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MINI REVIEW

Emerging Approaches in Membrane Preparation from Phase Separation to 3D Printing

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Membrane technologies are receiving increasing attention as effective solutions to global challenges such as water treatment, energy efficiency and environmental protection. This mini-review summarizes advances in polymeric materials and membrane fabrication methods. A wide variety of polymeric and non-polymeric materials, ranging from natural clays to synthetic polymers like polysulfone, are used to fabricate membranes for applications including reverse osmosis and ceramic separations. Fabrication techniques such as non-solvent induced phase separation (NIPS), thermally induced phase separation (TIPS), vapour-induced phase separation (VIPS), and liquid-induced phase separation (LIPS) enable the production of microporous membranes, while electrospinning is employed to create ultrathin fibrous structures. In addition, emerging 3D printing technologies allow precise control over membrane architecture, including pore size and porosity, supporting the development of next-generation membrane systems.

Keywords: Membranes, Materials, Phase separation, Electrospinning, 3D Printing technology.

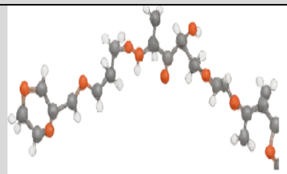
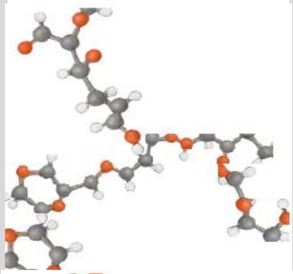
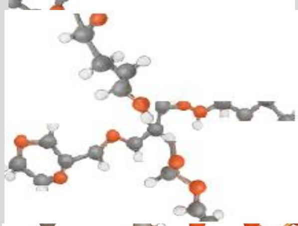
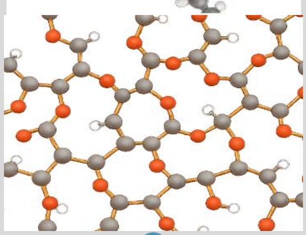
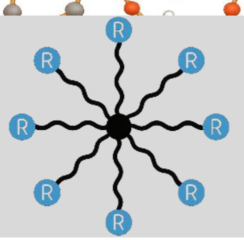
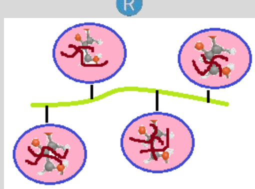
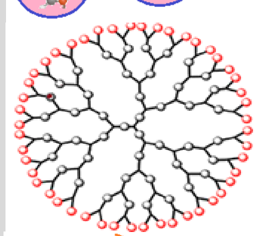
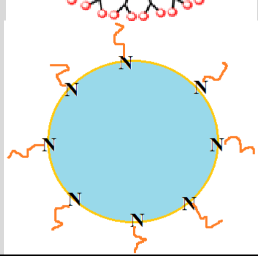
INTRODUCTION

As the world grapples with pollution, water scarcity and electromagnetic chaos, polymeric membranes are stepping out the lab and into the spotlight-powering solutions as thin as a micron but as vast as global challenges. Today, polymers are ubiquitous, encompassing plastics, fibers, rubbers and more. Polymeric materials used in water purification can be broadly categorised into several types, each with distinct characteristics and applications [1]. These include synthetic polymers, biopolymers and composite materials [2-4]. Synthetic polymers such as polyacrylonitrile (PAN), polystyrene (PS) and polyethylene glycol (PEG), are widely used due to their robustness and ease of modification [5-7]. Biopolymers like chitosan and alginate are derived from natural sources and offer biodegradability and low toxicity, making them environmentally friendly alternatives [8,9]. Polymeric composite materials have the advantages of superior mechanical properties, high thermal stability, impact resistance, outstanding abrasion resistance, exceptional electrical insulation and high rigidity [10]. Polymers having diverse applications makes huge impact on global economy as shown in Fig. 1, which illustrates the global polymers market forecast, highlighting the significant role of polymers in economic context [11].

