

CHARACTERISATION AND APPLICATION OF PBI

Submitted in partial fulfilment of the requirements for the award of

Bachelor of Science in Chemistry

by

SWETHA.S (39030039)

NANDHINI.E (39030016)



DEPARTMENT OF CHEMISTRY

SCHOOL OF SCIENCE AND HUMANITIES

SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

Accredited with Grade "A" by NAAC | 12B Status by UGC | Approved by AICTE

JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI - 600 119

April - 2022



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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **SWETHA.S (39030039)** and **NANDHINI.E (39030016)** who carried out the project entitled “**CHARACTERISATION AND APPLICATION OF PBI**” under our supervision from December 2021 to April 2022.

Internal Guide

Dr.K.CHENNAKESAVULU M.Sc., Ph.D.,

Head of the Department

Dr. J. KARTHIKEYAN, M.Sc.,Ph.D.

Submitted for Viva voce Examination held on _____

Internal Examiner

External Examiner

DECLARATION

We **SWETHA.S (39030039)** and **NANDHINI.E (39030016)** hereby declare that the Project Report entitled **CHARACTERIZATION AND APPLICATION OF PBI** done by us under the guidance of **Dr.K.CHENNAKESAVULU, M.Sc., Ph.D.**, Associate Professor, Department of Chemistry at **Sathyabama Institute of Science and Technology**, Jeppiaar Nagar, Rajiv Gandhi Salai, Chennai – 600119 is submitted in partial fulfillment of the requirements for the award of Bachelor of Science degree in Chemistry.

DATE:

PLACE:

SIGNATURE OF THE CANDIDATE

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SWETHA S

NANDHINI.E

ABSTRACT

Polybenzimidazoles (PBIs) and benzimidazole-linked polymers (BILPs) have exceptional thermal and chemical stability, and hence, their membranes were developed and used under harsh conditions. In this review, the formation, structures, and properties of these polymers are studied follow by the fabrication of membranes. Applications, such as gas separation, organic solvent nanofiltration, water treatment, pervaporation and proton exchange, are extensively reviewed. The relationship of membrane performance and structure is established, highlighting the importance of processing protocols and post treatments. Future directions are provided on the basis of the conclusions.

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LIST OF ABBREVIATION

FTIR	Fourier Transform Infrared Spectroscopy
FT RAMAN	Fourier Transform Raman Spectroscopy
TGA	Thermal gravimetric Analysis

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CHAPTER 1

INTRODUCTION

1.1 POLY BENZIMIDAZOLE (PBI):

Poly [2,2'- (m-phenylene)- 5,5'- bibenzimidazole], alluded to all through this article as PBI, is notable for its exceptional warm stability and synthetic obstruction. PBI is a thermoplastic; it has the most elevated T_g , 435°C (815°F), of any monetarily accessible polymer. It doesn't consume, contribute fuel to flares or produce a lot of smoke. These qualities are an outcome of the innate compound dependability of the benzimidazole usefulness found in the polymer spine[3].

