SEBS 블록 공중합체를 기반으로 한 수전해용 음이온 교환막에 대한 총설

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A Review on SEBS Block Copolymer based Anion Exchange Membranes for Water Electrolysis

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요 약: 재생에너지의 보급과 기후변화를 대응하기 위한 해결책으로 수소에너지에 대한 관심이 늘어나고 있다. 수소는 미이용 전력을 대용량 장주기로 저장하기에 가장 적합한 수단이며 이러한 수소를 생산하는 기술 중 수전해는 물에 전기에너지를 인가하여 수소를 생산하는 친환경적 수소생산 기술로 알려져 있다. 수전해의 구성 요소 중 분리막은 음극과 양극을 물리적으로 분리할 뿐만 아니라 생성되는 수소와 산소의 섞임 현상을 방지하며 이온의 전달을 가능하게 하는 복합적인 역할을 수행한다. 특히 기존의 수전해 기술들의 단점을 보완할 수 있는 차세대 음이온 교환막 수전해에서의 핵심은 우수한 음이온 교환막을 확보하는 것이다. 높은 이온 전도성과 알칼리 환경에서 우수한 내구성을 동시에 가지려는 많은 연구들이 진행되고 있으며 다양한 소재에 대한 탐색이 이루어지고 있다. 본 총설에서는 상용 블록 공중합체인 Polystyrene-b-poly(ethylene-co-butylene)-b-polystyrene (SEBS)를 기반으로 하는 음이온 교환막에 대한 연구에 대해 살펴보며 최신 연구 동향과 앞으로 나아가야할 점에 대해 논하고자 한다.

Abstract: Hydrogen energy has received much attention as a solution to the supply of renewable energy and to respond to climate change. Hydrogen is the most suitable candidate of storing unused electric power in a large-capacity long cycle. Among the technologies for producing hydrogen, water electrolysis is known as an eco-friendly hydrogen production technology that produces hydrogen without carbon dioxide generation by water splitting reaction. Membranes in water electrolysis system physically separate the anode and the cathode, but also prevent mixing of generated hydrogen and oxygen gases and facilitate ion transfer to complete circuit. In particular, the key to next-generation anion exchange membrane that can compensate for the shortcomings of conventional water electrolysis technologies is to develop high performance anion exchange membrane. Many studies are conducted to have high ion conductivity and excellent durability in an alkaline environment simultaneously, and various materials are being searched. In this review, we will discuss the research trends and points to move forward by looking at the research on anion exchange membranes based on commercial polystyrene-b-poly(ethylene-co-butylene)-b-polystyrene (SEBS) block copolymers.

Keywords: water electrolysis, energy, anion exchange membrane, SEBS, hydrogen, technology

1. Introduction

Water electrolysis is the process whereby water decomposes into hydrogen and oxygen through the application of electrical energy. Hydrogen production by water electrolysis is a good way to fully utilize surplus renewable energy[1-7]. Water electrolysis as energy storage and conversion electrochemical technology has

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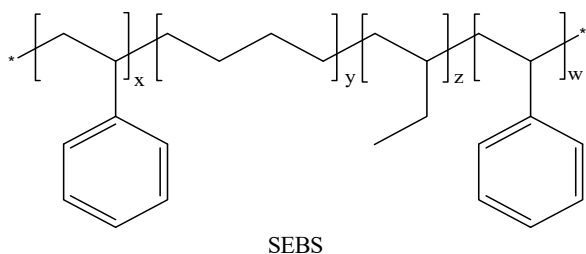
SEBS

Scheme 1. Chemical structure of polystyrene-b-poly(ethylene-co-butylene)-b-polystyrene (SEBS) triblock copolymer.

attracted considerable attention for their competitive advantages like environmentally friendly and easy to scale up[8].

