

Review

Footsteps of graphene filled polymer nanocomposites towards efficient membranes—Present and future

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Abstract: Due to rising global environmental challenges, air/water pollution treatment technologies, especially membrane techniques, have been focused on. In this context, air or purification membranes have been considered effective for environmental remediation. In the field of polymeric membranes, high-performance polymer/graphene nanocomposite membranes have gained increasing research attention. The polymer/graphene nanomaterials exposed several potential benefits when processed as membranes. This review explains the utilization of polymer and graphene-derived nanocomposites towards membrane formation and water or gas separation or decontamination properties. Here, different membrane designs have been developed depending upon the polymer types (poly(vinyl alcohol), poly(vinyl chloride), poly(dimethyl siloxane), polysulfone, poly(methyl methacrylate), etc.) and graphene functionalities. Including graphene in polymers influences membrane microstructure, physical features, molecular permeability or selectivity, and separations. Polysulfone/graphene oxide nanocomposite membranes have been found to be most efficient with an enhanced rejection rate of 90%–95%, a high water flux >180 L/m²/h, and a desirable water contact angle for water purification purposes. For gas separation membranes, efficient membranes have been reported as polysulfone/graphene oxide and poly(dimethyl siloxane)/graphene oxide nanocomposites. In these membranes, N₂, CO₂, and other gases permeability has been found to be higher than even >99.9%. Similarly, higher selectivity values for gases like CO₂/CH₄ have been observed. Thus, high-performance graphene-based nanocomposite membranes possess high potential to overcome the challenges related to water or gas molecular separations.

Keywords: graphene; nanocomposite; membrane; water purification; gas separation

1. Introduction

Generally, membrane-based technologies have been used to remove toxic nanomaterials from the environment [1]. Among membrane materials, polymeric-based materials and nanomaterials have been adopted for separation purposes [2,3]. Owing to the technical benefits of nanocarbons, graphene, fullerene, and carbon nanotubes have been adopted as unique and valuable nanostructures [4,5]. Graphene-derived nanomaterials possess fine tendencies toward separation applications [6]. Especially, graphene has been reinforced in polymers to form high-performance nanocomposite membranes [7]. Polymer- and graphene-derived nanocomposite membranes have been efficiently used for the separation of hazardous molecules [8]. For the fabrication of polymer/graphene membranes, facile processing approaches have been used [9]. Solution processing, the phase inversion method, the infiltration technique, and other facile methods have been reported [10,11]. Polymer/graphene

membranes have been developed using polymers like polyamides, polysulfone, poly(dimethyl siloxane), poly(methyl methacrylate), and several others [12]. The nanocomposite membranes possess superior nanofiller dispersion, pore sizes, molecular permeation, and selectivity properties [13]. Owing to their effective characteristics, polymer/graphene nanocomposite membranes have been applied for technological sectors focusing on water, gaseous, and chemical separations [14]. The resulting membranes have been applied in commercial-scale water purification systems, gas sensing, and separation systems, fuel cell systems, and a myriad of other technical areas [15,16]. Most importantly, polymer- and graphene-based nanocomposite membranes have been fabricated for gas and water purification. Graphene mostly develops torturous pathways in the matrices to facilitate gas- or water-based ionic or molecular diffusion processes [17]. Homogeneous graphene dispersion in polymeric membranes has been used to enhance the separation of impurities and toxic molecules from air mixtures or contaminated water [18,19]. In this regard, several membrane processes have been studied, like nanofiltration, microfiltration, ultrafiltration, and reverse osmosis [20–22]. Afterward, graphene-derived nanocomposite membranes have been efficiently used for removing pollutants [23]. The polymer/graphene nanocomposite membranes have structural advantages relative to reported nanocomposite membrane designs in terms of facile processing and resulting performance benefits [24]. Research progressions have led to the advancement of efficient air/water membranes [25].

For efficient graphene membrane fabrication, membranes, and mechanisms of molecular transport need to be thoroughly understood. The self-standing nanocomposite membranes must be researched for new design novelties [26]. In addition to graphene, graphene derivatives like graphene oxide, reduced graphene oxide, etc. may widen the potential of these membranes. The ultimate thinness of the membranes has been desirable to allow high flux [27]. The narrow pore size distribution and surface chemistry have been identified as desirable factors to promote molecular sieving and diffusion through the membranes. According to the literature, the interlayer spacing between graphene nanosheets can promote molecular transportation through the membrane [28]. To better withstand the high temperature, pressure, and humidity conditions, membrane support materials must be used [29]. Such efforts fill gaps between the membrane designs, large-scale productions, and commercialization of the novel graphene nanocomposite membranes.

This review article discusses the developments of graphene nanocomposites towards efficient membrane applications. The inclusion of graphene in polymers has improved the membranes' permeation and selectivity properties for water purification and gaseous molecular separation. In this article, the design, structure, and properties of polymeric membranes filled with graphene or graphene oxide nanofillers have been scrutinized. Subsequently, the microstructure, durability, stability, permeability, and other membrane properties have been used. The competence of nanocomposite membranes has been studied for gas or water purification, especially for the separation of toxins, pollutants, and unwanted species. The polymer and graphene-based nanomaterials have a high surface area and exceptional structure for efficient membrane performance. Accordingly, the formation of polymer/graphene membranes has extended the scope of air purification and water management. To the best of my

knowledge, this article is ground-breaking in presenting efficient graphene-based membranes. The review outline, including literature, as well as relevant discussions, is novel and based on recent research assumptions for graphene-based membranes. Moreover, hardly any recent topical comprehensive review reports have been observed on the polymer/graphene nanocomposite membranes. The need for this review article also arises due to remarkably increased research reports on graphene nanocomposite membranes in the past two to three years. Hence, there is the utmost need for a recent innovative review on polymer/graphene nanocomposite membranes. According to recent reports on polymer/graphene nanocomposite membranes, it can be stated that substantial progress has been made in this field up until now. This article will definitely be beneficial for field scientists/researchers to expand their research toward the future success of high-performance industrial-scale nanocomposite membranes.

2. Graphene

Graphene is a one-atom-thick nanosheet of sp^2 hybridized carbon atoms [30]. **Figure 1** shows the structure of graphene and related derivative forms. This remarkable nanocarbon was discovered in 2004 [31].

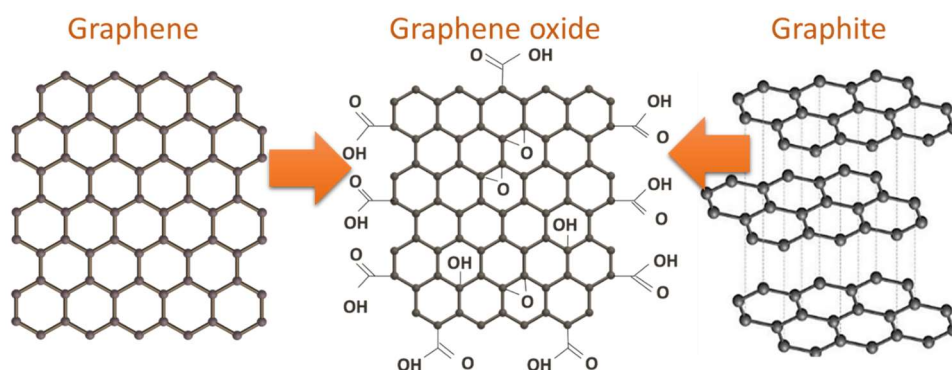


Figure 1. Graphene and related derivative forms.