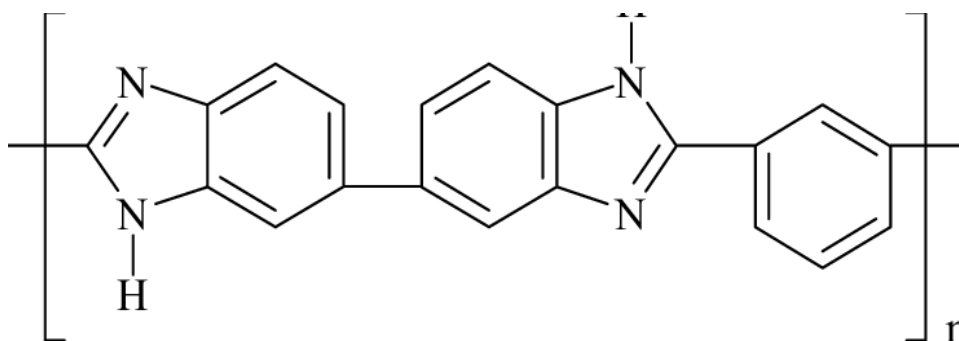


Fig 1.1: Structure of PBI

Polybenzimidazoles (PBI) are a class of heterocyclic polymers, which have uncommon warm, compound, and mechanical sound qualities at raised temperatures. When created into films and doped with low fume pressure proton guides like phosphoric corrosive (PA), the relating corrosive doped PBI films were accounted for as promising other options to customary perfluorosulfonic corrosive sort films (for example Nafion®). For the use of polymer electrolyte layer power modules (PEMFCs) such films give benefits like high functional temperatures (120-200 °C), quick anode energy, improved on water the board, and high resistance to fuel debasements (for example CO, H₂S) [1-5].

Among different PBI subordinates, meta-PBI (poly(2,2'-(m-phenylene)- 5,5'-bibenzimidazole)) is the most concentrated because of its business accessibility, yet it likewise has shortcomings, for example, powerless mechanical properties at high

corrosive stacking and unfortunate solvency in natural solvents. Another significant PBI variation is para-PBI (poly(2,2'-(p-phenylene)- 5,5'-bibenzimidazole)). Its acid-doped film was ready by an extraordinary sol-gel process what's more, displayed higher corrosive doping levels (>30 mol PA per PBI repeat unit) and better proton conductivity (>0.2 S cm $^{-1}$) than meta-PBI while as yet keeping up with strong mechanical strength.



Fig 1.2: Polybenzimidazole

Nonetheless, the solid chain normal for para-PBI brought about by a more inflexible para-arranged moiety makes the polymer practically insoluble in any natural solvents, which limits its handling window. Accordingly, as of late significant research has been centered around examining new PBI science, which could offer a superior mix of wanted properties for power device applications.[7]

Polybenzimidazoles have been accounted for in the writing since the mid 1960s. While being notable for their great thermo-oxidative security, PBIs were remembered to be unmanageable 'block ties'.

- (I) PBI can be made at high atomic weight,
- (II) PBI is flexible and
- (III) Benefit can be taken from PBI's solvency in dimethylacetamide (DMAc) considering the easy readiness of PBI lattice composites containing consistent fiber support.[3].

The amalgamation of a progression of polybenzimidazoles (PBI) with presentation of methylene bunches into the polymer tie to adjust the processability of the polymers was accounted for in the past paper.' As these underlying changes impacted the warm strength of the polymers also, we have completed a more exhaustive examination of the decay designs by thermogravimetric-mass spectrometric (TGA-MS) methods.

It was found that, not at all like the all-sweet-smelling PBI, the majority of the polymers arranged in this series have two areas of disintegration: one, between 450 what's more, 580°C, yielding generally strong items, the other, at higher temperatures, yielding just vaporous deterioration items. It was additionally found that preoxidation of specific methylene bunches in the polymer chain changes this deterioration design profoundly and improves the warm dependability of the polymers.[8]

1.2 BACKGROUND:

PBI is ready from the polycondensation of tetraaminobiphenyl, TAB, with diphenylisophthalate, DIPI. Two potential instruments have been proposed. One instrument hypothesizes the presence of a polyamic corrosive as an essential middle of the road, which then, at that point, goes through parchedness and cyclization to the imidazole. The subsequent system proposes the presence of a Schiffs base moderate that cyclizes to a benzimidazoline which thusly disposes of phenol after shaping the imidazole.[3].

1.3 SYNTHESIS OF PBI:

Polybenzimidazole3,-Diaminobenzidine tetrahydrochloridedihydrate was bought from Triton Chemicals. 2,2-Bis(4-carboxyphenyl) hexafluoropropane was bought from ABCR GmbH. Polyphosphoric corrosive (115%) and sulfuric corrosive (95-97%) were bought from Sigma-Aldrich. All synthetics were utilized as gotten.

