SULFONATED POLYETHERSULFONE AND FUNCTIONALIZED MULTIWALL CARBON NANOTUBES/ POLYVINYLPYRROLIDONE NANOCOMPOSITE BASED HEMODIALYSIS MEMBRANE

MUHAMMAD IRFAN

UNIVERSITY TEKNOLOGI MALAYSIA

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MUHAMMAD IRFAN

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DEDICATION

I would like to dedicate this thesis to my beloved wife,

MASOOMA

Lovely children

Zavier and Zimal,

And especially my **Parents** for their prayers, endless support and encouragement.

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In the name of ALLAH, the Almighty, the Most Gracious and the Most Merciful, Alhamdulillah, all praises be to Allah for His countless blessings and granting me the health, strength, and time for the completion of my PhD Thesis.

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ABSTRACT

Chemical modification of polymer and blending of suitable additives are the common methods used to improve the properties of polyethersulfone (PES) based hemodialysis membranes. In this research work, both methods are adopted and novel nanocomposite based additives were synthesized and blended with PES alone; and then with chemically modified PES (sulfonated PES (S-PES)). The whole research work was divided into three phases. In the first phase, the nanocomposites (NCs) were formed by mixing together the acid functionalized multiwall carbon nanotubes (f-MWCNT) and two different grades of polyvinylpyrrolidone (PVP-k90 and PVP-k30) in dimethylformamide and subsequently blended with PES. The f-MWCNT contained some hydrophilic functional groups (–COOH, and –OH) and heredity hydrophobic carbon part, which made it dual nature. On one side, it carbon part created sites for attachment for the hydrophobic polymer (PES) by hydrophobic–hydrophobic interaction and π–π stacking, whereas on the other side, its hydrophilic acid and hydroxyl groups attracted the hydrophilic sides of PVP by hydrogen bonding, dipole–dipole interaction and dispersion forces. Thus, f-MWCNT acted as the anchoring material between the PVP and PES in the membrane that also greatly reduced the leaching process of the additives and stabilize the membrane composition as shown by elution ratio test. The Fourier transform infrared spectroscopy spectra of fabricated membranes revealed that both types of NCs were physically bonded with PES by hydrogen bonding and the addition of NCs to PES, improved the internal capillary system of membranes as confirmed by field emission scanning electron microscope analysis. The results showed that f-MWCNT/PVP-k90 based membranes exhibited better performance than f-MWCNT/PVP-k30 based membranes in terms of flux rate, rejection rate and biocompatibility. The results from dialysis of uremic solutes unveiled that membrane formed by PVP-k90 based NCs demonstrated superior performance with 56.30%, 55.08% and 27.90% clearance ratio of urea, creatinine and lysozyme solutes, respectively. In the second phase, two best performance membranes of f-MWCNT/PVP-k90 NCs based were selected and then blended with variable ratio of S-PES. The outcome indicated that the blending of S-PES polymer, further enhanced the membrane biocompatibility and reduced the protein adsorption (bovine serum albumin, 55% and lysozyme, 65%), hemolysis process (74.80%) and illustrated longer clotting times than pristine and non-sulfonated membranes. The clearance ratio of uremic solutes was also improved and reached up to 57.3%, 57.1% and 32.4% of urea, creatinine and lysozyme, respectively. Thus, the blending of S-PES and NCs in the PES membrane greatly improved the biocompatibility and removal ability of uremic solutes. In the third and final phase, the hollow fiber (HF) membranes were spun using S-PES and PVPk90/f-MWCNT based NCs and the HF membrane characteristics and dialysis performances were evaluated. The results showed that HF membrane had a good flux rate (29.8l/h.m^2.bar), low molecular weight cut off (29-34 kDa) than pristine PES membranes. The dialysis tests confirmed that the HF membranes illustrated 72.7%, 75.1% and 35.4% clearance ratio of urea, creatinine and lysozyme solutes, especially. Thus, the blending of S-PES and NCs in the PES membrane highly improved the biocompatibility and removal ability of uremic solutes and it can be used in commercial grade dialyzers.

