Electronic Supplementary Material (ESI) for RSC Advances

Supporting information

Self-assembled silica nanocrystal based anti-biofouling nanofilter membranes

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S1. Synthesis of 3-(2-chloroethylthio)-5-(4-chlorophenyl)-4H-1,2,4-triazole-4-amine (TC)

Synthesis of 3-(2-chloroethylthio)-5-(4-chlorophenyl)-4H-1,2,4-triazole-4-amine was involved following steps:

(a) Preparation of methyl 4-chlorobenzoate

A mixture of *p*-chlorobenzoic acid (0.3 mol) in absolute methanol (105 cm^3) and concentrated sulphuric acid (50 cm^3) was refluxed in a flask for about 4 h on a steam bath. The solution was cooled and slowly poured with stirring in crushed ice. Ammonia solution was added to render the resulting solution strongly alkaline. The ester, so formed was extracted with ether and dried over anhydrous magnesium sulphate. The ether was removed and distilled under reduced pressure to give ester. Yield: 70%

(b) Preparation of 4-chlorobenzohydrazide

Hydrazine hydrate (1 mol) and ester (1 mol) were refluxed for about 4 h and then cooled. The crystals were separated out which were filtered off, washed with aqueous ethanol and then recrystallized from hot water. Yield: 68%

(c) Preparation of 4-amino-5-(4-chlorophenyl)-3-thia-1, 2, 4-triazoles

Methanolic solution of substituted acid hydrazide (0.01 mol), potassium hydroxide (0.01 mol) and carbon disulfide (0.01 mol) were stirred for 2 h. A solid mass was obtained which was refluxed with excess of hydrazine hydrate for *ca.* 4 h. The reaction mixture was cooled,

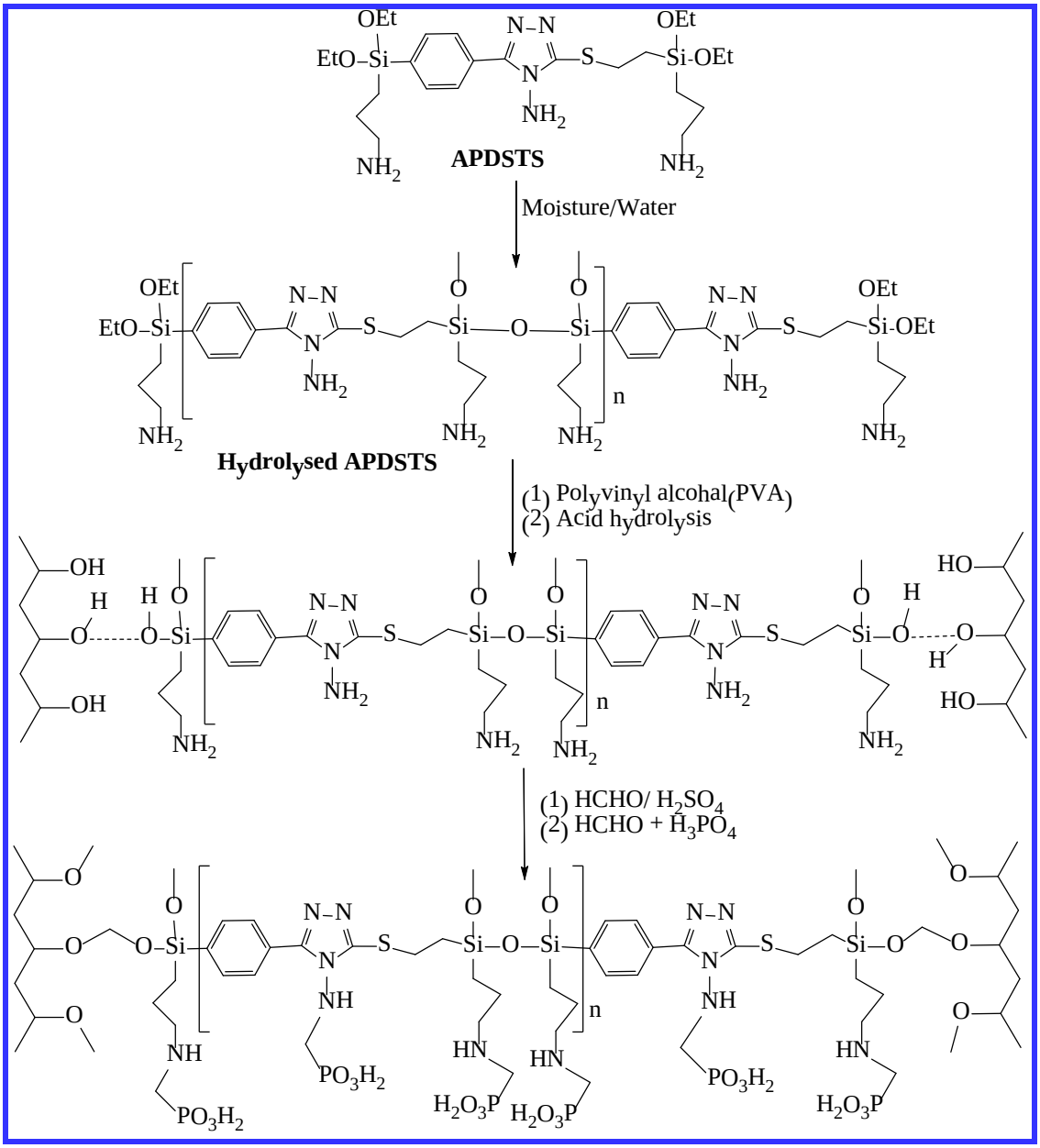
poured in cold water and neutralized with diluted hydrochloric acid. The product, thus obtained, was filtered off, washed with ether and crystallized from methanol. Yield: 60-70%

(d) Synthesis of 5-(2-chloroethylthio)-3-(4-chlorophenyl)-4H-1,2,4-triazole

A solution of appropriate 4-amino-5-(4-chlorophenyl)-3-thia-1, 2, 4-triazoles (0.1 mol), Potassium carbonate (0.1 mol) and 1,2-dichloroethane (0.1 mol) in methanol (50 cm^3) was refluxed for 6-8 h. The mixture was poured in water and made it slightly acidic with dilute hydrochloric acid. The crystals were separated out which were filtered off, washed with hot water and then recrystallized from ethanol. Yield: 70%

Table S1
WXRD data for different nanocomposite membranes (TS-x).

Membrane	2(θ) degree	Inter planer distance (d) nm
TS-30	18.81	0.2388
TS-50	19.23	0.2338
TS-60	19.37	0.2322



Scheme S1 Schematic structural diagram of synthesized NF membranes.