The word 'Polymer' is derived from the Greek words 'poly' meaning many and 'meros' meaning parts [12]. When considering the diverse properties and applications of polymers, it is essential to understand their various architectures [13]. Polymers can be classified based on origin, response to temperature, mechanical properties and other criteria. In Table-1, different polymer architectures are summarized along with their notable applications.

Within this broader polymer framework, membranes [42] represent one of the most important functional material classes. Membranes may be polymeric [43] or non-polymeric [44] in nature; however, polymer-based membranes dominate most industrial and research applications due to their structural versatility, ease of processing, and tunable physico-chemical properties. The intrinsic link between polymer architecture and membrane performance is evident as parameters such as pore size, permeability, selectivity and mechanical stability are strongly governed by the underlying polymer structure.

TABLE-1
LIST OF DIFFERENT POLYMER ARCHITECTURES AND THEIR APPLICATIONS

Type	Schematic architecture	Properties	Application	Example
Linear polymer [14]		Long chain, high crystalline, strong intermolecular forces [15]	Self-healing capability [16], prodrug chemistry [17]	Polystyrene
Branched polymer [18]		Degree of branching affects rheology, crystallisation, Tg and degradation [19]	Branching architecture offers a lever to tailor optical behaviour for optoelectronic applications [20]	Polyazomethines
Cross linked polymer [21]		High dimensional stability, low creep, solvent resistance, high heat distortion and softening temperature [22]	Useful in organic photovoltaic [23]. Hyper cross-linked polymer useful in gas storage, catalysis, separation [24]	Epoxy resin
Network polymer		Hard and brittle [25]	Dielectric elastomers, nanogenerators, energy storage device [26]	Bakelite
Star polymer [27]		Low viscosity on dilution, encapsulation capability, peripheral functionality, hydro dynamic volume, good flexibility [28]	Drug delivery applications, encapsulation of smaller molecules [29]	Pentaerythritol
Comb polymer [30]		Stretchability, low viscosity, lesser melting point, more compact [31]	Specific flow property applications [32], rheological & mechanical applications [33]	Poly(styrene-graft-butadiene)
Dendrimers [34]		Polyvalency, self-assembly, pharmacokinetic, electrostatic interaction, low viscosity [35]	In nanomedicine & drug delivery [36], genomic editing [37], biomedicine [38]	Polyamidoamine
Cyclic polymers [39]		Smaller hydrodynamic volume, higher T_g, lower intrinsic viscosity, self-assembly behaviour [40]	As biomaterials, viscosity modifiers, nanoelectronic applications [41]	Cyclic poly(dimethylsiloxane)