ABSTRAK

Pengubahsuaian kimia untuk polimer serta pengadunan bahan tambah yang bersesuaian adalah kaedah biasa yang dilakukan untuk menambahbaik ciri-ciri bagi membran hemodialisis yang berasaskan polietersulfon (PES). Dalam kerja penyelidikan ini, kedua-dua kaedah digunakan iaitu nano-komposit (NCs) novel berasaskan bahan tambah telah disintesis kemudian diadunkan dengan PES sahaja; dan kemudiannya PES diubahsuai secara kimia (PES tersulfonat, (S-PES)). Penyelidikan ini telah dibahagikan kepada tiga fasa. Dalam fasa pertama, NCs telah dibentuk dengan mencampurkan bersama-sama tiub nano karbon berbilang dinding yang difungsikan dengan f-MWCNT dan dua jenis polivinilpirolidon yang berbeza gred (PVP-k90 dan PVP-k30) dalam dimetilformamid dan seterusnya diadunkan dengan PES. Asid f-MWCNT mengandungi beberapa kumpulan berfungsi hidrofilik (-COOH dan -OH) dan bahagian karbon hidrofobik, yang menjadikan ia mengandungi dua sifat. Pada satu bahagian, karbon menyediakan tapak untuk lekatan bagi polimer hidrofobik (PES) dengan interaksi hidrofobik-hidrofobik dan penyusunan π-π, manakala di sisi lain, asid hidrofilik dan kumpulan hidroksil ditarik ke bahagian hidrofilik PVP oleh ikatan hidrogen, interaksi dwikutub-dwikutub dan penyebaran daya. Oleh itu, f-MWCNT telah bertindak sebagai bahan teras antara PVP dan PES dalam membran yang juga dengan banyaknya mengurangkan proses larut lesap bahan tambah dan menstabilkan komposisi membran seperti yang ditunjukkan oleh ujian nisbah elusi. Keputusan spektrum spektroskopi inframerah transformasi Fourier untuk membran yang terhasil mendedahkan bahawa kedua-dua jenis NCs ini secara fizikal terikat dengan PES oleh ikatan hidrogen dan penambahan mereka ke PES telah menambah baik sistem kapilari dalaman membran seperti yang disahkan oleh analisis mikroskop elektron pengimbas pancaran medan. Hasil kajian menunjukkan bahawa membran berasaskan f-MWCNT/PVP-k90 menunjukkan prestasi lebih baik daripada membran berasaskan f-MWCNT/PVP-k30 dari segi kadar fluks, kadar penolakan dan keserasian bio. Keputusan dialisis bahan larut uremik menunjukkan bahawa membran dibentuk dengan PVP-k90 berasaskan NCs telah menunjukkan prestasi yang lebih baik dengan 56.30%, 55.08% dan 27.90% masing-masing bagi nisbah kepelepasan bahan larut urea, kreatinin dan lisozim. Dalam fasa kedua, dua membran yang mempunyai prestasi terbaik berasaskan f-MWCNT/PVP-k90 NCs dipilih dan kemudian diadunkan dengan S-PES mengikut nisbah yang berlainan. Hasilnya menunjukkan bahawa pengadunan S-PES polimer telah meningkatkan lagi keserasian bio membran dan telah mengurangkan penjerapan protein (bovin serum albumin, 55% dan lisozim, 65%), proses hemolisis (74.80%) dan menunjukkan pembekuan lebih panjang berbanding asal dan membran tanpa sulfonat. Nisbah pelepasan bahan larut uremik juga bertambah baik dan mencapai sehingga 57.3%, 57.1% dan 32.4% masing-masing untuk urea, kreatinin dan lisozim masing masing. Oleh itu, pengadunan S-PES dan NCs di dalam membran PES telah menambah baik keserasian bio dan keupayaan penyingkiran bahan larut uremik. Dalam fasa ketiga iaitu fasa terakhir, gentian berongga (HF) membran telah dihasilkan oleh S-PES dan PVPk90/f-MWCNT berasaskan NCs dan ciri-ciri membran HF dan prestasi dialisis telah dinilai. Hasil kajian menunjukkan bahawa membran HF mempunyai kadar fluks yang baik (29.8 l / h.m^2.bar), berat molekul dengan had potongan yang rendah (29-34 kDa) berbanding dengan membran PES asli. Ujian dialisis mengesahkan bahawa membran HF menunjukkan 72.7%, 75.1% dan 35.4% masing-masing bagi nisbah pelepasan urea, kreatinin dan lisozim bahan larut. Oleh itu, pengadunan S-PES dan NCs dalam membran PES dapat menambah baik keserasian bio dan keupayaan penyingkiran bahan larut uremik dan ia boleh digunakan dalam dialisis gred komersial.

TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

AA	-	Acrylic acid
AFM	-	Atomic force microscopy
AN	-	Acrylonitrile
APTT	-	Activated partial thrombin time
ß2-m	-	Beta-2- microglobin
BSA	-	Bovine serum albumin
BPA	-	Bisphenol-A
BDO	-	1,4-butanediol
C	-	Carbon
CA	-	Contact angle
CNT	-	Carbon nanotube
-COOH	-	Carboxylic group
$ClSO_3H$	-	Chlorosulfuric acid
CKD	-	Chronic kidney disease
PEO	-	Polyethylene oxide
CPES	-	Carboxylic polyethersulfone
DNA	-	Deoxyribonucleic acid
DI	-	De-ionized
DMF	-	Dimethyleformamide
DNA	-	Deoxyribonuclic acid

DG	-	Degree of grafting
DS	-	Degree of sulfonation
EDX	-	Energy dispersive X-ray spectroscopy
EG	-	Ethylene glycol
FESEM	-	Field emission scanning electron microscopy
FDA	-	Food and Drug Administration
f-MWCNTs	-	Functionalized multiwall carbon nanotubes
FTIR	-	Fourier transforms infrared spectroscopy
FRSP	-	Free radical solution polymerization
GO	-	Graphene oxide
HD	-	Hemodialysis
HP	-	Hemolytic percentage
IEC	-	Ion exchange capacity
Max	-	Maximum height
mPEG	-	Methoxylpoly (ethylene glycol)
M_{dry}	-	Dry membrane
M_{wet}	-	Wet membrane
MF	-	Microfiltration
Mid	-	Average between the minimum and maximum height
Min	-	Minimum height
MPC	-	Methacryloyloxyethyl-phosphoryl-choline
MWCNT	-	Multiwall carbon nanotube
N	-	Nitrogen
NCs	-	Nanocomposites
NF	-	Nanofiltration

Nps	-	Nanoparticles
O	-	Oxygen
OH	-	Hydroxyl
P	-	Poly
PA	-	Polyamides
PAN	-	Polyacrylonitrile
PBS	-	Phosphate-buffered solution
PC	-	Phosphorylcholine
PES	-	Polyethersulfone
PEG	-	Polyethylene glycol
PEO	-	Poly ethyleneoxide
PMMA	-	Polymethyl methacrylate
Pmp	-	Persons per million populations
PP	-	Polypropylene
PT	-	Prothrombin time
PRT	-	Plasma recalcification time
PSf	-	Polysulfone
PU	-	Polyurethanes
PVP	-	Polyvinylpyrrolidone
RAFT	-	Reversible addition fragmentation chain transfer polymerization
RO	-	Reverse osmosis
SO3	-	Sulpher trioxide
$-SO_3H$	-	Sulfonic group
SiO_2	-	Silicon dioxide

SDS	-	Sodium dodesylesulphate
Semi-IPN	-	Semi-interpenetrating network
SMA	-	Poly (styrene-alt-maleic anhydride)
S-PES	-	Sulfonated polyethersulfone
SPEES	-	Sulfonated polyether-ethersulfone
St	-	Styrene
SWCNTs	-	Single-walled carbon nanotubes
TGA	-	Thermogravimetric analysis
TiO_2	-	Titanium dioxide
TT	-	Thrombin time
UF	-	Ultrafiltration
VP	-	Vinylpyrrolidone
WBCT	-	Whole blood clotting time
XRD	-	X-Ray Diffraction
ZrO_2	-	Zirconium dioxide

