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Hydrophilic Modification of Polymeric Membrane using Graft Polymerization Method: A Mini Review

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Abstract. The separation performance of polymeric membrane is strongly influenced by its surface characteristics. In water treatment applications, it is generally accepted that a hydrophilic membrane surface is favorable to enhance water permeate flux and mitigate membrane fouling. Numerous methods, both chemical and physical modification, have been proposed to improve the hydrophilicity of polymeric membranes. Graft polymerization is one of the promising methods to modify polymer membranes by tethering hydrophilic polymer chains onto membrane surface. The existence of hydrophilic chains on the membrane surface facilitates the reduction of interfacial tension with water, thus higher water flux can be obtained. In addition, the increase of membrane hydrophilicity also improves the antifouling resistance. This review provides a concise summary and discussion of the modification of polymeric membranes using graft polymerization method. The effects of graft polymerization on the membrane hydrophilicity as well as the antifouling property are discussed.

Keywords: antifouling, graft polymerization, hydrophilic, polymeric membrane

1. Introduction

The interest in technologies for water treatment applications is now growing amidst the concern over drinking water security issues and environmental impact. Wide range of water treatment technologies have been developed or are currently developed to overtake traditional technologies, i.e. chemical clarification, granular media filtration, and chlorination. One of the most interesting processes for water treatment is polymeric membrane. It offers some advantages, such as strict solid-liquid separation, high quality of product, and simple operation [1, 2].



The separation performance of polymeric membrane is strongly influenced by its surface characteristics. In water treatment applications, it is generally accepted that a hydrophilic membrane surface is favorable to enhance water permeate flux. Besides, the impurities components in surface water are mostly hydrophobic (i.e. microbes, particles, humic acids, etc.), thus their interaction with hydrophobic membranes causes the membranes to be more prone to the fouling than hydrophilic membranes [3-8]. It has been proved in the studies by Laine et al. [9] and Malgorzata et al. [10] where better permeability occurred for more hydrophilic membranes (cellulose) than hydrophobic ones (acrylic, polyaramide, or polysulfone).

Fouling not only leads to productivity decline, but also increases the energy requirement to keep the membrane performance constant [11-15]. Therefore, it is necessary to improve the antifouling resistance by increasing membrane surface hydrophilicity. Various methods to increase hydrophilicity of polymeric membranes have been developed either by chemical or physical modification. Hydrophilization by chemical modification can be done by blending polymers with more suitable properties or by addition of polymer layers on the active surface of membrane through ozone treatment, sulfonating treatment, or grafting of strong hydrophilic monomers [16-19]. In the most cases, chemical modifications lead to destruction or a change in the porous structure of the membrane, and typically require expensive instruments and/or complicated process [20]. Meanwhile, physical modification such as wetting or dip-coating is a mild technique and relatively inexpensive. However, the hydrophilicity from physical modification is not stable, thus the water flux decrease over time.

