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Synthesis and characterization of polymer (sulfonated poly-ether-ether-ketone) based nanocomposite (h-boron nitride) membrane for hydrogen storage

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ABSTRACT

The development of light weight and compact hydrogen storage materials is still prerequisite to fuel-cell technology to be fully competitive. The present experimental study reports the hydrogen storage capability of sulfonated poly-ether-ether-ketone (SPEEK)-hexagonal boron nitride (h-BN) (SPEEK-h-BN) nanocomposite membranes. The nanocomposite membranes are prepared by considering various amount of h-BN (0, 1, 3 and 5 wt. %) by phase inversion technique. The degree of sulfonation of the PEEK (SPEEK) is found to be 65% by Proton Nuclear Magnetic Resonance (¹H NMR) spectroscopy. Hydrogen adsorption studies have been carried out using a Seiverts-like hydrogenation setup. The membranes are characterized by X-ray Diffractometer (XRD), Micro-Raman spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy, Scanning Electron Microscopy (SEM), Atomic Force Microscopy (AFM), CHN-elemental analysis and Thermo Gravimetric Analysis (TGA). It is observed that the SPEEK-5% h-BN membrane performs better than pure SPEEK membrane, has maximum storage capacity of 2.98 wt. % at 150 °C and the adsorbed hydrogen has an average binding energy of 0.38 eV. The TGA study shows the dehydrogenation behavior of hydrogenated SPEEK-h-BN nanocomposite membrane and it is found to be in the temperature range of 214–218 °C for SPEEK-5% h-BN membrane.

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Introduction

The major challenges in the recent years are the reduction of fossil fuels and global warming [1]. Necessity is the driving force for finding innovative technologies. Alternative energy

sources like wind, solar, super capacitor and fuel cells have been developed recently and are used effectively. Nowadays hydrogen is considered as the most promising and potential alternative energy carrier that can facilitate the transition from fossil fuels to sources of clean energy [2,3]. The development of fuel cell power systems has enabled hydrogen as an

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alternative to conventional systems [4]. Although hydrogen is often presented as the fuel of the future, the development of a hydrogen-based fuel is rather difficult. Hydrogen has to be produced, stored, distributed and used, but each of these steps is hindered due to several factors [5]. Multiple challenges must be overcome to achieve the vision of secure, abundant, inexpensive, and clean hydrogen production with low carbon emissions [4,6,7].

In the prospect of a hydrogen-based economy, the major issue is to address how to reversibly store hydrogen while minimizing energy consumption. Three major approaches [8,9], compression for storage in tank, storage by purely physical absorption and storage via chemical reaction or chemical storage are in the forefront of current research efforts. Direct use of compressed hydrogen as a fuel encounters major constraints due to large tank volumes, short lifetimes of the storage system and stringent safety measures, in addition to high investment costs and portability.

A lot of research works have been carried out on hydrogen rich materials like chemical hydrides [10], complex hydrides [11] and metal hydrides [12] that store hydrogen by chemisorption. In the last two decades, over 350 distinct materials have been investigated experimentally [13] and more than 70% have been discontinued as potential candidates for hydrogen-fuel cells. Although some complex metal hydrides (such as, MgH_2 , $NaBH_4$, $Ca(BH_3)_2$, AlH_3 , K_2LiAlH_6 , etc.) have advantages of high gravimetric density (~15–20 wt. % of H_2) and high volumetric density (about 150 g H_2/L), major disadvantages were found to be high desorption energy, slow kinetics and toxic byproducts, besides cyclability as a major issue.

Large specific surface area materials such as carbon nanotubes [14], metalorganic frameworks [15,16] and zeolites [15,17] store hydrogen by physisorption and in most of these cases hydrogen uptake are not so high. Recent reports have demonstrated that boron nitride (BN) nanomaterial can be used as a potential hydrogen storage medium [18–22]. Hexagonal boron nitride (h-BN) is an analog of graphite, has unique physical and chemical properties, including high specific surface area, low density, high mechanical strength and oxidation resistance [22–24]. Weng et al. [18] reported the ability of BN porous microbelts to exhibit high and reversible hydrogen uptake of 2.3 wt. % at 77 K and 1 MPa. Wang et al. [19] reported a hydrogen storage capacity of 2.6 wt. % in h-BN powders under the hydrogen pressure of 1.0 MPa. The conventional BN nanopowder, multiwalled BNNT and bamboo-like BNNTs can absorb hydrogen up to 0.2, 1.8 and 2.6 wt. %, respectively at room temperature and at 10 MPa [20]. The hydrogen storage capacity of 4.2 wt. % at 10 MPa in BNNTs collapsed walls [21] and 5.6 wt. % at 3 MPa at room temperature in BN porous whiskers were noticed [22].

The other promising route is the use of polymers/polymer nanocomposites that have also been considered for hydrogen storage [4,6,25–35]. Keown et al. [25] found that polymer of intrinsic microporosity (PIMs) stores 1.7 wt. % of hydrogen at 77 K and 10 bar. The hypercrosslinked polystyrene could store 2.75 wt. % of hydrogen at 77 K and 10 bar [26]. The hydrogen storage capacity of HCl treated conducting polymers like polyaniline (PANI) and polypyrrole (Ppy) were found to be 6 and 8 wt. %, respectively at room temperature and at 9.3 MPa [27]. The hydrogen storage ability of the hypercrosslinked

polymers with platinum (Pt) nanoparticles was analyzed and a maximum storage capacity of 0.21 wt. % at 298.15 K and 19 bar was noticed [28]. Hypercrosslinked polystyrene – Pt nanoparticles display a hydrogen uptake of 0.36 wt. % at 294 K and 100 atm [29]. The PANI impregnated with tin oxide, multiwall carbon nanotubes (MWCNTs) and aluminum powder (Al) exhibit 0.31, 0.38, 0.5 wt. % of hydrogen adsorption at 115 °C and 70 bar [30]. Hydrogen storage capacity of halloysite nanotubes (HNT) – PANI nanocomposite was examined by Attia et al. [31] and noticed 0.78 wt. % of hydrogen was stored at room temperature and a pressure of 0.5 MPa. The hydrogen storage capacity of poly(ether-ether-ketone) functionalized in situ by manganese oxide formation, showed a hydrogen storage capacity of 1.2 wt. % at 77 K [6]. The hydrogen storage capacity of acid treated water-soluble polymers such as polyvinyl alcohol (PVA) and polyvinyl pyrrolidone (PVP) doped single-walled carbon nanotubes (SWCNTs) has shown to be 1.2 and 1.5 wt. %, respectively at 50 °C [32]. Kim et al. [33] reported that the PANI – vanadium oxide nanocomposites (VONC) showed storage capacity of 1.8 wt. % at 77 K and 70 bar. The activated rectangular PANI-based carbon tubes (ARP-CTs) exhibited the hydrogen adsorption of 5.2 wt. % at 77 K and 5 MPa and 0.62 wt. % at 293 K and 7.5 MPa respectively [34]. Makridis et al. [4] studied Mg doped polymethylmethacrylate (PMMA) which exhibited 6 wt. % at 250 °C. The Li-conjugated microporous polymers could able to store more than 6 wt. % of hydrogen at 77.3 K and at 1 bar [35].

