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Synthesis and Characterization of Various Electrodes for Heterogeneous Ion Exchange Membrane

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ABSTRACT

Ion exchange membranes (IEMs) are generally used as proper media for separation and extractors in diverse electrically driven techniques such as electro dialysis for desalting brackish waters, deconcentrating brine of seawater and production of table salt. They are also efficient tools in extraction efficiency of significant metals of effluents industries and food and pharmacy processing as well as manufacturing basic chemical products. Furthermore, ion exchange membranes play some important part in media protection, treating effluents and many further processes. The homogenous IEMs usually indicate good electrochemical factors but are weak in their mechanical properties, whereas heterogeneous types have acceptable mechanical property but by inadequate electrochemical parameters. In the research, various coated electrodes in heterogeneous ion exchange membrane were synthesized. Also, the effect of various parameter such as multi walled carbon nanotubes (MWCNTs), single walled carbon nanotube (SWCNT), polyvinyl chloride (PVC) and H^+ and Cl^- resin on properties of these electrodes was studied. Based on BET analysis, FSWCNT+ electrode have the higher surface area in coated electrodes and so can be more performance in separation of target compounds in various environment. Field emission scanning electron microscope, and cyclic voltammetry used for investigating of the coated electrodes and their properties. Results of cyclic voltammetry excessed that electrodes electrochemical properties were improved with utilizing single wall carbon nanotube (SWCNT) in electrode. In additional, the modified electrode such as functionalized single wall CNT by resin H^+ (FSWCNT1+) showed further proper electrochemical characteristics and property in comparison to others.

INTRODUCTION

Up to day, ion exchange membranes (IEMs) are generally applied as suitable media for separation and extractors in diverse electrically driven methods including electro dialysis for desalting brackish waters, deconcentrating brine of seawater and production of table salt. They are also efficient tools in extraction efficiency of important metals of effluents industries and food and pharmacy processing as well as manufacturing basic chemical products. Furthermore, ion exchange membranes play some significant part in media protection, treating effluents and many further processes [1-5]. In the processes, the ion interactions by membrane, water and by each other occur in difficult fashions. Knowledge of the electro kinetic properties are large obtaining parameter behind decisions about their usage in separation methods [6-8].

The power production, resource recovery and pollution monitoring are all significant parameters enumerated for the IEMs development and utilization [9,10]. Preparing inexpensive membranes by special adapted physico-chemical characteristics may be as vital step in future

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chemical and waste treatment utilizations [4,11]. Variation of functional groups, selection of various polymeric media, polymers blending, utilize of different additives, alteration of cross-link density, nature of surface layer and further uniform distribution of functional groups are significant ways to obtain suitable IEMs. The homogenous IEMs generally indicate excellent electrochemical conditions but are weak in their mechanical properties, whereas heterogeneous types have acceptable mechanical property but by inadequate electrochemical parameters. The heterogeneous ion-exchange membranes explained which these membranes can be obtained with calendaring ion exchange materials into an inert plastic film of inert film forming polymers and ion exchange materials and subsequently milling the format stock in another way using applying dispersion of resin materials in polymeric solution and casting protocol [1,2,4].

Synthesis of the heterogeneous IEMs using proper properties for the utilization in electro dialysis goals correspond to water recovery and waste water treatment was the aim of researches. For the goal, polycarbonate (PC)/styrene-butadiene-rubber (SBR) blend heterogeneous IEMs by various electrodes were synthesized using solution casting protocols applying ion exchange resin powder as functional groups agent and tetrahydrofuran as solvent.

Additionally, multi-walled carbon nanotubes (MWCNTs) single walled carbon nanotube (SWCNT), polyvinyl chloride (PVC) and resin H^+ and Cl^- were applied as inorganic filler additive in the membrane to improve of properties. MWCNTs are filler particles by very interesting features and capacity including superior electrical and mechanical properties. The incorporation of inorganic materials or fillers especially carbon nanotubes into polymeric particles has been evaluated in many usages to increase the mechanical and chemical stabilities of polymer media in high temperature and strongly oxidizing environment and also to increase the separation properties [12, 13]. So, adding these materials such as carbon nanotubes and resin to heterogeneous IEMs can be improve chemical stabilities for separation and extractors in diverse electrically driven methods including electro dialysis for desalting brackish waters, deconcentrating brine of seawater and production of table salt.