Graphene has been developed using various techniques such as graphite exfoliation, plasma processes, chemical vapor deposition, and chemical or organic synthetic strategies [32]. It is a transparent carbon nanostructure [33] having a high thermal conductivity of 3000–5000 W/mK [34], Young's modulus of ~ 1 TPa [35], and weak van der Waals forces [36]. Graphene oxide is a graphene-based nanocarbon that is usually formed through the oxidation and stripping of graphite. This graphene derivative has a graphene nanosheet structure with carboxylic, hydroxyl, carbonyl, or other oxygen-containing surface groups [37]. Graphene and graphene oxide have been employed to form nanocomposite materials [38]. Graphene nanocomposites have been studied for their high electron conductivity, thermal and chemical stability, and physical properties [39]. In addition, the high-tech potential of graphene has been developed for coatings, membranes, energy devices, and biomedical sectors [40].

Graphene is composed of a single carbon atom layer arranged in a two-dimensional honeycomb lattice. A wide range of two-dimensional nanomaterials like graphene, nanoclays, MXenes, silicane, hexagonal boron nitride, transition metal dichalcogenides, etc. have been reported. A two-dimensional carbon nanostructure like graphene has probabilities to tailor and functionalize through

surface defects, modified groups, number of layers, morphology, etc. Graphene has been explored for doping, modification, strength, conductivity, and other physical characteristics, relative to other two-dimensional nanomaterials. Consequently, graphene has been found to be stronger than other zero-, one-, or two-dimensional materials due to its structural strength. Especially compared with other carbon nanomaterials like carbon nanotubes (one-dimensional) and fullerene (zero-dimensional), graphene nanosheets have revealed remarkable potential due to their high surface area nanostructures and better compatibility with polymer matrices. Research has realized the importance of graphene two-dimensional monolayers in several fields, with a special emphasis on their benefit to our society.

3. Polymer nanocomposite based membranes

Polymer-based membranes have been produced using a range of polymer matrices and preparation approaches [41]. Numerous carbonaceous nanoparticles and inorganic nanofillers were filled in the different polymeric matrices to develop the nanocomposite membranes [42]. The combination of these nanoparticles with polymers resulted in the formation of membranes with significant physical characteristics [43]. The resulting properties depend upon the nanoparticle type, nanoparticle amount, and polymer types as well [44]. These nanocomposite membranes have fine microstructure and separation properties for different types of molecules [45,46]. In other words, polymeric nanocomposite membranes act as molecular sieves for molecular separations [47]. The molecular permeation mechanisms depend on the interactions between the polymer and nanoparticles and their mutual effects [48]. In inorganic nanoparticle-filled membranes, silica nanoparticles have been used as nanofillers [49,50]. For silica-filled membranes, permeation and selective separation of O₂, CO₂, and N₂ gaseous molecules have been studied. Functional silica nanoparticle-filled poly(vinylidene-fluoride-hexafluoropropylene) membranes were prepared through the phase separation method [51]. The molecular separation of CO₂ molecules was studied. The nanocomposite membrane with 40 wt.% nanoparticles revealed a CO₂ uptake of 33.75 mg/g [52,53]. Silica nanoparticles developed fine pathways for gas diffusion [54,55]. Titania nanofiller [56] and zinc oxide nanoparticles [57] have also been used with the polymers. Such membranes may have high structural robustness and CO₂/H₂ selectivity of 2.77.

In addition, carbon nanoparticles have been reinforced in the polymeric membranes [58]. Various nanocarbon nanoparticles have been used as efficient nanofillers with polymers like carbon nanotube, nanodiamond, fullerene, etc. to form nanocomposite membranes. Graphene-based nanomaterials have a high surface area-to-volume ratio, light weight, facile processing, and structural flexibility [59]. The inclusion of very minor amounts of graphene nanofiller in the nanocomposites has been found to enhance the physical features due to their interfacial properties [60]. Interfacial bonding has been found to directly affect the mechanical and thermal properties of the nanocomposites. As compared to zero- and one-dimensional nanocarbons like fullerene and carbon nanotubes, graphene nanostructure has an advantageous two-dimensional nanostructure with a light and strong nanosheet

nanostructure and intrinsic charge mobility and permeability features [61]. Therefore, graphene nanocomposites reveal a range of potential applications and remarkable properties, from high-performance nanocomposites to technical nanostructures like membranes. In nanocomposites membranes, graphene has a better alignment, dispersion, porosity, and tortuous pathway formation than one-dimensional nanostructures for better molecular permeability [62]. The polymer-based nanocomposite membranes have been applied for water purification and gas permeation purposes [63–65].

4. Efficiency of graphene nanocomposite membranes for water or gas separations

Owing to the lack of inherent robustness, and structural, and fouling drawbacks, polymeric membranes have been continuously replaced with nanocomposite membranes for better performance [66,67]. Consequently, nanocomposite membranes have been recognized for their controlled and advantageous thermal stability, selectivity, and permeability features [68]. For water remediation applications, solution processing, blading, phase separation, and related membrane fabrication strategies have been focused on [69]. Membrane matrices like poly(vinyl alcohol), polysulfones, nylons, and numerous others [70,71]. Graphene and graphene oxide nanofillers have been used for the development of efficient membranes. Polystyrene, polysulfone, and polyethersulfone have been widely used as ultrafiltration membranes due to their fine strength, durability, pH operating range, and chemical stability [72]. However, their uses in water treatment have been restricted due to their hydrophobicity and related reduced permeability properties. Widely used ultrafiltration polymeric membrane materials have hydrophobic properties. Poly(vinyl fluoride), poly(vinyl chloride), and poly(methyl acrylic acid) have been adopted for these membranes. Membrane hydrophobicity has been found to decrease the water flux due to the organic compound accumulation on the membrane surface. In this regard, polymer modification has been suggested to induce membrane hydrophilicity to enhance the membrane antifouling properties for enhanced water filtration processes. The future of polymer-based water treatment membranes relies on the adoption of new modified polymer matrices as well as nanoparticle nanofillers in the nanocomposite matrices.