Up to date, none of the proposed hydrogen storage systems appears able to match US-DOE targets. Recent studies showed that the polymer nanocomposites could be an attractive material for hydrogen storage. The present work is aimed at the synthesis and characterization of light weight and compact hydrogen storage material based on polymer nanocomposite using sulfonated poly(ether-ether-ketone) (SPEEK) and hexagonal boron nitride (h-BN) nanoparticles, where the PEEK has excellent mechanical and chemical properties and thermal stability [36,37]. The dipolar nature of B–N bonds in hexagonal boron nitride (h-BN) can have stronger absorption of hydrogen from hydrocarbons. where phase inversion technique is employed for the preparation of microporous SPEEK-h-BN nanocomposite membranes. So it is expected that the light weight and more microporous nature of SPEEK could accommodate more h-BN nanoparticles which in turn can adsorb more hydrogen molecules.

Experimental methods

Materials

The PEEK polymer from Sigma Aldrich and h-BN (99.99% purity) with average particle size of 70 nm from Sisco Research Laboratories were purchased. Concentrated sulfuric acid (H_2SO_4) (98%) and N, N-Dimethylformamide (DMF) were received from Merck.

Preparation of SPEEK

The present work involves preparation of sulfonated PEEK and SPEEK-h-BN nanocomposite membranes for the hydrogen

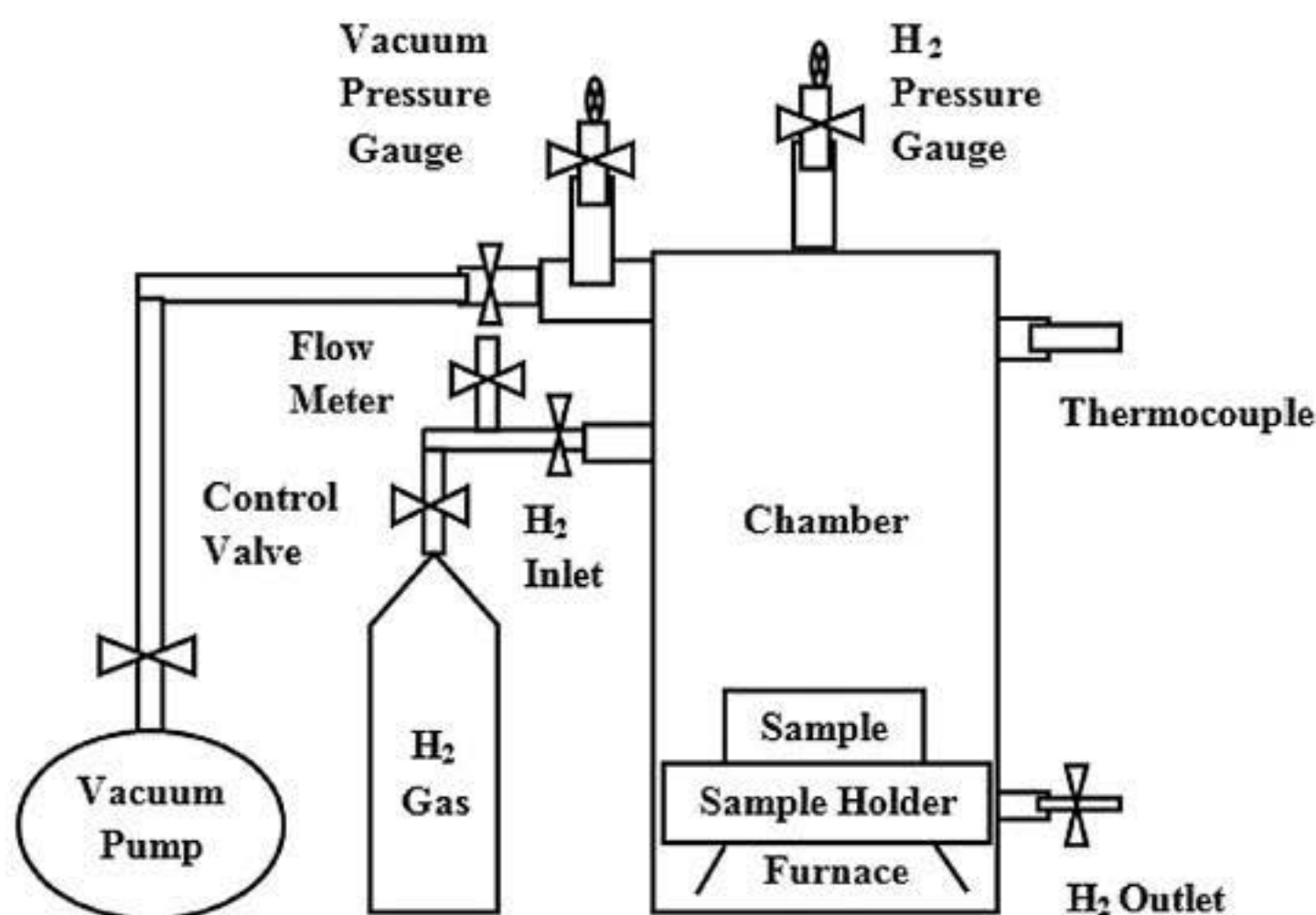


Fig. 1 – Schematic diagram of Sieverts-like hydrogenation setup.

adsorption studies. Poly (ether-ether ketone) (PEEK) was sulfonated as described in literature [36,38,39]. The pure PEEK polymer was dried in a vacuum oven at 100 °C for 12 h, then dissolved in concentrated sulfuric acid (98%) and heated to 40 °C under constant stirring for 24 h. This solution was decanted into large excess of ice-cold water to precipitate the acid polymer. The precipitate obtained was repeatedly washed with distilled water until the rinsed water has a pH value above 6. The washed precipitate was further dried at 100 °C for 12 h. The final product obtained was sulfonated PEEK, which has the desired degree of sulfonation.

Preparation of SPEEK-h-BN nanocomposite membranes

The SPEEK-h-BN nanocomposite membranes were prepared with 0, 1, 3 and 5 wt % of h-BN using a phase inversion technique [38,40,41]. To fabricate SPEEK-h-BN nanocomposite membranes, SPEEK polymer was dissolved in N, N-Dimethylformamide and water (non-solvent). Then h-BN nanoparticles were taken with N, N-Dimethylformamide at 100 mg/5 ml concentration. To allow h-BN nanoparticles to be dispersed uniformly the mixture was kept in an ultrasonic bath at room temperature for 1 h. Then both the solutions were magnetic stirred. After getting homogeneous solution it was casted on a glass plate. Then it was heated at 60 °C for 5 h and dried at 100 °C for 20 h in vacuum in order to avoid moisture. The thickness of the resulting membranes was found to be in the range of $52 \pm 10 \mu\text{m}$.