The substance of adsorbed water in 3-diaminobenzidine tetrahydrochloride not entirely settled from the weight reduction of an example treated in vacuum at room temperature. All responses were performed under nitrogen and the

polyphosphoriccorrosive as the response dissolvable was deoxygenated at 140 °C before expansion of monomers. Inborn viscosities of the polymer items were estimated in a Group Ubbelohde viscometer in 95-97% sulfuric corrosive at 30 °C.[2].

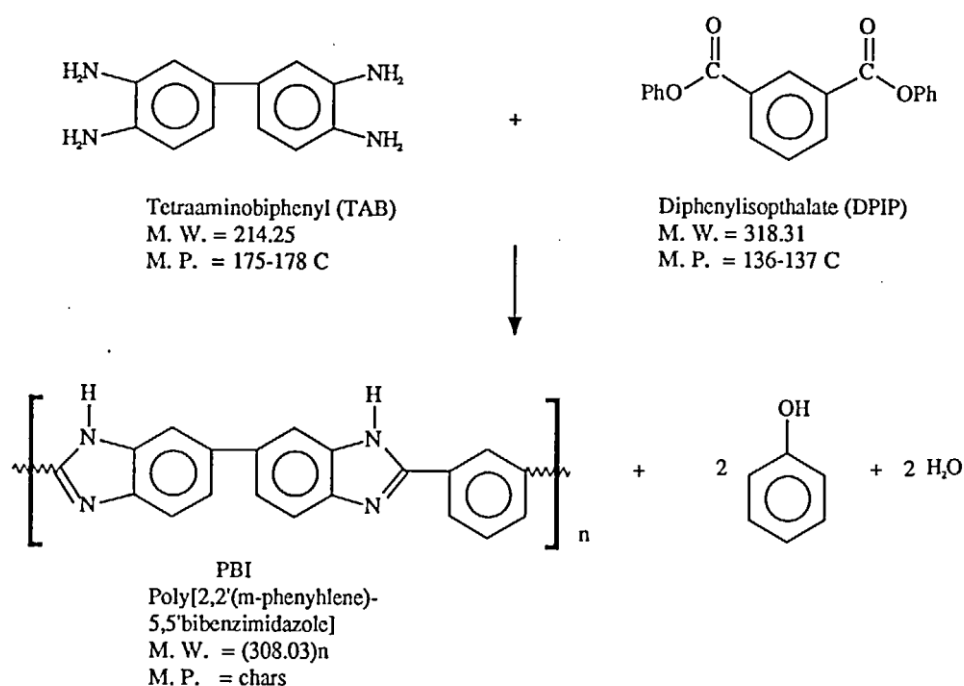


Fig 1.2: Synthesis of PBI

Poly [2,2'- (m-phenylene)- 5,5'- bibenzimidazole], PBI is the just economically accessible polybenzimidazole and is made at Hoechst Celanese's Rock Hill office (South Carolina, USA). The business polymerization happens in two phases, the two of which are done in an idle air. In the principal stage, the DPIP liquefies and solubilizes the TAB. As the temperature increases, polymerization starts and phenol and water are produced. The development of the buildup results causes the arrangement of a friable froth. In the subsequent stage, the froth is squashed and the polymer sub-atomic weight is progressed in the strong state.[3].

1.3 PBI FIBER AND ITS APPLICATIONS

The fiber is dry turned from a - 25% arrangement of PBI in DMAc. Expansion of a little level of lithium chloride protracts the arrangement's timeframe of realistic usability from days to months. The arrangement is ready under tension at temperatures over the limit of DMAc.

Particulates are taken out by filtration and the arrangement is then expelled into a warmed nitrogen air where most of dissolvable vanishes. The remaining dissolvable and lithium chloride are removed from the fiber by washing in water.