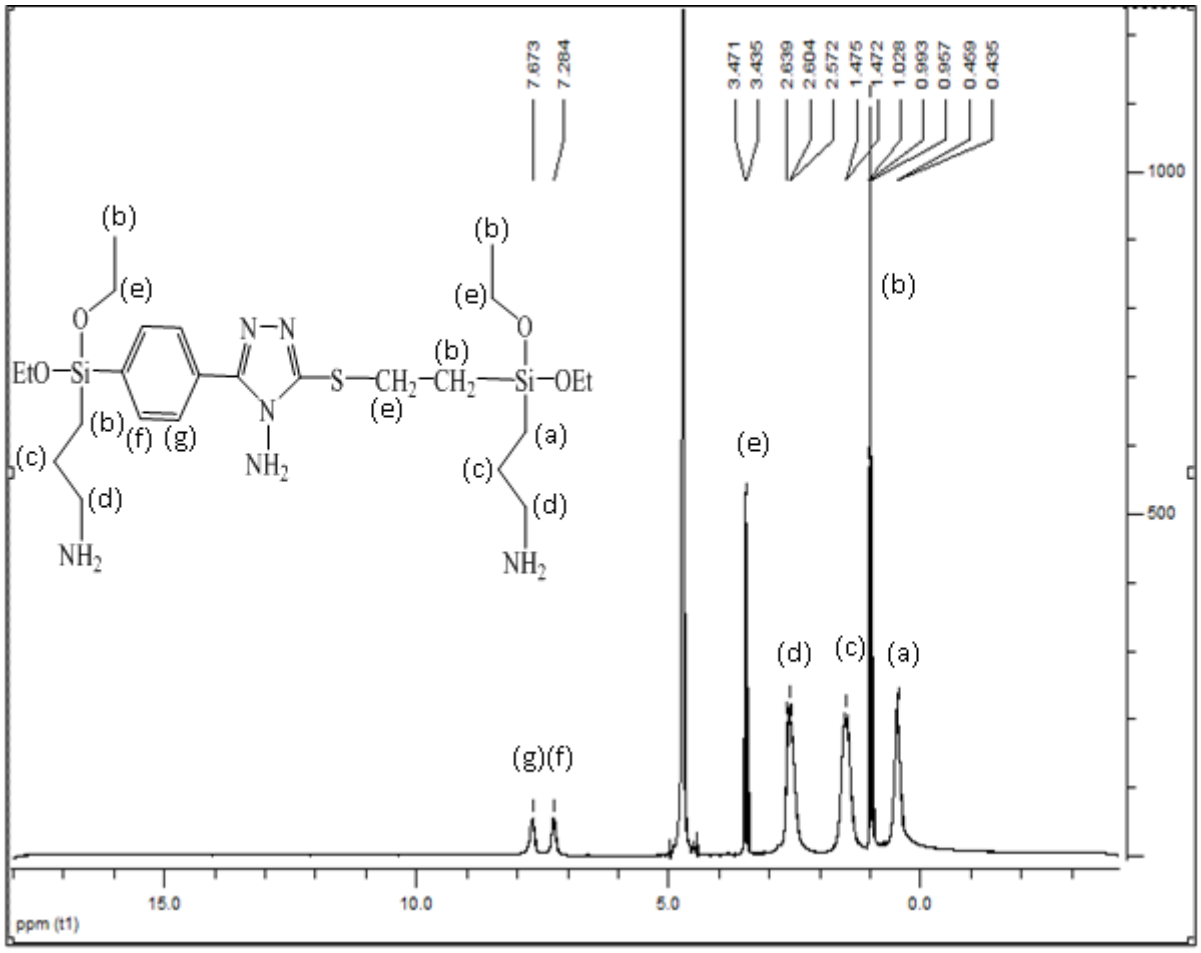


Fig. S1 ^{1}H NMR spectra organosiloxane (TS) in D_2O.

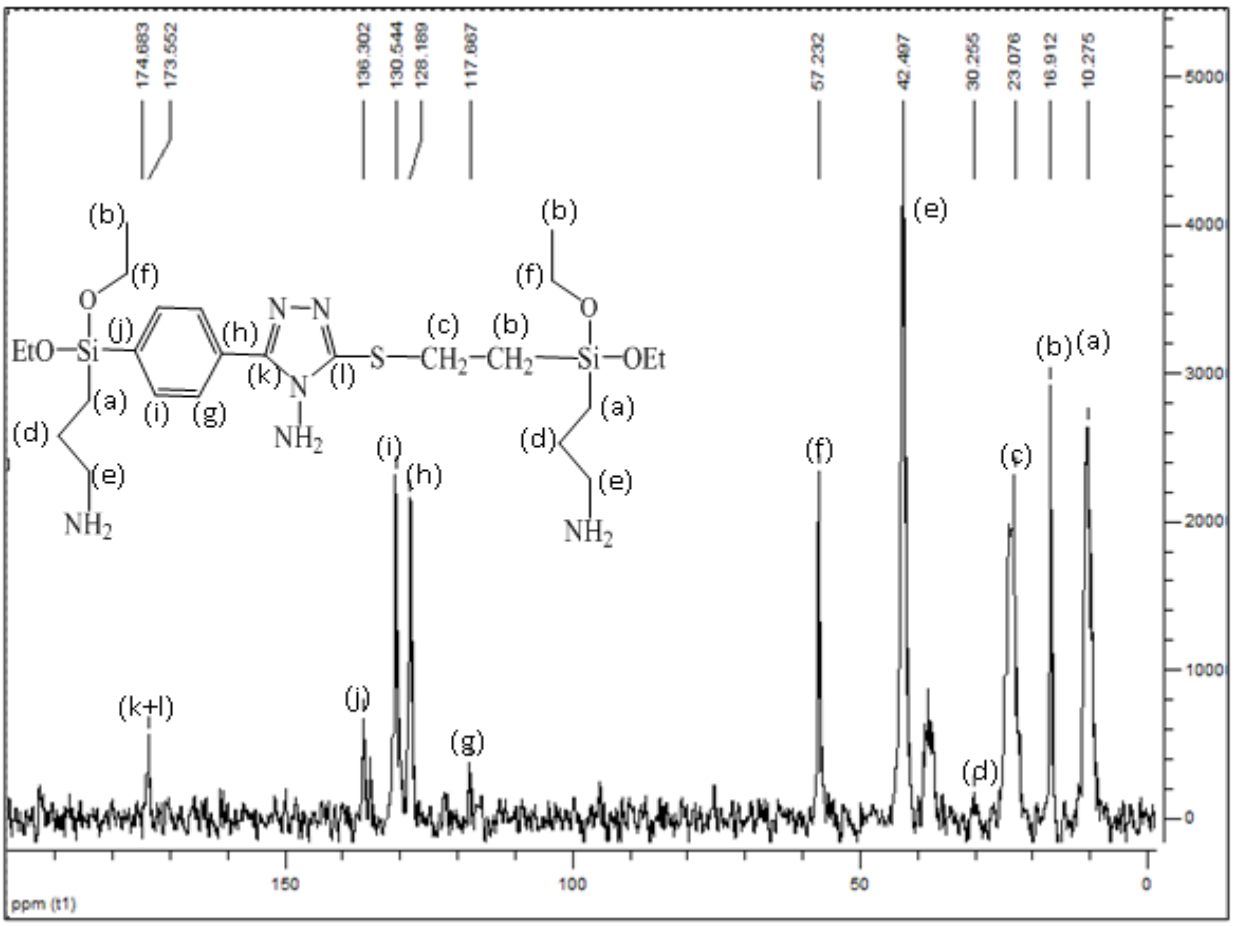


Fig. S2 ^{13}C NMR spectra organosiloxane (TS) in D_2O.