Hydrogenation studies

Hydrogen adsorption studies have been carried out for the developed SPEEK-h-BN nanocomposite membranes using Sievert's-like hydrogenation setup and its schematic diagram is shown in Fig. 1. The details of hydrogenation procedure are

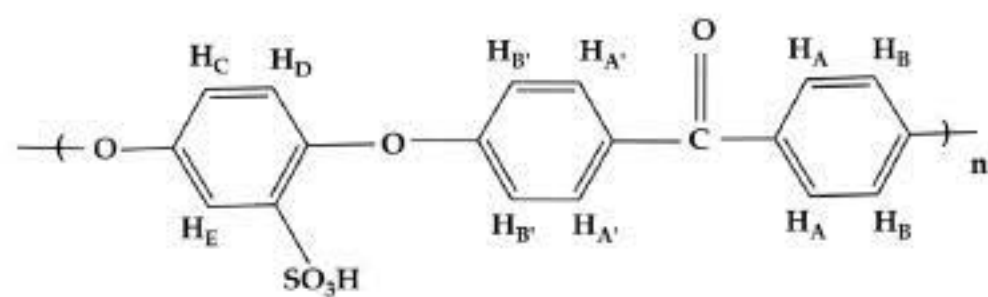
as follows. Before hydrogenation, the chamber should be free from leak. The prepared membranes were loaded in the sample container and the hydrogen was flushed few times within the chamber for proper flow. The membranes were maintained in a vacuum at 150 °C for 1 h. Then the hydrogen was allowed to react with these membranes at a constant flow rate of 0.5 L/min for 15 min, where the equilibrium pressure of the chamber was maintained at 1 kg/cm². The hydrogenated membranes were kept in the chamber to attain room temperature.

Characterization

Powder X-ray diffraction (XRD) technique (X'Pert PAN analytical diffractometer), Micro-Raman spectroscopy (Labram HR800), Fourier Transform Infrared (FTIR) spectroscopy (FTIR-8400, CE, Shimadzu model), Scanning Electron Microscopy (SEM, JEOL-MODEL 6390), Atomic force microscopy (AFM, A-100 SGS), CHN – elemental analysis (Elementar Vario EL III), Thermo Gravimetric Analysis (TGA, SII EXSTAR 6000), Proton Nuclear Magnetic Resonance (¹H NMR) spectroscopy (Bruker 300 MHz) were used to examine the structure, composition, functional groups, desorption temperature and degree of sulfonation of the prepared nanocomposite membranes.

Results and discussion

The Degree of Sulfonation (DS) is estimated from ¹H NMR spectrum by finding the ratio of peak area of the distinct H_e signal (A_{H_e}) to the peak area of all the other aromatic hydrogens (A_{H_{A,A',B,B',C,D}}). The aromatic hydrogens are denoted by the symbol "H_x" in the SPEEK structure, where x = A, A', B, B', C, D [38]. Scheme 1 shows the presence of various aromatic protons in the SPEEK structure [37,38].



Scheme 1 – Structure of SPEEK.

Fig. 2 displays the ^1H NMR spectrum of SPEEK and has a distinct signal at 7.5 ppm that indicates the presence of protons of the $-\text{SO}_3\text{H}$ group. DS can be calculated from the following formula [36,37,39].

$$\frac{n}{12 - 2n} = \frac{A_{\text{H}_E}}{\sum A_{\text{H}_{\text{A,A}',\text{B,B}',\text{C,D}}}}$$

where 'n' is the number of H_E per repeat unit

$$\text{DS}(\%) = n \times 100$$

The estimated degree of sulfonation of our prepared SPEEK is 65%. In general, the DS of 40–70 % produces a more mechanically and thermally stable polymer of SPEEK [39,42].

Fig. 3 shows the XRD patterns of SPEEK-0%h-BN, SPEEK-1%h-BN, SPEEK-3%h-BN and SPEEK-5%h-BN nanocomposite membranes. Fig. 3(a) shows a very broad peak centered at 22° , which indicates the amorphous nature [43] of SPEEK membrane. A characteristic sharp reflection at $2\theta = 27^\circ$ in Fig. 3(b–d) confirms the presence of h-BN in the prepared nanocomposite membranes [44]. It is noticed that the intensity of h-BN peak increases with increase of h-BN content in the nanocomposite membranes and it is the clear evidence for the crystalline nature of nanocomposite membranes [45,46]. The absence of any other peak except SPEEK and h-BN proves that the prepared nanocomposite membranes are free from impurities.

Fig. 4 shows the Raman spectra of SPEEK-0%h-BN, SPEEK-1%h-BN, SPEEK-3%h-BN and SPEEK-5%h-BN nanocomposite membranes. Fig. 4(a) indicates a Raman mode for SPEEK polymer at 1149 cm^{-1} and is attributed to the sulfonation [47]. In Fig. 4(b–d), the sharp peak of E_{2g} mode appears at 1367 cm^{-1} corresponding to the h-BN and is due to the in-plane atomic displacement of B and N atoms against each other [48]. The intensity of the h-BN mode (1367 cm^{-1}) increases compared to SPEEK mode (1149 cm^{-1}) as the content of h-BN nanoparticles increases in SPEEK and enhances the crystalline nature of the nanocomposite membranes and it is confirmed by XRD results.

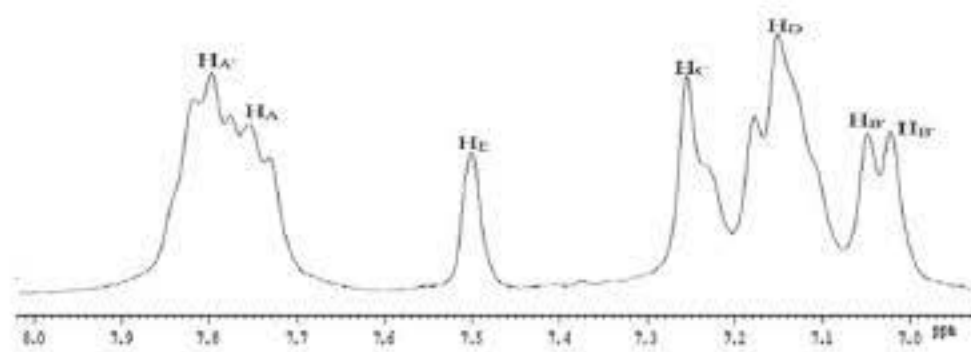
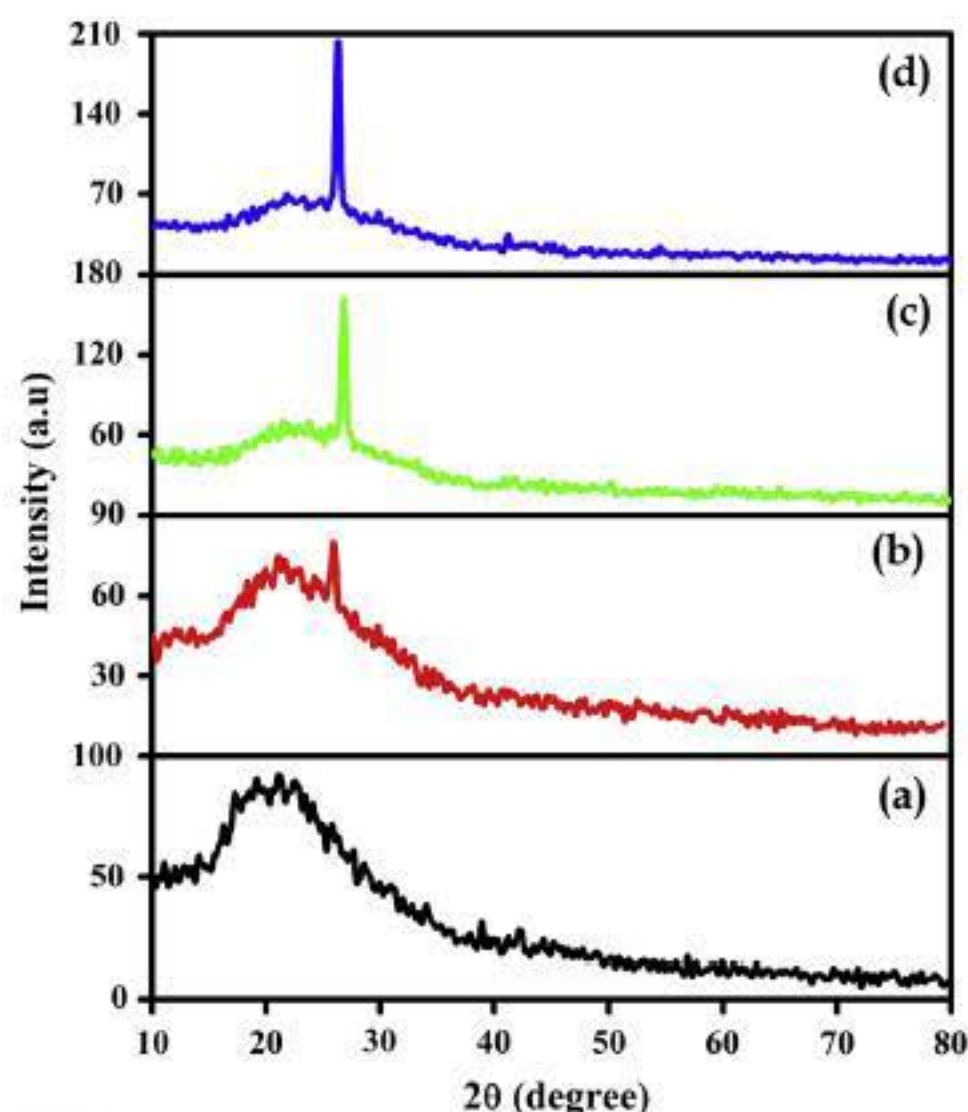
Fig. 2 – ^1H NMR spectrum of Sulfonated PEEK in DMSO-d_6 .