Various electrodes in the structure of membrane such as Pt [2], wire-shaped and carbon electrode [14], and Ag/AgCl were used [15]. In the paper to investigate of the desalting and chemical properties of the membrane, various electrodes of graphite electrode were synthesized. The main aim of this research is to fabricate heterogeneous ion exchange membranes with electrochemical properties suitable for use in the electro dialysis process and for water and wastewater treatment. In this study, carbon nanotubes were used to improve the electrochemical properties of heterogeneous cation exchange membranes. Novelty of the method is introducing suitable coating of graphite electrode by functionalized single wall carbon nanotube by resin H+ and providing its chemical better performance.

MATERIALS AND METHODS

Chemical

All chemical and reagent were of analytical grade and achieved of Merck (Darmstadt, Germany). Ultrapure water was applied for the experiment. Carbon nano-tube (Single wall) with Plasmachem GmbH, Berlin, Germany as modifier of electrode.

Apparatus

A field emission scanning electron microscope (FESEM) (KY KY EM8000F, China) was applied for taking electron microscope images. The Brauner-Emmett-Teller (BET) surface areas, pore volume, average pore size and nitrogen adsorption/desorption analysis of the electrode were determined with nitrogen physisorption applying a Quanta Chrome Nova 2000 (USA). Degassing of each sample was performed in nitrogen at 300 ^{0}C for 4 h for to achieve the BET surface areas, pore volumes and average pore sizes.

Coating of electrodes

Considering that improving the physical and electrical properties of the electrodes can improve the performance of the capacitive ionizer cell, therefore, according to Table 1, some modifications were made to improve the electrodes. For this purpose, two methods used in research [1,2] have been used.

Electrode of number of 1 was prepared with heating of graphite rod for 30 min in 350 ^{0}C [1] (called Heated). Electrode 2 was synthesized by adding 30 mg of single walled carbon nanotube (SWCNT) and 20 mL distilled water to graphite rod for 30 min (first dried in temperature of 40^{0}C and then placed in 350 ^{0}C for more stability) and then ultrasound (2 h) (named SWCNTDW) [1]. Electrode 3 was obtained by dipping of graphite rod in 33 mg SWCNT, 1g polyvinyl chloride (PVC) and 20 mL tetrahydrofuran (THF) for 30 min in 350 ^{0}C and then stirred (for 4 h) and ultrasound (1 h) (called SWCNTPVC) [2]. Electrode 4 was achieved by immerging in 60 mg Multi wall CNT (MWCNT) and 20 mL THF for 30 min in 350 ^{0}C (called MWCNT). Electrode 5 prepared by mixing in 50 mg functionalized single wall CNT (FSWCNT), 1g resin H^+, 1g PVC and 20 mL THF for 30 min in 350 ^{0}C and subsequently stirred (4 h) and ultrasonic bath (1 h) (named functionalized single wall CNT by resin H^+ ($FSWCNT^+$)). Electrode 6 synthesized by adding to mixture of 50 mg functionalized single wall CNT, 1g resin Cl^-, 1g PVC and 20 mL THF for 30 min in 350 ^{0}C and subsequently stirred (4 h) and ultrasonic bath (1 h) (named $FSWCNT^-$). Table 1 shown the various method for modification of electrode.