Most membranes have been used to remove soluble and non-soluble impurities through the processes of ultra-filtration, reverse osmosis, nanofiltration, microfiltration, etc. [73]. Ultrafiltration membranes have pore sizes of 0.01–0.1 μm , which are smaller than microfiltration membranes. However, these pores are larger than the pores of nanofiltration (0.0001 μm) and reverse osmosis membranes. Nanofiltration has been used to remove small organic molecules, like viruses. Ultrafiltration has been found to remove bacteria, microbes, and suspended solids from the water. Reverse osmosis works like filter media which attracts contaminants. The efficiency of the membrane filtration processes depends on the polymer type, surface functional groups, and physical characteristics of the polymeric membranes. The polymer modification has been used to attain efficient membrane separation processes and desired membrane performance. The modification may involve the incorporation

of copolymers and nanoparticles into the polymeric membranes to form blends or nanocomposites.

For the fabrication of polymer/graphene nanocomposite membranes, efficient techniques have been used [74,75]. The solution-casting technique follows the principle of Stokes' law [76]. In this technique, the polymer is dissolved in a solvent. The nanoparticles are also dispersed in a solvent. Both the polymer solution and the nanoparticle solution are mixed to form a homogeneous phase. Later, the solution phase is evaporated to form a polymer film or membrane. The phase inversion technique has also been focused on polymer/graphene nanocomposite membranes [77]. In this process, the controlled transformation of polymers is performed from the liquid to the solid phase. Consequently, steps like precipitation, controlled evaporation, and immersion precipitation are involved in this method. Furthermore, the polymer/graphene nanocomposites have been formed by interfacial polymerization [78]. Interfacial polymerization involves steps like the oil phase, emulsification, and solvent evaporation. All these technologies have been used to form nanocomposite membranes with finely dispersed graphene and derived nanofillers.

Consequently, poly(vinyl alcohol) and poly(vinyl chloride) matrices have been considered important as important matrices for graphene nanofillers [79–81]. Production and properties of poly(vinyl chloride) and graphene oxide nanocomposite membranes have been produced through the phase inversion method [82]. According to microscopic studies, these membranes revealed a macro-void structure. The nanocomposite membranes were investigated to remove bovine serum albumin from water. The separation performance was observed due to the hydrophilic nature of the membranes. Poly(vinyl alcohol) matrix has been filled with graphene or graphene oxide nanoparticles [83]. These nanomaterials based on poly(vinyl alcohol) and graphene oxide possess hydrogen and hydrophilic binding interactions. Moreover, the membrane design based on poly(vinyl alcohol) and graphene oxide was reported by Castro-Muñoz and researchers [84]. The poly(vinyl alcohol)/graphene oxide-based mixed matrix membranes were prepared using dense-film casting and solvent evaporation methods. The inclusion of 1 wt.% graphene oxide in the membrane resulted in a permeate flux of $0.14 \text{ kg m}^{-2}\text{h}^{-1}$. Sun and co-workers [85] used the pressure-assisted filtration process for the production of a poly(vinyl alcohol)/graphene oxide system. Including 10 wt.% nanoparticles caused superior water flux and salt rejection of $98 \text{ kgm}^{-2}\text{h}^{-1}$ and 99.9%, respectively. Thakur and co-researchers [86] utilized the direct laser writing method to form laser-induced graphene-based three-dimensional porous carbon nanomaterial. Three-dimensional laser-induced graphene had superior electron conductivity. Then, nanocomposite of poly(vinyl alcohol) and laser-induced graphene have been prepared for the formation of membranes. The poly(vinyl alcohol)/laser-induced graphene nanocomposites own fine mechanical, physical, and surface wettability characteristics. **Figure 2** displays a scheme for the development of poly(vinyl alcohol) and laser-induced graphene nanocomposite derived nanocomposite based water treatment membranes for nt. Consequently, the ultrafiltration poly(vinyl alcohol)/laser-induced graphene nanocomposite membranes showed separations of solute particles and bacterial species. **Figure 3** specifies the construction of poly(vinyl alcohol)/laser-induced graphene nanocomposite membranes.

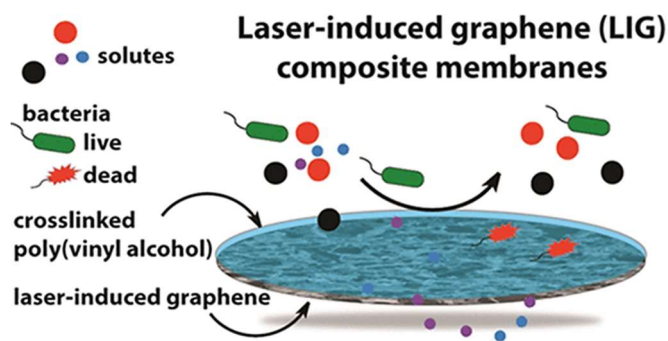


Figure 2. Schematic of poly(vinyl alcohol) and laser induced graphene nanocomposite membranes for water remediation [86]. Reproduced with permission from ACS.

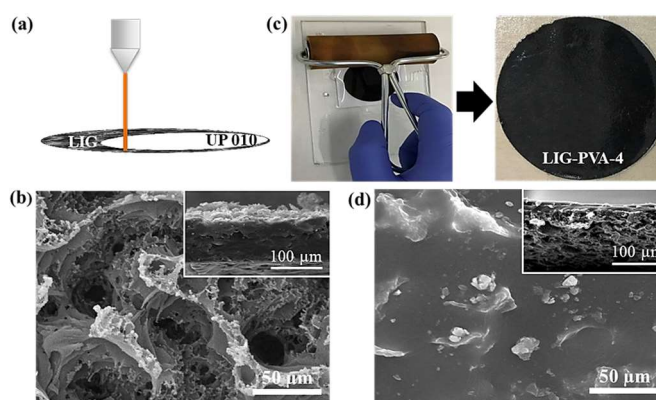


Figure 3. (a) Laser-induced graphene (LIG) is generated on UP 010 membranes through 10.6 μm CO_2 laser; (b) scanning electron microscopy images of as-prepared LIG including cross-section (inset); (c) fabrication technique for laser-induced graphene and poly(vinyl alcohol) (LIGPVA-4) membrane showing excess of poly(vinyl alcohol) (PVA) solution removal with a rubber roller; and (d) scanning electron microscopy images of LIG-PVA-4, including cross-section (inset) [86]. Reproduced with permission from ACS.

Initially, laser-induced graphene was coated on a polyethersulfone substrate. Then, the laser-induced graphene was coated with poly(vinyl alcohol) to form the nanocomposite membrane. Scanning electron microscopic studies on laser-induced graphene and poly(vinyl alcohol)/laser-induced graphene nanocomposite membranes revealed the development of a porous three-dimensional network with consistent pore size distributions. The rejection rate was found to be 99.9%.