Fig. 3 – XRD profile of (a) SPEEK-0%h-BN (b) SPEEK-1%h-BN (c) SPEEK-3%h-BN and (d) SPEEK-5%h-BN nanocomposite membranes.

The presence of functional groups has been determined from the bands appearing in the FTIR spectrum and Fig. 5 shows the FTIR spectra of SPEEK and SPEEK-h-BN nanocomposites. The IR spectrum of SPEEK (Fig. 5(a)) has the

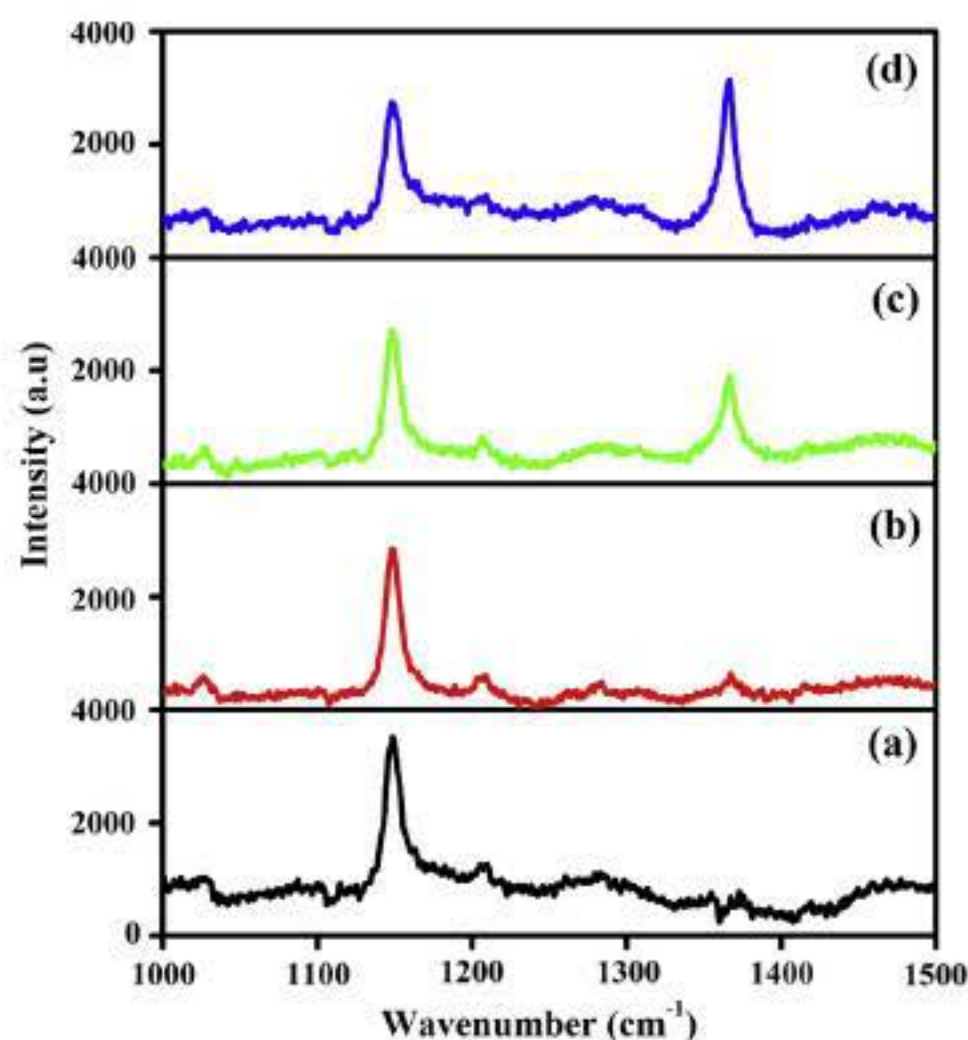


Fig. 4 – Raman spectra of (a) SPEEK-0%h-BN (b) SPEEK-1%h-BN (c) SPEEK-3%h-BN and (d) SPEEK-5%h-BN nanocomposite membranes.

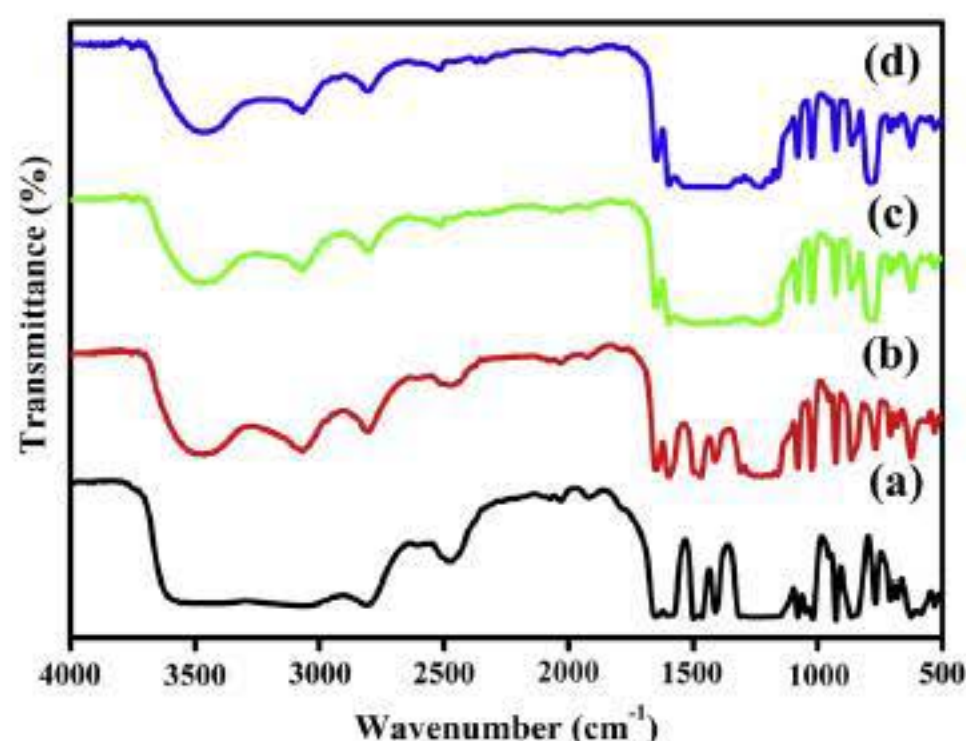


Fig. 5 – FTIR spectra of (a) SPEEK-0%h-BN (b) SPEEK-1%h-BN (c) SPEEK-3%h-BN and (d) SPEEK-5%h-BN nanocomposite membranes.