Water electrolysis is an environmentally friendly technology because there are no side reactions that could produce undesired byproducts which can accelerate climate change. Water electrolysis technology can be divided into high-temperature and low-temperature water electrolytes depending on the operating temperature[9,10]. Low-temperature water electrolytes can be classified into alkaline water electrolysis (ALKWE), proton exchange membrane water electrolysis (PEMWE)

and anion exchange membrane water electrolysis (AEMWE) depending on the operating condition and membranes (Fig. 1). AEMWE is the most ideal water electrolysis technology with economic feasibility of ALKWE and the fast response and high efficiency of PEMWE (Table 1). AEMWE is considered as one of the promising technologies because of the use of non-precious metal catalysts (such as Fe, Co and Ni) which can reduce the manufacturing cost of water electrolysis device.

Anion exchange membranes (AEMs) is the critical components of water electrolysis system which is used to not only separate produced hydrogen and oxygen gases, but also transport ions between the cathode and anode. AEMs can determine the efficiency and safety of the water electrolysis system. Amphiphilic polymers which consisted of hydrophobic backbone and hydrophilic ion conducting groups can be used as starting materials for AEMs. Amphiphilic property of polymer induced microphase-separated morphology that can act as ion facilitate channels in membrane matrix[11,12].
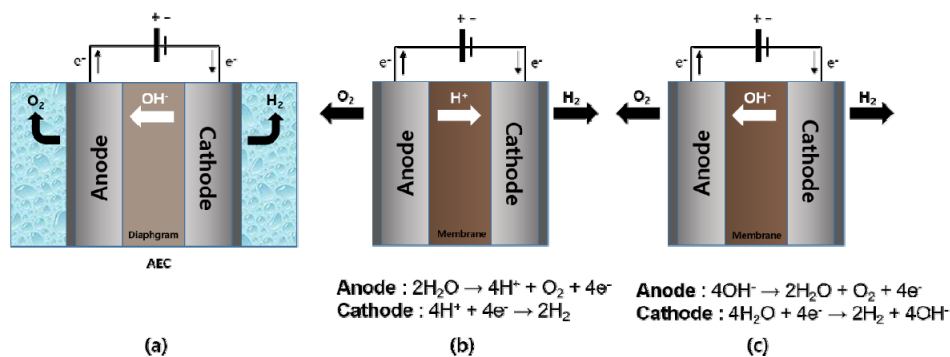

Anode : $2H_2O \rightarrow 4H^+ + O_2 + 4e^-$
Cathode : $4H^+ + 4e^- \rightarrow 2H_2$

Anode : $4OH^- \rightarrow 2H_2O + O_2 + 4e^-$
Cathode : $4H_2O + 4e^- \rightarrow 2H_2 + 4OH^-$

Fig. 1. Schemes of (a): ALKWE, (b): PEMWE, (c): AEMWE.

Table 1. Advantages and Disadvantages of ALKWE, PEMWE and AEMWE

	ALKWE	PEMWE	AEMWE
Advantages	- Mature technology - Non-PGM catalyst - Long term stability - Low cost	- Higher performance - Rapid response - Compact cell design - Dynamic operation	- Non-PGM catalyst - Rapid response - Compact cell design - Low cost
Disadvantages	- Gas crossover - Low operating pressure	- High cost (membrane/catalyst)	- Laboratory stage - Low ion conductivity and long-term stability