LIST OF SYMBOLS

A	-	Area of the flat sheet membrane (m^2)
C_f	-	Concentrations in feed solution (%)
C_p	-	Concentrations in permeate solution (%)
C_0	-	Concentration, when time = 0 (%)
C_t	-	Concentration, when time = 1-4 hours (%)
d_o	-	Outer diameter of fiber (μm)
D_{pc}	-	Absorbance of the positive control
D_t	-	Absorbance of the negative control
Dw	-	Solute diffusion coefficient
δ	-	Membrane thickness (μm)
€	-	Porosity
f(q)	-	Friction coefficients
–ΔGSL	-	Surface free energy
Jp	-	Protein flux ($Lm^{-2}h^{-1}$)
Jw	-	Water flux ($Lm^{-2}h^{-1}$)
L	-	Hydraulic permeability
ι	-	Effective length of fiber (m)
M	-	Mole
N	-	Number of the fiber
n	-	Number of membranes

π	-	Pi
P_m	-	Diffusive permeability
R	-	Protein rejection (%)
Ra	-	Roughness average (nm)
R_{FR}	-	Flux recovery ($Lm^{-2}h^{-1}$)
R_{ir}	-	Irreversible resistance
r_p	-	Pore radius (nm)
Rpv	-	Peak-to-valley line (nm)
Rq	-	Root-mean-squared roughness (nm)
R_r	-	Reversible resistance (%)
R_t	-	Total resistance (%)
r_m	-	Average pore radius (nm)
r_s	-	Radius of solute (nm)
Rz	-	Ten points average roughness (nm)
S	-	Surface area of the membrane (cm^2)
SF	-	Factor of steric hindrance
σ	-	Staverman reflection coefficient
μ	-	Micro
V	-	Volume (m^3)
δ_w	-	Density of water (g/cm^3)
γ_L^T	-	Total surface tension of water

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Overview

The developments in science and technology have assisted mankind to live longer and consumed a large amount of world's resources. However, the need to sustain life requires a good health condition with better and advance curing methods and therapy. Human life is invaluable and there are various factors that affect the survival and quality of life. Amongst them are diseases that damaged the human kidneys. Kidney, is a bean shaped organ, made up by functional units, called nephrons, which received the blood from the renal arteries and after processing forced the waste solutes and fluids to move into the urinary bladder and return the purified blood to the body circulation. The two key mechanisms are served by the kidney. One is the plasma filtration via UF technique that separate the extracellular fluid in the glomeruli and the second is the removal of extra water through active and passive tubular transport system that also contain dissolved uremic solutes and electrolytes (Findlay et al., 2015).

The origin of kidney disease may be metabolic, vascular, immunologic, degenerative, infectious or genetic. The renal failure might be chronic that last up to months or years or acute stay for some days or weeks. Acute failure is generally related with tubular necrosis, acute glomerulonephritis, ischemia or poisoning with nephrotoxins that involved excessive loads of free hemoglobin, aminoglycosides and heavy metals. The chronic renal failure is typically initiated by hypertension, vascular disease (e.g. diabetes) or chronic glomerulonephritis. If the proper treatment

is not taken for acute renal failure, then it will cause the chronic renal failure. The human kidney can continue to work until 90% of its function has been lost and keep deteriorating and renal failure exceeds 95%. At this stage, survival becomes impossible without the replacement therapy (Findlay et al., 2015).

Supportive therapy like kidney transplant, peritoneal dialysis or hemodialysis (HD) are required to prolong life when the human kidney fails to work either as a result of painful injury or any other disease. Globally, the number of ESRD patients was estimated to be 3,010,000 with ≅ 7% growth rate at the end of 2012. Based on a previous report, 89% of dialysis patients opted for HD and only 11% chose peritoneal dialysis (National Institute of Diabetes and Digestive and Kidney Diseases).

Hemodialysis (HD) method is a significant clinical therapy that can eliminate toxic metabolites from the blood of patients whose kidney fail to perform and the patient suffered into end stage renal diseases (ESRD) (Miller et al., 2010). This method has benefited approximately more than one million people per year all over the world and the prevalence rate of ESRD is increasing globally (Saran et al., 2015). The annual renal data report of United State has covered 57 countries and showed that the ESRD incidence rates varied significantly across the globe. United States, Mexico and Taiwan described the maximum occurrence of treated ESRD at 363-458 persons per million population (pmp). Indonesia, Thailand, Portugal, Republic of Korea, Japan and Singapore demonstrated 208–308 pmp, whereas other countries showed less than 96 reported incidence rates of treated ESRD patients. The highest growths in the treatment of ESRD were observed in Mexico (122%), Malaysia (176%), Philippines (185%), Russia (249%), Bangladesh (629%) and Thailand (1210%) in 2012/2013 period (Saran et al., 2015).