prominent absorption peaks at 3470, 1652, 1078 1016 and 864 cm^{-1} . The characteristic peak at 3470 cm^{-1} is assigned to the O–H vibration of sulfonic acid groups. In addition the peak at 1652 cm^{-1} is assigned to the carbonyl band of SPEEK. The absorption bands at 1078, 1016 and 864 cm^{-1} correspond to the O=S=O symmetric vibration, symmetric stretching of sulfonated S–O modes and out-of-plane C–H bending of an isolated hydrogen in a 1,2,4-trisubstituted phenyl ring respectively. These characteristic modes confirmed the presence of sulfonic acid groups in the prepared SPEEK [49–52].

In addition to the peaks observed for SPEEK, some new prominent peaks are noticed (Fig. 5 (b–d)) at 3460, 2367 and 1190 cm^{-1} in the case of prepared polymer nanocomposite membranes. These new characteristic peaks are due to the presence of h-BN nanoparticles in SPEEK. The peak at 3460, 2367 and 1190 cm^{-1} correspond to N–H stretching vibration, asymmetric stretching mode of B–H and bending mode of B–H of SPEEK-h-BN nanocomposite and this confirms the incorporation of h-BN nanoparticles in the SPEEK matrix [53–55].

Fig. 6 shows the SEM images which are helpful to examine the presence and distribution of h-BN nanoparticles in SPEEK-h-BN nanocomposite membranes. The SPEEK has smooth and

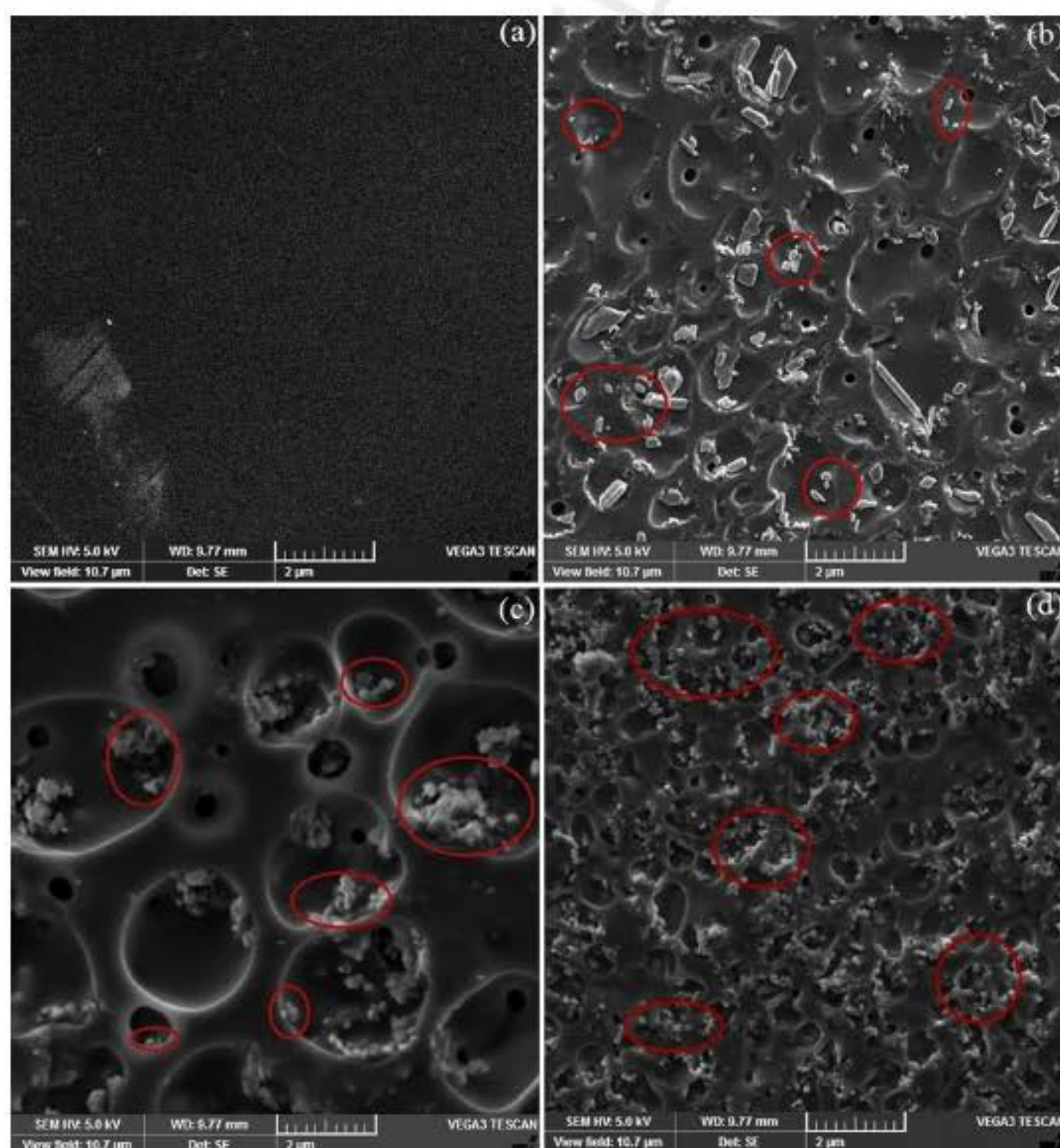


Fig. 6 – SEM photograph of (a) SPEEK-0%h-BN (b) SPEEK-1%h-BN (c) SPEEK-3%h-BN and (d) SPEEK-5%h-BN nanocomposite membranes.