Following the wash, the fiber is dried, attracted to expand strength and afterward treated with sulfuric corrosive. Sulfonation of PBI lessens the fire shrinkage of the drawn fiber from >half to <10% [5]. At long last, the fiber is creased and cut, giving a material customary material handling qualities. [3].

CHAPTER 2

LITERATURE SURVEY

1.TITLE:Properties, degradation and high temperature fuel cell test of different types of PBI and PBI blend membrane.

AUTHOR:Q.F. Li, H.C. Rudbeck^b, A. Chromik^c, J.O. Jensen^a, C. Pana^a, T. Steenberg^b,M. Calverley^a, N.J. Bjerrum^a, J. Kerres^c.

JOURNAL AND YEAR:Journal of membrane Science

INFERENCE:Polybenzimidazoles (PBIs) with synthetically modified structures and their blends with a partially fluorinated sulfonated aromatic polyether have been prepared and characterized for high temperature proton exchange membrane fuel cells. Significant improvement in the polymer chemical stability in terms of the oxidative weight loss, molecular weight decrease and onset temperatures for the thermal SO₂ and CO splitting-off was achieved with the electron-deficient polybenzimidazoles containing –S(O)₂– and –C(CF₃)₂– bridging groups. Ionic cross-linking in the form of acid–base blends was found to further improve the polymer stability and assist maintaining the membrane integrity. Upon acid doping the membrane swelling was reduced for the modified PBI and their blend membranes, which, in turn, results in enhancement of the mechanical strength, proton conductivity and high temperature fuel cell performance.

2.TITLE:Synthesis and Characterization of a New Fluorine-Containing Polybenzimidazole (PBI) for Proton-Conducting Membranes in Fuel Cells

AUTHOR:X. Li¹, G. Qian¹, X. Chen², B. C. Benicewicz

JOURNAL AND YEAR:30th August 2013

INFERENCE:A new type of fluorine-containing polybenzimidazole, namely poly(2,2'-(2,2'-bis(trifluoromethyl)-4,4'-biphenylene)-5,5'-bibenzimidazole) (BTBP-PBI), was developed as a candidate for proton-conducting membranes in fuel cells. Polymerization conditions were experimentally investigated to achieve high molecular

weight polymers with an inherent viscosity (IV) up to 1.60 dl g⁻¹. The introduction of the highly twisted 2,2'-disubstituted biphenyl moiety into the polymer backbone suppressed the polymer chain packing efficiency and improved polymer solubility in certain polar organic solvents. The polymer also exhibited excellent thermal and oxidative stability. Phosphoric acid (PA)-doped BTBP-PBI membranes were prepared by the conventional acid imbibing procedure and their corresponding properties such as mechanical properties and proton conductivity were carefully studied. The maximum membrane proton conductivity was approximately 0.02 S cm⁻¹ at 180 °C with a PA doping level of 7.08 PA/RU. The fuel cell performance of BTBP-PBI membranes was also evaluated in membrane electrode assemblies (MEA) in single cells at elevated temperatures. The testing results showed reliable performance at 180 °C and confirmed the material as a candidate for high temperature polymer electrolyte membrane fuel cell (PEMFC) applications.

3.TITLE:Raman study of the polybenzimidazole–phosphoric acid interactions in membranes for fuel cells

AUTHOR:Fosca Conti, Anne Majerus,^a Vito Di Noto,^b Carsten Korte,^a Werner Lehnert^a and Detlef Stolten^a

JOURNAL AND YEAR:6th May 2012

INFERENCE:Poly(2,5-benzimidazole) (AB-PBI) membranes are investigated by studying the FT-Raman signals due to the benzimidazole ring vibration together with the C–C and C–H out-of- and in-plane ring deformations. By immersion in aqueous ortho-phosphoric acid for different time periods, membranes with various doping degrees, i.e. different molar fractions of acid, are prepared. The chemical–physical interactions between polymer and acid are studied through band shifting and intensity change of diagnostic peaks in the 500–2000 cm⁻¹ spectral range. The formation of hydrogen bonding networks surrounding the polymer seems to be the main reason for the observed interactions.