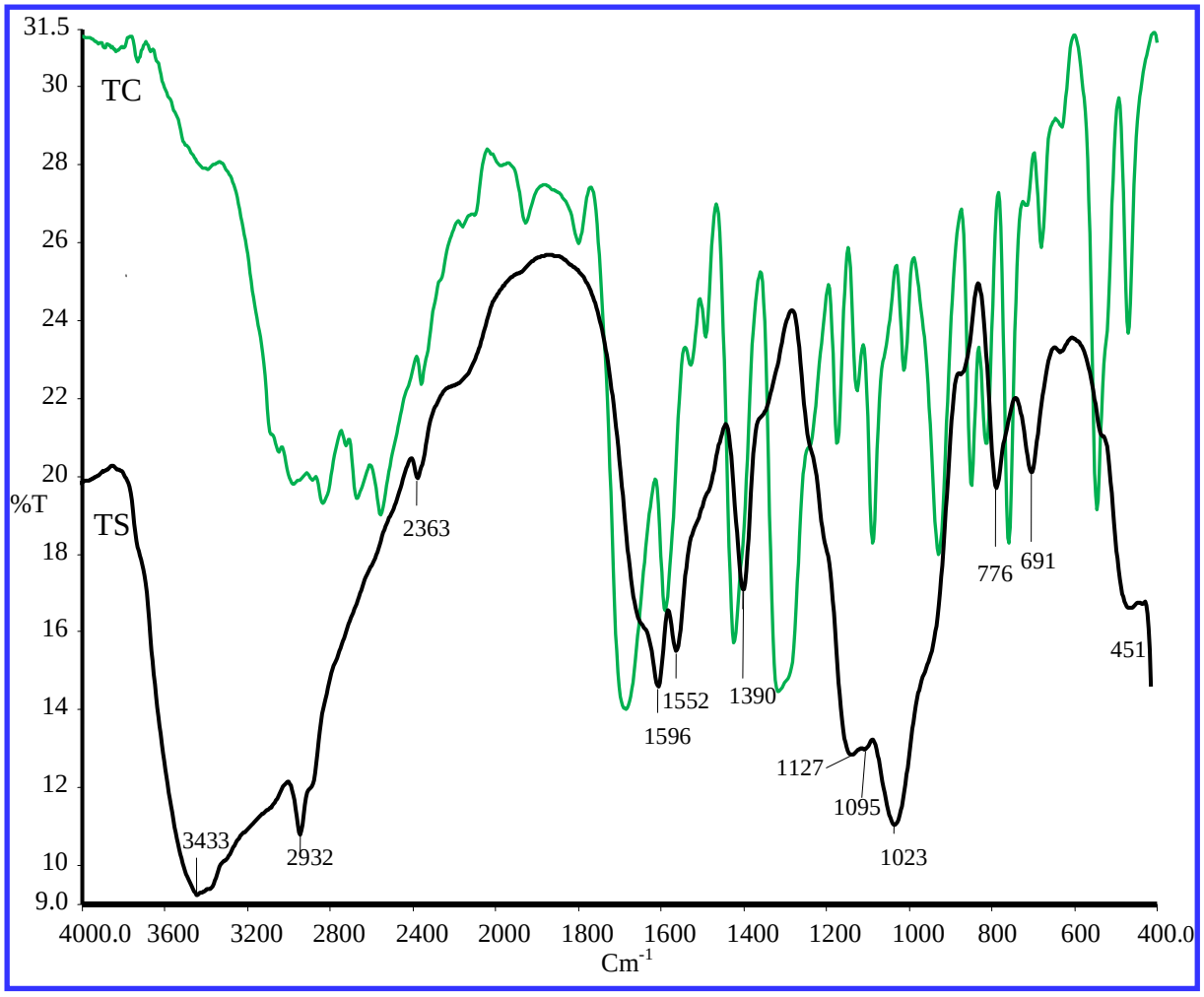


Fig. S3 FT-IR absorption spectra of organosiloxane (TS) and intermediate product 3-(2-chloroethylthio)-5-(4-chlorophenyl)-4H-1,2,4-triazole-4-amine (TC).

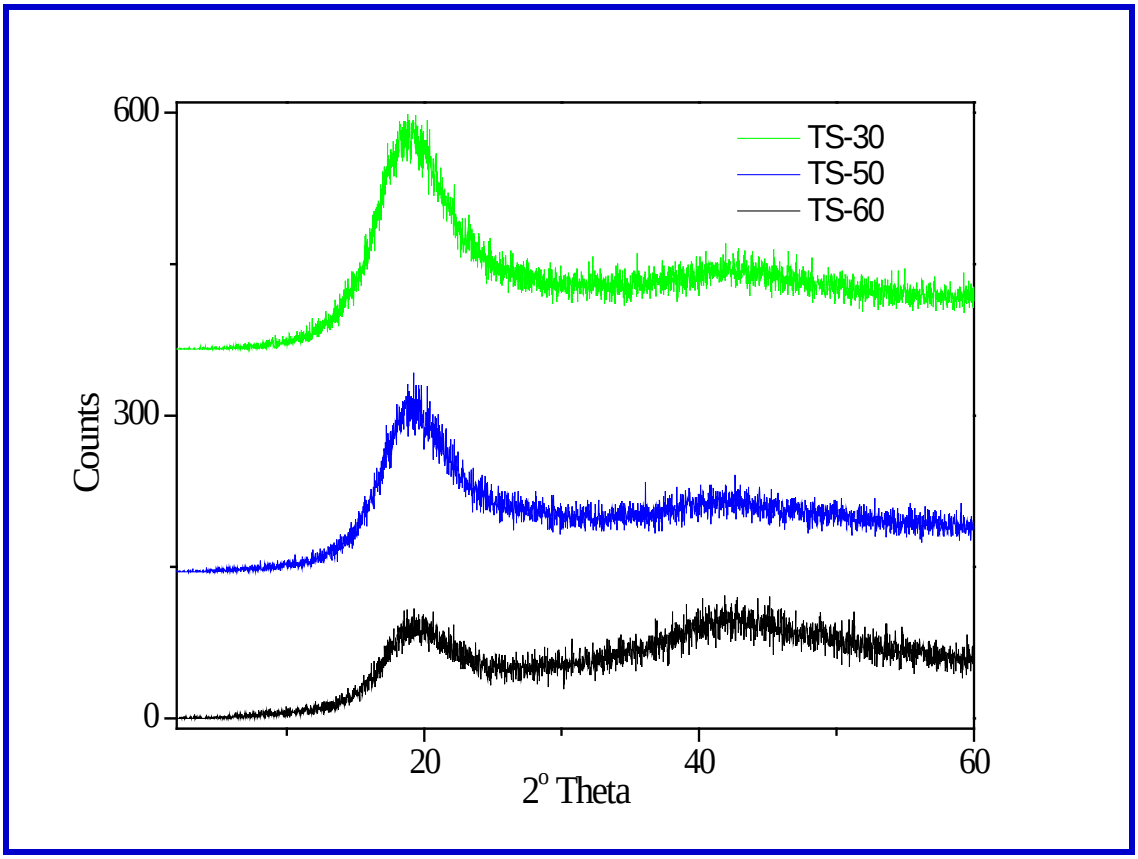


Fig. S4 XRD patterns of the nanocomposite membrane.