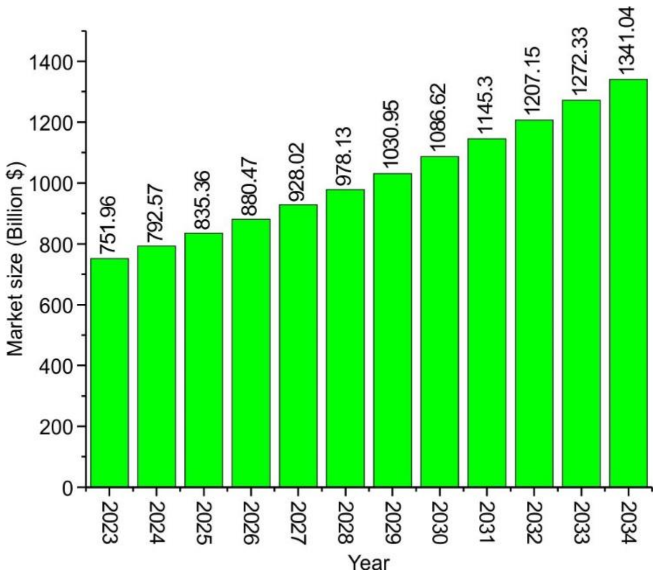


Fig. 1. Global polymers market size and forecast 2023 to 2034

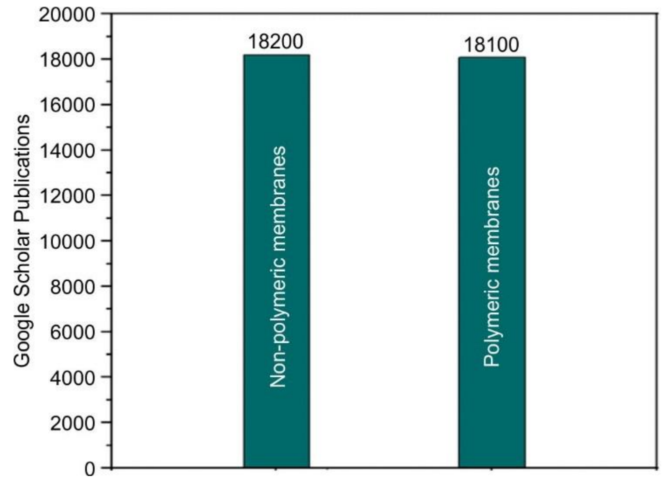


Fig. 2. Trends in membrane preparation methods based on a survey of Google Scholar publications

Consequently, a wide range of polymers are specifically designed and processed into membranes for targeted applications, as summarized in Table-2.

Preparation of membranes: Table-2 indicates that membranes can be prepared from diverse materials. Interestingly, when considering last decade (2015-2025) the prompt 'membranes prepared from non-polymer' shows more publications on Google Scholar than the prompt 'membranes prepared from polymer,' as shown in Fig. 2 [67,68]. According to Lakshminarayanaiah (1965), in simple terms, membranes refer to a typically heterogenous phase that serve as a barrier, restricting the movement of molecular and ionic species found in the liquids or vapours interacting with both surfaces [69]. Lonsdale [70] further defines membranes as essentially a semi-permeable barrier that allows some components to pass through while rejecting others. From Nollet's [71] experiments on animal membrane semi-permeability to the ground breaking development of modern membranes by Loeb & Sourirajan [72], the field of membrane science has achieved remarkable milestones. Membranes can be prepared by many ways. Extensive literature is available on the fabrication of microporous membranes using techniques such as phase separation (commonly referred to as phase inversion), extrusion, electrospinning and stretching methods [73-77].

Phase separation methods: Wang *et al.* [78] reported the formation of microporous membranes by thermally induced phase separation (TIPS) and non-solvent induced phase separation (NIPS) technique using solvent mixtures of dimethyl acetamide, triethyl phosphate mixed with non-solvent polyethylene glycol [78].

TIPS method has few advantages such as preparation of anisotropic and isotropic structure with controlled pore size and easy to scale up [79]. The original work of Castro [80] in the development of crystalline polymer by cooling discreetly concentrated polymeric solution in non-solvent gave birth to a new innovative method of TIPS [80]. Zhao *et al.* [81] developed PVDF membranes by diffusion-mediated TIPS method for enhanced macromolecule separation. Zhang *et al.* [82] using TIPS method fabricated hydrophilic HDPE/PE-β-PEG blend membrane for protein solution separation [82]. Chen *et al.* [83] studied that TIPS method allows precise control of morphology of membrane by tuning polymer concertation, quenching time and water content. Kim *et al.* [84] created high tensile strength (8.4 MPa) and permeability (538 L/m^2/ h/bar) microporous PVDF hollow fibre membranes using TIPS technique. Hu *et al.* [85] presented a facile method of fabrica-