4.TITLE:Alkali doped polybenzimidazole membrane for high performance alkaline direct ethanol fuel cell

AUTHOR:HongyingHou^{a,c}, GongquanSun^a, RonghuanHe^b, ZhimouWu^{a,c}, Baoying Sun.

JOURNAL AND YEAR:Journal of power sources.

INFERENCE:An anion exchange membrane for alkaline direct ethanol fuel cell (ADEFC) was prepared by doping KOH in polybenzimidazole (PBI) membrane. The distributions of nitrogen, oxygen and potassium in the membrane were analyzed by means of XRD and SEM-EDX, respectively. It was found that free or combined KOH molecules may exist in the PBI matrix, which was helpful for the ionic conductivity of PBI/KOH. Ethanol permeability through this membrane was much lower than that of Nafion. For ADEFC based on this PBI/KOH membrane electrolyte, the power density was 3 to 6 times of the results in literatures. In addition, the micro-structure of alkali doped PBI and the interaction between KOH and PBI matrix were also speculated logically.

5.TITLE:PBI (Polybenzimidazole): Synthesis, Properties and Applications .

AUTHOR:R.B. Sandor

JOURNAL AND YEAR:Polymers. Vol.2, 1990

INFERENCE: (polybenzimidazole) is a thermally stable high performance polymer. In a variety of forms, e.g. fibers, neat resin and composites, PBI finds use in a wide range of high temperature applications. The synthesis of PBI, and properties, and applications of fiber and molded resin are discussed. Also included are recent developments in composite fabrication technology and PBI composite applications in carbon/carbon composites.

CHAPTER 3

AIM AND SCOPE

- (PBI) A made fiber where the fiber-framing substance is a long chain fragrant polymer having intermittent imidazole bunches as an indispensable piece of the polymer chain (FTC definition).
- The polymer is produced using tetra aminobiphenyl and diphenyl isophthalate and is dry turned from a bonehead with dimethylacetamide as a dissolvable. Qualities:
- An elite exhibition fiber with high synthetic opposition that doesn't consume in air. It has no liquefying point and doesn't trickle when presented to fire.
- The fiber and textures from PBI hold their adaptability, layered solidness, and critical strength without embrittlement in any event, when presented to fire or outrageous intensity.
- The fiber radiates little smoke in outrageous circumstances. It processes well on customary material hardware, having handling qualities like polyester.
- It tends to be utilized in 100 percent structure or mixed with different filaments.
- It has a high dampness recover and low modulus with solace properties like cotton.

CHAPTER 4

MATERIALS AND METHODS

4.1 MATERIALS

The materials used for this work are Poly Benz Imidazole, Fourier Transform Infr-red spectroscopy, Raman spectroscopy and Thermo Gravimetric Analysis.

4.2 CHARACTERIZATION TECHNIQUES

4.2.1. Fourier Transform Infrared Spectroscopy (FTIR)

Infrared spectroscopy takes advantage of the way that atoms retain frequencies that are normal for their design. These retentions happen at thunderous frequencies, for example the recurrence of the retained radiation matches the vibrational recurrence.

The energies are impacted by the state of the sub-atomic potential energy surfaces, the majority of the iotas, and the related vibronic coupling. Specifically, in the Born-Oppenheimer and symphonious approximations, for example when the sub-atomic Hamiltonian relating to the electronic ground state can be approximated by a symphonious oscillator in the neighborhood of the harmony sub-atomic math, the full frequencies are related with the typical modes comparing to the sub-atomic electronic ground state potential energy surface.

The thunderous frequencies are additionally connected with the strength of the bond and the mass of the particles at one or the flip side of it. In this way, the recurrence of the vibrations is related with a specific typical method of movement and a specific bond type.