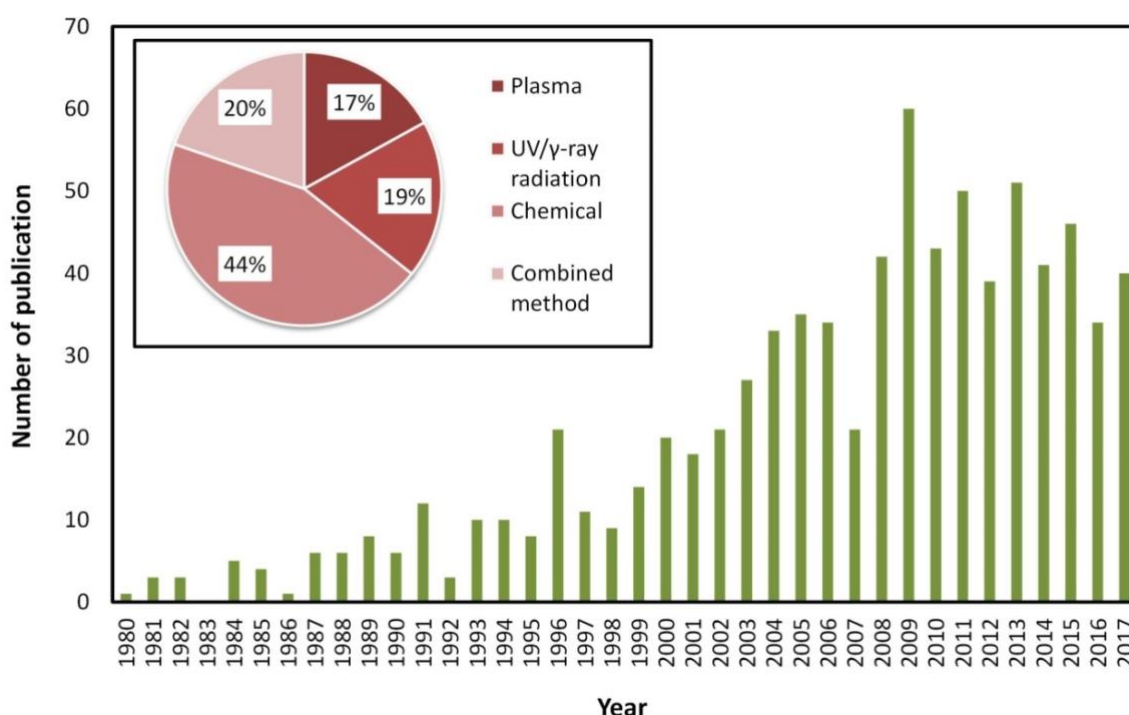


Figure 1 The number of published papers related to the search term of graft polymerization on polymeric membrane. Indexed by Scopus (TITTLE-ABS-KEY (graft polymerization AND polymer membrane); August 2018)

Among the developed hydrophilic modifications of polymeric membrane, graft polymerization is the most promising method. As shown in Figure 1, the number of publications for graft polymerization on polymeric membranes has been increased over year and the chemical-induced graft polymerization has the highest number of publications. Various studies showed that the hydrophilicity and water flux of grafted membranes were

stable after several times of operation. To provide an insight for further development of hydrophilic polymeric membrane, this paper gives a review of surface hydrophilization of polymeric membrane using graft polymerization. The effects of graft polymerization on the membrane hydrophilicity as well as the antifouling property are discussed.

2. Methods of Graft Polymerization

Graft polymerization is the promising alternative to increase polymeric membrane hydrophilicity and improves the antifouling resistance by tethering hydrophilic polymer chains onto membrane surface [21, 22]. This method offers advantages of stable hydrophilicity and high water flux. The comparison of graft polymerization to other modification methods is shown in Table 1.

Table 1 Advantages and disadvantages of hydrophilic modification methods [23, 24]

Modification method	Water flux	Simplicity	Hydrophilic stability	Environmental aspect	Cost effectiveness
Coating	Low	High	Low	High	High
Blending	High	High	High	High	High
Composite	High	High	High	High	High
Plasma treatment	High	Low	Low	High	High
Graft polymerization					
• Chemical-induced	High	High	High	Low	High
• Plasma-induced	High	Low	High	High	Low
• Irradiation induced	High	Low	High	Low	Low

In general, graft polymerization can be divided into three main procedures, i.e. chemical-, plasma-, and irradiation- induced graft polymerization [25-28]. For membrane material with reactive groups, it can be easily grafted through the free radical graft polymerization or the chemical reaction. Meanwhile, membrane material without reactive groups needs plasma, γ -ray, or UV to induce graft polymerizations [29].

2.1 Chemical-Induced Graft Polymerization

In chemical-induced graft polymerization, a radical for the polymerization process is formed on the membrane surface through reaction with chemical compounds. One of the most attractive techniques for oxidizing a substrate surface is ozonation [27, 30]. It can be performed either in the gaseous phase or aqueous phase. Ozone acts as a precursor providing radical elements that will initiate the polymerization process [31, 32].