Preparation of home-made membranes

Preparation of home-made membranes was performed similar Hosseini et al. [2]. The heterogeneous IEMs were synthesized with casting solution protocol and inversion protocol. In order to undertake the membranes preparation, resin was dried in oven (in 30^{0}C for 48 h) and subsequently pulverized into fine materials in a ball mill and sieved to the desired mesh size. The ion exchange resin by suitable materials size (-300 + 400 mesh) was applied in

membranes preparation. The preparation proceeded with dissolving the polymers binder into THF solvent in a glass reactor equipped by a mechanical mixer for 6 h. This was followed with dispersing a specific quantity of grinded resin material as functional groups agents and carbon nanotube as filler additive in polymeric solution. The solution was mixed vigorously at 25 ^{0}C to achieve uniform material distribution in the polymeric solution. Also, for better dispersion of materials and breaking up their aggregates, the mixture was placed for 2 h in ultrasonic bath. Subsequently, the mixing process was repeated for 30 min applying a stirrer. The mixture was cast on a glass plate in room temperature. The membranes were dried at 25^{0}C and immersed in ultra-pure water. Finally, the membranes were pretreated utilizing immersing in HCl and NaCl solutions.

Table 1
Coating of electrodes

No	Processes	Method	Compounds	Type of added compounds	Ref.
1	30 min in 350 ^{0}C	-	-	heated	[1]
2	30 min in 350 ^{0}C	Ultrasonic bath (2 h)	30mg SWCNT +20 mL distilled water	SWCNT DW	[1]
3	30 min in 350 ^{0}C	Stirred (4 h)-Ultrasonic bath (1 h)	33 mg SWCNT+ 1g PVC + 20 mL THF	SWCNT PVC	[2]
4	30 min in 350 ^{0}C		60 mg Multi wall CNT (MWCNT) + 20 mL THF	MWCNT	[2]
5	30 min in 350 ^{0}C	Stirred (4 h)-Ultrasonic bath (1 h)	50 mg FSWCNT + 1g resin H + 1g PVC + 20 mL THF	FSWCNT +	[2]
6	30 min in 350 ^{0}C	Stirred (4 h)-Ultrasonic bath (1 h)	50 mg FSWCNT + 1g resin Cl + 1g PVC + 20 mL THF	FSWCNT-	[2]

In order for the particles to settle well in the coated electrodes, it is necessary to heat treat them [3]. For this purpose, the stabilization operation should be done for about half an hour and at a temperature between 300 and 900 degrees Celsius. In most cases, the selected temperature was about 350 degrees Celsius [3, 4]. In order to be able to observe the effect of the heat treatment on the electrodes alone, some simple electrodes (uncoated) were placed in the furnace.

Laboratory Analyses

For a detailed review of the cases related to the coatings, the results of surface analysis such as light microscope images, FESEM, cyclic voltammetry (CV), etc. are given below.

FESEM

Figs 1 to 7 indicates FESEM images of the modified graphite electrodes such as Reference, Heated, MWCNT, SWCNTDW, SWCNTPVC, FSWCNT+, and FSWCNT-. It can be clearly seen that the images of the modified electrodes are different of reference electrode. It is apparent from the images which the dispersion of FSWCNT+ is acceptably uniform and regular. So, it will be possible that better performance of FSWCNT+ observed.

BET analysis

BET analysis was performed for surface areas of the electrodes. The results shown that surface area of electrodes are: MWCNT (4.7668 m^2/g), SWCNTDW (3.6907 m^2/g), SWCNTPVC (3.6612 m^2/g), FSWCNT+ (5.3251 m^2/g), FSWCNT- (3.9238 m^2/g), (FSWCNT2- (3.7837 m^2/g) and Reference (2.228 m^2/g). FSWCNT+ showed the better surface area.