According to the U.S. Renal data system (2014) that the medical expenditure of ESRD patients in the USA increased from $30.4 billion to $30.9 billion from 2012 to 2013 with 1.6% growth rate. Whereas, the total global expenditures for ESRD patients reached up to $437 billion. It is also estimated that the growth rate of ESRD patients will increase in the future due to different diseases, poor quality lifestyles

and improper medical treatments (Saran et al., 2015). Since the necessity of HD dialysis will be increasing in the future, thus the dialysis therapy is considered as a multi-million dollar industry (National Institute of Diabetes and Digestive and Kidney Diseases).

In Malaysia in 2014, the total registered patients undergoing dialysis were 34,767 and 6107 new HD cases were registered during the same year. In the last ten years, the rate of dialysis acceptance was doubled and reached 203 pmp. Table 1.1 represents the short summary of the registered and treatment-type of dialysis patients in Malaysia. Moreover, there are 758 dialysis centers in the whole country and 53.2% were funded by government, 12.9% and 31.2% by charity and self supported, respectively (Goh et al., 2016).

Table 1.1: The registered dialysis patients and the type of treatment supplied to ESRD in Malaysia (Goh et al., 2016).

Year	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014
New Dialysis patients	3167	3709	4103	4640	4952	5305	6073	6690	6985	7055
New Transplants	172	151	112	131	141	128	127	107	98	81
Dialysis deaths	1515	1820	1987	2191	2601	3047	3292	3645	4001	4015
Transplant deaths	49	58	47	59	49	48	55	64	56	45
Dialyzing at 31st December	13356	15080	17084	19388	21590	23709	26328	29223	32026	34767
Functioning transplant at 31st December	1716	1771	1788	1808	1852	1881	1907	1891	1870	1844

During the last decades, membrane technology plays a central role in the purification and separation of the biotechnological products. The use of membrane based modules progressively enhances both industrial processes and academic science of engineering (van Reis et al., 2007). The membrane filtration system offers robust performance, easy availability, less processing time and low cost compared to

the other available techniques. These benefits extend the microfiltration (MF) and ultrafiltration (UF) membrane systems into various biomedical applications like hemofiltration, hemodiafiltration, hemodialysis, plasma collection and plasmapheresis (Samtleben et al., 2003; Tullis et al., 2002; Werner et al., 1996; Zhao et al., 2013).

The innovation of asymmetry membrane structures introduced the new era. Nowadays, a number of new polymeric materials are available whose properties can be upgraded by the use of proper additives and new formulation. In 2004, Membrana GmbH launched a performance enhancement technology, in which they used spacer yarns woven pattern in HF bundles and optimized the dialysate flow (Membrana, 2004). Asahi Kasei Medicals Co. Ltd. had launched a PSf dialyzer modified by vitamin-E to reduce the oxidative stress, which reduced the tissue damaging and also shows the good biocompatibility to the body immune system during HD therapy (Sasaki, 2006). Some studies suggested that high flux dialyzer membrane was better due to a lower death rate than the low flux dialyzers (Bloembergen et al., 1999; Woods et al., 2000). The Eknoyan et al. (2002) reported a HD study of 72 patients in 15 clinical centers and checked the mortality and morbidity. He found that neither the high flux rate nor higher dialysis dose enhanced the mortality and morbidity rate among patients. Besides, no much difference was found in the clearance rate of urea solute for the both high and low flux membranes (Eknoyan et al., 2002). The previous study of Bonomini et al. (1995) also provided the same results as Eknoyan et al. (2002) (Bonomini et al., 1995). Moreover, House et al., (2000) found that high flux membranes, increased the cardiovascular risk in the ESRD patients.