Alkalin degradation of polymer backbones

Alkaline degradation of quaternary ammonium groups

nucleophilic substitution
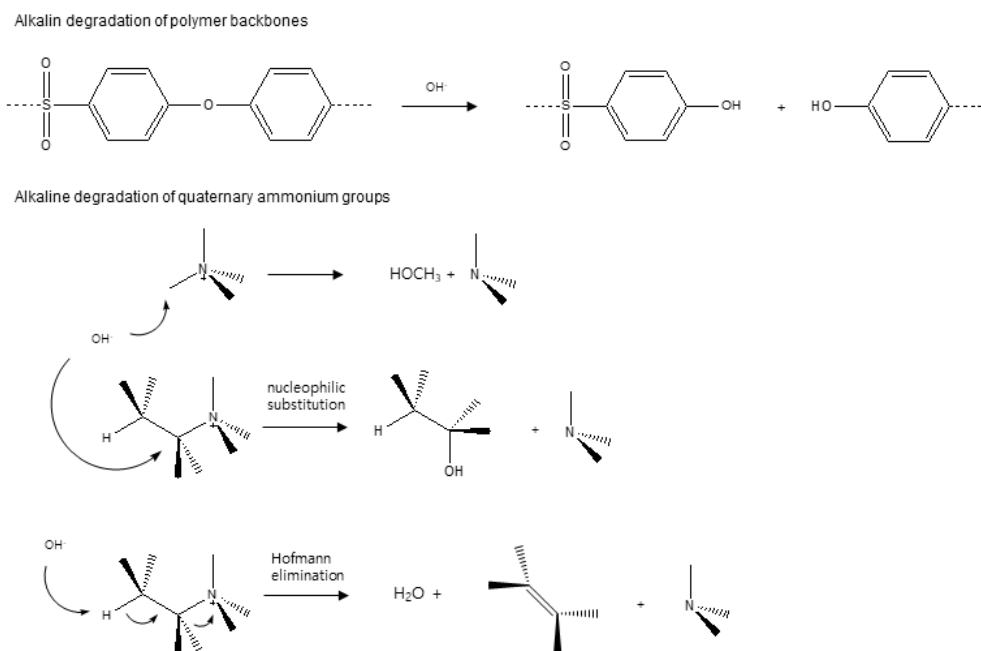
Hofmann elimination

Fig. 2. Alkaline degradation mechanism of AEMs.

In alkaline environment, the polymer backbone and functional group of AEMs can be degraded by OH⁻ ions or radicals, that decreases long-term alkaline stability of membrane. (Fig. 2) Two main mechanisms including nucleophilic attack of hydroxide ion on N-alkyl groups (SN₂ mechanism) and Hofmann elimination lead to degradation of ion exchange groups. The normal quaternary ammonium (QA) group suffer degradation in the alkaline condition due to the direct nucleophilic substitution at α-carbon. The electron-withdrawing group in the polymer backbone increase the steric hindrance of the electron-deficient domain and decrease the electron density of the ion-conducting group, resulting in the attack of OH⁻ ions and the decreased alkaline stability of AEMs[13]. Other degradation mechanisms were also identified such as the electrochemical oxidation of the phenyl group on oxygen evolution catalysts. Especially, poly(phenylene oxide)(PPO)-based AEMs suffered degradation during fuel cell operation mainly because of the degradation of cation group. Furthermore, Amel *et al.* noted that the sulfone linkages also had a negative effect on the

alkaline instability of AEMs. Considering the alkaline stability of AEMs, it is necessary to develop highly stable polymer backbone and ion-conducting functional groups[14].

The aromatic polymers have been widely used as backbone materials in AEMs because they show good mechanical and thermal stability and are easy to modify and synthesize. So far, poly(ether sulfone)s, poly(ether ether ketone)s, PPO, poly(phenylene)s have been widely used for the preparation of AEMs. And up to now, ion conducting groups such as quaternary ammonium (QA), imidazolium, guanidinium and phosphonium are introduced into the polymer backbone. Unfortunately, the C-O linkages in aromatic polymer backbone which inevitably generated as a result of condensation polymerization often undergoes cleavage via the attack of OH⁻ ions when the ion conducting groups are adjacent to the polymer backbone[15,16].

Polystyrene-b-poly(ethylene-co-butylene)-b-polystyrene (SEBS) triblock copolymer have been considered as alternative of other polymer backbone recently due to their high chemical and thermal stability and amend-

able mechanical properties.(Scheme 1) SEBS is not only commercially available but also easy to modify because various alkyl group can be introduced and the length of the alkyl chain can be adjusted. Especially attaching the quaternary ammonium (QA) group to the SEBS triblock copolymer free of C-O linkage via long flexible alkyl chain is responsible for the robust alkaline stability of the AEMs[17-19]. In this review, we will focus on the research trends and points on anion exchange membranes based on SEBS triblock copolymers.