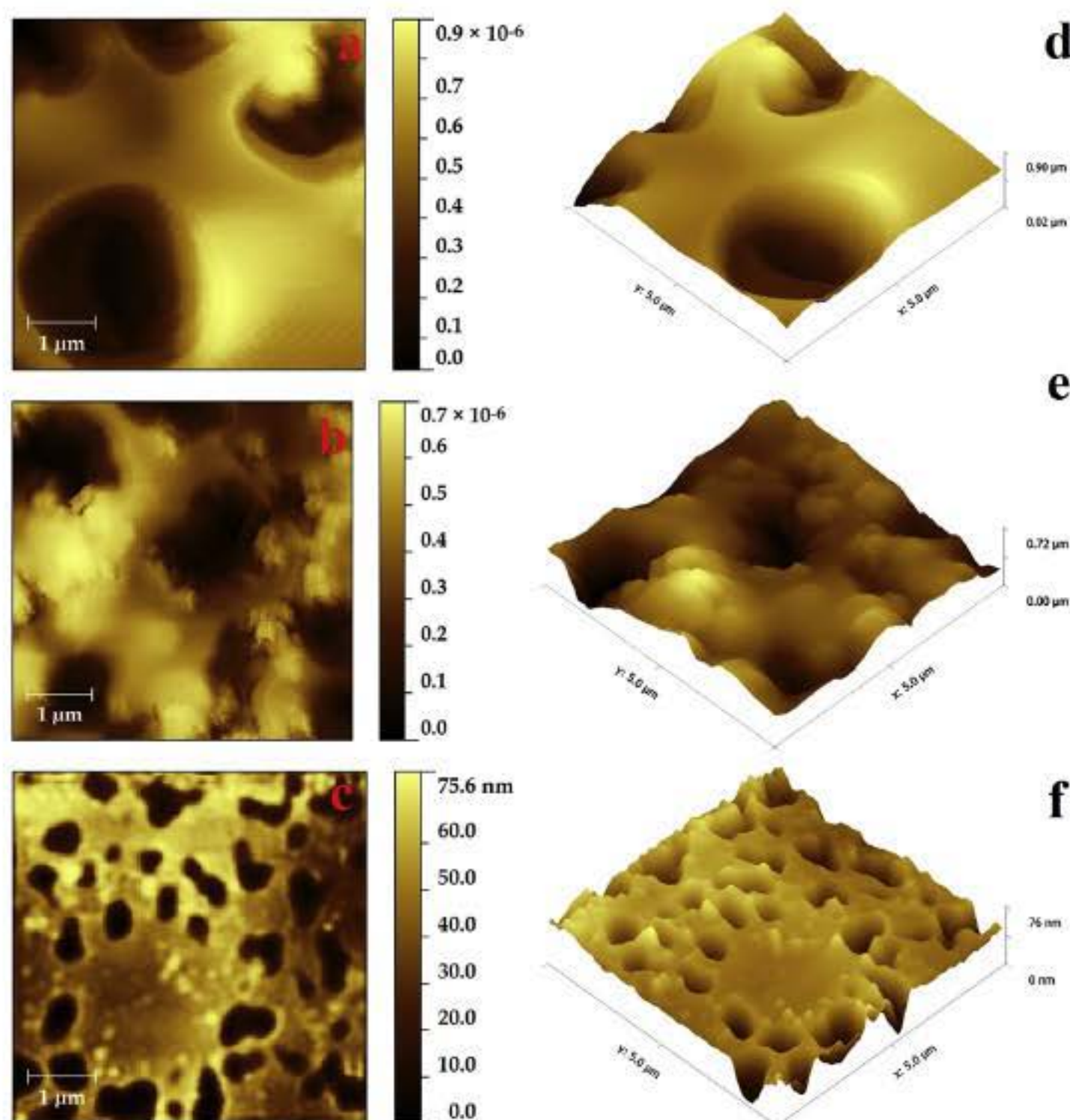


Fig. 7 – AFM images of (a) SPEEK-1%h-BN (b) SPEEK-3%h-BN (c) SPEEK-5%h-BN nanocomposite membranes (Two dimensions) (d) SPEEK-1%h-BN (e) SPEEK-3%h-BN (f) SPEEK-5%h-BN nanocomposite membranes (Three Dimensions).

uniform surface morphology (see Fig. 6a). This smooth morphology confirms the amorphous nature of SPEEK polymer which agrees well with our XRD results. The remaining micrograph depicts the porous structure, indicating the

distribution of h-BN nanoparticles with the particle size in the range of 70–90 nm and some of h-BN nanoparticles agglomerated in SPEEK matrix. The presence of pores in the microstructure is mainly due to the non-solvent removal and

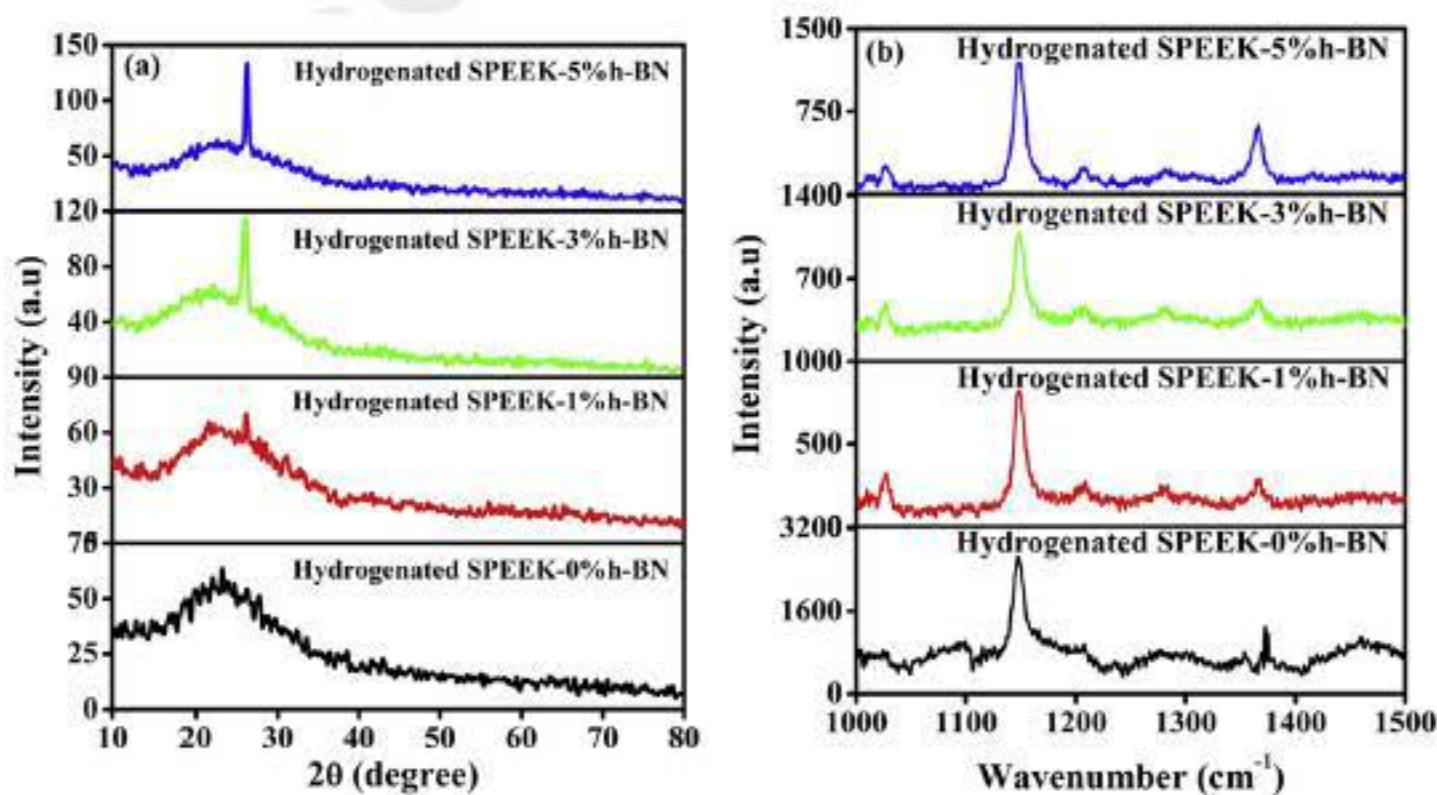


Fig. 8 – (a) XRD spectra (b) Raman spectra of hydrogenated SPEEK-h-BN nanocomposite membranes.