The polysulfone and graphene-derived nanocomposites formed some advantageous membrane design combinations [87]. Zinadini et al. [88] reported on polysulfone and graphene oxide-derived membranes. The addition of nanoparticles to the membrane systems resulted in a unique microstructure and high water flux [89]. The polysulfone/graphene oxide-derived membranes revealed a contact angle of 55° – 65° and a water flux of $>20 \text{ kg/m}^2\text{h}$. Hydrogen bonding interactions have also been observed between the polysulfone matrix and graphene oxide, leading to the formation of efficient hydrophilic membranes. Here, the wet phase inversion method has been preferred for the fabrication of polysulfone and graphene oxide-derived membranes [90]. Rezaee and co-workers [91] reported on polysulfone/graphene oxide

nanocomposite membranes using the solution technique. **Table 1** demonstrates the influence of adding graphene oxide amounts on the pure water flux, porosity, and pore structure of the membranes. Enhancing the graphene oxide contents from 0.5 to 1 wt.% enhanced the pure water flux from 20 to 50 L/m²h. The membrane porosity was also enhanced from 78% to 87%.

Table 1. Effect of GO content on pure water flux and pore structure parameters of the prepared membranes [91]. GO = graphene oxide; PSF = polysulfone; PSF/GO = polysulfone/graphene oxide. Reproduced with permission from Springer (Creative Commons CC BY).

Membrane	Pure water flux (L/m ² h)	Porosity (%)	Pore diameter (nm)
Pure PSF	19.7 ± 3.2	48.3 ± 2.6	6.9 ± 0.56
PSF/GO 0.5	32.3 ± 3.5	77.9 ± 2.2	8.3 ± 0.31
PSF/GO 1	49.9 ± 2.6	86.5 ± 1.8	9.1 ± 0.63
PSF/GO 2	46.4 ± 2.0	82.1 ± 2.6	8.7 ± 0.42

Adding 1 wt.% graphene oxide caused higher pure water flux, porosity, and pore diameter properties relative to the neat polymer and other nanofiller loaded membranes. The effect of a change in pH on the rejection rate was studied for membranes with different nanofiller contents (**Figure 4**). Better arsenate rejection performance was observed for 1 and 2 wt.% nanoparticle loading. Consequently, the nanofiller addition caused high separation efficiency due to homogeneous membrane structure, morphology, pore sizes, and optimum porosities [92]. Hence, polymer/graphene and polymer/graphene oxide nanocomposites have been studied for water remediation and filtration [93]. However, these membranes need further research efforts to resolve the challenges of low membrane stability and fouling effects.

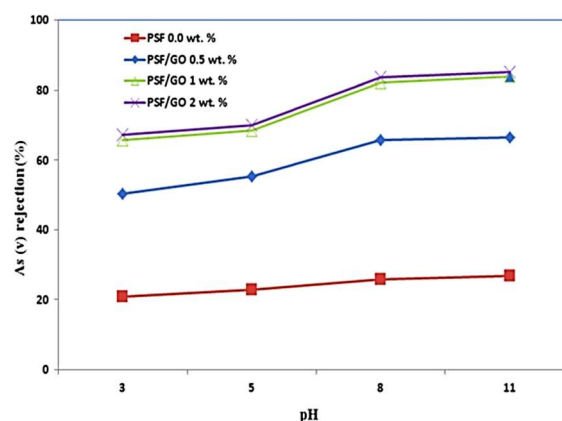


Figure 4. Percentage rejection of As (V) at different pHs by prepared membranes with various graphene oxide (GO) contents. (Operating pressure = 4 bar; initial As (V) concentration = 300 ± 10 µg/L; feed temperature = 25 ± 0.5 °C) [91]. As = arsenate; PSF = polysulfone; PSF/GO = polysulfone/graphene oxide. Reproduced with permission from Springer (Creative Commons CC BY).

Tulugan et al. [94] formed polysulfone/graphene nanocomposite-derived nanofiltration membranes. The water flux of the neat polysulfone membrane (33.2

L/m²/h) was improved with the inclusion of graphene to 183.6 L/m²/h. Moreover, the nanofiltration membranes have a high adsorption rate of 79.8%, relative to neat polymer membranes (26.7%). Alshahrani et al. [95] used the interfacial polymerization method for polysulfone/reduced graphene oxide membrane fabrication. Including 0.015% nanofiller in polyamide led to a water permeability of 48.9 L/m² h, higher than the neat polyamide membrane (25.0 L/m² h). In addition, these membranes have a high salt rejection of 80–95%. Yu et al. [96] developed polyamide-polysulfone membranes through interfacial polymerization. The water permeability of membranes was found to be 48.90 L/m²h at 22 bar, which was superior to the neat polyamide membrane of 25.0 L/m²h. Salt rejection was observed in the range of 80%–95%.

In addition to water permeation or desalination membranes, polymer/graphene membranes have been frequently investigated for gas molecule transportation [97]. Koenig and colleagues [98] formed pristine graphene membranes on a silicon substrate for the separation of H₂ and CO₂ gases. The structure and morphology of membranes have been reported. In addition, membranes have been studied for CO₂/CH₄, CO₂/O₂, and CO₂/N₂ permeation and separation processes [99]. The performances were found to be related to membrane pore sizes as well as affinity towards different molecular species [100]. Subsequently, graphene designs have been investigated for fine gas separations [101]. To improve the properties of graphene towards gas permeation, polymer and graphene-based membranes have been reported [102]. Li and researchers [103] fabricated the polymer/graphene nanocomposite membranes with a pore size of 0.34 nm. The membranes were tested for high selectivity for H₂/CO₂ and H₂/N₂ gases. These membranes still need to be focused on improving pore sizes towards CO₂ sieving [104]. For gas separation membranes, poly(dimethyl siloxane) has been considered [105]. Ultrathin membranes of poly(dimethyl siloxane) have been designed to focus on the carbon dioxide and other toxic gases removal [106]. Nevertheless, pristine poly(dimethyl siloxane) membranes have certain drawbacks due to a lack of structural robustness. In this regard, reports on poly(dimethyl siloxane) and graphene oxide-derived nanocomposite membranes have been found in the literature [107]. Such nanocomposite membranes have been prepared using ultrasonication and solvent-based methods. The poly(dimethyl siloxane)/graphene oxide membranes have fine CO₂ permeability and CO₂/CH₄ separation characteristics. Poly(methyl methacrylate) is a thermoplastic polymer widely applied for membrane applications [108]. Baldanza and researchers [109] produced poly(methyl methacrylate) and graphene-based nanocomposite membranes through a wet deposition technique. The ‘lift-off/float-on’ method was used for the formation of these membranes [110]. **Figure 5** shows the formation of poly(methyl methacrylate) and twenty layers of graphene-based nanocomposite membrane. The membrane thickness and nominal volume fraction were observed at around 550 nm and 0.06%, respectively. According to scanning electron microscopy, a regular lamination sequence was observed. Neat poly(methyl methacrylate) and poly(methyl methacrylate)/graphene membranes were studied for the permeability coefficients of humidified CO₂ and O₂ (**Figure 6**). The nanocomposite membrane had significantly low permeability properties. Adding graphene nanofiller reduced the CO₂ and O₂ permeability coefficients of the membrane to 1.30×10^{-17} and 0.21×10^{-17} mol·m·m⁻²·Pa⁻¹·s⁻¹, respectively (**Table 2**). The property was declined owing to the development of diffusion pathways in the

membrane. These membranes had high permeability coefficients suitable for commercial scale uses of poly(methyl methacrylate)/graphene membranes [111].

