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1. Introduction

Modified membrane technology is widely used in the petrochemical sector, notably for gas purifications and separations. The main advantages of membrane technology over more conventional separation techniques, such gas purification and separation, are energy efficiency and environmental considerations. To increase gas permeability and selectivity, several novel families of membrane material modifications have been developed. As a result, selecting a membrane material for the gas permeation process is an important step. As polymeric materials are used in this research, several alterations may be made to the membrane materials to increase their performance with regard to the suggested application. Even the membrane-based separation and purification process uses less energy and is less expensive to run. A trade-off between the selectivity and permeability of gases dependent on a polymeric membrane and enhancing both the permeability and selectivity of parameters has generally limited polymeric films. The majority of research on polymeric gas separation membranes has improved gas permeability or selectivity. Typically, rather than the other way around, glassy polymers find it challenging to improve permeability without compromising their outstanding inherent selectivity. Thermally rearranged (TR) polymers and membrane films for gas separation from precursor polyimides with ortho-positioned hydroxyl groups [1]. Due to their high-free volume topologies, TR polymers have a specific free-volume element architecture. These polymers excel at producing membranes with excellent permeability, selectivity, and resistance to harsh environments, which are required for a wide range of gas separation applications This study focuses on employing different precursors or thermal treatment methods to incorporate inorganic nanoparticles into polymer structures. They are produced by heating aromatic polyimides with functional groups positioned orthogonally and have a rigid aromatic structure.

It has also been used as a carrier of energy in transportation and power generation and heating solutions. For this reason, hydrogen is seen as a sustainable energy carrier that may facilitate the much-needed transition from fossil to renewable energy sources, and it is a crucial element in achieving net-zero emissions in the next 20 years. Although hydrogen is the future's cleanest, most sustainable, and renewable energy source. Thus, using a filter, hydrogen must be separated from other gas mixtures like CO_2 or CH_4. Membrane technology may provide economic, environmental, and high-performance benefits as a low-cost, energy-efficient, and high-performance method of gas separation. Utilizing inorganic elements like graphene oxide and carbon nanotube sieves in a polymer matrix to improve polymer membrane performance for gas permeation has been the subject of much study. Due to the enormous increase in demand for ultra-pure hydrogen over the last several years, new methods of hydrogen purification have been under pressure to be developed. [2]. The ability for chemically modifying nanofillers to interact better with polymer matrix has garnered a lot of interest recently. Graphene, due to its outstanding structural, mechanical, thermal, optical, and electrical properties, graphene makes a good two-dimensional filler nanomaterial for polymer nanocomposites. Consequently, further efforts to develop new membranes that perform better in terms of energy efficiency should be made in addition to the emphasis on graphene-based materials. Porous graphene and graphene oxide (GO) nanocomposite membranes have been the subject of much study in recent years for use in gas separation applications [3]. GO substrates have the potential to increase membrane thickness and penetrating flux while still offering an appropriate level of mechanical strength for the production of composite membranes [4]. In comparison to glass or metal, plastic packaging is more permeable to small molecules, such as gases, water vapour, and organic vapour, as well as other low molecular weight components like food additives, flavours, and smells. Along with other characteristics like strong heat stability and superior mechanical capabilities, polymer science researchers seem to be concentrating more on developing suitable materials for the food packaging industry [5]. Nitrogen, carbon dioxide, and oxygen are the gases often used in packaging for controlled environments (N_2, O_2, CO_2). Other gases, such as Ar and H_2, are being researched in food packing. However, by including GO nanoparticles into the polymer matrix, the significant change in gas permeability with the mechanical and thermal stability of both composite membranes was enhanced [6].

2. Experimental Techniques for synthesis and characterizations

To measure permeate flux in terms of height gain of mercury slug by the mercury flow metre and bubble flow meter linked to the gas permeability cell, a constant pressure variable volume system has also been developed. Additionally, permeate flow is measured using a constant volume variable pressure method by utilising a pressure transducer (Pirani gauge) to record the increase in pressure of collected permeate gas in a sealed container. The permeability of gases including H_2, CO_2, CH_4, O_2, Ar, and N_2 has been measured using both methods. Additionally, additional membrane characteristics like mechanical characteristics and thermal stability are taken into consideration. The characteristics of the membrane are further analysed using techniques including scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), universal testing machine (UTM), Water

Contact Angle Analysis, Thermogravimetric Analysis (TGA) and differential scanning calorimeter (DSC). At the time of thesis submission, some additional characterisation methods will be used.