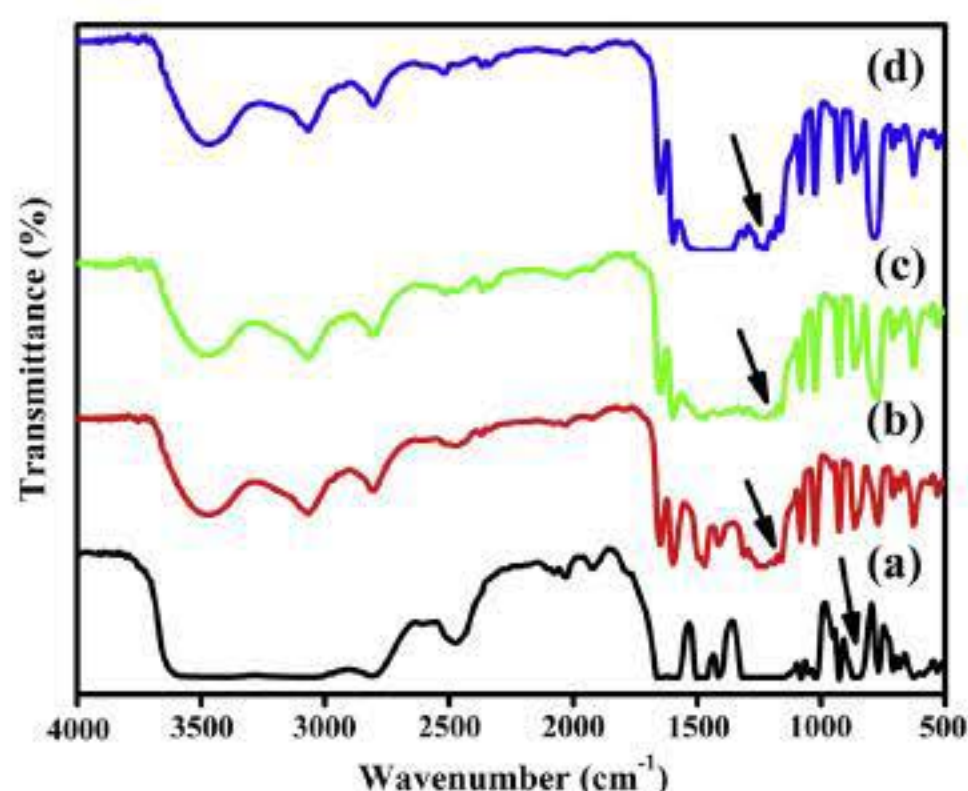


Fig. 9 – FTIR spectra of hydrogenated (a) SPEEK-0%h-BN (b) SPEEK-1%h-BN (c) SPEEK-3%h-BN and (d) SPEEK-5%h-BN nanocomposite membranes.

solvent retention ability of the system [56]. Moreover, some of h-BN nanoparticles are distributed uniformly and are highlighted (see Fig. 6b–d).

The topographic images of 2D and 3D of SPEEK-h-BN nanocomposite membranes are captured by AFM and are presented in Fig. 7. From the AFM image, it is evident that the smoothness of membrane surface decreases as the content of h-BN increases in the prepared nanocomposite membranes. Since the phase inversion method has been adopted in the present study, a porous top-layer is formed due to non-solvent removal and creates more pores in the prepared polymer nanocomposite membrane [48]. It is observed that, as the quantity of h-BN increases, the number of pores increases and simultaneously the pore size decreases. Our AFM observations are consistent with the SEM results.

Adsorption analysis

It is expected that hydrogenated nanocomposite membranes exhibit peculiar behavior. The XRD/Raman spectra (Fig. 8) of hydrogenated SPEEK-h-BN nanocomposites are lower in intensity compared to unhydrogenated membranes and confirm the adsorption of hydrogen. The FTIR spectra of the hydrogenated SPEEK-h-BN nanocomposite membranes are shown in Fig. 9. Due to hydrogenation, some prominent modes are noted that are indicated by arrows in the spectra.

CHN-elemental analysis is performed to find the quantity of hydrogen incorporated in hydrogenated SPEEK-h-BN

nanocomposite membranes. The hydrogen storage capacity of the prepared nanocomposite membrane is estimated by finding the difference of hydrogen content in the membranes before and after hydrogenation. The storage capacity of SPEEK-0%h-BN, SPEEK-1%h-BN, SPEEK-3%h-BN and SPEEK-5%h-BN nanocomposite membranes are found to be 0.66, 0.91, 1.66 and 2.98 wt. %, respectively. The presence of more pores in SPEEK matrix and more adsorption sites in the h-BN nanoparticles could be the reason for increased storage capacity in nanocomposite membranes. Therefore, it is concluded that the SPEEK-5%h-BN membrane is a more effective hydrogen storage medium than other membranes. The results obtained are compared with earlier reports and are given in Table 2. However, most of the hydrogenation experiments reported in Table 2 have been conducted either at low temperature or at different pressure conditions. Since the present work is conducted at 150 °C, a proper interpretation of our results with earlier works could not be possible. Moreover, the storage of hydrogen at low temperature and high pressure conditions are not realistic for automobile application.

Desorption analysis

Fig. 10 displays the TG plot of hydrogenated nanocomposite membranes and the inset shows the region where hydrogen desorption takes place. The TGA spectrum of SPEEK-5%h-BN (see Fig. 10d) exhibits two weight loss, one at the temperature interval of 214–218 °C and another above 250 °C. The initial weight loss of 2.98% is attributed to desorption of stored hydrogen in the hydrogenated membrane. The second weight loss starts above 250 °C which corresponds to the disintegration of SPEEK [36,39,57]. Hence, one can confirm that the initial weight loss is due to the release of stored hydrogen and not by the hydrogen in SPEEK. From the desorption temperature, the activation energy of desorption (E_d) was calculated using the following equation [54,58].

$$\ln\left(\frac{T_m^2}{\beta}\right) = \frac{E_d}{RT_m}$$

where, T_m is the desorption temperature, β is the heating rate (10 °C/min) and R is the universal gas constant. Furthermore, the binding energy (E_b) of hydrogen is calculated using the van't Hoff equation [59,60]. The calculated values of desorption temperature of hydrogen, activation energy of desorption and binding energy are displayed in Table 1.

In order to make use of these prepared light weight and compact membranes for fuel cell applications, the recommended binding energy of stored hydrogen should be

Table 1 – The desorption parameters in hydrogenated SPEEK-h-BN nanocomposite.

Polymer nanocomposite	H ₂ Weight loss (wt. %)	Desorption temperature range (°C)	Desorption activation energy range (kJ/mol)	Binding energy range (eV)
SPEEK-0%h-BN	0.66	190–192	25.53–25.67	0.362–0.364
SPEEK-1%h-BN	0.91	201–204	26.32–26.54	0.371–0.373
SPEEK-3%h-BN	1.66	204–214	26.54–27.26	0.373–0.381
SPEEK-5%h-BN	2.98	214–218	27.26–27.56	0.381–0.384

Table 2 – Hydrogen storage capacity of various polymer composites.