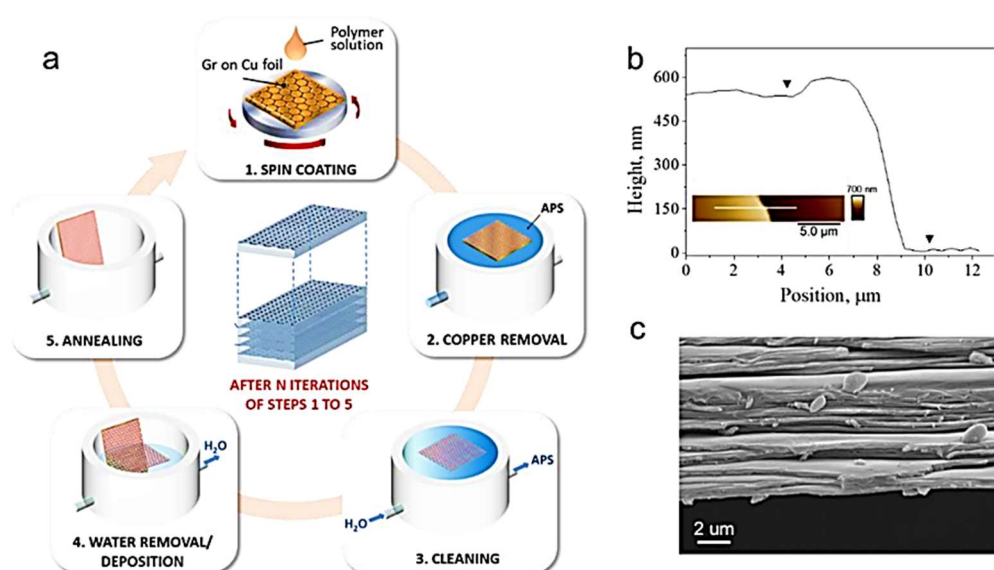


Figure 5. (a) Schematic illustration of the iterative ‘lift-off/float-on’ process combined with wet depositions adopted to produce the Gr-PMMA nanolaminates; (b) Thickness evaluation of the single Gr-PMMA layer deposited on a Si wafer: representative cross-section of the scratch and atomic force microscopy image as inset; and (c) scanning electron microscopy image in the cross-section plane of the nanolaminate [109]. Gr = graphene; Gr-PMMA = poly(methyl methacrylate/graphene nanocomposite); APS = ammonium peroxydisulfate. Reproduced with permission from MDPI.

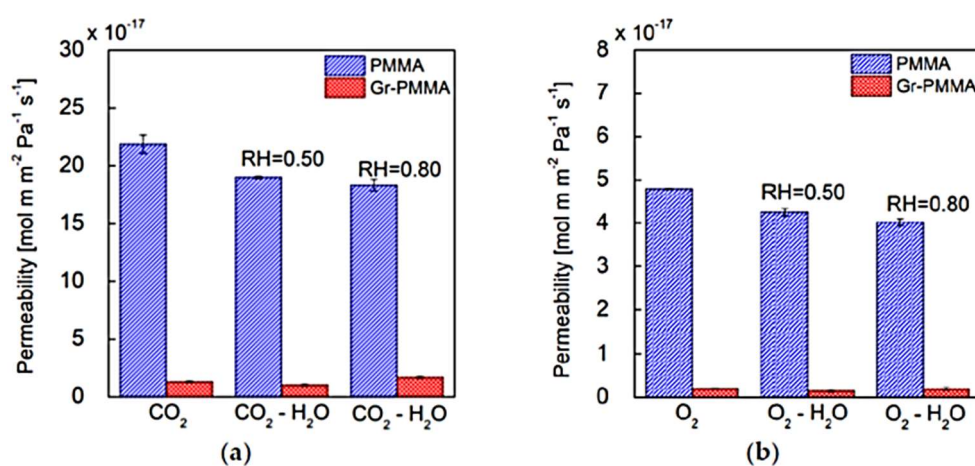


Figure 6. Gas permeability coefficients at 25 °C through poly(methyl methacrylate) (PMMA) (blue bars) and Gr-PMMA (poly(methyl methacrylate/graphene nanocomposite)) (red bars) for (a) CO₂ and humidified CO₂ and (b) O₂ and humidified O₂ [109]. Reproduced with permission from MDPI.

Table 2. Permeability coefficients of CO₂ or O₂ through the nanocomposite at different R.H. levels [109]. PMMA = poly(methyl methacrylate); Gr-PMMA = poly(methyl methacrylate/graphene nanocomposite). Reproduced with permission from MDPI.

Nanolaminate/ permeating gas	P [mol·m·m ⁻² ·Pa ⁻¹ ·s ⁻¹]	P [Barrer]
PMMA/CO ₂	21.9 (±0.8) × 10 ⁻¹⁷	6.5 (±0.2) × 10 ⁻¹
Gr-PMMA/CO ₂	1.30 (±0.1) × 10 ⁻¹⁷	0.39 (±0.03) × 10 ⁻¹
PMMA/O ₂	4.79 (±0.01) × 10 ⁻¹⁷	1.434 (±0.003) × 10 ⁻¹
Gr-PMMA/O ₂	0.21 (±0.01) × 10 ⁻¹⁷	0.063 (±0.003) × 10 ⁻¹

Polysulfone is also an important thermoplastic polymer for gas purification membrane systems [112]. In this context, the mixed matrix membranes of polysulfone have been reported [113]. These membranes have been reported for the separation or selective separation of CO₂ and other noxious gases [114]. Sainath and co-workers [115] produced the mixed matrix polysulfone/graphene oxide membrane for the selective separation of gases. Adding 0.25 wt.% graphene oxide caused 3–4 times higher CO₂/CH₄ selectivity relative to the pristine membrane. The results were obtained due to better nanofiller dispersion and the formation of diffusing routes in the nanocomposite membranes [116]. Gas separation membranes of copolymers have also gained success in gas separation applications such as poly(1-trimethylsilyl-1-propyne)/graphene oxide [117–119] and poly(phenyl sulfonepyridine)/graphene oxide nanocomposites [120]. Similarly, poly(2,6-dimethyl-1,4-phenylene oxide) has been adopted for gas separation membrane matrix [121–123]. Rea and co-workers [124] fabricated the poly(2,6-dimethyl-1,4-phenylene oxide) and graphene-based nanocomposite membranes. The 0.3–15 wt.% nanoparticle contents were filled in the membranes. **Figure 7** shows the morphology of the poly(2,6-dimethyl-1,4-phenylene oxide)/graphene nanocomposite membrane. A few layers of graphene were observed in the polymer matrix, showing layered morphology and fine dispersion.