Polypropylene (PP) membranes have been successfully modified using ozone or oxidizing agents such as benzoyl peroxide (BPO) and benzophenonyl bromoisobutyrate (BPBB) as precursor for grafting of hydroxyethyl methacrylate (HEMA) [31, 33], N,N-dimethylaminoethyl methacrylate (DMAEMA) [34] and acrylic acid [35]. Meanwhile, for polyethersulfone (PES) membranes, peroxydisulfate and metabisulfite oxidizing agents have been used to initiate free radical polymerization grafting of methacrylic acid, polyethyleneglycol-methacrylate, and sulfopropylmethacrylate in aqueous solution at ambient temperature [36, 37]. Chemical-induced graft polymerization also has been used to increase hydrophilicity of polysulfone (PSf) membranes [38-40], polyacrylonitrile (PAN) membranes [41], and Poly-vinylidene fluoride (PVDF) membranes [42, 43], as shown in Table 2. In general, polymeric membranes that have been modified by chemical-induced graft

polymerization to be less sensitive to fouling due to the presence of the hydrophilic monomer grafted.

2.2 Plasma-Induced Graft Polymerization

Plasma-induced graft polymerization can be achieved through two different procedures, i.e. peroxy radical (P) method and alkyl radical (R) method [44]. In the (P) method, the membrane is introduced to plasma reactor without working gas, then air is added to make peroxy radical that can initiate the grafting process. Meanwhile, the (R) method can be achieved in the absence of working gas or using noble gases such as argon, helium, etc. [44, 45]. This method is operated under vacuum condition where the alkyl radical will initiate the grafting process.

Table 2 Graft polymerization of polymeric membranes

Membrane material	Grafting method	Polymer/additive	Induction	Ref.
PP	Chemical-induced	Acrylic acid, Polyvinyl alcohol, Polyethylene amine, PEGMA, DMAEMA, PNVP, HEMA, TiO ₂	Ozone, BPO, BPBB	[28, 30, 31, 46-52]
	Plasma-induced	Acrylic acid, Alkyl glycoside, PNVP, PNIPAM, PSLG, TiO ₂	Plasma: Air, N ₂ , NH ₃ , Argon	[53-60]
	Irradiation-induced	Acrylic acid, Alkyl glycoside, Acrylamide, PEGMA, HEMA, GMA, PNIPAM, GAMA, SBMA, MPDSAH, TiO ₂	UV, γ -ray	[25, 29, 61-73]
PES	Chemical-induced	TiO ₂ , PEGMA	Peroxydisulfate, Metabisulfite	[36, 37, 74, 75]
	Plasma-induced	Acrylic acid, Acrylamide, PNVP	Plasma: Argon, Helium	[76, 77]
	Irradiation-induced	Acrylic acid, Ethylene diamine, HEMA, PNVP	UV	[78, 79]
PSf	Chemical-induced	PES, PEG, Acrylamide	-	[38-40]
PAN	Chemical-induced	PEO	-	[41]
	Irradiation-induced	Acrylic acid, PEGMA	UV	[80]
PAS	Irradiation-induced	Methacrylic acid, GMA, HEMA	UV	[81]
PVDF	Chemical-induced	PEGMA, PVP	-	[42, 43]
	Irradiation-induced	PES	UV	[82]

Abbreviation: BPBB = benzophenonyl bromoisobutyrate, BPO = benzoyl peroxide, DMAEMA = N-dimethylaminoethyl methacrylate, GAMA = Gluconamidoethyl methacrylate, GMA = Glycidylmethacrylate, HEMA = Hydroxyethyl methacrylate, MPDSAH = [3-(methacryloylamino)propyl]-dimethyl(3-sulfopropyl) ammonium hydroxide, PAN = Polyacrylonitrile, PAS = Polyarylsulfone, PE = Polyethylene, PEG = Poly(ethylene glycol), PEGMA = Poly(ethylene glycol) methacrylate, PEO = Polyethylene oxide, PES = Polyethersulfone, PNIPAM = Poly(N-isopropylacrylamide), PNVP = Poly(N-vinyl-2-pyrrolidone), PP = Polypropylene, PSf = Polysulfone, PSLG = Poly(c-stearyl-L-glutamate), PVDF = Poly(vinylidene fluoride), PVP = Poly(vinyl pyrrolidone), SBMA = Sulfobetaine methacrylate

Several studies of plasma-induced graft polymerization in order to increase the hydrophilicity of polymeric membrane have been conducted using gases such as ammonia [55, 83], oxygen [84], nitrogen [53, 54, 85, 86], hydrogen [87], water [76, 88, 89], argon [58, 77, 90, 91], or air [48, 49, 57]. Meanwhile, the grafting substances consisted of N,N-dimethyl acrylamide (NDMAm) [44], N-isopropyl acrylamide (NIPAm) [53], polyethylene glycol (PEG) [87], acrylamide [92], acrylic acid [58, 90], sulfonated glycidyl methacrylate [91], and tetraethoxysilane (TEOS) [93], as shown in Table 2.