2. Results and Discussion

2.1. Non-crosslinked AEM

Benzyl-trimethyl ammonium (BTMA) was used as ion conducting group. But it is noted that the BTMA based AEMs were fragile to disassemble under alkaline condition due to the displacement of the QA group by OH$^-$ ions via direct nucleophilic substitution. To solve this problem, many strategies such as introducing long flexible alkyl chain between the QA groups and backbone or using benzyl-substituted quaternary ammonium (QA) group were proposed because the steric hindrance effect is effective at preventing nucleophilic attack from OH$^-$ ions than alkyl-substituted quaternary ammonium (QA) group.

Z. Wang *et al.* synthesized SEBS-based AEMs with quaternary trimethylammonium (TMA) via chloromethylation, ketone reduction and quaternization. The cationic group was linked to the SEBS polymer backbone via a long flexible alkyl chains. It was demonstrated that introducing a long alkyl chain between QA group and polymer backbone was efficient to weakening the electron-withdrawing effect of cation group on the polymer backbone and improving the mobility of ionic group. The hydrophilic domain formed ion conductive domain with sizes ranging from 57 nm to 95 nm. As a comparative group, PPO-based AEMs were also synthesized and compared various properties between SEBS-based AEMs. In the case of PPO-based AEMs, 28% of IEC were lost after 30 days in a 1M KOH (60°C) solution whereas SEBS-based AEMs lost

Table 2. Ion-exchange Capacity, Elemental Analysis, Water Uptake, and Ion Conductivity Data of Various SEBS-QA Membranes[21]

SEBS-QA	IEC (mequiv/g)	WU (%)	OH$^-$ σ (mS/cm)	
			30°C	60°C
SEBS-TMA	2.41 (2.19)	211	45	89
SEBS-DMP	2.06 (1.96)	249	19	33
SEBS-MCH	1.77 (0.71)	68	8	10
SEBS-MiPr	2.07 (0.54)	49	7	9
SEBS-TMHA	2.01 (1.95)	236	39	59
SEBS-DMHA	2.01 (1.91)	194	28	34

10% of IEC and the mechanical properties of SEBS-based AEMs were initially maintained[20].

A. D. Mohanty *et al.* also synthesized SEBS-based AEMs functionalized with various benzyl- and alkyl-substituted quaternary ammonium (QA) groups for fuel cell applications. Six QA group with different structures such as TMA, dimethyl piperazinium (DMP), benzyldicyclohexyl methylammonium (MCH), benzyldiisopropyl methylammonium (MiPr), trimethyl hexylammonium (TMHA), benzyldimethyl hexylammonium (DMHA) was incorporated to the polystyrene units of SEBS via transition metal (Ir)-catalyzed C－H borylation and Suzuki coupling. Properties of AEMs according to the results of attaching six QA group with different structures under alkaline conditions were investigated. (Table 2) AEMs containing the trimethylammonium pendants which is the smallest QA group in six different structured QA exhibited the most significant improvement in water uptake and hydrophilic domain spacing to offer the highest ion conductivity. And both SEBS-TMA and SEBS-TMHA remained mechanically and chemically stable even at 60~80°C. It was found that incorporating stable cation structures composed of solely C-C bonds and C-H to a polymer backbone resulted in AEMs with improved long term alkaline stability by inhibiting nucleophilic substitution reaction. Although sterically bulky QAs (e.g., MCH, MiPr) were found to suffer lower ion conductivity and incomplete quaternization, they have higher stability than SEBS-BTMA under alkaline conditions[21].
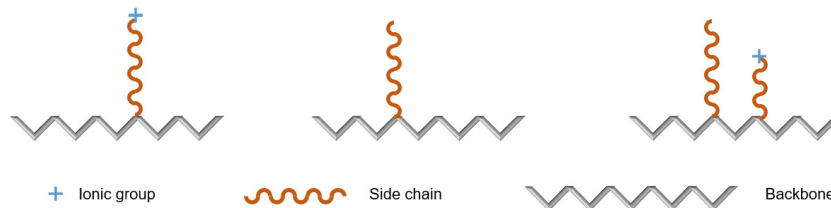
Fig. 3. Schematic illusion of the (a) hydrophilic comb-type polymers with side chain between the ionic group and backbone, (b) hydrophobic comb-type polymers with side chain, and (c) hydrophilic-hydrophobic comb-type polymers having both the side chain and the side chain having ionic group[22-25].