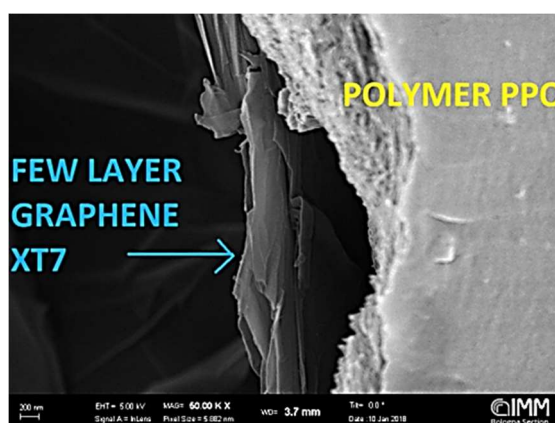


Figure 7. Scanning electron microscopy image of PPO and a few-layer graphene membrane [124]. PPO = poly(2,6-dimethyl-1,4-phenylene oxide). Reproduced with permission from MDPI.

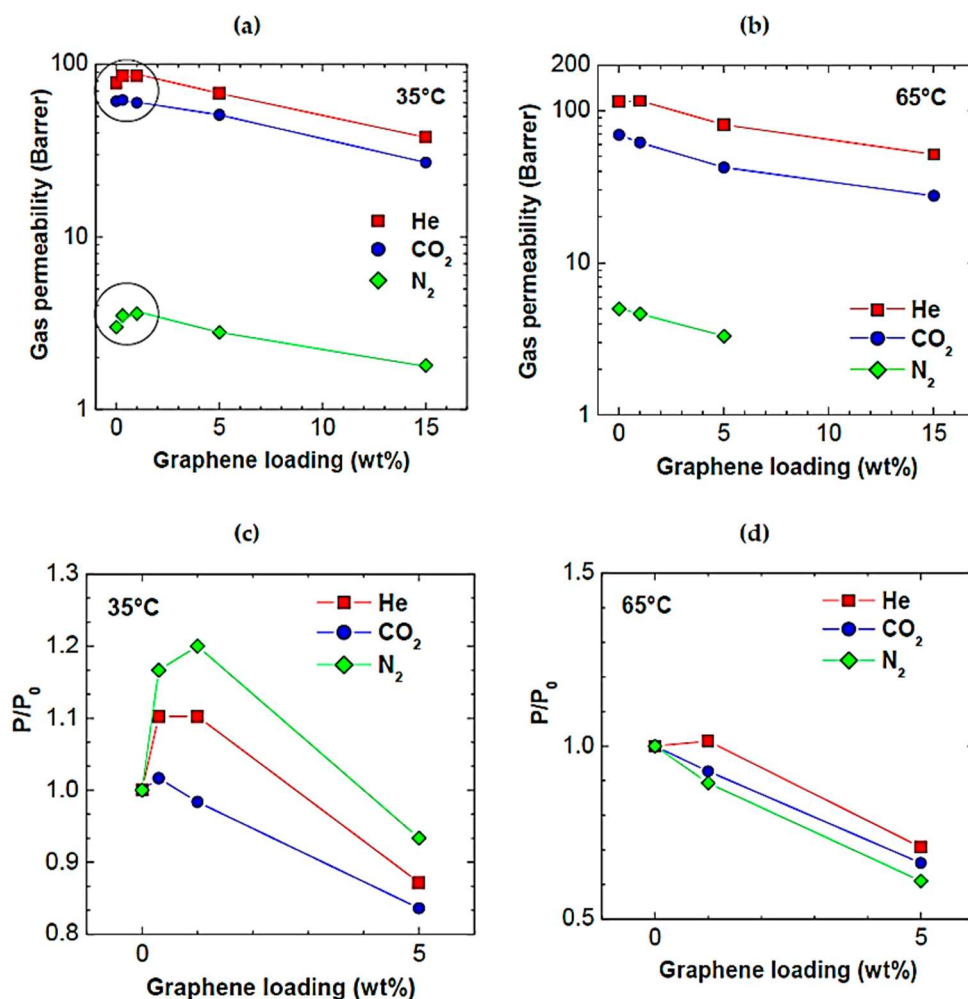


Figure 8. Gas permeability before (a) 35 °C; (b) 65 °C; and after graphene addition (c) 35 °C; (d) 65 °C, as a function of graphene loading in the poly(1-trimethylsilyl-1-propyne) matrix [124]. Reproduced with permission from MDPI.

The gas permeability was found to decrease at 35 and 65 °C with the addition of graphene (Figure 8). The effect on the permeation properties was observed due to the variation in loading level and dispersion in the polymer matrix. Hence, numerous polymeric membranes have been prepared with graphene or graphene oxide nanofillers for gas permeation [125]. The gas permeability and selectivity properties rely on the nanoparticle alignment in the matrices, which may affect the membrane pore sizes and microstructures [126,127].

5. Important prospects

Numerous polymer/graphene nanocomposite membrane systems have been proposed for superior water permeation, desalination, gas separation, and selective separation characteristics. Key points of this review article include the (i) fundamentals of graphene; (ii) fundamentals of polymer membranes; (iii) efficiency of graphene nanocomposite membranes for water remediation or gas separations; (iv) important aspects of the nanocomposite membranes; (v) graphene amount and dispersion; (vi) graphene interactions with membrane matrix; (vii) membrane porosity, morphology, and surface properties; and (viii) membrane permeability and selectivity

properties. Additionally, graphene-based systems have advanced mechanical stability and thermal stability properties. Generally, fine graphene dispersion in matrices has been considered for fine molecular transportation characteristics such as barrier, permeation, and selective separation (**Figure 9**). These membrane properties can be limited due to poor nanoparticle dispersion, surface properties, and imperfect membrane pore formation. Another limiting factor is the fabrication of graphene-based membranes on industrial or commercial levels. The large-scale processing depends upon technique, polymer/nanofiller types, and functionalization affecting the microstructure, durability, and water/gaseous molecular transportation.

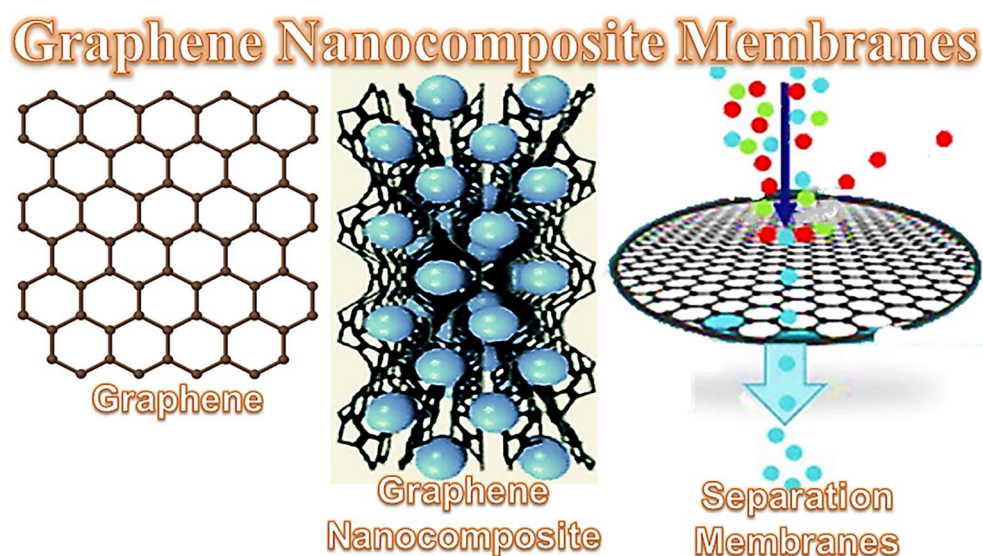


Figure 9. Graphene for gas separation membranes.