TABLE-2
DETAILS OF DIVERSE MATERIALS USED FOR MEMBRANE PREPARATION

Membrane type	Materials	Polymer	Common applications
Polysulfone	Polysulfone [45]	Yes	Ultrafiltration [46], haemodialysis [47]
Proton exchange	Nafion [48]	Yes	Fuel cell [49]
Chitosan	Chitosan (Natural Polymer) [50]	Yes	Water treatment [51]
Silicone	Silicone rubber [52]	Yes	Gas separation [53], electroactive actuator [54]
Zeolite	Aluminosilicate [55]	No	Pervaporation [56]
Metal oxide	Silicon dioxide, aluminium oxide, iron oxide [57]	No	Wastewater treatment [58]
Metal alkoxide	Alumina substrate, tetraethyl orthosilicate, ethanol, water, acetic acid, 3-(trimethoxysilyl)propane [59]	No	Permselective cation exchanger [60]
Metal organic frameworks	Zirconium chloride octahydrate, fumaric acid, graphene oxide [61]	No	Desalination [62]
Inorganic composite	Graphene oxide, titanium dioxide, alumina [63]	No	Water purification & separation [64]
Coated ceramic	White clay, silica flour, arabic gum, marble powder, glass powder [65]	No	Wastewater filtration [66]

ting superhydrophobic (water contact angle 161º) isotactic polypropylene membranes by combining micro molding and TIPS methods. Fan *et al.* [86] reported patterning of PVDF membrane by TIPS process in the presence of green solvent tributyl O-acetyl citrate. In their research work, Tang *et al.* [87] employed Hansen solubility parameter (HSP) to evaluate polymer-diluent interactions and determine optimal diluents for membrane preparation *via* the TIPS method. Arundati *et al.* [88] fabricated porous cellulose acetate membrane for the application of lithium-ion battery separator using combination method of nonsolvent and TIPS. Several researchers successfully utilised TIPS technique in the polyvinylidene fluoride, polypropylene, poly(ethylene chlorotrifluoroethylene), high-density polyethylene and cellulose acetate membrane fabrication [89-93]. TIPS further can be classified into two types namely liquid-liquid (L-L) phase separation and solid-liquid (S-L) phase separation [94,95]. These two methods refer to different phase separation mechanisms that determine membrane structure. In L-L type, upon cooling polymer solution separates into two polymers rich and diluent rich liquid phases creating porous, sponge like membranes [96]. In contrast, S-L mechanism upon cooling polymer crystallizes (solidified) while diluent remains in liquid resulting in denser, less porous with higher mechanical strength membranes [97]. A review article by Osali *et al.* [98] provided an in-depth analysis of membranes prepared *via* L-L and S-L phase separation techniques, highlighting their diverse applications. Membranes prepared by TIPS method of S-L or L-L phase separation technique has variety applications like sewage treatment [99], microfiltration [100], oil/water separation [101], battery separator [102], chemical resister [103].

Since the pioneering work of Loeb & Sourirajan in 1963 [72], NIPS has remained one of the most widely adopted techniques for the fabrication of commercial membranes, particularly for reverse osmosis [104,105], nanofiltration [106,107], gas separation [108], face mask [109]. In contrast to TIPS, solvents employed for NIPS are usually able to dissolve the selected polymer at room temperature [110,111]. NIPS also known as immersion precipitation due to its versality, simplicity and capacity to form high through-put processing make it one of the widely used technique of membrane preparation [112-115]. NIPS method technically involves following steps, polymer solution preparation, spreading it to a thin film and submerging it in the non-solvent bath which triggers the demixing and coarsening mechanism leading to the solidification of polymer into membrane [116-118]. Key factors which to be taken care while preparing membrane through NIPS technique are polymer concentration, film casting condition, types of solvent and non-solvent, nature of the polymer and additives [119,120]. Pochivalov *et al.* [121] studied manipulation of membrane morphology by controlling temperature parameter in NIPS process. Kong *et al.* [122] demonstrated that regulations of morphology and permeability of surfactant facilitated NIPS prepared membranes without compromising membranes pore size and tensile strength. Jin *et al.* [123] prepared ultrafiltration membrane by combined technique of NIPS and TIPS to form uniform sponge like structure with increased tensile strength. Several researchers [124-128] explored possibility of finger like pore structured membranes by NIPS technique. A major drawback of the NIPS method is its heavy dependence on hazardous dipolar aprotic solvents (NMP, DMF, DMA) which pose reproductive toxicity and environmental concerns [129,130]. Besides NIPS and TIPS, there are many other techniques of phase separation to prepare membranes shown in Table-3.