While using plasma-induced graft polymerization, the operating condition should be maintained at an appropriate level. As temperature increased, there will be more active sites on the membrane surface, and monomer diffusion is also enhanced due to an increase of grafting degree. Conversely, a high-concentration monomer can decrease the degree of grafting due to increased viscosity that hinders the monomer diffusion to the membrane surface [85] or prevents the plasma treating to reach the surface of the PP membrane, and then no active sites are produced [94].

2.3 Irradiation-Induced Graft Polymerization

Irradiation-induced graft polymerization on PP membrane involves monomer and PP membrane simultaneously to react through radical reaction, with ultraviolet (UV) or γ -ray as the initiator in irradiation induced. UV irradiation is able to cross-link polymer chains and cleave polymer bonds, thus forming functional groups such as hydroxyls, carbonyls, or carboxylic acids on the surface [23].

γ -ray has been used for grafting acrylic acid [69], HEMA [62, 95], N-vinyl-2-pyrrolidone (NVP) [29], and NIPAm/acrylic acid [96] onto PP membrane surfaces. These substances have been successfully grafted onto PP membranes using γ -ray irradiation, resulting hydrophilic surfaces with WCA < 65°. Meanwhile, by UV irradiation, several substances have been grafted onto PP membrane surface, such as acrylic acid [66, 67, 97-99], poly-(ethylene glycol)-200-monomethacrylate (PEG200MA) [97], glycidylmethacrylate (GMA) [63], 3-hydroxy-2,2-bis(hydroxymethyl) propylacrylate (PEA) [100], methoxyl polyethylene glycol (MPEG) [101], acrylamide [67], D-gluconamidoethyl methacrylate (GAMA) [68, 102], sulfobetaine methacrylate (SBMA) [71], and α -allyl glucoside [61, 103].

UV-irradiation has also been used to graft several hydrophilic monomers onto PES membranes, i.e. Nvinyl-2-pyrrolidinone, N-vinylcaprolactam, and N-vinylformamide. The results showed there were 25% increase in PES membranes hydrophilicity and 50% decrease in BSA fouling [104-106]. The same method also successfully increased the hydrophilicity of PAN [80], polyarylsulfone (PAS) [81], and PVDF membranes [82].

3. The Influence of Graft Polymerization

Graft polymerization is the promising method to increase hydrophilicity of polymeric membrane. The hydrophilicity of polymeric membrane is mainly measured by water contact angle (WCA). Membrane with WCA less than 90° is classified as hydrophilic membrane while hydrophobic membrane has WCA greater than 90°. Figure 2 shows the effect of graft polymerization on WCA of polymeric membranes from various studies. It can be seen that graft polymerization is able to produce hydrophilic membrane with low WCA using various procedures and chemicals.

