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Electrospinning Polymers of Intrinsic Microporosity (PIMs) ultrafine fibers; preparations, applications and future perspectives

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Polymers of Intrinsic Microporosity (PIMs) are increasingly recognized as membrane materials for molecular separation applications due to their unique structural and functional properties. Development of electrospun PIMs further improved the practical use of PIM polymers. PIM nanofibers produced by electrospinning could be an effective membrane material to handle various environmental concerns owing to their high surface area, high hydrophobicity and high adsorption abilities. In addition, highly selective electrospun PIMs could be produced by simple modification methods to obtain specific interactions with desired species. Versatility of electrospun PIMs provides a unique advantage of producing various novel fibrous membranes by electrospinning method for a range of potential applications. Therefore, this review aims to discuss the recent progress of electrospun PIMs, their properties and future directions in various applications.

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Introduction

The invention of Polymers of Intrinsic Microporosity (PIMs) was achieved almost two decades ago, and since that time they have sparked a significant interest in developing functional polymer membranes [1]. PIMs are a class of polymers that could be produced by incorporating highly rigid contortion centers into polymer chains while preventing the conformational freedom. This unusual molecular design enables the production of solution processable polymers with high free volumes and interconnect micropores [2]. PIM-1, is the first synthesized PIM polymer, shows a decent separation ability in various membrane separation applications [3,4]. The success of PIM-1 in separation facilitates a considerable achievement in the synthesis of numerous PIM polymers [5–9]. While the major focus was producing dense membranes of PIMs until 2014, a new focus has emerged after the introduction of PIM-1 fibrous membrane by electrospinning technique [10].

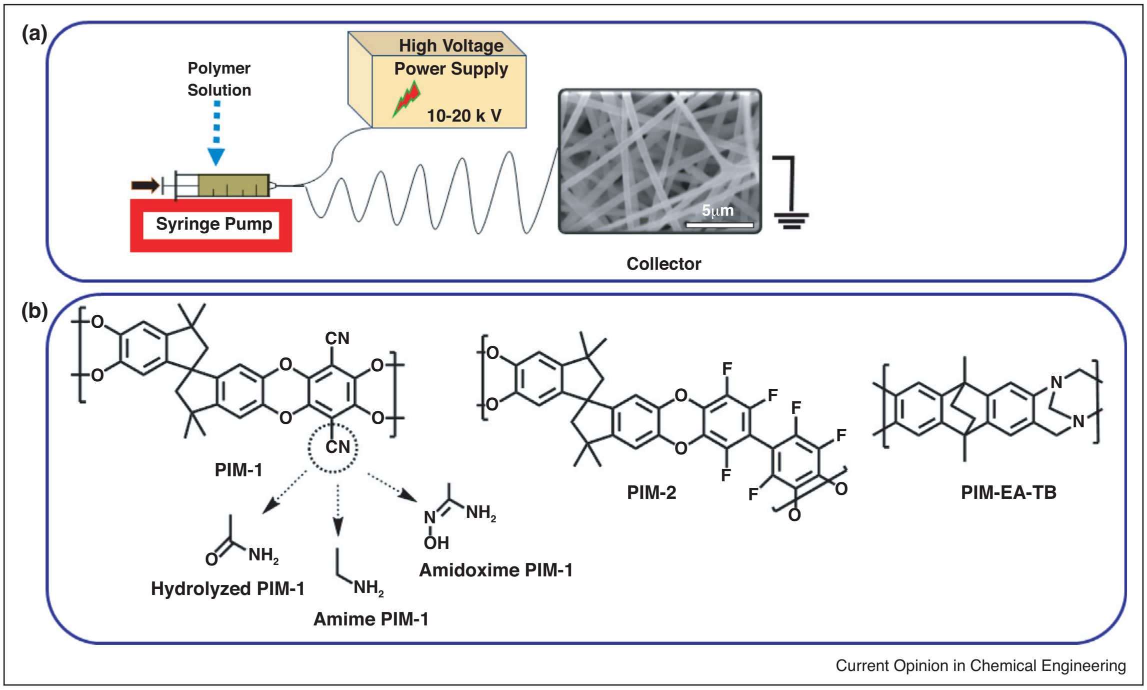
Electrospinning is a straightforward method to produce ultrafine nanofibers from a range of polymers [11]. Polymer nanofibers produced by electrospinning could have a huge potential in dealing with various challenges, including environmental, health and energy owing to their characteristic features such as high interconnected porosity, high surface area and lightweight [12]. The development of electrospun PIM nanofibers has become a research hotspot since the remarkable features of PIMs such as high porosity, hydrophobicity, and the affinity for organic species could be exploited more efficiently in the fibrous membrane form than that of dense membrane form [13–15]. Hence, electrospun PIM nanofibers have enhanced the practical use of PIMs in the past two-three years. This review will overview the recent progress of electrospun PIM nanofibers, their preparations, applications and future perspectives.