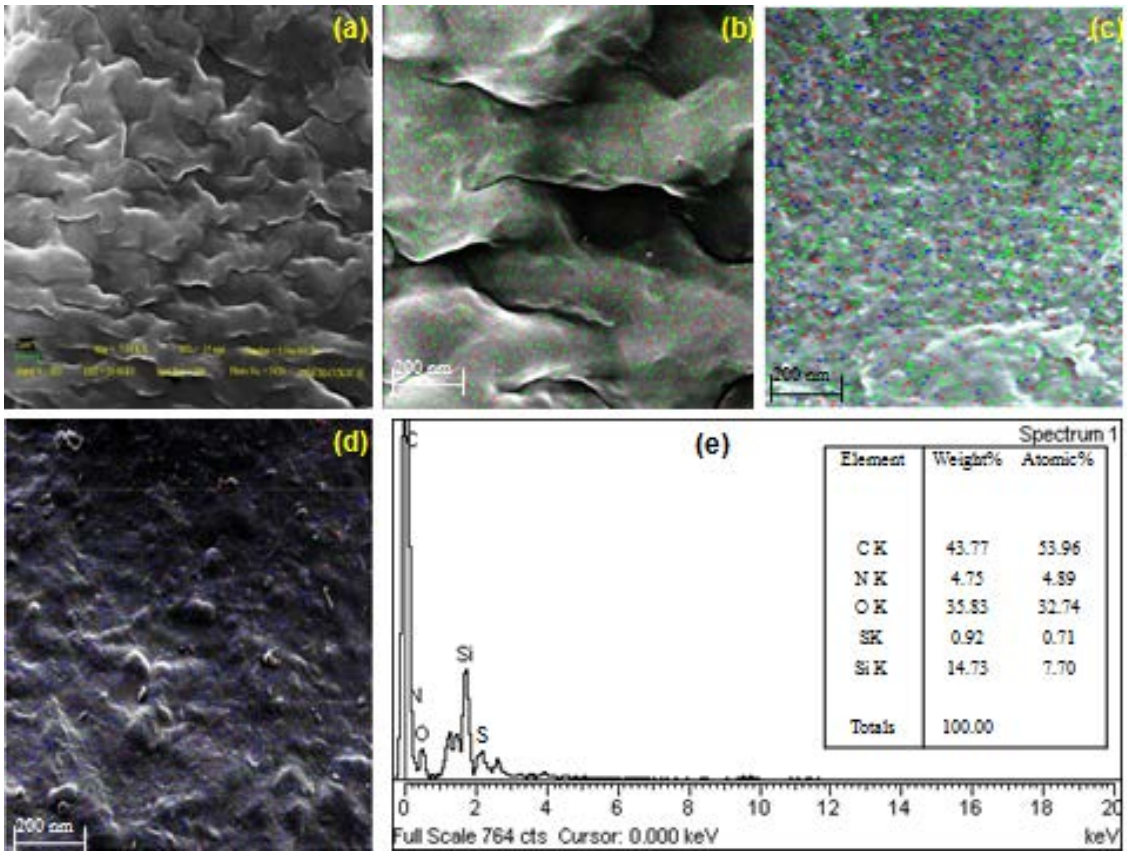


Fig. S5 SEM and EDX performance results of (a) morphology of TS; (b) elemental mapping of TS carbon present red dot and Si present by green dot; (c&d) elemental mapping of TS-60a membrane and TS-30 (green, red, and blue colour dots represents P, Si, and C); (e) EDX performed result of organosiloxane (TS).

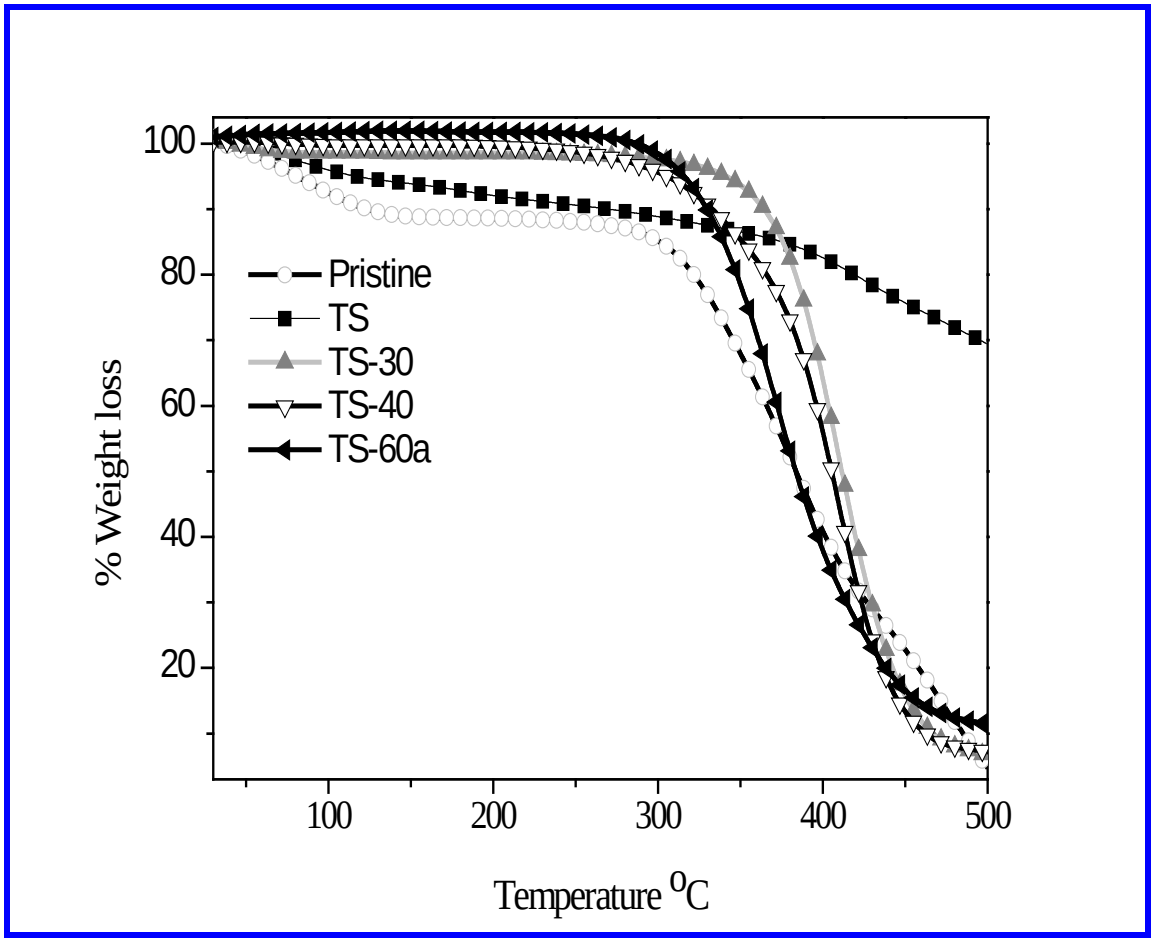


Fig. S6 Thermal gravimetrical analysis (TGA) profiles of the thermolysis of organosiloxane (TS) and their polymeric membranes.