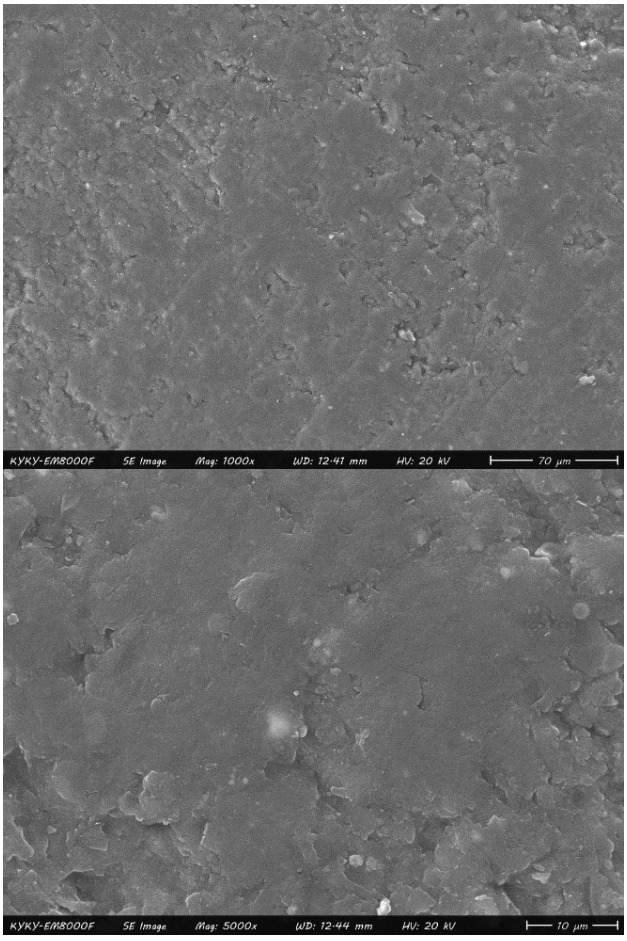

Fig. 1. Reference graphite the electrode

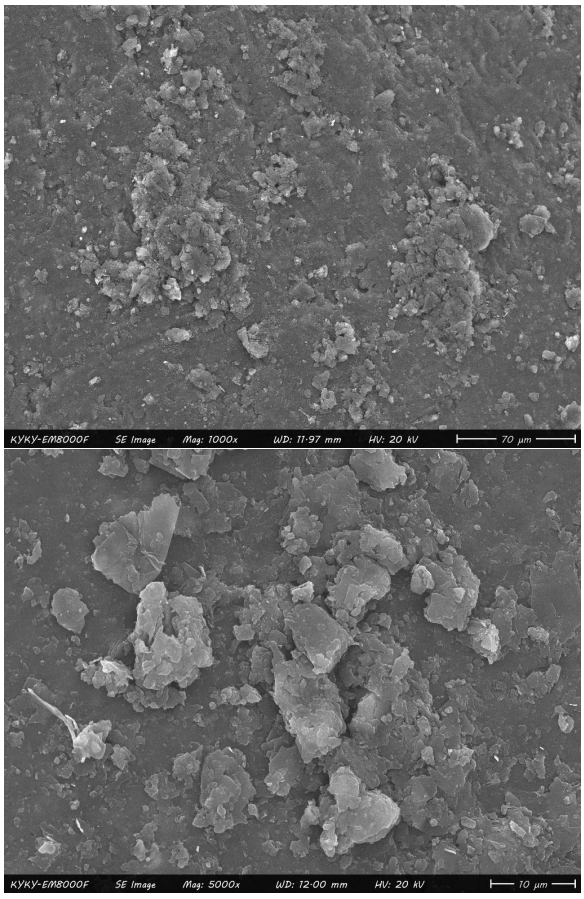

Fig. 2. Heated graphite the electrode

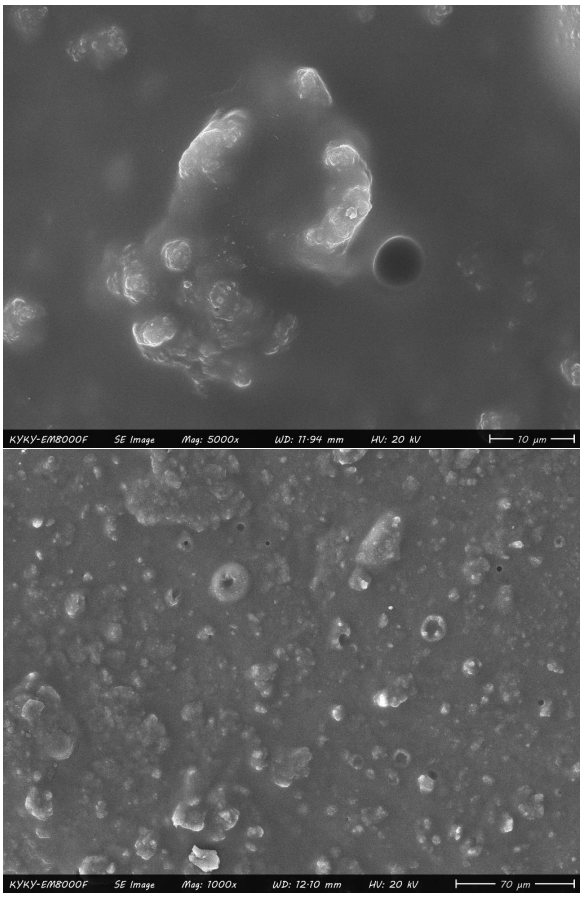