Electrospinning Polymers of Intrinsic Microporosity (PIM) nanofibers

Electrospinning is a simple and cost-effective method that exploits the interaction between the liquid and the electrical field to produce fibers with diameters from a few nanometers to micrometers within a short time. Basic electrospinning set-up is composed of high voltage power supply, syringe pump, a spinneret with needle nozzle and a collector (Figure 1a). Several parameters could affect the properties of nanofibers, such as polymer solubility and molecular weight, solution concentration and conductivity, applied voltage, flow rate, distance, temperature and humidity. These parameters are strongly related to each other and, thus, changing one parameter may require changing a range of parameters. Using an appropriate solvent has the paramount importance to produce nanofibers as it directly influences the surface tension and the conductivity of the polymer solutions [11].

PIMs exhibit excellent solubility in several common organic solvents, and they could be modified for a specific

Figure 1



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(a) Basic electrospinning set up, **(b)** chemical structures of electrospun PIMs.

target by straightforward chemical modifications [16,17]. These features make them promising candidates for electrospinning. Although various PIM structures have been reported in the form of dense membranes for a broad range of applications, only a few PIMs have been produced in the form of electrospun fibrous membranes [10,13–15,18]. Electrospinning Polymers of Intrinsic Microporosity was first achieved by Bonso *et al.* [10]. They managed to produce electrospun PIM-1 nanofibers using tetrachloroethane (TCE) solvent. Afterwards, other groups focused on the electrospinning of PIM-1 by using same solvent as well as using different solvent mixtures due to the toxic nature of TCE solvent [19,20]. Convenient synthesis of PIM-1 from commercially available monomers made it appealing for various studies in a short time [21–24]. Meanwhile, electrospinning modified PIM-1 has also attracted attention, as the affinity of PIM-1 could be easily tailored by modifying the nitrile group in the polymer backbone [13,15,18,25]. Additionally, electrospinning PIM-2 and a new generation PIM polymer PIM-EA-TB have been accomplished recently [14,26]. Chemical structures of electrospun PIMs are depicted in Figure 1b and Table 1 summarizes the electrospinning studies performed on PIM polymers, electrospinning parameters and the properties of electrospun PIMs.

PIMs exhibit high surface area and hydrophobic nature, which makes them suitable for adsorption and separation applications. These properties could be further improved by producing nanofibers with a smaller diameter. PIM nanofibers obtained from various studies reveal a broad range of average fiber diameters from 0.7 to 10 μm as displayed in Table 1. This significant disparity is mainly originated from the different precursor polymers. Depending on the molecular weight of the polymer, electrospun PIM-1s and modified PIM-1s could be produced between 0.5 and 2.5 μm diameter range without the necessity of any additives. Average fiber diameters of electrospun PIM nanofibers could be further reduced up to 160 nm by the addition of tetraethyl ammonium bromide salt into spinning solution [33]. Moreover, the temperature and the relative humidity have a negligible effect on the electrospinning PIM polymers, facilitating convenient use of these polymers in the electrospinning applications [33].