The chief constituent of a hemodialysis instrument is a semipermeable membrane, whose chemical composition has great effect on a patient's health either in terms of membrane biocompatibility or uremic waste removal (Daugirdas et al., 2012; Kumar et al., 2014; Nie et al., 2012; Zhao et al., 2013). Some of the most commonly used materials to make HD membranes include PSf, PES, polyamide, ethylene vinyl alcohol copolymers, cellulose triacetate, polymethylmethacrylate and polyacrylonitrile (Matsuda, 2011). Currently, synthetic polymers are used in most of the dialyzer membranes, 93% of which are derived from the parent polyarylsulfone

family, with 22% produced from PES and 71% from PSF (Bowry et al., 2010). Each polymeric membrane has its own advantages or disadvantages and complications may arise, when the membranes are judged only by their polymer names. Due to the varying membrane compositions, membranes with the same polymer names may differ in their adsorption, flux properties and hemocompatibility characteristics. Membranes in the new super high-flux dialyzers are primarily PSf and PES and future trends to use PES as main hemodialyzer materials are increasing because PES has an equivalent property to that of PSf, but it is considered as a bisphenol-A free membrane (Matzke et al., 2011; Yamasaki et al., 2001).

PES has a higher atomic weight ratio of sulfone groups, which makes it more mechanical resistant, heat resistant and additional hydrophilic than that of PSf (Abe et al., 2011). PES is highly amorphous, transparent thermoplastic, comparatively less flammable, chemical resistant, relatively hydrophobic and has less water sorption (0.8% at 50% relative humidity) (Kesting, 1985). In biomedical fields PES membranes are broadly employed for artificial organs and medical devices used for purification of blood like hemofiltration, hemodiafiltration, hemodialysis, plasma collection and plasmapheresis (Samtleben et al., 2003; Tullis et al., 2002; Werner et al., 1996; Zhao et al., 2013).

In order to improve the performance of PES membrane in terms of biocompatibility and uremic solute removal, PVP was frequently used with PES and very little work was reported relating to PVP-K30. PVP is a highly hydrophilic and water-soluble polymer that prevents the protein adsorption and act as a pore forming agent on the membrane surface (Yang et al., 2008). The high affinity of PVP may cause it to swell in aqueous media and then it is eluded during phase inversion and blood dialysis. Thus, it may be possible that HD property of the membrane is altered during dialysis therapy (Sun et al., 2009). In order to maintain the composition of the membrane and retention of PVP as an additive in PES, PVP was used in the form of mono, di and tri-blocks such as poly(styrene-co-acrylic acid)-b-poly(vinyl pyrrolidone)-b-poly(styrene-co-acrylic acid) (Remes et al., 1992), poly(vinyl pyrrolidone)–b-poly(methyl methacrylate)–b-poly(vinyl pyrrolidone) (Ran et al., 2011), poly(vinyl pyrrolidone-acrylonitrile-vinyl pyrrolidone) (Yin et al., 2012),

poly(acrylonitrile-co-acrylic acid) (Fang et al., 2009), PVP nanoparticles (Weifeng-Zhao et al., 2011) and PVP-k90 alone (Barzin et al., 2004).

The use of nanoparticles and nanotubes has revealed outstanding potential in biological systems and membranes. In view of this MWCNTs are verified and reported as a revolutionary choice in biomedical field such as biomolecular transporters and recognition devices (Shi Kam et al., 2004; Wilson et al., 2009), biosensors (Krauss, 2009), cancer therapy and diagnoses (De La Zerda et al., 2008; Liu et al., 2009). It is believed that the high surface to volume ratio of MWCNT can improve the overall performance of many polymer-CNT composite membranes. Moreover, MWCNT can be easily functionalized with different functional groups and provide sites for attachment or bonding to other incoming molecules and open up new applications (Vivekchand et al., 2002). Both covalent and non-covalent alterations of the MWCNT surfaces have been employed to improve the solubility and wetting of MWCNT. The non-covalent approach includes surfactant modifiers (Kang et al., 2003), polymer absorption (Gómez et al., 2003), and polymer wrapping (Star et al., 2001). The benefit of noncovalent connection is that the perfect structure of the MWCNT is maintained and the wrapped or absorbed compound is not damaged, and thus most of the properties remained intact (Chen et al., 2005).

1.2 Problem Statement

Koopman et al., (2008) reported that current HD therapy failed to replace the complicated functions of the kidney in ESRD patients. Numerous research has been carried out in this field, however, patients suffer in chronic kidney disease were still suffering from low sickness situations, normal life quality and low death rate. The ESRD patients also suffered from chronic malnutrition, short life period and high cardiovascular risks (Koopman et al., 2008). Although, the HD technology had been used as a replacement therapy for renal failure for a few decades, it still has a number of unsolved issues.