Fig. 4. The SWCNTPVC electrode

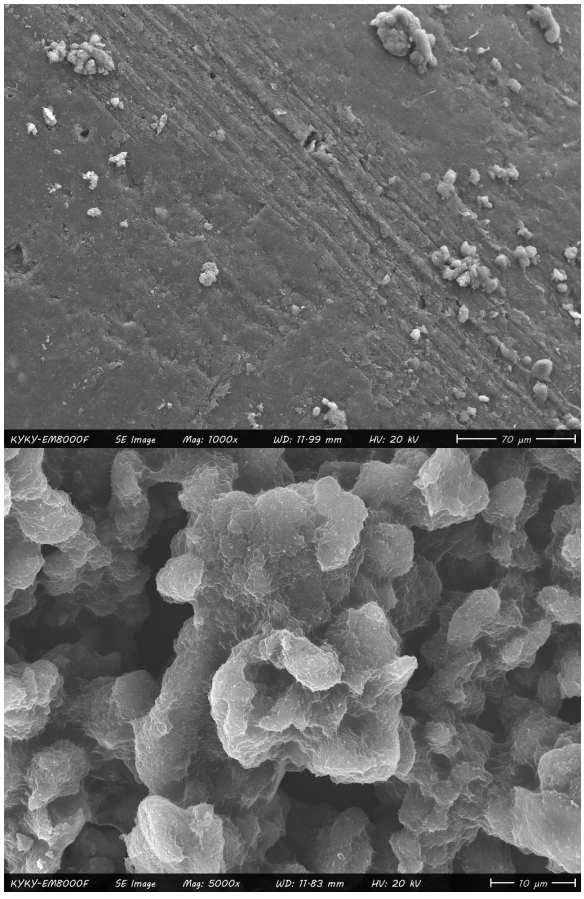

Fig. 3. Added MWCNTs (in THF) to the electrode

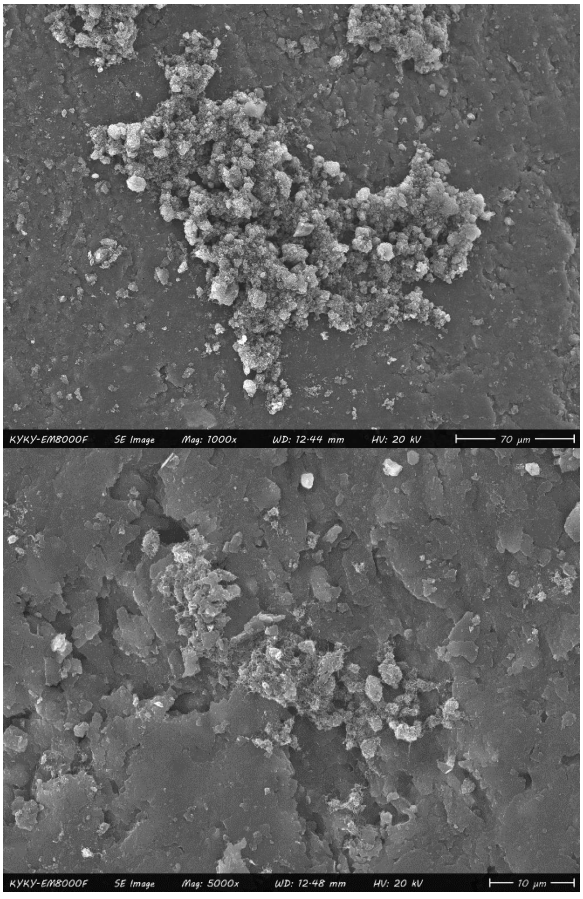

Fig. 5. The SWCNTDW electrode