3. Gas Transport Through Thermally Rearranged Polyimide Nanocomposite Membranes

3.1 Synthesis and characteristics of HAB-6FDA thermally rearranged polyimide nanocomposite membranes

In this study, HAB-6FDA polyimide was synthesized from 3,3 dihydroxy-4,4-diamino-biphenyl (HAB) and 2,2-bis-(3,4-dicarboxyphenyl) hexafluoropropane dianhydride (6FDA) by chemical imidization. A sample was modified from a pure polymer to silica nanoparticle composition. Furthermore, a modification was carried out by thermal rearrangement reaction at temperatures of 350 °C, 400 °C, and 450 °C. Permeability decreases with an increase in the kinetic diameter of gasses, which is normal behavior for glassy polymers. The composition of silica nanoparticles slightly changes the permeability in the polyimide. The combined effect of silica nanoparticles and thermal rearrangement of the HAB-6FDA membrane has shown an excellent performance. The thermal rearrangement with nanocomposite shows a significant impact on a larger effect on permeation for lighter gases, that is, H_2, CO_2, and O_2, compared with N_2 and CH_4. Particularly for H_2/CH_4 gas pair, it lies over Robeson's 2008 upper bound limit, which fits the composition in the novel class for the gas separation membranes [7].

3.2 Transport, Spectroscopic, and Electrical Properties of Thermally Rearranged Nanocomposite Membranes

Nanocomposite membranes are a class of innovative filtering materials consisting of nanofillers embedded in a polymeric or inorganic oxide matrix that functionalizes the membrane. Thermally rearranged (TR) polymers have a good blend of selectivity and permeability. Nanocomposite membranes were tested after applying a TR process. The selectivity decreases with increasing permeability, showing a trade-off relationship between permeability and selectivity. TR polymer nanocomposite exhibits higher gas permeability than silica-doped and pure polymer. The selectivity for H_2/N_2 and H_2/CO_2 gas pairs exceeds the Robeson upper-bound limit in the case of the H_2/CH_4 gas pair. The selectivity for H_2/N_2, H_2/CH_4 and H_2/CO_2 gas pairs exceeds the Robeson upper-bound limit [8].

4. Study of Characterization of Glassy Polymer Blends and Composite Membranes

4.1 Transport Properties of Polymer Blends and Composites Membranes for Selective Permeation of Hydrogen

This paper reports a study of blends of PMMA and PS that were prepared in different ratios of weight percentage for PMMA: PS (80:20, 50:50 and 60:40) composite with 1 wt% of GO and 2 wt% of GO. The permeability measurements indicate that the GO nanofillers in blends of PS/PMMA have shown higher permeability for hydrogen gas than that of pure polymers. The gases used for the permeation measurements were H_2, CO_2, N_2 and CH_4. Selectivity has been calculated for H_2/CO_2, H_2/N_2 and H_2/CH_4 gas pairs and plotted to show Robeson's 2008 upper bound and compared with reported data. The transport properties of these gases have been compared with that of a neat membrane. The permeability of all gases has increased to that of the unmodified polymer membrane. The selectivity measurements show that GO composite with PS/PMMA blend membranes is highly selective for hydrogen gas from different gas pairs, therefore, these composite membranes can be used for hydrogen purification. There is a trade-off between permeability and selectivity parameters; GO nanofillers keep selectivity constant as permeability increases, which the nanogap theory could explain [9].

4.2 Study of Thermal Behavior of Various PMMA-PS / GO Nanocomposite Blends Membrane

The thermal conductivity of polymer nanocomposites made of PS/GO and PMMA/GO is studied with crystallinity and free volume impact. This paper reports a DSC study of PMMA/GO (1 wt%) & PS/GO (1 wt%) and nanocomposite blends of PMMA and PS prepared in different ratios by weight percentage for all blend ratios used for PMMA: PS (80:20, 60:40, 50:50, 60:40, 20:80) composite with 1 wt% of graphene oxide using the solution cast method. The samples were analysed by DSC instrument at different heating rates from 5 ºC/min, 10 ºC/min and 20 ºC/min in the temperature range of 50 ºC to 160 ºC. We see significant shifts in FT-IR, XRD, and DSC analyses, DSC and TGA thermogram of nanocomposite blend supported the confirmation of the formation of PS/PMMA blend and demonstrated a significant change in its thermal behavior with blend ratio. Similar results have been obtained when calculating the activation energy of crystallization using either the isokinetic or isoconversional methods. The activation has determined pure and blended polymers through their glass transition using different models of the isoconversional method [11].