A. Z. A. Munsur et al. synthesized SEBS with 50% hexyl quaternary ammonium (HQA) group and 20% alkyl chain of different alkyl chain lengths C_4 and C_{12} (Fig. 3). The HQA-functionalized SEBS membrane with hydrophobic alkyl group and no-hydrophobic membrane were compared, and the effect of the alkyl lengths was investigated. The alkyl chain which introduced between the ion-conducting group and polymer backbone played a role as a hydrophilic spacer and additional alkyl chain grafted onto the SEBS triblock copolymer backbone played a role as hydrophobic group. Well-connected morphology, enhanced water uptake, and ion conductivity as well as improved chemical properties of the membrane were obtained as a result of additional introduction of hydrophobic alkyl chain[22-25].

2.2. Crosslinked AEM

Ion conductivity of AEMs can be improved by attaching more ionic groups such as quaternary ammonium (QA) group into SEBS polymer backbone. But increasing the amount of ionic group leads to higher water uptake of membrane, which results in high swelling behavior of AEMs or gelation of SEBS polymer solution. It is contributed to weakening mechanical property of AEMs and results in failure of film formation. This is called trade-off relationship between dimensional stability and ionic conductivity. To overcome trade-off limitation, crosslinking process could be applied to polymer matrix.

Crosslinking is an effective option to improve the dimensional stability for the purpose of solving the trade-off of the conductivity and swelling behavior of AEMs because it can improve the mechanical property for AEMs by forming a connecting between polymer chains. Introduction of crosslinking agents occupies conduction sites and can make reduction in ion conductivity of AEMs. Since high crosslinking degree also makes the membrane brittle causing the membrane to break, it is necessary to consider the amount and structure of the crosslinker agent carefully.

C. Bae et al. synthesized elastomeric AEMs by acid-catalyzed Friedel−Crafts alkylation of SEBS polymer backbone using bromoalkylated tertiary alcohols and triflic acid as a catalyst followed by amination with trimethylamine. The 1,6-hexanediamine was additionally used as a crosslinker agent. Properties between crosslinked TMA-functionalized membrane (XLy-SEBS-Cn-TMA-x) and non-crosslinked TMA-functionalized membrane (SEBS-Cn-TMA-x) were compared. As the degree of crosslinking was increased, in-plane swelling and water uptake were remarkably reduced. The in-plane swelling ratios were decreased from 26% to 15%, 13%, and 10% and water uptake were 155%, 70%, 46%, and 28% and for SEBSC5-TMA-0.8 as the degree of cross-linking increased to 0%, 20%, 60%, and 100%, respectively (Table 3). It was demonstrated that the crosslinked SEBS-based AEMs showed similar ion conductivity with non-crosslinked SEBS-C5-TMA-0.8 despite their lower water uptake values and mechanical strength also improved after crosslinking[26].

Zhang and Liu et al. used long flexible chains multi-cation crosslinker for AEMs, which can not only increase the ion conductivity, but help to inhibit the

Table 3. Ion-exchange Capacity, Water Uptake, and Ion Conductivity of TMA-functionalized SEBS- AEMs and Crosslinked SEBS-AEMs[26]

samples	IEC (meq/g)		WU	OH⁻ σ (mS/cm)		
	^{1}H NMR	Titration		30°C	60°C	80°C
SEBS-C$_3$-TMA-0.5	1.12	1.09	110	21	36	50
SEBS-C$_4$-TMA-0.5	1.10	1.12	123	22	37	55
SEBS-C$_5$-TMA-0.5	1.09	1.02	120	20	35	-
SEBS-C$_3$-TMA-0.8	1.62	1.55	150	47	72	93
SEBS-C$_4$-TMA-0.8	1.58	1.51	155	36	60	-
SEBS-C$_5$-TMA-0.8	1.55	1.45	155	23	41	-
XL20-SEBS-C$_5$-TMA-0.8	1.54	1.46	70	30	42	62
XL60-SEBS-C$_5$-TMA-0.8	1.52	1.47	46	33	45	65
XL100-SEBS-C$_5$-TMA-0.8	1.50	1.46	28	29	41	65