According to a literature comparison (**Table 3**), polysulfone nanocomposite membranes have been prepared with carbon nanotubes [128], zeolites [129], and silicon dioxide [130]. The polysulfone/graphene or graphene oxide-based membranes revealed better nanoparticle dispersion, antifouling, water flux, and permeability properties. By comparing the utilization of graphene or graphene oxide nanofillers in the water purification membranes, most of the membranes have been prepared using the graphene oxide nanofiller. The inclusion of graphene oxide or reduced graphene oxide in membranes led to superior water flux, permeability, and rejection properties. The reason seems to be the functionalization of graphene nanosheets, which causes better interactions and dispersion with the polymers relative to neat graphene nanofillers.

Solution casting, phase inversion, and ultrasonication. Techniques that have been frequently used include gas separation polymer/graphene and polymer graphene oxide membranes. As compared to polysulfone/graphene and polysulfone/graphene oxide membranes [115,131], lower CO_2 permeability and ideal CO_2/CH_4 selectivity of 4.2% and 2.7%, respectively, were observed for commercial polysulfone/zeolite membranes [132]. For gas separation membranes, both graphene, graphene oxide, and modified graphene oxide have been applied. A number of studies have been reported on graphene and derivative-based membranes [133]. By comparing various studies on graphene and graphene-derived membranes (**Table 4**), graphene oxide-based

membranes were found to have higher selectivity and permeability values than graphene-based systems. For example, the polysulfone [103] system revealed much higher gas selectivity than the corresponding graphene-based membranes [115,131]. The reason seems to be the nanostructure of graphene, which has impermeability towards molecular passage. However, the formation of graphene oxide or modified graphene nanostructures may cause surface defects, leading to better compatibility, interactions, and interface formation. Consequently, superior permeability and selectivity of graphene oxide-based membranes have been observed. Thus, the polymer/graphene oxide membranes reveal better gas separation properties to overcome the tradeoffs between permeability and selectivity of the nanocomposite membranes. Nevertheless, these membranes are still in their developmental stages, and further studies have been desirable to understand the transport mechanisms as well as the structural specifications.

Table 3. Specifications of polymer/graphene nanocomposite membranes for water separation.

Polymer	Nanofiller	Fabrication route	Membrane properties	Ref
Poly(vinyl alcohol)	Graphene oxide	Mixed matrix membranes; dense-film casting technique; solvent evaporation	Permeate flux 0.14 kg m ⁻² h ⁻¹ ; permeation rate 75%	[84]
Poly(vinyl alcohol)	Graphene oxide	Pressure-assisted filtration process	water flux 98 k gm ⁻² h ⁻¹ ; salt rejection 99.9%	[85]
Poly(vinyl alcohol)	Laser-induced graphene	Ultrafiltration, direct laser writing method	Three-dimensional network; consistent pore size distribution; rejection rate 99.9%	[86]
Polysulfone	Graphene oxide	Phase inversion method	Water flux >20 kg/m ² h; contact angle 55°–65°	[89]
Polysulfone	Graphene oxide	Solution casting	Pure water flux 20–50 L/m ² h; porosity 78%–87%.	[91]
Polysulfone	Graphene oxide	Nanofiltration	Contact angle 7°; water flux 33.2 183.6 L/m ² /h; adsorption rate 79.8%	[94]
Polysulfone	Reduced graphene oxide	Interfacial polymerization	Water permeability 48.9 L/m ² h; permeability 25.0 L/m ² h; salt rejection 80%–95%	[95]
Polysulfone-polyamide	Sulfonated graphene oxide	Interfacial polymerization	Fluorine treatment; F ⁻ retention effect; water flux 83.2%	[96]

Here, research progress in the field of polymer/graphene nanocomposite membranes needs to be analyzed according to the membrane design, type, and specific end application in order to assess the crucial difficulties in this field. Research progress in the field of polymer/graphene membranes can be primarily categorized as desalination or separation membranes for the removal of salts, biological, and organic pollutants by attaining optimally high water flux. The design and essential characteristics of polymer/graphene nanocomposite-based water permeation membranes have been studied.

The nanocomposite membranes have been investigated for their morphological properties, permeability, flux, desalination, and toxin removal.

Table 4. Specs of polymer/graphene nanocomposite membranes for gas separation.

Polymer	Nanofiller	Fabrication route	Physicochemical properties	Membrane properties	Ref
Polymer	Graphene or graphene oxide	Solution casting	Ion-molecule interaction; 1.8–20 nm thickness	H ₂ /N ₂ selectivity 900; H ₂ /CO ₂ selectivity 3400; pore size ~0.34 nm	[103]
Polysulfone	Graphene oxide	Solution route; N-Methyl-2-pyrrolidone solvent	Physical interaction between oxygenated functional groups of graphene oxide and polymer; Interactions between functional groups of nanocomposites and gas molecules	CO ₂ /CH ₄ selectivity ~45	[115]
Polysulfone	Graphene	Phase inversion; hollow fiber mixed matrix membrane	Nanosize synthesized graphene; Interfacial interaction between graphene and polymer matrix	CO ₂ /N ₂ selectivity 158%; CO ₂ /CH ₄ selectivity 74%	[131]
Polysulfone	Zeolite	Mixed matrix membranes	Interlinked morphology	CO ₂ permeability and ideal CO ₂ /CH ₄ selectivity were slightly 4.2% and 2.7%, respectively	[132]
Polyphenylsulfone-pyridine	Graphene oxide	Vacuum infiltration technique	Wettability and surface charge response to pH; acidic pH = 3 form hydrophilic state contact angle 63.3°; alkaline pH = 11 form hydrophobic state contact angle 106.5°; charge-density-tunable nanoporous; power of $\approx 0.76 \text{ W m}^{-2}$	Dispersion; morphology	[120]
Poly(dimethyl siloxane)	Graphene	Solution casting; p-xylene solvent	π - π interactions in matrix-nanofiller	0.2 wt.% nanofiller; N ₂ , CO ₂ , Ar, and CH ₄ permeation 60%; CO ₂ /CH ₄ selectivity 4.2	[134]
Poly(dimethyl siloxane)	Graphene oxide	Solution/ultrasonication methods; tetrahydrofuran solvent	Interfacial interactions between functional groups of graphene oxide and polymer; density 1.09–1.12; Thickness 1.9–2.8 nm	5 wt.% nanofiller; CO ₂ /CH ₄ selectivity 112%; CO ₂ permeability 29%.	[107]
Poly(dimethyl siloxane)	Graphene oxide	Solution casting	Matrix-nanofiller interactions; interaction between graphene oxide and polymer	8 wt.% nanofiller; H ₂ , O ₂ , N ₂ , CH ₄ and CO ₂ permeability 99.9%	[135]
Poly(methyl methacrylate)	Graphene	Wet deposition method	Water adsorption, membrane wrinkles; degree of dispersion/orientation of the graphene nanosheet, structure organization of polymeric chains at the interface with graphene nanosheet	CO ₂ permeability coefficient $1.30 \times 10^{-17} \text{ mol} \cdot \text{m} \cdot \text{m}^{-2} \cdot \text{Pa}^{-1} \cdot \text{s}^{-1}$; O ₂ permeability coefficient $0.21 \times 10^{-17} \text{ mol} \cdot \text{m} \cdot \text{m}^{-2} \cdot \text{Pa}^{-1} \cdot \text{s}^{-1}$	[109]