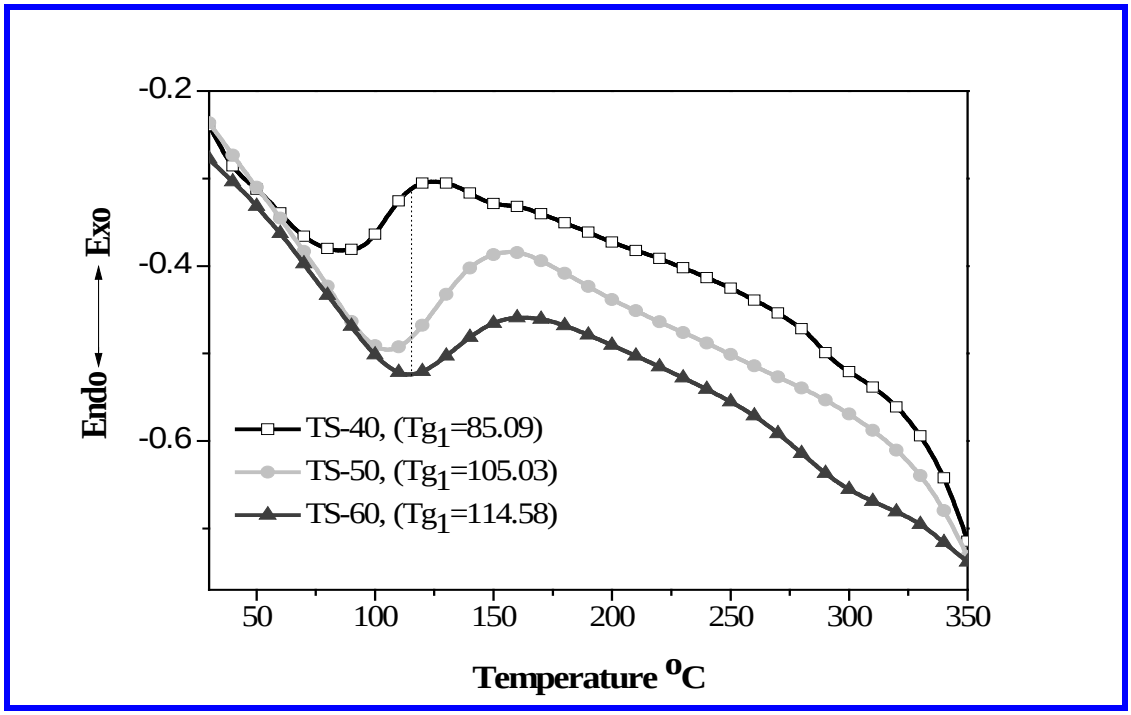


Fig. S7 DSC profiles of the polymeric membranes with change of temperature.

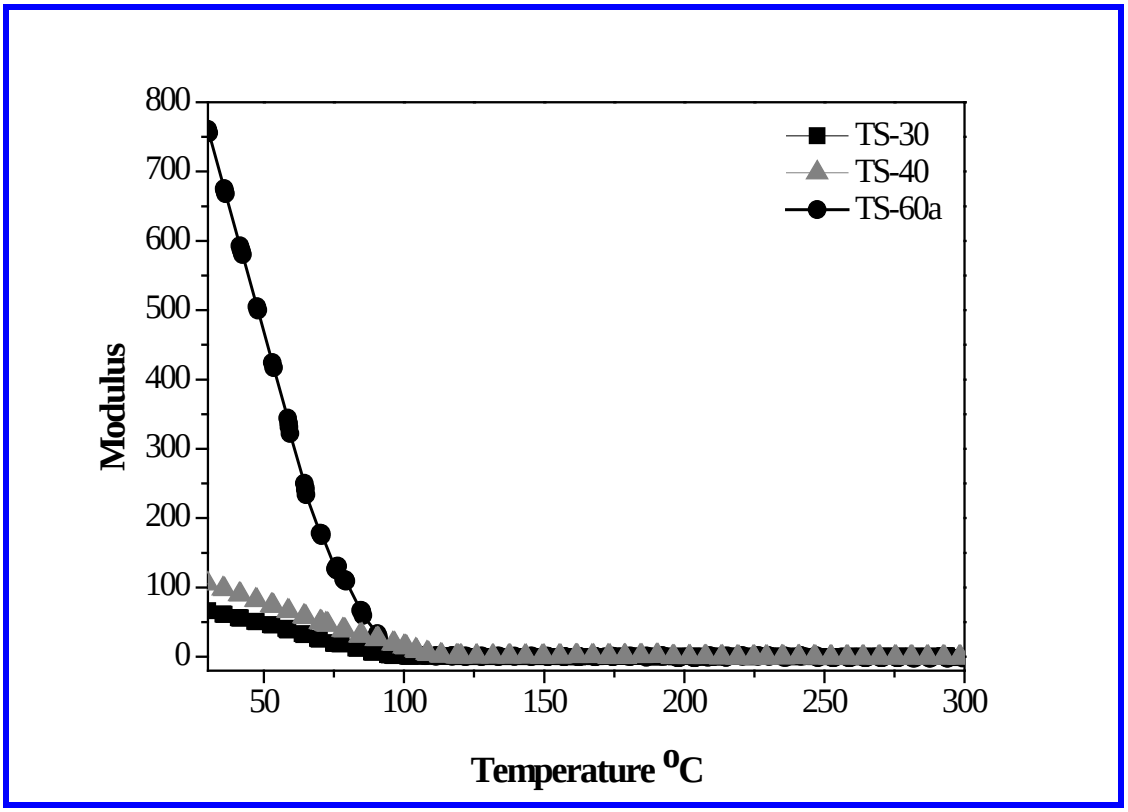


Fig. S8 Dimensional mechanical analysis (DMA) profiles of the polymeric membranes.

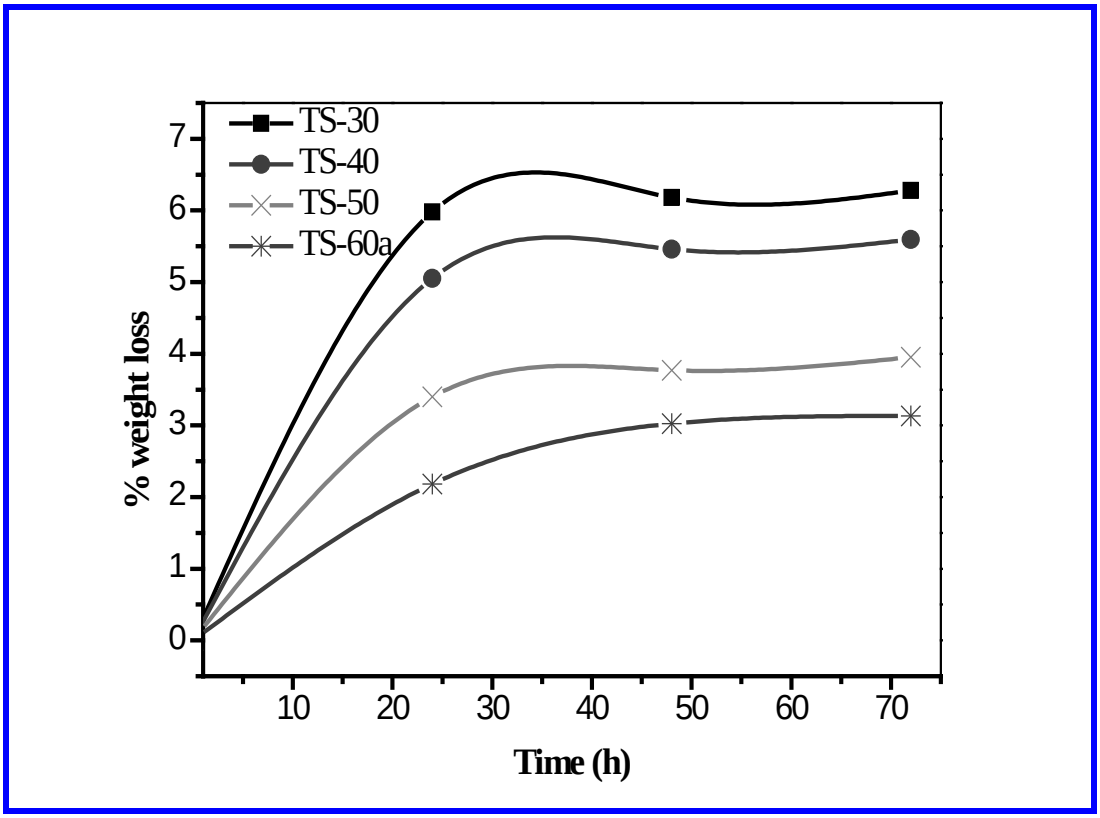


Fig. S9 Chlorine stability of different membrane in term of percentage weight loss vs varied time.