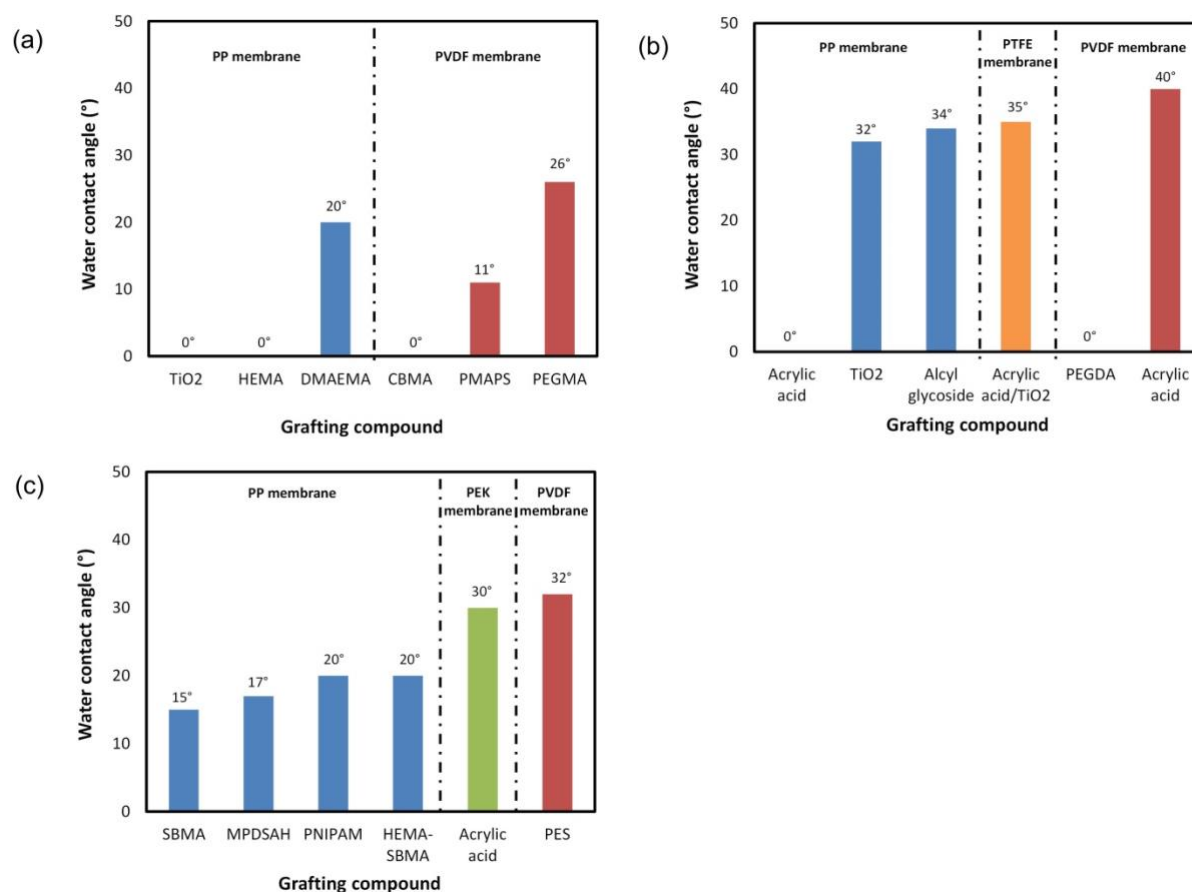


Figure 2 Effect of (a) chemical induced [48, 50, 52, 107-109], (b) plasma induced [54, 59, 60, 110-112], and (c) irradiation induced [70-73, 113, 114] graft polymerization on WCA

Furthermore, hydrophilic modification of polymeric membranes not only improves the water flux, but also endows the membranes with fouling-resistant properties. It makes the modified membranes exhibit higher performance when applied in water and wastewater treatment. Hydrophilic modifications also leads to the increase of biocompatibility of polymeric membranes, thus widen their applications in biotechnology and biomedical applications. The results summary of hydrophilic modification of polymeric membranes via graft polymerization is presented in Table 3. It can be seen that UV irradiation was the most used induction for grafting polymers or additives onto polymeric membrane surface. It is due to the ability of UV to induce cross-linking in the polymer matrix, thus the membrane selectivity was increased [115].

In general, the characteristics of modified membrane mainly depend on the grafting degree. The higher degree of graft polymerization provides a strong driving force to reduce the interfacial free energy by migrating and reorienting of the hydrophilic monomers [51]. As the result, higher grafting degree with a large coverage surface decreases the water contact angle. The study of Yu et al. [67] showed that the water contact angle of PP membrane was decreased from 135° to 42° by increasing the grafting degree to 120%. It is in agreement with the results from other studies [62, 116, 117]. The water flux on modified membrane remained almost unchanged when the grafting degree was low, however the pore-covering soon become the major problem with the increase of the grafting degree, causing the water flux dropping severely. Therefore, the precise design of a graft layer and well-controlled graft architecture is a very important to minimize the membrane fouling.