Blood proteins have a propensity to rapidly adsorb onto the surface of the polymer, when PES-based hemodialysis membranes come into contact with blood (Klinkmann et al., 1987). Consequently the adsorbed protein layer demonstrated unwanted results such as higher platelet adhesion, fast blood coagulation and aggregation (Fang et al., 2009; et al., 2009; Zhu et al., 2007). The biocompatibility of the pristine PES was not satisfying (Klinkmann et al., 1987; Liu et al., 2009; Samtleben et al., 2003; Tullis et al., 2002; Zhao et al., 2013), thus the quest for simple additives and modification methods to improve such property continued and is a challenge for membranologists.

Most of the results obtained from di or tri block of PVP suggested that they can be used in hemodialysis, but their actual dialysis performance in terms of blood compatibility are not well reported in the literature. Moreover, the preparation of the covalently combined di or tri block of PVP require highly specific and controlled conditions. Among the various modification techniques, the blending of PVP, is the simplest method to improve the biocompatibility of PES membranes, but pure PVP cannot be used directly and need slight modification for its permanent residence on the HD membrane (Matsuda et al., 2008).

The MWCNTs are highly hydrophobic and non-polar material and its direct use without surface modification or surfactants may cause agglomeration and poor dispersion in membrane matrix, leading to defective product. The properties of MWCNT can be easily improved or upgraded by the attachment of different functional groups (Bahr et al., 2001; Georgakilas et al., 2002; Pekker et al., 2001; Valcárcel et al., 2008; Ying et al., 2003). Acid treatment is the simplest chemical method to create the polarity and attachment of the hydrophilic functional groups (carboxyl and hydroxyl) with MWCNT surface. This hydrophilic-hydrophobic nature of acid functionalized f-MWCNT provide a dual character, that can attract and bind both hydrophobic (like PES polymer) and hydrophilic types of chemicals (e.g. PVP type hydrophilic compounds) by π-π stacking and dipole-dipole interactions, respectively (Dyke et al., 2004a; Lu et al., 2011; Ma et al., 2010; Qian et al., 2000; Xie et al., 2005).

Most of the blood proteins are negatively charged and thus exhibit reduced adsorption behavior towards anionic character polymers. Sulfonation of PES is the bulk modification method which can increase the percentage of negative charge polarity due to the addition of sulfonated (-SO_3H) group. The presence of sulfonate functional groups in the polymer reduced the blood and membrane interaction via steric repulsion and showed good anticoagulant activity. Sulfonation is a chemical modification method of the PES that enhanced its biocompatibility and hydrophilicity. Many studies had focused on the sulfonation of polymers that increased the anionic character (Nie et al., 2014; Wang et al., 2009). Gertz et al., (2005) found that S-PES polymer reduced the contact activation of blood, whereas Wang et al., (2009) reported that blending of S-PES reduced the protein adsorption and elevated the blood coagulation time.

There are many dialysis membranes on the market, but the high death ratio of ESRD patients is suggesting that more research need to be done on this issue (Saran et al., 2015). Moreover, a few researchers reported the self-synthesized performance of HD membranes and most of the published work are focused on the performance investigation of commercial dialyzer membranes (Jalal Barzin et al., 2004; Leypoldt et al., 2006; Li et al., 2012; Su et al., 2008). In this research work, keeping in view the above discussion and literature review, an effort has been made to develop a highly biocompatible HD membrane composed of PES as a basic polymer with improved uremic solute permeability. Since the biocompatibility of PES HD membranes does not depend on a particular factor, hence different techniques are utilized to improve this property. Some of the researchers have focussed to chemical modification of PES polymer and others have used different additives (Ran et al., 2011; Wang and Yang, et al., 2009). The use of both methods, polymer modification and blending of additives together; have attracted much attention to improving the PES biocompatibility and uremic solute removal ability.