4.3 Energy Bandgap and Thermal Analysis of Polymer PC/GO Nanocomposites Membrane Films

Nanocomposites of Polycarbonate (PC) polymer with graphene oxide (GO) were made using the solution casting method. This work reports a DSC study of PC/GO ((2, 5, 7, 10 and 15 wt%) and the thermal stability of PC nanocomposite with the different wt% ratio of graphene oxide was studied using DSC analysis, which showed an increase in thermal stability due to the presence of graphene oxide. The samples were analysed at different heating rates from 5 ºC/min, 10 ºC/min and 20 ºC/min in the temperature range of 50 ºC to 160 ºC. It was found that the experimental glass transition temperature is very close to the theoretical values. FTIR spectroscopy and X-ray diffraction analyses revealed that the crystalline phases of PC/ GO composite depended on the graphene oxide content in mixed solvents. Significant changes in SEM, TGA and DSC analysis are observed, which reveals that interactions between the PC/GO nanocomposites had well certain miscibility. UV spectroscopy shows the change in transmission at higher wavelengths and calculated energy bandgap from Tauc's plot. The activation energy of crystallization has been evaluated using both isokinetic and isoconversional approaches, and similar findings have been founds [12].

4.4 Thermally Stable PET/PS Polymer Blend Membranes with Enhanced Properties for Food Packing Applications

The purpose of this paper was to make that type of polymer blend that gives low permeability properties for the polymer packaging used in the food business. In the present work, we have analysed various thermograms of Polyethylene terephthalate (PET), Polystyrene (PS) and their blends using DSC and TGA to check the glass transition temperature, weight loss and thermal stability of materials. A study of gas permeability, thermal stability and mechanical properties of PET and PS polymer blends with different weight percentage ratios of PET: PS (80:20, 60:40, 50:50, 60:40, 20:80) synthesized by solution cast method. According to the permeability tests, a 1 wt% of GO composite with PET/PS blends has shown a change in permeability than pure polymers. H_2, CO_2, N_2, Ar and O_2 were the gases used for the penetration measurements. This paper describes the blending of PET/PS with enhanced barrier characteristics to O_2, CO_2, N_2, Ar and H_2, especially improving the thermal and mechanical properties of PET/PS blends. When compared to the results obtained with PET/PS blend, we can determine that with a PS polymer concentration of 50%, the permeability of all gases was decreased by around 40% as compared to pure PET membrane. Porosity analysis and contact angle are also analysed. In this study, we focused on developing packaging materials' mechanical, degradability, thermal stability, and antibacterial activity for various packaging applications [10].

5. Gas Transport Through A Blend of Glassy and Rubbery Polymer Nanocomposites

The blending of polymers has been studied in different amount for different polymeric materials. However, very few works have been reported for mixture of inorganic nanoparticles with blending of two different polymers. In this chapter, the transport behavior of various gases such as H_2, CO_2, O_2, N_2, CH_4 and Ar across PS/PDMS blend membranes as well as blend composites with GO and CNT has been studied.

5.1 CO_2 Separation through Different Blends of PS/PDMS Polymers for Food and Beverage Industry

The efficiency of the ideal CO_2/N_2 and CO_2/CH_4 membrane separation was examined in relation to the polymer backbone (i.e., polystyrene (PS), Polydimethylsiloxane (PDMS) and functional cationic substituent (i.e., alkyl, fluoroalkyl, oligo(ethylene glycol), and disiloxane). However, these polymers experience physical degradation, which decreases gas permeability over time, as well as competitive sorption by heavy hydrocarbons, which increases CO_2 permeability. A strong CO_2 gas solubility selectivity that is unaffected by heavy hydrocarbons in the raw natural gas is the result of the ether/ester oxygen groups' attractive interactions with CO_2 but not CH_4. By using PS and PDMS, we develop a polymer blend of glassy- rubbery, solubility-selective polymers with a high ratio of ether/ester oxygen to carbon to get around these challenges. More free volume and weaker size-sieving properties result from increased CO_2/CH_4 selectivity in polymers with higher CO_2 permeability. By inhibiting competitive sorption, hydrocarbons (such as CH_4 and heavy hydrocarbons) in natural gas might increase CO_2 permeability by inhibiting CO_2 sorption in glassy polymers. Due to the decrease in non equilibrium volume with time, which may significantly increase gas permeability, thin films of glassy polymers experience physical aging. By developing glassy-rubbery, solubility-selective polymers for CO_2/CH_4 separation for raw natural gas, we provide a significantly new approach. The permeability/selectivity trade-off has effectively broken by increasing solubility selectivity since the trade-off requires the absence of any particular interactions between polymers and gases. Most importantly, in the presence of high pressure and heavy hydrocarbons, solubility selectivity may be achieved much well than diffusivity selectivity. These rubbery, solubility-selective polymers exhibit strong separation properties near the upper bound when treating simulated natural gas, in contrast to conventional glassy polymers with a high diffusivity selectivity. They also show robust sorption challenges by heavy hydrocarbons and the physical aging of thin films [13].