The hydrophobic nature of electrospun PIMs could be further improved by using highly fluorinated PIM polymer, PIM-2, which shows a superhydrophobicity with a water contact angle of 155 ± 6°. Superhydrophobicity could also be attained by chemical modification [34]. These membranes have an excellent ability to repel water and separate organic compounds and oils from water mixtures. High surface area usually arises from the structure of PIMs; however, it could be affected from the spinning conditions. While some groups claimed the BET surface area of electrospun PIMs are higher than that of powder forms. In some studies, electrospun PIMs

Table 1

Summary of the electrospinning studies performed on PIM nanofibers and the properties of electrospun PIMs

Electrospun PIM sample	Solvent[a]	Concentration (wt %)	Applied voltage (kV)	Distance (cm)	Flow rate (mL/h)	Collector (S/R-rpm)[b]	Average fiber diameter (μm)	Contact angle (θ)	BET surface area ($m^2 g^{-1}$)	Reference
PIM-1	TCE	10	10–15	n.a.	1–2	R-300	1.7 ± 0.3	n.a.	546	[10]
		10	10–12	17	0.6	R-100	1.7	132 ± 8	n.a.	[21]
		7–10	10–12	17	0.6	R-100	1.7	n.a.	1114	[22]
		10	10–12	17	0.6	R-100	n.a.	n.a.	n.a.	[27]
		23	11–12	18	0.5	R-2000	2.07 ± 0.5	134 ± 8	767	[23]
		10–15	10–15	15–20	3.6	n.a.	5–7	n.a.	545	[24]
		8–12	14–18.2	20	1	R-600	1–10	n.a.	n.a.	[28]
	THF/DMF (9:1)	5–10	15–25	8–16	5–10	S	2–5	135	n.a.	[19]
		10	15	15	2	S	n.a.	n.a.	650	[29•]
	TCE/THF(7:3)	n.a.	16	15	3	S	n.a.	n.a.	712	[30•]
	THF/toluene	5	10–15	15	1–2	S	1.8–4.6	n.a.	660–745	[20]
Hydrolyzed PIM-1	DMF	40–120	10–15	10–15	0.3–0.6	S	0.76 ± 0.09–1.21 ± 0.15	n.a.	25–320	[18]
Amidoxime PIM-1	DMF	40	12	15	0.5	S	1.69 ± 0.34	128 ± 7	306	[13]
		40	10–20	20	n.a.	S	1.7	n.a.	605	[31•]
	DMSO	15–30	15–25	10–20	0.12–0.9	S	0.7–1.4	121–132	n.a.	[32]
PIM-2	TCE	43	12	18	0.6	R-1000	5.5 ± 1.5	155 ± 6	580	[14]
PIM-EA-TB	$CHCl_3$/n-Propyl lactate (10:0–5:5)	20	16–25	10–30	0.6–6	S	4.7–7.9	126	n.a.	[26]

[a] Tetrachloroethane (TCE), tetrahydrofuran (THF), dimethylformamide (DMF), dimethyl sulfoxide (DMSO).

[b] S; Stationary collector, R; Rotating Collector and the numbers represent rotation speed of the collector (rpm).

show similar or lower BET surface area compared to their powder forms [13,18,23]. The discrepancy is possibly due to the difference in spinning conditions; thus, a range of BET surface have been reported for electrospun PIMs as shown in Table 1. In addition to these remarkable properties, PIMs exhibit high thermal stability and high char yields which make them particularly interesting candidate for all types of electrochemical applications [10,35,36]. Therefore, research interest has also been directed to the production of carbon electrodes from electrospun PIM nanofibers [37–39,40•].

Applications of electrospun PIMs

PIMs have attained considerable attention because of their exceptional adsorption and separation abilities, and PIM-1 has always engrossed most of this attention due to its remarkable affinity to organic species. It takes up a significant amount of gas molecules and small molecules from liquid media. This affinity can be tailored for desired molecules with the proper modification methods. Satilmis *et al.* [17] revealed that neutral affinity of PIM-1 could be directed towards cationic species by simple hydrolysis and it could also be shifted to anionic species with amine and ethanolamine modifications [6,41]. The idea of producing a solution processable adsorbent with high adsorption capacity along with a high selectivity makes these polymers perfect candidates for water treatment applications in various forms. However, as it is the case for most polymeric membranes, dense membranes have intrinsic limitations, as they tend to show low flux, high energy cost, and high fouling potential. Electrospun fibrous membranes could overcome these limitations and exceed the performance of conventional dense membranes [12]. Therefore, the major focus became the adsorption applications for electrospun PIMs in their as-spun forms. Additionally, their thermal stability coupled with high surface area enabled their use in carbonized forms for electrochemical applications. Table 2 presents the studies performed on electrospun PIMs, the composition of electrospun fibrous membranes and their applications in their as-spun and carbonized forms.