In this work, effort was made to improve the performance and biocompatibilty of PES membranes by the chemically modified sulfonated PES and blending of novel nanocomposites (NCs) additives. The acid functionalized MWCNT and lower and higher molecular weight of PVP grades (PVP-k30 and PVP-

k90) was used for NCs preparation and then they were incorporated into PES and S-PES/PES based membranes. The hydrophilic part of f-MWCNT contributed to the -COOH and –OH groups, whereas sulfonated polymer provided the $–SO_3H$ group in the membrane composition.The S-PES increased the anionic character and hydrophilicity of the HD membrane and favor the reduced protein adhesion. While in the NC's, the PVP tended to enhance the biocompatibility and hydrophilicity and f-MWCNT provided the mechanical strength, reduced the PVP leaching and act as a bridging material between PVP and PES. Moreover, the PVP was also reported as a surfactant for carbon nanotubes that improved its dispersibility and reduced the agglomeration in different solvents. Therefore, PVP in situ with acid treated MWCNT might also improve the dispersion properties of MWCNT in the formulated membranes (Vatanpour et al., 2011).

Thus, the purpose of this research work is to contribute in the development of a new HD membrane with a combination of additives, which might be able to reduce biocompatible issues and exhibits higher rates of uremic solute clearances

1.3 Objective of the Study

The main objective of the study is to develop a high performance, biocompatible dialysis membrane with notable uremic solute sieving properties by blending f-MWCNT into the polymer matrix. In order to achieve this the following objectives, need to be addressed;

1. To formulate and synthesize various flat sheet HD membranes consisting of i) PES as base polymer and NC's which consists of f-MWCNT/PVP-k90 and f-MWCNT/PVP-k30. ii) Various ratios of PES and S-PES as the base polymer and the NCs which comprise of f-MWCNT/PVP-k90.

2. To characterize the synthesized membranes in terms of chemical composition, biocompatibility and to evaluate their dialysis performance using uremic solute model solution.

3. To fabricate HD hollow fiber membrane using the best formulation and analyse its dialysis performance.

1.4 Scope of the Study

The scope of research is as follows

i. Acid functionalization of MWCNT and NC's synthesis by blending technique and covalent attachment of sulfonated group to the PES to develop S-PES polymer. The FTIR and XRD were used to characterize the MWCNT, f-MWCNT, NC's and S-PES polymer.

ii. Preparation of i) various concentrations of NC's and PES alone, ii) various ratios of S-PES and PES polymer with NC's via microwave technique.

iii. Flat sheet membranes were casted and their chemical properties were analysed by FTIR, whereas FESEM was used for morphological studies.

iv. The performance of HD membranes was initially evaluated in terms of pure water permeation, the molecular weight cutoff (MWCO) (should be <60kDa), mean pore size, porosity and flux recovery ratio.

v. The leaching tests were performed to observe the stability of the NC's in the fabricated membranes

vi. The membrane hydrophilicity were determined by contact angle and water uptake measurements. In addition, AFM technology was utilized for the surface roughness study and 3D micrographs.

vii. In order to evaluate the biocompatibility of HD membranes, various experiments are performed; protein resistance ability of FSMs, hemolysis (destruction of red blood cells), blood coagulation factors, including Thrombin time (TT), Prothrombin time (PT), Activated partial thrombin time (APTT), fibrin formation, and the plasma re-calcification time.

viii. The single layer dialysis cell is used to estimate the dialysis properties formulated membranes using a model solution of uremic solutes consisting of urea, creatinine and lysozyme against distilled water as dialysate.

ix. Finally, hollow fiber membranes were fabricated using the best FSM formulation. Their performances were evaluated in terms of molecular weight cutoff, pure water permeation, pore size, antifouling properties and dialysis ability against uremic solutes.

1.5 Significance of the Study

In this research a novel HD membrane with excellent biocompatibility, good flux rate, better antifouling properties and excellent dialysis performance was synthesized and produced. PVP and the f-MWCNT based di-block nonocomposites were developed by simple blending method and its influence on PES and combine PES/S-PES polymeric membranes were thoroughly investigated with special emphasis on membrane biocompatibility, consistency of chemical composition, dialysis and typical HD performances. The biocompatibility tests include the study of protein adhesion, red blood cell behavior and fibrin formation towards the formulated membranes. Although previous researchers have used different modification techniques to enhance the performance of PES, but none of them has reported a comprehensive study in terms of typical membrane performance, biocompatible studies and dialysis of uremic solutions. Finally, the performance data

produced were then compared to the other commercial hemodialysis membranes reported in literature.

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