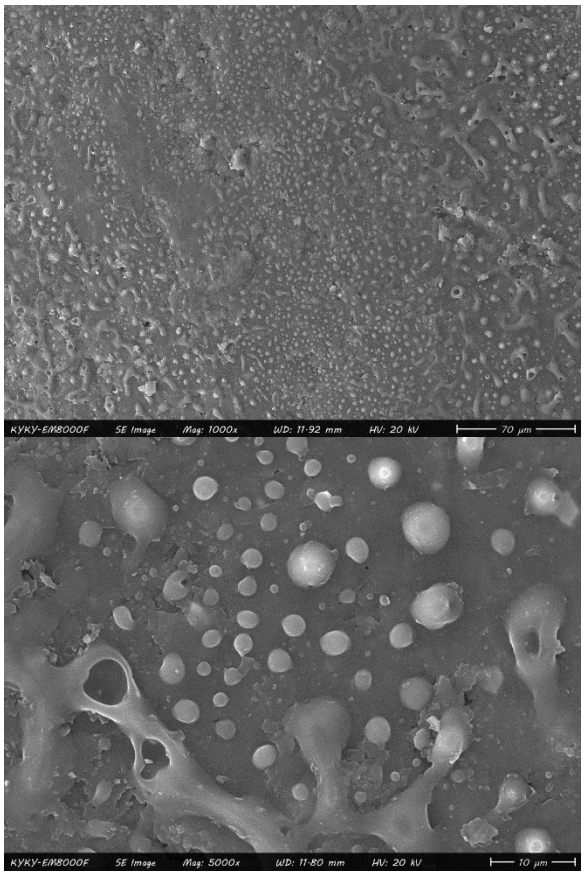

Fig. 6. The FSWCNT+ electrode

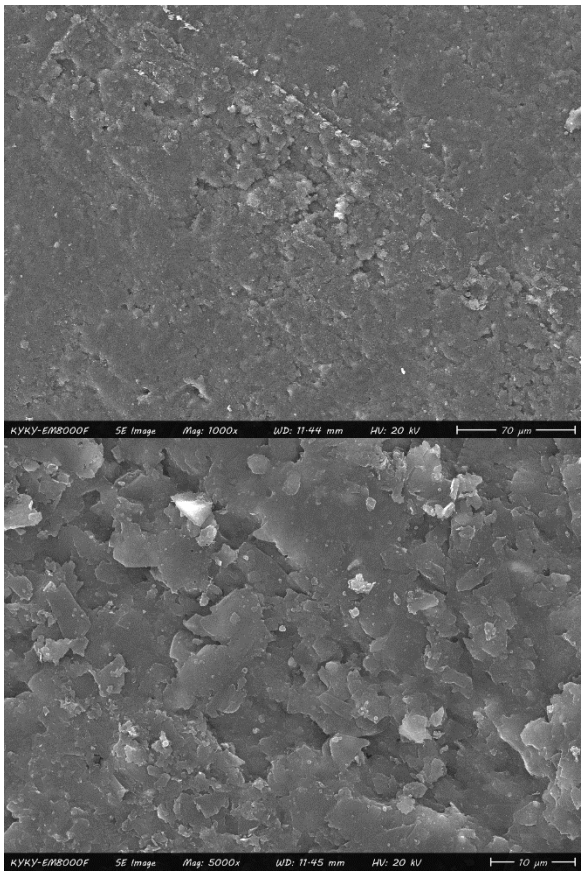

Fig. 7. The FSWCNT- electrode

Effect of the electrodes on cyclic voltammetry (CV)