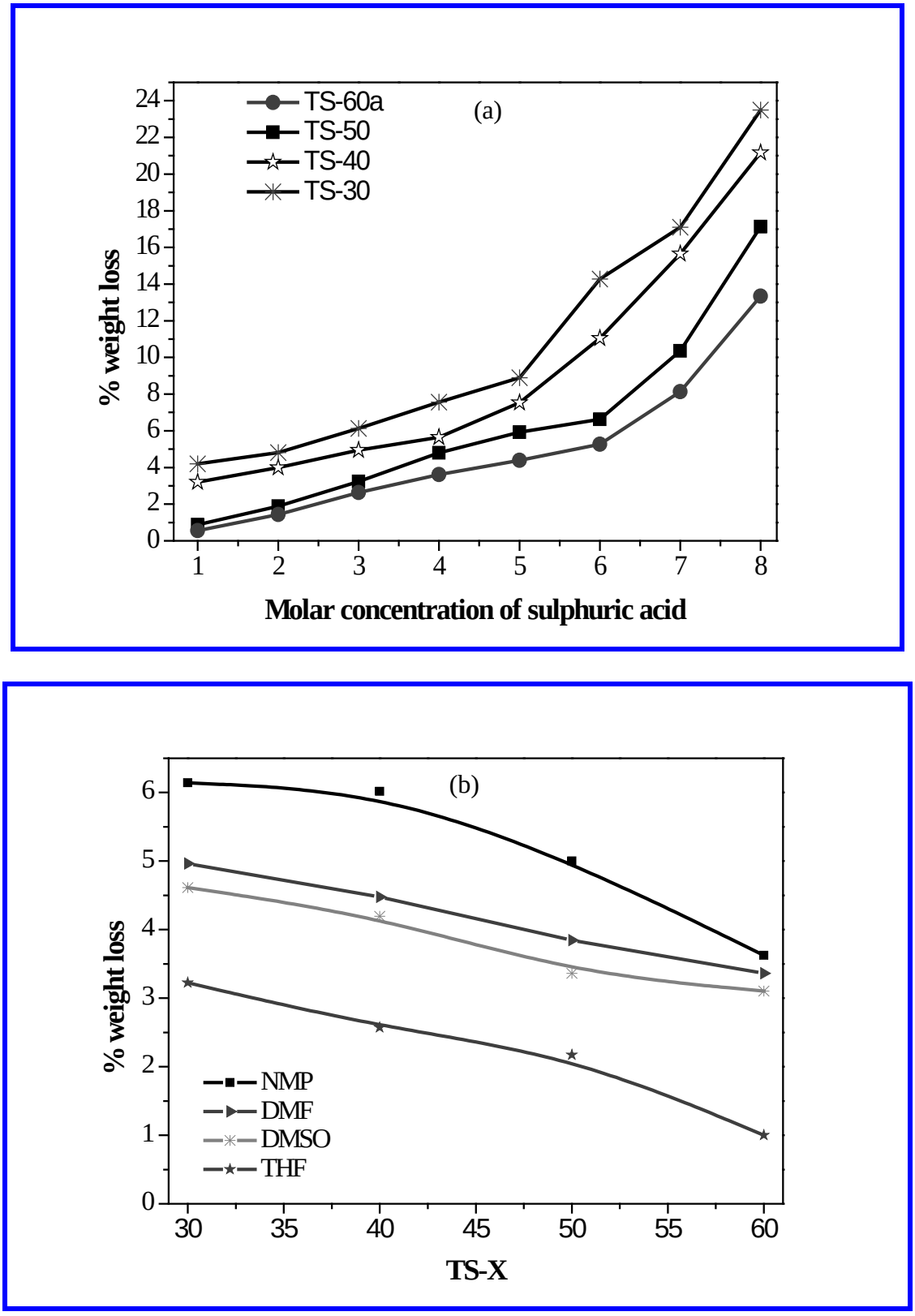


Fig. S10 Chemical stability of membranes; (a) stability in sulphuric acid; and (b) stability in different solvents in term of percentage weight loss.

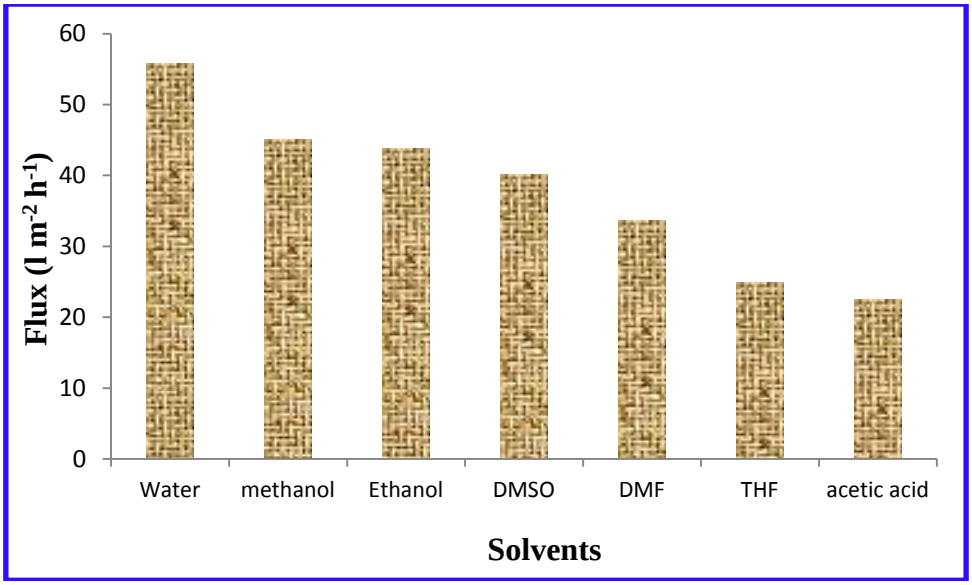


Fig. S11 Flux performance of TS-60a membrane in different solvent at 1.2 MPa and 25 ^{o}C.