5.2 CO_2 Removal from Natural Gases specifically for lighter gases N_2 and CH_4 through CNT doped PS/PDMS nanocomposite blend Membranes

Membrane materials with a high CO_2/CH_4 diffusivity selectivity have based on glassy polymers for CO_2 removal from natural gas. In this study, we provide a cross-linking method that is less damaging and more effective, based on a composite containing CNT nanofillers, to increase the stability of the TFN membrane for CO_2/CH_4 separation. CNT nanoparticles (diameters of 50 nm) are used as fillers in the selective layer, which is composed of a PS/PDMS blends. Moreover, the cross-linked membranes had greater plasticization pressure than the majority of reported membranes. The PS/PDMS membranes were characterized for single (CO_2, H_2, CH_4, and N_2) and mixed (CO_2/N_2 and CO_2/CH_4) gas permeability. The resultant composite with CNT membranes have increased the selectivities for CO_2/N_2 and CO_2/CH_4, respectively, with a high CO_2 permeance. The performance of MMMs for gas separation has evaluated using H_2, CO_2, O_2, CO_2, and CH_4. Comparing composites membrane to blends membrane, CO_2 permeance increased. As the PDMS weight percentages has increased in the PS phase, gas permeability of all gasses enhances. It shows a huge change in the CO_2 permeability as the PDMS weight percentage has gained up to 25% of total PS weight percentage. What is more, CNT nanofillers plays a vital role to improve permeability in the same blend composition. Further, the permeability has improved by increasing the filler weight percentage. PDMS being a rubbery polymer, adds porosity in the final component which provides a large surface area for the gas molecules to be soluble within the membrane thickness. Last but not the least, such a hybrid membrane composition, has enhanced the separation factor of some of the gas pairs in such a way that it has tend towards the Robeson upper bound limits of both 1991 and 2008. For gas pairs, N_2/CH_4, H_2/CH_4 and CO_2/CH_4, the membrane compositions have crossed the upper bound limits [14].

5.3 Mechanically and thermally improved DES/GO doped PS/PDMS nanocomposite membranes to enhance the stability of CO_2/CH_4 separations for flue gas application

Thin film composite (TFC) membrane development presents a possibility of achieving the permeability/selectivity requirements for the highest CO_2 separation performance. However, poor mechanical characteristics and significant CO_2 plasticization are the greatest challenges to the performance and longevity of thin film gas separation membranes. Here, we developed novel PS/PDMS blend composite with GO nanofillers with substantial aromatic chain extenders that provide the ideal mechanical characteristics for the creation of ultra-thin films. GO nanofillers based PS/PDMS blends showed an improvement in Young's modulus and hardness, over only blends of PS/PDMS membranes. In order to synthesize PS/PDMS blends with GO nanofillers with constant thicknesses between 70 and 75 nm from liquid polymer solutions [15]. DES/GO nanofillers based PS/PDMS have improved mechanical characteristics, resulting in high-performance thin membranes with selectivity comparable to the bulk polymer. In comparison to DES/GO blend membranes wit pure GO nanofillers, the thin film membranes made from DES/GO nanofillers based PS/PDMS blends also demonstrated a twofold increased plasticization strength. The obtained nanocomposite blends were characterised using a variety of methods, including XRD, SEM, FTIR, and TGA, to comprehend their compositional and structural characteristics. The addition of these DES/GO nanofillers improves CO_2 permeability and CO_2/N_2 selectivity in mixed matrix membranes, according to the results [16].