Table 3 Hydrophilic modification of polymer membranes via graft polymerization

Membrane material	Grafting induction	Polymer/additive	Influence of graft polymerization	Ref.
PP	Chemical	Polyvinyl alcohol/4-vinylpyridine	Water fluxes were increased up to 500.4 L/m ² .h at operating pressure 40 kPa.	[47]
	Nitrogen plasma	α -allyl glucoside	Water fluxes were increased up to 3826 \pm 80 kg/m ² .h at operating pressure 80 kPa.	[54]
	UV	Acrylic acid, Acrylamide	The modified membranes showed better filtration performances in the submerged membrane bioreactor (SMBR) than the unmodified one. Acrylic acid-grafted membrane presented the best antifouling characteristics.	[92]
		GAMA	After continuous operation in the MBR for about 70 h, reduction of water flux was 90.7% for the initial membrane and 80.8-87.2% for the modified membranes, increasing with an increase of a length of the grafted chains.	[68]
		HEMA	The modified membrane showed better protein resistance as well as hemo-compatibility due to the enhancement of surface hydrophilicity. The static water contact angle of the membrane surface decreased from 145° to 42°, with the extent of grafting increasing from 0 to 35.67 wt.%.	[65]
		HEMA, SBMA	Fouling was significantly reduced and most of the fouling on the modified PP membrane surface was reversible.	[72]
PES	Argon plasma	Polyacrylamide	The grafting yield for polyacrylamide on the membrane surfaces increased nearly linearly with the Argon-plasma pretreatment time, with grafting yields (GY) higher than 100 μ g/cm ² . The membranes obtained a permanent hydrophilicity (almost no change in contact angle after 1 year), and BSA adsorption was reduced to less than half that of the control membrane (306 to 148 μ g/cm ²).	[77]
	UV	Polyvinyl alcohol/4-vinylpyridine	Membranes modified with N-vinyl-2-pyrrolidinone (25% increase in hydrophilicity) exhibited the best combination of low fouling (50% decrease in BSA fouling) and high flux, although membrane permeability was significantly decreased because the grafted polymer chains blocked the membrane pores (over 25% reduction in flux due to modification).	[105]

Table 3 (continue)

Membrane material	Grafting induction	Polymer/additive	Influence of graft polymerization	Ref.
PES	UV	PNVP	The fouling tendency was reduced for membranes irradiated for 60 s comparing with those irradiated for 180 s. Enlargement of pore structure was observed under long irradiation times facilitating the membrane fouling with a high molecular weight fraction of natural organic matters.	[104]
		PEGMA	Modified membranes showed more resistance to fouling and higher rejection than unmodified membranes.	[118]
PES and PVDF	UV	DMAEMA	The modified membranes were more resistant to biofouling.	[119, 120]
PES/PVDF blend	UV	PNVP	Bovine serum albumin (BSA) adsorption on the membranes decreased from 159 ± 2 to 13 ± 2 $\mu\text{g}/\text{cm}^2$ after 10 min of the grafting. The fouling extent of the blend membrane with 7 min grafting was reduced by 66% and the flux recovery after chemical cleaning increased by 32%.	[82]
PVDF	UV	HEMA, Polydopamine, Acrylic acid	Antifouling properties, e.g., flux recovery, irreversible flux loss, total flux loss, and fouling resistance of modified membranes were enhanced due to an increase in membrane hydrophilicity.	[121]
PSf	UV	MPDSAH	Hydrophilicity and antifouling properties of the modified membranes were enhanced with the increase of the extent of grafting.	[122]
		PNVP, HEMA, Acrylic acid	Modified membranes showed high protein retention, high solution flux, and low irreversible fouling.	[123]
PAN	UV	HEMA, PEGMA, Acrylic acid	Protein-polymer surface interactions were diminished by the membrane modification. Adsorption and fouling were reduced both for the modified membranes.	[80]

4. Conclusions

The application of polymeric membrane is mostly restricted by fouling formation. Hydrophilic modification through graft polymerization is one of the promising methods to enhance membrane hydrophilicity and antifouling properties. This method can be conducted through chemical-induced, plasma-induced, and irradiation-induced using UV or γ -ray. Graft polymerization was able to decrease water contact angle and BSA adsorption on polymeric membranes. In addition, this method offers advantage of stable hydrophilicity and high water flux compared to other hydrophilic modifications.

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