost analysis studies should be performed to determine the feasibility of applying adsorptive membranes at an industrial scale. In addition to thorough investigations regarding maintenance, materials' regeneration, and accessibility remain ambiguous in the available studies published. Another challenge that requires research is the energy requirements of this technology when applied at a larger scale. In particular, operating and backwashing pressure requirements remain undiscussed. Fouling issues and agglomeration are additional crucial factors that are always a concern in the membranes' industry. To date, no final solution has been provided. A future recommendation would be to study the dispersion mechanism of nanoparticles within the membrane matrix to prevent agglomeration as this is still an obstacle in utilizing nano-materials in membranes. Future research should focus on studying several materials' types like ceramic adsorptive membranes since past studies focused mostly on polymeric adsorptive membranes. The use of natural adsorptive materials instead of synthetic ones appears to be a novel and sustainable trend in this aspect. Finally, the published research studies still lack a proper discussion on the fate and utilization of these removed pollutants. For example, heavy metals removed can be reused in manufacturing, pharmaceuticals can be regenerated to be used in producing new medicines, and dyes can be sent to textile industries. However, the feasibility of such a recommendation remains unknown.

3. Conclusions

The appearance of various and emerging pollutants in wastewater is a serious problem that impose negative impacts on the environment and amplifies the existing water scarcity issue. Adsorptive membranes possess the dual advantages of adsorption and separation, thus, demonstrating broad prospects in environmental applications. They were proposed in several studies as promising technologies for the adsorption of trace pollutants from wastewater while possessing fast kinetics. The merits of utilizing adsorptive membranes for the removal of several emerging contaminants from wastewater have been also highlighted in several studies. They specifically proved their efficiency in removing dye materials, heavy metal ions, and pharmaceutical substances. In addition, adsorptive membranes have shown enhancement in water permeability and flux, rejection rates, and selectivity. However, there are still some challenges and drawbacks associated with applying them on an industrial scale. There is also a lack in the number of studies regarding utilizing them to remove pharmaceutical compounds. It appears that the application of this technology is still premature and efforts should be directed towards improving the adsorption capacity, the cost, the reusability and anti-fouling properties.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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