Extrusion: Another method of membrane preparation was extrusion, which involves forcing a polymer melt or concentrated polymer solution through a die under pressure, followed by cooling or solvent removal to solidify the membrane. This method is ecofriendly as it avoids use of large amount of hazardous solvent [152,153], scalable for large scale production [154], low cost [155] and faster [156-158]. Fard *et al.* [159] reported preparation of membranes through extrusion process for the application of desalination. Armstrong *et al.* [160] demonstrated production of gas separation membranes by extrusion technique. Deka *et al.* [161] and Kumar *et al.* [162] prepared membranes from extrusion technique for dairy wastewater and oily wastewater treatment, respectively. Roshni *et al.* [163] described applications of membranes prepared from extrusion process for clinical application. Biron *et al.* [164] reported the fabrication of hollow fiber membranes through an integrated extrusion followed by phase separation technique, demonstrating their suitability for water treatment applications. Wang *et al.* [165] described prepara-

TABLE-3
VARIOUS PHASE SEPARATION TECHNIQUES OF MEMBRANE PREPARATION

Method	TIPS	NIPS	VIPS [131,132]	EIPS [133,134]	LIPS [135]
Abbreviation	Thermally induced phase separation	Non-solvent induced phase separation	Vapour induced phase separation	Evaporation induced phase separation [136]	Light induced phase separation
Mechanism	Thermal quenching	Non-solvent immersion	Vapour exposure	Solvent evaporation [137]	UV/visible exposure [138]
Components	Polymer solvent	Polymer solvent, non-solvent	Polymer, solvent vapour	Polymer, solvent, non-solvent	Polymer, solvent, light
General solvent toxicity	High [139]	High [140]	High [141]	High [142]	Less
Advantages	Easy control & uniformity [143]	Diverse porous structure [143]	Crystallisability [144]	Good consistency [145]	Patterned structure [146]
Drawback	High energy consumption [147]	Poor pore size control at low exposure time [148]	Takes longer time [149]	Difficult choice of solvent & non-solvent [150]	Relatively new method need optimisation [151]

tion of perfluorosulfonate ion-exchange membranes by melt extrusion technique. Lee *et al.* [166] tested membranes, which were prepared by extrusion technique for the separation of oil droplets from oily wastewater. Wang *et al.* [167] provided crucial crystallographic insights for optimising the PTFE paste extrusion process. Xiang *et al.* [168] investigated enhanced fouling resistance polyketone membranes, fabricated using solvent co-extrusion and chemical reduction. Xu *et al.* [169] fabricated novel janus hollow fibre membrane by extrusion and grafting technique for greenhouse gas removal study. Nguyen *et al.* [170] designed haemodialysis membranes *via* advanced co-extrusion technology. Huang *et al.* [171] developed an efficient and robust support structure *via* co-extrusion process for high performance zeolite membranes used in liquid separation. Raji *et al.* [172] effectively removed persistent organic pollutants from wastewater using TiO_2-embedded dual-layer mullite hollow fiber membranes which were fabricated *via* co-extrusion and co-sintering techniques. Finally, Guo *et al.* [173] comprehensively reviewed various extrusion-based membrane fabrication strategies, including vesicle extrusion, membrane emulsification, precipitation extrusion and biological membrane extrusion, high-lighting the versatility and continued evolution of extrusion technologies in membrane science.

Electrospinning: Another membrane preparation method is electrospinning (ES). With its unmatched versatility in material compatibility, nanostructure control ES stands as the gold standard for next generation functional membranes [174]. Patent work of Kiyohiko [175] on producing the artificial silk and other filaments by electrically spinning a raw material solution marked a crucial milestone in the development of electrospinning process.