Adsorptive properties of electrospun PIMs was first investigated by Zhang *et al.* [21]. They prepared a series of electrospun PIM-1/polyhedral oligomeric silsesquioxane (POSS) membranes to enhance the hydrophobicity of PIM-1 and successfully used these membranes to remove oily products from water. They also showed that both PIM-1 and PIM-1/POSS membranes could adsorb organic contaminants from organic solvents [21,22]. Following that, Satilmis *et al.* [23] investigated the removal of aniline from air and water by using various forms of PIM-1 samples including powder, dense and fibrous membranes. The study showed that the aniline adsorption capacity of PIM-1 fibers was greater than that of PIM-1 powder and dense membranes from air. The maximum aniline adsorption capacity of PIM-1 fibers was found to be 818 mg g^{-1} from air. Besides, they also demonstrated that the adsorption occurs much faster in PIM-1 fibers than the PIM-1 dense membrane when the adsorption is performed in water. Aniline removal ability of PIM-1 fibers

Table 2

Composition of electrospun PIMs and their applications

Form of the electrospun PIMs	Composition of fibrous membrane	Application	Reference
As-spun	PIM-1/POSS	Adsorption of oil soluble contaminants, oil–water separation	[21]
	PIM-1	Adsorption of dyes from non-aqueous media	[22]
	PIM-1	Adsorption of aniline from air and water	[23]
	PIM-1	Adsorption of carbendazim and phenol from methanol	[24]
	Pd coated PIM-1	Reduction of nitroaromatic compounds	[28]
	PIM-1/PAN/MOF	Adsorption/filtration and catalysis	[29•]
	PIM-1	CO_2/N_2 adsorption	[20]
	PIM-1/MOF	Hydrogen adsorption/storage	[30•]
	HPIM-1	Adsorption of dyes and heavy metals from water	[27]
	HPIM-1	Adsorption of dyes from water	[18]
	HPIM-1/HMDI	Adsorption of organic compounds and oil/water separation	[34]
	ZnO decorated HPIM-1	Adsorption and photocatalytic degradation of organic compounds from water	[42,43••]
	Amine PIM-1	Adsorption of organic compounds from water	[15]
	Amidoxime PIM-1	Adsorption of uranyl ions from water	[13]
	Amidoxime PIM-1	Air filtration	[32]
	Amidoxime PIM-1	Detoxifying organophosphorus and SO_2 adsorption	[31•]
	PIM-2	Adsorption of organic compounds	[14]
	PIM-EA-TB	Air filtration	[26]
Carbonized	c-PIM-1	Supercapacitor	[10]
	Pt decorated c-PIM-1	Gas diffusion electrode for polymer electrolyte membrane fuel cells	[37,39]
	NiOOH/Ni(OH)2 decorated c-PIM-1	Electrochemical water splitting	[38]
	c-PIM-1, c-Hydrolyzed PIM-1, c-Amine PIM-1, c-Amidoxime PIM-1	Catalysis of oxygen reduction reaction	[40•]