Polymer nanocomposite	H ₂ (wt %)	Conditions	Reference
Hypercrosslinked polymers – Pt	0.21	298.15 K and 19 bar	[28]
Hypercrosslinked Polystyrene – Pt	0.36	294 K and 100 atm	[29]
PANI – SnO ₂	0.31	115 °C and 70 bar	[30]
PANI – MWCNTs	0.38	115 °C and 70 bar	[30]
PANI – Al	0.5	115 °C and 70 bar	[30]
ARP-CTs	0.62	293 K and 7.5 MPa	[34]
HNT – PANI	0.78	RT and 0.5 MPa	[31]
PEEK-Manganese oxide	1.2	77 K	[6]
PVA – SWCNTs	1.2	50 °C	[32]
PVP – SWCNTs	1.5	50 °C	[32]
PANI – VONC	1.8	77 K and 70 bar	[33]
SPEEK/h-BN	2.8	150 °C	Present work
ARP-CTs	5.2	77 K and 5 MPa	[34]
Li-conjugated microporous polymers	6	77.3 K and 1 bar	[35]
PMMA – Mg	6	250 °C	[4]

0.2–0.4 eV [54,58,59,61]. The obtained binding energy of hydrogenated SPEEK-h-BN nanocomposite membranes lies within this range. Moreover the nanocomposite membranes exhibit 100% desorption which is estimated by comparing the amount of stored hydrogen from CHN-elemental analysis and desorption of stored hydrogen from TG analysis, where the amount of stored hydrogen is equal to the amount of desorbed hydrogen. Moreover, the hydrogenated SPEEK-h-BN

nanocomposite membranes are stable at room temperature. Hence, it is expected that the SPEEK-h-BN nanocomposite membranes could be an effective hydrogen storage medium in near future.

Conclusion

A lightweight and compact hydrogen storage medium using SPEEK and h-BN was prepared by phase inversion technique. The prepared hydrogen storage medium was subjected to various characterization studies like XRD, micro-Raman, FTIR, SEM, AFM, CHN and TGA. The XRD and micro-Raman results demonstrated the presence of h-BN nanoparticles in SPEEK polymer. The distribution of h-BN nanoparticles on the surface of SPEEK was confirmed by SEM and the AFM images indicating the porous nature of membranes. The hydrogenated SPEEK-h-BN nanocomposite membranes (hydrogen storage medium) were characterized by XRD, micro-Raman, FTIR, CHN and TGA. The XRD, micro-Raman and FTIR confirm the adsorption of hydrogen in the synthesized SPEEK-h-BN nanocomposite membranes. The hydrogen storage capacity of SPEEK-h-BN nanocomposite membrane increases with increasing h-BN and is due to the larger number of adsorption sites offered by h-BN. The hydrogenated membranes are stable at room temperature. A 2.98 wt. % of hydrogen storage capacity was achieved in SPEEK-5%h-BN nanocomposite membrane. The average binding energy of hydrogen was found to be 0.38 eV and the nature of hydrogen binding is weak chemisorption. Moreover the desorption temperature was found to be 214–218 °C for the stored hydrogen to be released and SPEEK-h-

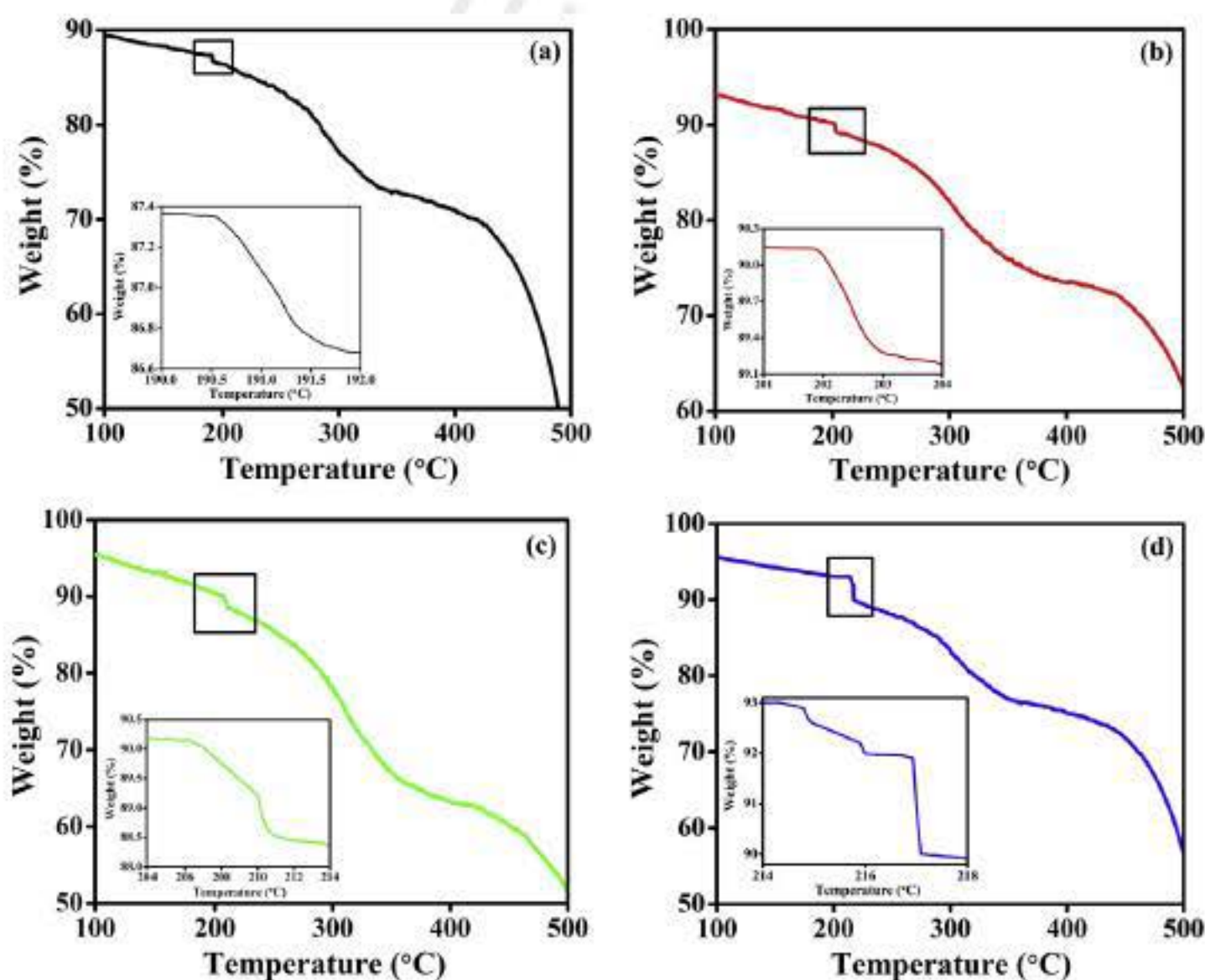


Fig. 10 – TGA spectra of hydrogenated (a) SPEEK-0%h-BN (b) SPEEK-1%h-BN (c) SPEEK-3%h-BN and (d) SPEEK-5%h-BN nanocomposite membranes.

BN membrane shows 100% desorption. It is clear from the present investigation that the SPEEK-h-BN nanocomposite could be a potential candidate for hydrogen storage in near future for fuel cell application.

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