In electrospinning, a polymer solution is fed through metallic needle *via* a syringe pump and a high voltage (1-30 kV) power supply electrifies the droplets at the needle tip to form a Taylor cone ejecting a jet that thins into nanofibers [176]. Broadly there are five types of ES, namely blend electrospinning, coaxial electrospinning, emulsion electrospinning, gas-nanofibre jet electrospinning and melt electrospinning [177]. Behroozi *et al.* [178] presented a comprehensive review of electrospinning method of NF membrane preparation for the application of water treatment. Ray *et al.* [179] comprehensively reviewed membrane fabrication and surface modification by ES for the water treatment applications. Meng *et al.* [180] reported uses of electrospun membranes in membrane distillation, oil/water separation, nanofiltration and haemodialysis. Hu *et al.* [181] reported fabrication of nano-netted membranes by electrospinning technique, which would be useful in ultrafiltration, catalyst support. Wang *et al.* [182] demonstrated the application of electrospun membranes as a stent cover. Some well-known electrospinning approaches for the fabrications of membranes are listed in Table-4.

Stretching method: Membranes can be also prepared/fabricated using stretching method. In this technique, a porous membranes are prepared by mechanical stretching of a polymer film the direction of the extrusion. Stretching method optimizes pore control, making it more efficient for industrial microporous membrane preparation by reducing energy consumption and improving material properties [196]. Stretching is relatively green membrane preparation process as it needs no organic solvent [197]. Castejon *et al.* [198] reported preparation of multilayer membranes by extrusion followed by stretching process. Primachenko *et al.* [199] studied possibilities of controlled electrochemical and physical-chemical properties of the proton conducting membranes as prepared by orientational stretching method. Sadeghi *et al.* [200] examined the influence of the stretching method on the structure and performance of the polypropylene microporous membranes. Saffar *et al.* [201] investigated the evolution of pore structure during the fabrication of microporous membranes using the stretching method. Kim *et al.* [202] observed importance of annealing prior to stretching as it improves membranes properties like orientation, crystallite size and crystallinity. Wei *et al.* [203] presented a biaxially stretched anion exchange membrane, which is suitable for fuel cell application. Table-5 summarizes a few membranes improvised by the stretching process and their important research outcome.

3D printing: Additive manufacturing, commonly known as 3D printing, is a fabrication process in which complex and customized objects are constructed layer by layer directly from a digital computer-aided design (CAD) model. The CAD file is typically converted into a stereolithography (STL) format, which is subsequently processed to define the necessary printing parameters for producing the final object [214]. The conceptual foundation of 3D printing technology was first proposed in the 1980s by the Japanese researcher Hideo Kodama, who introduced the idea of layer-by-layer fabrication [215].

TABLE-4
DIFFERENT ELECTROSPINNING (ES) APPROACHES FOR THE FABRICATIONS OF MEMBRANES

Parameter	Single-spinneret ES	Multi-spinneret ES	Coaxial ES	Melt ES
Setup [183]	One spinneret/nozzle	Multiple spinnerets	Dual-fluid channels	No solvent, high temp
Materials used	Polymer solution	Multiple polymers/solutions	Core-shell materials	Thermoplastics
Fiber morphology	Fibers with distinct micro-morphology [184]	Mixed/composite fibers [185]	Core-shell or hollow fibers [186]	Ultrafine, solvent-free fibers [187]
Production rate	Low	Higher	Low to moderate	Moderate (no drying needed)
Applications	Filters, tissue scaffolds	Multi-functional membranes	Drug delivery, encapsulation	Medical implants, industrial mats
Solvent required?	Yes	Yes	Core/sheath optional	No (melt-based)
Advantages	Simple protocol [188]	Scalability [189]	Encapsulation capability [190]	Non-solvent method, eco-friendly [191]
Limitations	Low throughput [192]	Clogging [193]	Multifaceted parameter tuning [194]	High temperature required [195]