from water was found to be 161.2 mg g^{-1} which indicates that the adsorption performance of PIM-1 fibers could compete with some high-performance resins in aniline adsorption. Decontamination studies further continued using PIM-1 fibers to remove carbendazim and phenol contaminants from liquid media [24]. High adsorption capacity of PIM-1 fibers was further facilitated by decorating PIM-1 fibers with palladium nanoparticles to catalyze the p-nitrophenol reduction into p-aminophenol structure. This approach has shown that catalytic activity of Pd decorated PIM-1 fibers was greater than that of two high-performance commercial polymers [28]. Wang *et al.* [29•] used layer by layer spinning method to produce PIM-1/PAN/UiO-66 fibrous membranes to obtain excellent particle filtration efficiency. The same group also produced porous PIM-1 fibers using solvent/non-solvent mixtures during the electrospinning [20]. The produced porous PIM-1 fibers showed excellent CO_2 adsorption/desorption stability indicating the potential of PIM-1 fibers in gas capture. On the other hand, Bambalaza *et al.* [30•] have performed a different approach to exploit PIM-1 fiber in gas capture. They produced a monolith structure by compressing UiO-66 particles with PIM-1 fibers under high pressure and the resulting product showed enhanced H_2 uptake at high pressure.

Electrospinning PIM-1 fibers has still some limitations due to the toxic spinning solvent (TCE) used to obtain smooth fibers. At this point, electrospun modified PIM-1s were introduced not only to avoid toxic spinning solvent but also to tailor the adsorptive properties of PIM-1 fibers. Satilmis *et al.* [18] reported the systematic hydrolysis of PIM-1 (HPIM-1) and electrospinning HPIM-1 fibers with various degree of hydrolysis. The membranes were subsequently used to filtrate cationic dye; methylene blue from water by only using gravity as a driving force. They have also tailored the selectivity towards anionic species by producing electrospun amine modified PIM-1 fibrous membrane [15]. Morphology of amine PIM-1 fibers showed extreme stability after several adsorption/desorption cycles due to the insolubility of the fibers. The same group also used a crosslinker during the electrospinning to improve the properties of HPIM-1 fibrous membranes [34]. Resulting insoluble membranes showed superhydrophobic characters and they were successively used in organic adsorption and oil–water separation applications. HPIM-1 fibers were further used as a template to grow ZnO nanorods by atomic layer deposition (ALD) method. Ranjith *et al.* [43••] facilitated the adsorptive properties of HPIM-1 in photocatalytic degradation of organic contaminants. Electrospun amidoxime PIM-1 fibers also showed promising results in uranyl ion removal from water and air filtration studies [13,31•,32]. Recently, highly fluorinated superhydrophobic PIM-2 fibrous membrane was also introduced [14]. The organic and oil adsorption capacity of fibrous membrane was greater than that of dense membrane in liquid adsorption. Furthermore, it is also possible to produce flexible, self-standing and high surface area carbon nanofibers from electrospun PIMs [10,37,38,40•]. Electrospun carbonized PIMs have been used in various electrochemical applications as summarized in Table 2. The applications of electrospun PIMs are not limited to these examples. It could be further improved by blending with other organic/inorganic molecules, polymers, and nanoparticles to meet the requirements of various other industrial applications such as biotechnology, food processing, textile and sensor.

Conclusions and future directions

Electrospun PIM nanofibers have great potential to provide some unique solutions in dealing with emerging environmental challenges such as air and water pollutions owing to their excellent molecular separation abilities. Although the development of electrospun PIMs is still in its early stages, recent studies have shown the potential of electrospun PIMs in various separation and purification applications. Highly porous, self-standing and flexible fibrous membranes of PIMs could also have a bright future in electrochemical applications. Developing electrospun PIMs with superior properties could be possible by incorporating other high-performance materials with PIM structures in the future. Currently, the major limitation of these membranes is their lab scale productions. Industrial productions and applications of these membranes are still prevented as they do not fully correspond to the requirements of green chemistry. Further studies should be performed to produce simple, cost-effective and large-scale synthesis of PIMs in a green solvent. Also, electrospinning PIM nanofibers from environmentally friendly solvents should be explored along with their performance, stability and reusability evaluations for industrial applications.

Conflict of interest statement

Nothing declared.

Data availability

No data was used for the research described in the article. Data will be made available on request.

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- •• of outstanding interest

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In this paper, the authors successfully decorated HPIM fibers with ZnO nanorods by using atomic layer deposition technique. Resulting composite membrane not only adsorbs organic contaminants from water but also enables the photocatalytic degradation of those contaminants.