Table 4. (Continued).

Polymer	Nanofiller	Fabrication route	Physicochemical properties	Membrane properties	Ref
Poly(1-trimethylsilyl-1-propyne)	Graphene oxide	Solution casting; chloroform solvent	Anchoring of graphene oxide nanosheets lowers membrane flexibility; and less free volume; covalent cross-linking of polymer	1 wt.% graphene; diffusion coefficient of CO ₂ (25%); N ₂ (14); CH ₄ (9%)	[118]
Poly(1-trimethylsilyl-1-propyne)	Graphene	Solution route	Interaction between filler and polymer matrix; 0.93–1.36 MPa; 38–44 MPa	0.05 wt.% nanofiller; CO ₂ permeability 3.5×10^3 Barrer	[119]
Poly(2,6-dimethyl-1,4-phenylene oxide)	Graphene	Solution route	Void formation at the interface; glassy polymer filled with graphene; graphene inclusion for the physical constraint to relaxation of polymer chains	0.3–15 wt.% nanofiller reduced permeability	[124]

The mechanical properties like flexibility, strength, toughness, and other important properties of membranes have been deliberated. Graphene nanoparticle dispersion has been found to be important in enhancing the matrix-nanofiller interactions and improving the final membrane characteristics. In this context, compatibility between the polymer and graphene nanoparticles may cause better nanoparticle dispersion and miscibility effects. The molecular diffusion and permeability properties rely on the pore size, shape, and nanoparticle dispersal in the polymeric matrices. All these properties not only affect the selectivity/permeability features but also the membrane strength and functional life for membrane applications. Major challenges identified in this sector have been found to be complications owing to poor nanoparticle dispersion, phase separation, optimum fabrication parameters, and the identification of perfect membrane designs for commercial-scale production of these membranes. In this way, desirable barrier effects can be achieved for selective molecular transportation through the membranes to separate the salts, toxic ions, biological species, and other toxins. Thus, not much research has been observed regarding the separation mechanisms and overcoming the challenges of fabricating well-defined designs for commercial-level use. Future research in the mentioned directions will be beneficial for the formation of efficient water separation membranes.

Secondly, an important application of polymer/graphene nanocomposite membranes has been observed for gas separation. Here, matrices like polysulfone, poly(dimethyl siloxane), poly(methyl methacrylate), and other block copolymers have been used and filled with graphene nanofillers using facile solution, sonication, phase inversion, infiltration, and other techniques. For this application, efficient design combinations have been observed for separating the toxic or desired gas from gaseous mixtures. The resulting membrane must have optimum porosity, permeability, selectivity, and other membrane features for the separation of molecular species. As discussed above, all the membrane characteristics have been found to be dependent upon the processing, nanoparticle alignment, functionality, and compatibility with the polymer phases. By controlling all these features, complex gas mixtures can be separated using novel membrane designs. In addition, membrane thicknesses have also

been found to be important to control not only the gas transport and flux characteristics, but also the membrane durability and cyclic performance. For gas separation, identification of the perfect processing technique, membrane parameters, and ultrathin polymer/graphene nanocomposite membrane formation have been found to be limiting factors or challenges. In this field, there is a lack of research regarding the separation mechanisms, structure-property relationships, and well-defined membrane designs for the separation of specific gaseous pollutants. Hence, due to a lack of targeted research in this area, desired permeability, selectivity, and working life have been found challenging. More focused research efforts have definitely been needed in these directions to form high-performance membranes through facile processing with well-defined parameters.

6. Conclusions

Hence, this article presents the gas separation performance of polymer/graphene nanocomposite membranes, keeping in view the important literature reports. Graphene as well as modified graphene nanoparticles have been filled in the nanocomposite membranes. The resulting membrane systems have been analyzed for fine water and gas molecular separation as well as permeation properties. The polymer/graphene nanocomposite membranes have been examined for the nature of pores, microstructure, sturdiness, and gas or water molecular separation efficiencies. Various combinations of polymers and graphene, or modified graphene, have been developed for the formation of efficient membrane systems. Nevertheless, there are several challenges in the way of the formation and application of polymer/graphene nanocomposite membranes. The related challenges may be comprised of polymer type, nanoparticle modification, nanoparticle dispersion, and nanoparticle interaction with the polymer. Despite the advantages, there are numerous problems limiting the rapid development of graphene-based nanocomposite membranes. Even though facile solution methods have been used, no perfect design with all the defined parameters, high surface area, and even thickness has been identified so far for large-scale functioning. Facile methods have been found to be ineffective in producing membranes with all defined membrane parameters on a large scale. Pore clogging and membrane fouling (biofouling, micro fouling, macrofouling) due to the presence of organic/inorganic pollutants (dyes, metal particles, microbes, bacteria, etc.) have been found to prevent rapid water purification. Hence, the development of evenly structured ultrathin, enduring, lightweight, low price, antifouling, and extended life polymer/graphene membranes has been found difficult to attain for large-scale commercial systems. Overcoming all these challenges may yield fine future opportunities for high-tech, commercial-grade graphene-filled membranes.

Thus, the research progress on graphene nanocomposite membranes led to several advances in types, designs, and applications to overcome the challenges in this field. For an increase in physical properties, nanoparticle dispersion has been found to be important for matrix-nanofiller interactions, microstructure, mechanical features, and for advanced membrane characteristics. Consequently, the compatibility of matrix graphene has been recommended to improve for better miscibility and reinforcing effects. The membrane performance also depends upon the pore shapes, sizes, and

distribution in the matrices. The random nanofiller dispersion or pore distribution in membranes may influence the strength, durability, and life of the membranes. For commercial-scale membrane production, membrane design features must be analyzed. Hence, future research must resolve the challenge of identifying directions for high-performance gas separation membranes.

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