TABLE-5
SUMMARISATION OF STRETCHING PROCESS OF MEMBRANES

Method	Material/membrane	Basic study conducted	Ref
Melt-spinning & hot-stretching method	Polyphenylene sulfide hollow fiber membrane	Protein separation and D19 dye rejection rate. Chemical resistance to acid, alkali, high temperature and polar solvents.	[204]
Biaxially stretching	Polypropylene sheet with $CaCO_3$ filler	N_2 gas permeability	[205]
Melt-spun & axial stretching	Polysulfone-block-poly(ethylene glycol) hollow fiber membrane	Enhanced water permeance and rejection rate studied	[206]
Stretching extruded	Expanded polytetrafluoroethylene membranes	Impact of stretching condition & air filtration performance study	[207]
Melt-spinning and stretching	PVDF hollow fiber membrane	Pure water flux, rejection rate and turbidity are investigated	[208]
Uniaxial stretching	Polyimide membranes	He/CH_4, H_2/CH_4 and CO_2/CH_4 gas perm-selectivity were demonstrated	[209]
Heat-stretching	PVDF hollow fiber membrane	Permeability & stability of direct contact membrane distillation process investigated	[210]
Biaxial stretching	Ultrahigh-molecular-weight polyethylene membrane	Variation of surface potential of the membranes studied	[211]
Uniaxial/biaxial stretching	PTFE membrane	Node-fibril structure of the membrane investigated	[212]
Biaxial stretching	$CaCO_3$/polyolefin composite membranes	Membrane porosity & interconnecting pores were studied	[213]

While 3D printing has material limitations, its accuracy, customisation and potential for nature-mimicking designs make it promising for next generation membranes [216]. Advancement in 3D printable membranes could bridge the performance gap with conventional approaches [217]. 3D printed membranes have many advantages like surface area, geometry, thickness, enhanced antifouling property, removal efficiency and overall membrane performance [218]. Membranes made from biopolymers like polyhydroxybutyrate can be recycled and remanufactured using 3D printing technology contributing to a circular economy [219]. Additive printing technology is helpful in creating membrane with accurately adjusted pore size, thickness and wettability [220]. Few applications of 3D printed membranes are water desalination [221], oil/water separation [222], heavy metal removal [223], organic pollutant removal from water [224], fouling reduction [225], membrane spacers [226], biosensor [227], hemodialysis [228], gas separation [229], catalysis [230]. Depending on the available literature a comparison of 3D printed membranes with conventional membranes are listed in Table-6.

Conclusion

With the membrane market projected to reach a $ 14.21 billion by 2032, there is strong demand for research and development in this field. In this review, three areas of membranes have been discussed. Firstly, membranes are primarily prepared from polymeric materials. Secondly, different well-known methods of membranes preparation. Thirdly, membranes applications in electromagnetic shielding. Different polymers are utilised to prepare membrane, from linear structured polysulfone to cyclic poly(dimethylsiloxane). The polymers' architecture enables them to cast into films and modify their mechanical strength. Significant progress has been done on membrane preparation to achieve particular characteristic. This review insights into membrane preparation methods like phase separation, extrusion, 3D printing, electrospinning and stretching. Phase separation technique is ideal for the synthesis of asymmetric or microporous membranes with controlled pore structure. Electrospun nanofibrous membranes are highly porous, may be find useful in air filtration and biomedical applications. Track-etched membranes with great mechanical strength can be fabricated with extrusion methods. 3D-printed membranes offer customizable, complex pore designs, making them suitable for lab-on-a-chip and drug delivery systems. Every method of preparation tailor's membrane characteristics such as selectivity, porosity, pore size and geometry for the specific applications.

TABLE-6
COMPARISON BETWEEN 3D-PRINTED MEMBRANES AND CONVENTIONAL METHOD

Parameters	3D printed membranes	Conventional membranes
Manufacturing/preparation process	Layer by layer deposition	Phase inversion, ES, stretching, *etc*.
Customisation [231]	High (tailored pore structure, precise geometry)	Limited shapes, pore size
Membrane materials [232]	Limited (requires printable polymers)	Broad (wide range of polymers)
Material efficiency [233]	Minimal waste	Higher waste
Production speed [234]	Production speed controllable	Production speed not controllable
Cost [235]	Higher	Lower
Scalability [236]	Challenging for large scale manufacturing	Established process for large scale production
Sustainability	Better (potential remanufacturing)	Less (chemical waste)

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this work the author(s) used Microsoft Copilot in order to Assist in Proofreading. After using this tool/service, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the published article.

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