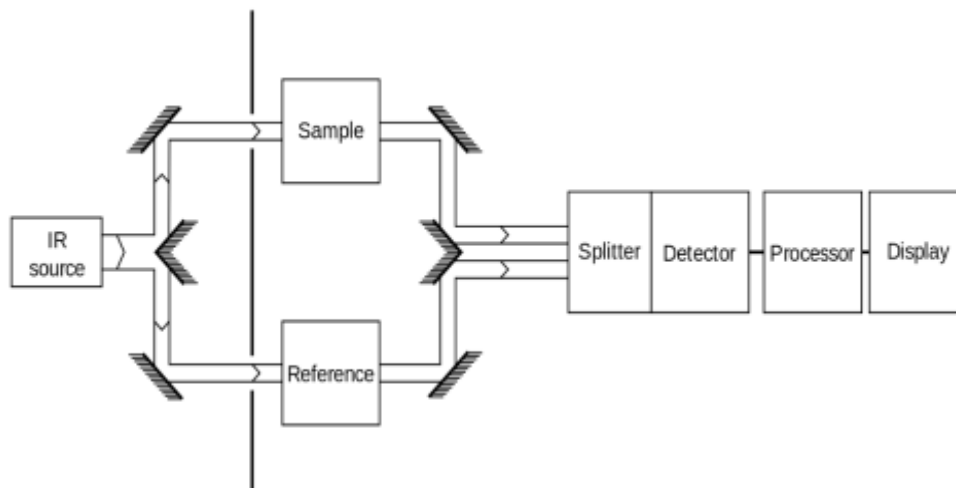


Fig.4.1. FTIR two beam absorption spectrometer

In order for a vibrational mode in a sample to be "IR active", it must be associated with changes in the dipole moment. A permanent dipole is not necessary, as the rule requires only a change in dipole moment. Six of these vibrations involve only the CH₂ portion: symmetric and anti- symmetric stretching, scissoring, rocking, and twisting.



Fig:4.2: FTIR Instrument

4.2.2. Raman Spectroscopy

The FT-Raman spectroscopy is a specific Raman configuration designed to collect fluorescence-free and wavelength-stable measurements from a wide range of samples, spanning from crystals to biological tissues (Hirschfeld and Chase, 1986).

A classical FT-Raman spectrometer is composed of an excitation source, an Nd:YAG laser, to produce photons in the NIR region (1064 nm), a sampling unit with proper collection optics, an interferometer comprising a fixed and moving mirrors, and a detecting unit.

A schematic representation of a conventional. A He Ne laser (633 nm “red” excitation) is made collinear to the excitation laser, to bring the system to the most effective optical alignment condition. In addition to guiding the Nd:YAG laser, the HeNe laser in an FT-Raman instrumentation functions as the reference laser for the operation of the Michelson interferometer. A line filter is used to filter out all unwanted plasma lines and to aid focusing of the incident light (1064 nm beam) on a sample sitting in a sample compartment.

In FT-Raman experiments, there is almost no need for sample pretreatment and good quality signals can be collected virtually from food samples at any physical state. The scattered light from the sample can then be collected using either 180 or 90 degrees back-scattering geometry, and passed through a dielectric filter to cut off the 1064- and 633-nm laser lines before entering the detector, which can either be nitrogen cooled Ge (germanium) or room temperature operating InGaAs (indium gallium arsenide).