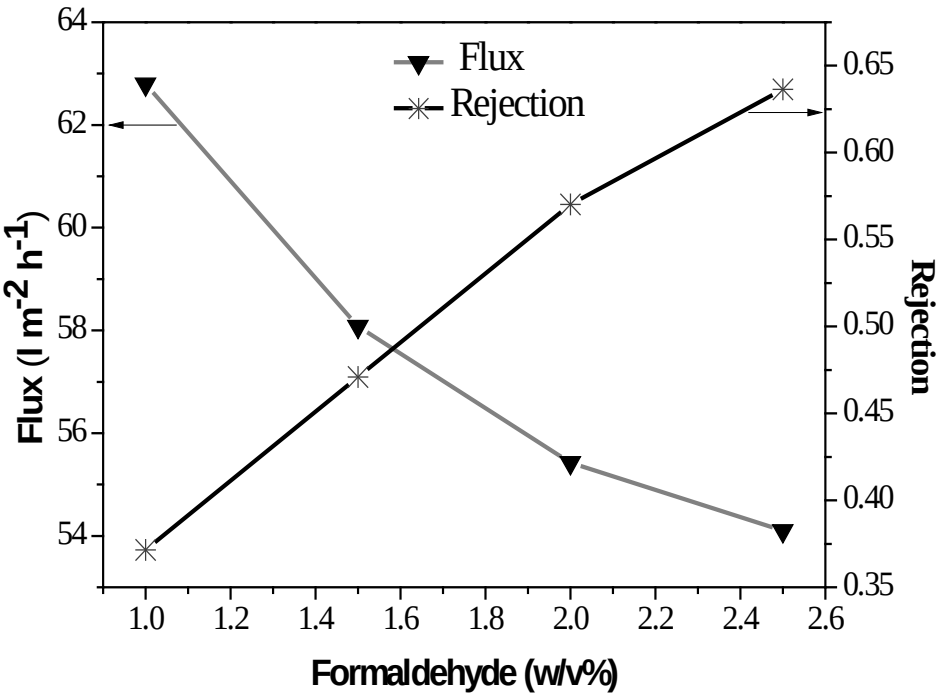


Fig. S12 Effect of formaldehyde concentration on the NaCl rejection and water flux of TC-60 (a-d) membranes testing with 1 gl^{-1} NaCl aqueous solution at 1.2 MPa and 25 ^{o}C.

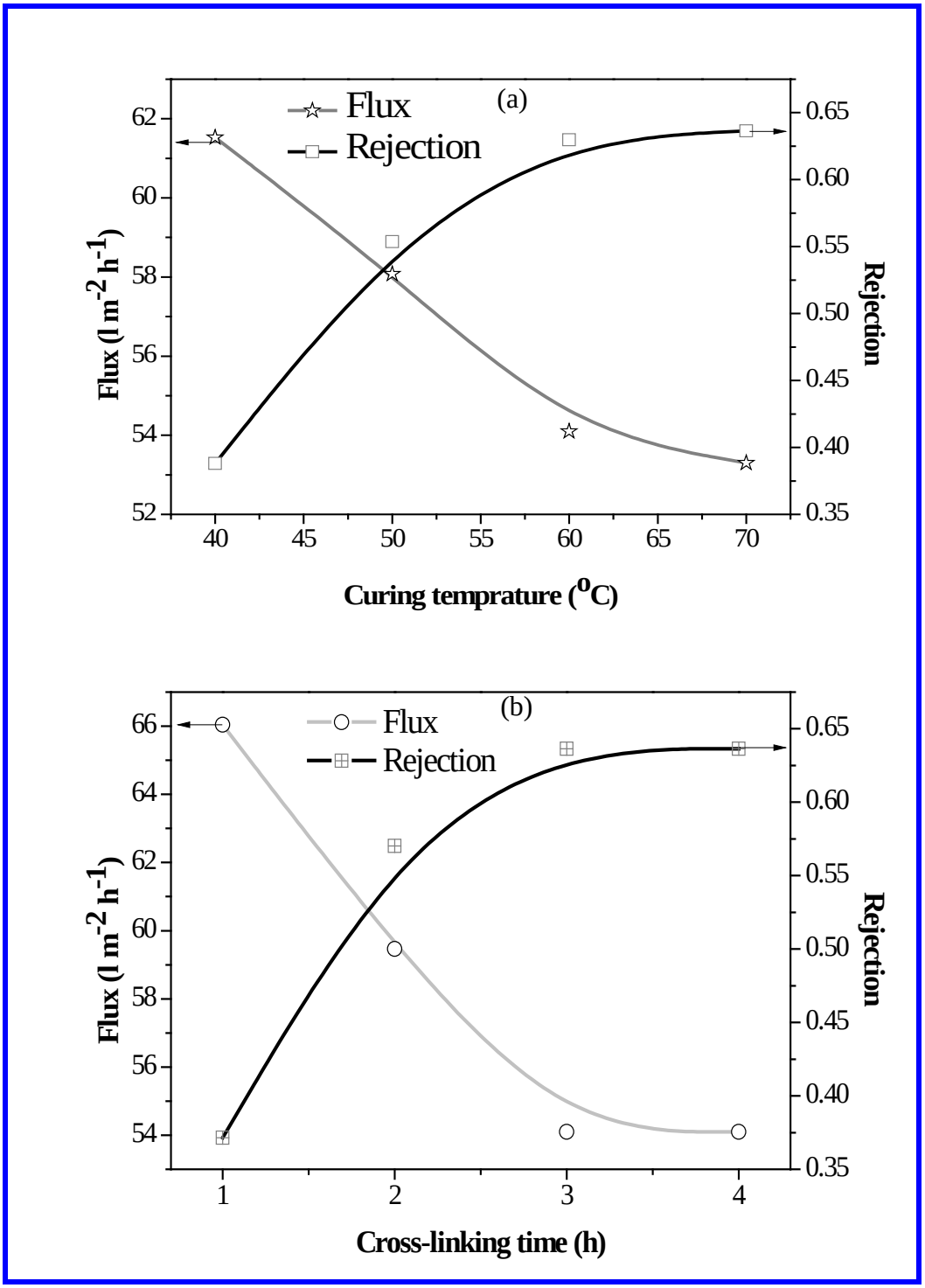


Fig. S13 Effect of (a) curing temperature, (b) cross-linking time on the NaCl rejection, water flux of TC-60 (a, e-g) and TS-60 (a, l-n) testing with 1 g L^{-1} NaCl aqueous solution at 1.2 MPa and 25 ^{o}C.