6. Summary of work and Future Scope

This chapter summarises the studies reported in the thesis and describes the scope of upcoming studies into the Glassy and Rubbery polymer with different types of nanocomposite membranes.

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List of Publications:

1. **H. D. Patel**, N. K. Acharya, Synthesis and Characteristics of HAB-6FDA thermally rearranged (TR) polyimides nanocomposite membranes, Polymer Engineering and Science, WILEY, V- 61, Issue -11,P :2782–91, Aug. 2021, **I.F. 2.573**
2. **H. D. Patel,** N. K. Acharya, Transport Properties of Polymer Blends and Composites Membranes for Selective Permeation of Hydrogen, International Journal of Hydrogen Energy, Elsevier, June 2022, (Article in Press), **I.F. 7.2**
3. **H. D. Patel**, N. K. Acharya, Transport, Spectroscopic, and Electrical Properties of Thermally Rearranged Nanocomposite Membranes, Chemical Engineering & Technology, V- 45, Issue -12, P :2223–2233, Oct 2022, **I.F. 2.215**
4. **H. D. Patel,** N. K. Acharya, Thermally Stable PET/PS-GO Polymer Blend Membranes with Enhanced Properties for Food Packing Applications
   (Manuscript to be communicated)
5. **H. D. Patel**, N. K. Acharya, Energy Bandgap and Thermal Analysis of Polymer PC/GO Nanocomposites Membrane Films
   (Manuscript to be communicated)
6. **H. D. Patel**, N. K. Acharya, Study of Thermal Behavior of Various PMMA-PS/ GO Nanocomposite Blends Membrane
7. **H. D. Patel**, A. K. Patel, N. K. Acharya, A Significant Permeability and Selectivity of PS/ PDMS Blends to Enrich the Upper Bound Visualization
   (Manuscript to be communicated)
8. **H. D. Patel**, A. K. Patel, N. K. Acharya, CO_2 Removal from Natural Gas specifically for lighter gases N_2/CH_4 through PS/PDMS Blend Membranes composite with CNT nanofillers
   (Manuscript to be communicated)
9. **H. D. Patel**, A. K. Patel, N. K. Acharya, Mechanically and thermally improved DES/GO doped PS/PDMS nanocomposite membranes to enhance the stability of CO_2/CH_4 separations for flue gas application
   (Manuscript to be communicated)

Papers presented in the conferences:

1. A New Way to Determine the Melting Behaviour of Polymer Blends,
   H. D. Patel, N. K. Acharya,
   22nd Workshop and Symposium on Thermal Analysis THERMANS-2020, Jan 28-Feb 01, 2020, BARC, Mumbai.

2. Gas permeation and thermal properties of thermally rearranged (TR) HAB-6FDA polymer nanocomposite membranes,
   H. D. Patel, N. K. Acharya,
   4th International Conference on Polymers for Energy conversion and storage, Dec 13-18, 2020, SMRS, Jaipur.

3. Synthesis & characterization of polyimides nanocomposite membranes
   H. D. Patel, N. K. Acharya,
   International conference on Recent Advances in Material Science (ICRAMS- 2021), May 15 – 17, 2022, HNB, Uttrakhand.

4. Spectroscopic and Transport Behavior of HAB - 6FDA Thermally Rearranged (TR) Nanocomposite Membranes
   H. D. Patel, N. K. Acharya,
   Frontline Area in Chemical Science (FACS 2021), Oct 3-4, 2021, Pacific University, Udaipur.

5. Thermal Analysis of various PMMA/PS polymer blends
   H. D. Patel, N. K. Acharya,
   International Symposium on Applications of Thermal Analysis and Calorimetry (SATAC- 2021), Dec 21-22, 2021, Magadh University, Bodh Gaya.

6. Transport Properties of Polymer Blends and Composites Membranes for Selective Permeation of Hydrogen
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   International Conference on Renewable Energy (ICRE 2022), Feb 25 – 27, 2022, Rajasthan University, Jaipur

7. Thermally Stable PET/PS Polymer Blend Membranes with Enhanced Properties for Food Packing Applications
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8. CO_2 Removal from Natural Gases specifically for lighter gases N_2 and CH_4 through CNT doped PS/PDMS nanocomposite blend Membranes
   H. D. Patel, N. K. Acharya,
   International Online Conference on Nano Materials, Mahatma Gandhi University, Kottayam, Kerala, India,
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