swelling behavior of membrane because the hydrophilic/hydrophobic microphase separation morphology builds high speed ion transport channel to achieve more rapid ion transportation. The activation energy reduced with the increasing amount of the multi-cation crosslinkers and it represents that the less energy was required to transport hydroxide in the AEMs as crosslinking degree increases[27].

Z. Li *et al.* applied N, N-dimethyl-1-hexadecylamine as multi-cation crosslinker onto the comb-shaped SEBS polymer backbone synthesized via chloromethylation to improve solubility. The SEBS-C$_{16}$-20C$_4$ membrane displayed the hydroxide conductivity ranging from 35.30 to 77.78 mS/cm at changing temperature. This value was relatively high compared to the previously synthesized benzyltrimethylammonium bearing SEBS membranes, crosslinked quaternized SEBS membranes, quaternized ammonium SEBS membranes with flexible alkyl chain and piperidinium functionalized SEBS membrane[28].

Z. Wang *et al.* addressed an effective method to enhance properties of AEMs by fabricating a crosslinked structure between SEBS backbone and poly (2,6-dimethyl-1,4-phenylene oxide) (PPO). AEMs overcame the trade-off between the ion conductivity and gelation issues of SEBS polymer which was the main disadvantage of SEBS and also showed excellent flexible mechanical properties and chemical stability of

these two outstanding polymers by using this strategy. Multi-cations group and the hard/soft segments of the SEBS backbone helped to form a distinct hydrophilic/hydrophobic morphology, which can significantly improve the ion conductivity. The crosslinked membranes show improved physicochemical properties like relative high hydroxide conductivity or restrained swelling behavior by combining the advantages of these two different polymer backbones. The swelling ratio and water uptake of membranes reduced as increasing content of SEBS and these results demonstrated that introduction of SEBS as well as the crosslinking structure can help control the water uptake and swelling ratio of the membrane. AEMs showed better alkaline stability after a long-term alkaline test than AEMs which used only PPO polymer backbone due to the absence of excellent chemical stability of SEBS polymer backbone[29].

Y. Shi *et al.* fabricated robust SEBS-based AEMs by hydrogenation, chloromethylation, quaternization and alkalization of SBS copolymers. The physically crosslinked structures which is contributed to the mechanical stability of AEMs were formed via the crystalline behavior(T_m = ~ 80°C and ~ 20% of crystalline) of poly(ethylene-co-butylene) phase after hydrogenation of 1,4-butadiene units. The ionic conductivity of AEMs reached 80.5 mS/cm at 80°C and also showed low SR and WU values even at high temperature[30].

3. Conclusions

In this review, anion exchange membranes (AEMs) based on polystyrene-b-poly(ethylene-co-butylene)-b-polystyrene (SEBS) triblock copolymer was mainly discussed. AEMWE are considered as a promising candidate for next-generation water electrolysis because this system uses non-platinum group metal (non-PGM) catalysts which can reduce the manufacturing cost of water electrolysis devices and the high performance. The key component of AEMWE system, anion exchange membranes (AEM), is extensively studied with various materials and strategies. The SEBS copolymer is great candidate for starting materials of AEM due excellent ionic conductivity, easy availability and chemical stability due to less OH- ion attacks in an alkaline environment. Many researches have been conducted to enhance electrochemical and physical properties of AEMs such as ion-conductivity, alkaline stability, water uptake and dimensional stability. So different kinds of aliphatic or aromatic functional groups were introduced onto SEBS polymer backbone. There are still many debates that which structure of functional group and backbone is best for water electrolysis system, but SEBS-based AEM could be a solution for low-cost hydrocarbon backbone based AEM.

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