Effect of the mentioned electrodes (Reference, Heated, MWCNT, SWCNTDW, SWCNTPVC, FSWCNT+, and FSWCNT-) on response of cyclic voltammetry (CV) were investigated. Fig. 8 shown that the best performance (response of CV) related to FSWCNT+ due to increase of capacitance of the electrode (the best current).

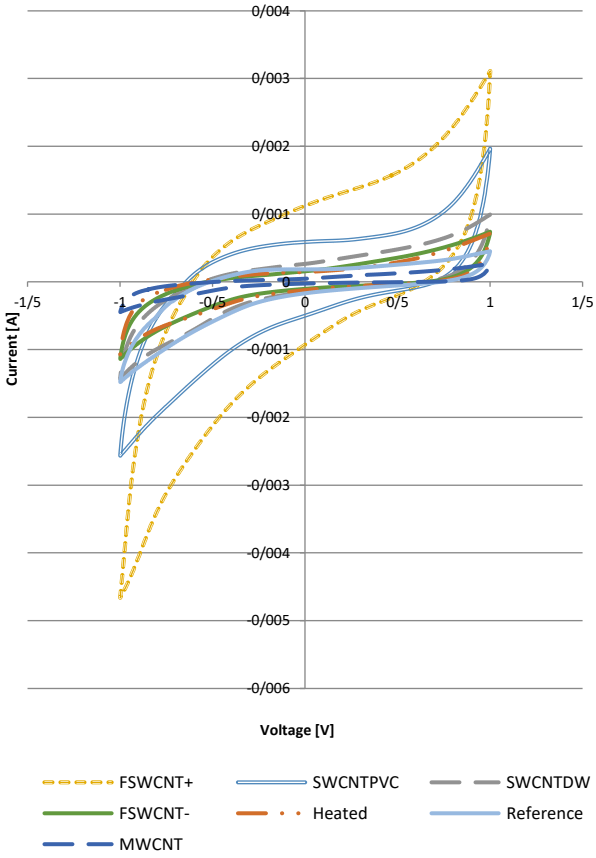

Fig. 8. Effect of the electrodes on cyclic voltammetry (CV)

DISCUSSION

Based on BET analysis, FSWCNT+ electrode have the higher surface area in coated electrodes and so can be more performance in separation of target compounds in various environment. Additionally, FSWCNT+ electrode has separation media via hydrophobic interaction in carbon nanotubes (CNTs) for nonpolar compounds. Also, with existence resin H^+ in the coated electrode, it can be used for separation of various ions and ionic compounds from media. So, FSWCNT+ electrode has suitable separation media for isolation of various molecules and targets. The transport mechanism in diffusion dialysis is more complex than in conventional dialysis due to the electrostatic interaction between positive and negative charges and the electroneutrality requirement. The process and equipment design is very similar to that used in conventional dialysis. In diffusion dialysis the driving forces for the transport of ions through the ion-exchange membrane are gradients in their chemical potentials only. There is no external electrical potential applied.

CONCLUSION

In the research, various electrodes including Reference, Heated, MWCNT, SWCNTDW, SWCNTPVC, FSWCNT+, and FSWCNT- were synthesized and characterized by field emission scanning electron microscope and cyclic voltammetry. The modification performed a significant effect in chemical and electrochemical properties on used electrodes in ion exchange membranes. As a conclusion, the result of FSEM and cyclic voltammetry shown that FSWCNT+ electrode can be possible better performance in comparison with other electrodes.

ACKNOWLEDGEMENT

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CONFLICT OF INTEREST

The authors have declared no conflict of interest.

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