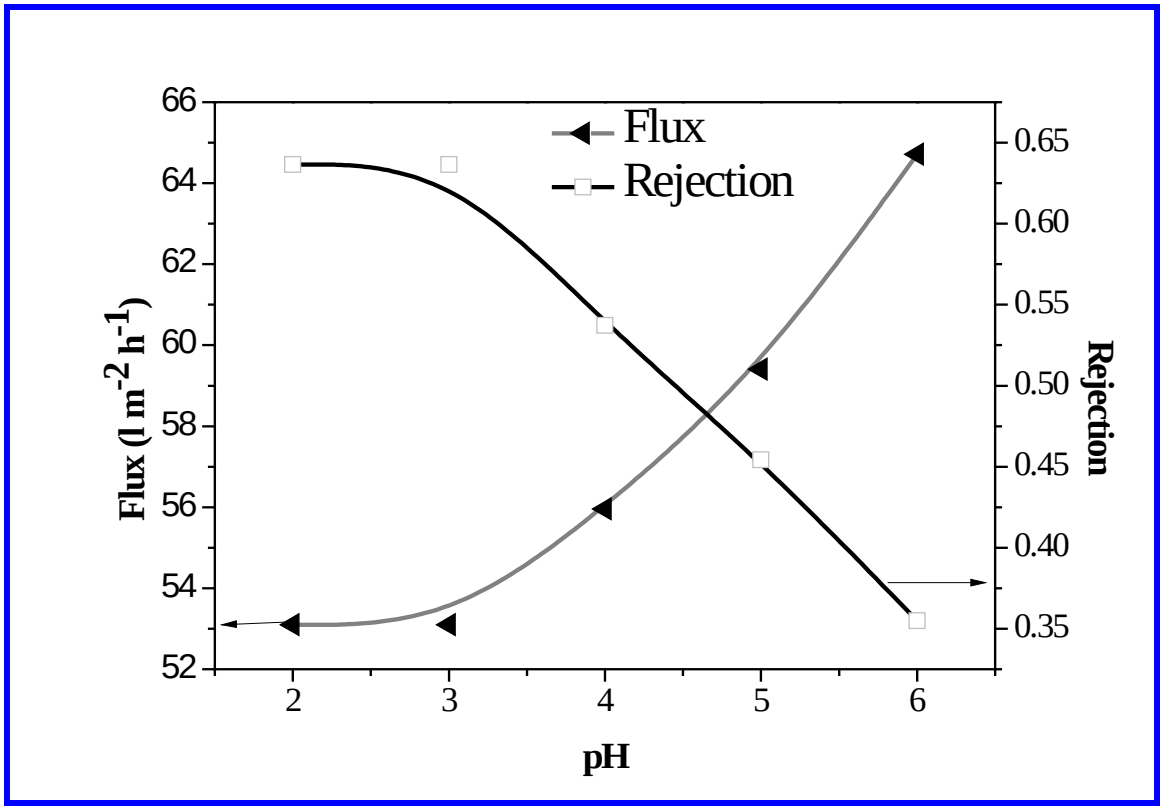


Fig. S14 Effect of casting solution pH value on the NaCl rejection and water flux of TC-60(a, h-j) testing with 1 gl^{-1} NaCl aqueous solution at 1.2 MPa and 25 ^{o}C temperature.

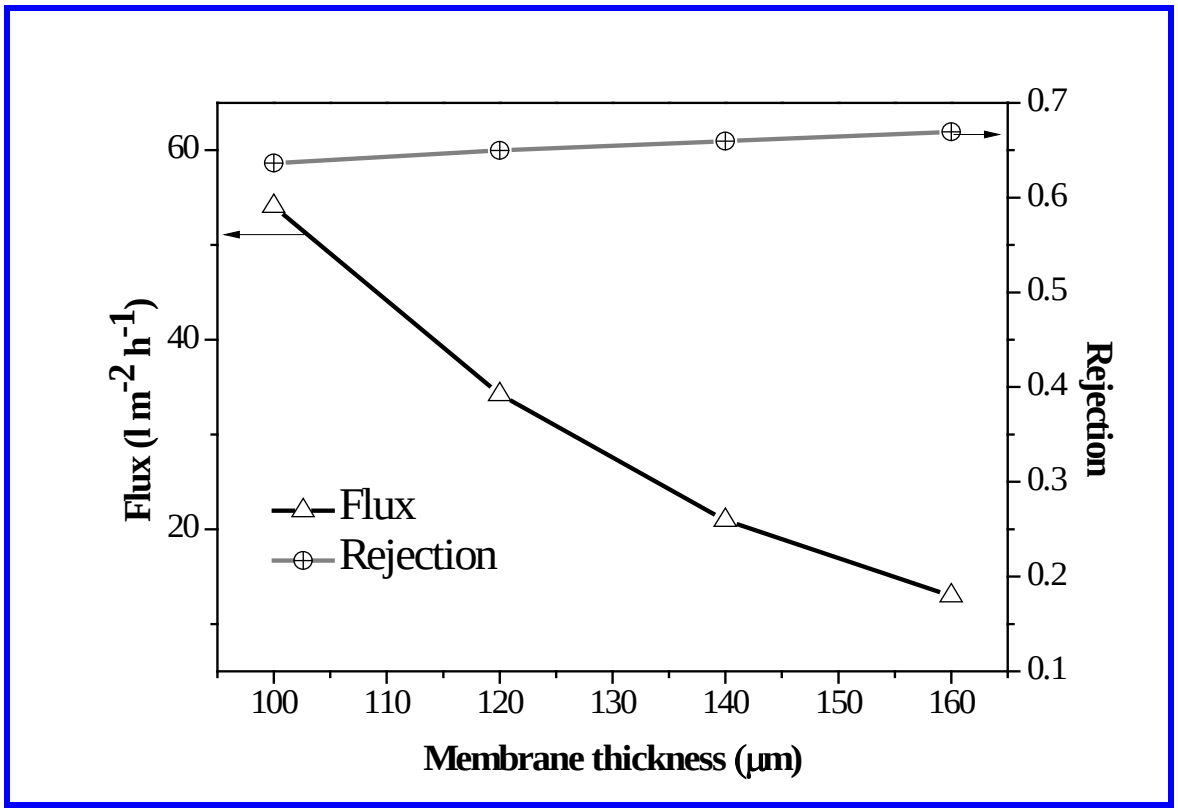


Fig. 15 Effect of membrane thickness on the NaCl rejection and water flux of TC-60a testing with 1 gL^{-1} NaCl aqueous solution at 1.2 MPa and 25 ^{o}C.

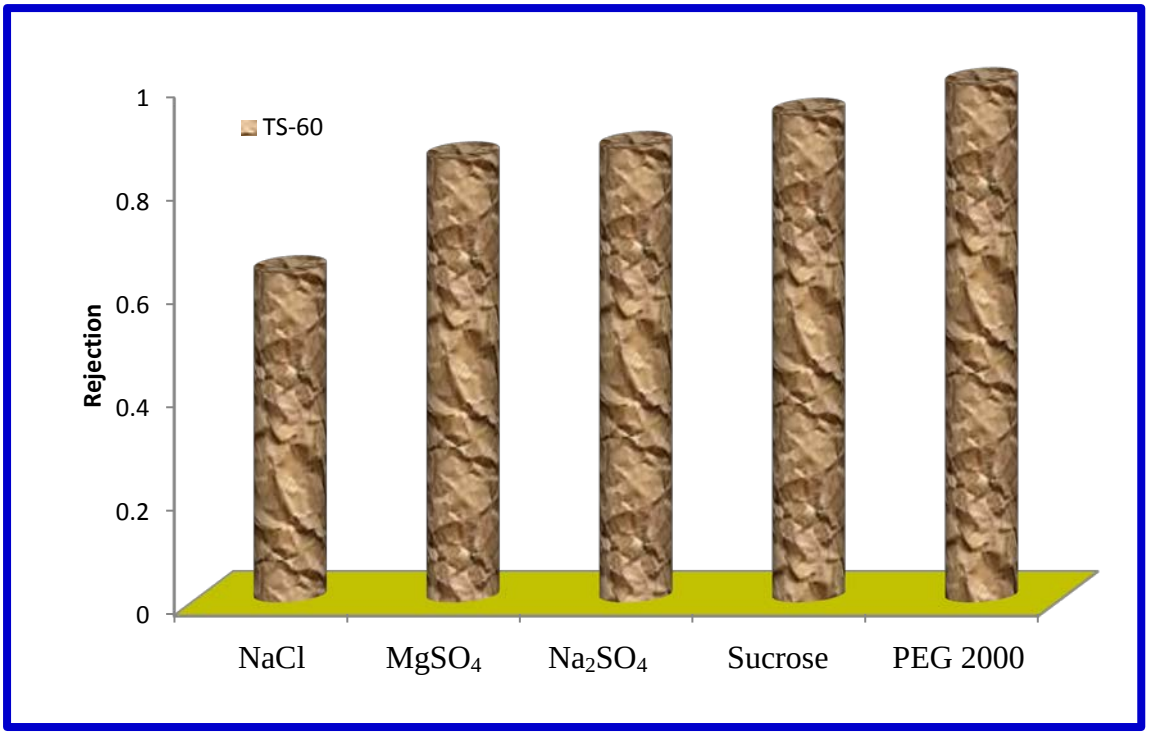


Fig. S16 Rejection of TC-60a to different solute testing with 1 gL^{-1} inorganic salt and organic neutral probe molecule aqueous solution at 1.2 MPa and 25 ^{o}C.