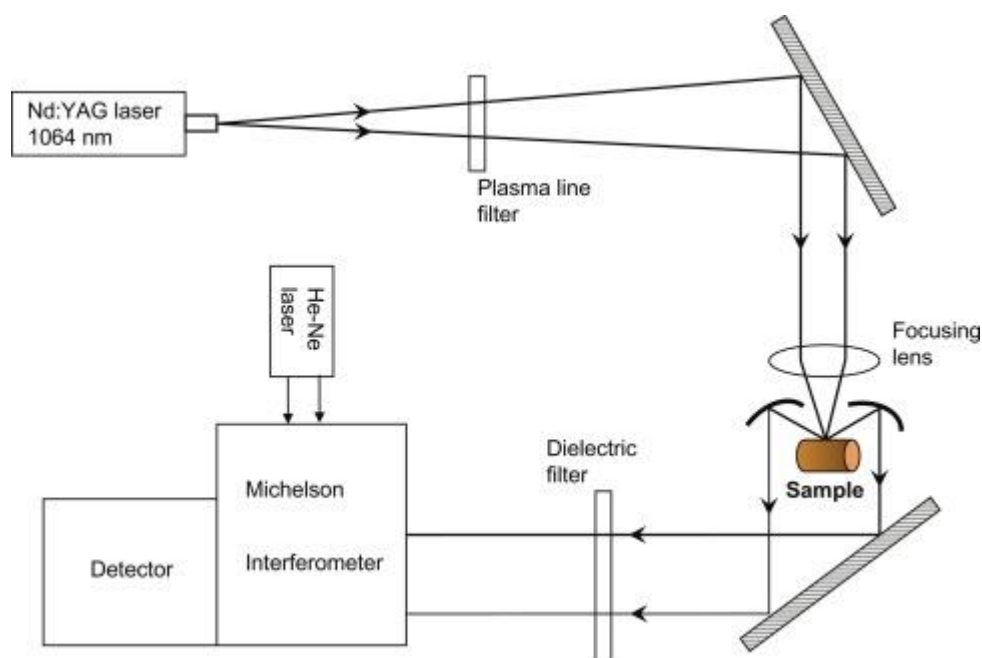


Fig:4.3: FT Raman Instrumentation

The central part of an FT-Raman spectrometer is the Michelson interferometer, composed of two perpendicular plane mirrors; one is fixed (or known as *dynamically aligned mirror*) and the other is the moving mirror (or known as the *scan mirror*). A beam splitter is used to transmit one part of the incident light partially (often 50%) toward the fixed mirror and reflect the other part toward the moving mirror.

Since the moving mirror travels at a constant velocity there will be an optical path difference (OPD) between the reflecting beams from these two mirrors. Interferogram as a trace of light intensity is produced as a function of OPD. Since OPD depends on the velocity of the moving mirror, it is the critical component of the interferometer. The beam transmitted from the fixed mirror and the other beam reflected from the moving mirror are combined into the detector, which detects the interference pattern between these two beams and transfers it into an electric signal.



Fig:4.4. FT Raman

4.2.3. Thermo Gravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) is a powerful technique for the measurement of thermal stability of materials including polymers. In this method, changes in the weight of a specimen are measured while its temperature is increased. Moisture and volatile contents of a sample can be measured by TGA. The apparatus consists of a highly sensitive scale to measure weight changes and a programmable furnace to control the heat of the sample. The balance is located above the furnace and is thermally isolated from the heat. A high precision hang-down wire is suspended from the balance down into the furnace. At the end of the hang-down wire is the sample pan, the position of which must be reproducible. The balance must be isolated from the thermal effects (e.g., by use of a thermostatic chamber) to maximize the sensitivity, accuracy, and precision of weighing. Addition of an infrared spectrometer to TGA allows analysis and identification of gases generated by the degradation of the sample.

The TGA apparatus is equipped with a micro-furnace that can be rapidly cooled. The heating element is made of platinum (reliable up to 1,000°C). An external

furnace with a heating element made of an alloy of platinum and 30% rhodium can extend the temperature range to 1,500°C.

A modern apparatus is usually equipped with a computer that calculates the weight-loss fraction or percentage. A commercial TGA is capable of >1,000°C, 0.1 µg balance sensitivity, and a variable controlled heat-up rate under an atmosphere of air or another gas. The heat-up rate capability of TGA can vary from 0.1°C–200°C/min. [7]



Fig:4.5. TGA

CHAPTER 5

RESULTS AND DISCUSSION

As a result of this review, the PBI was characterized using FTIR, Raman spectroscopy and Thermo Gravimetric Analysis. And with the outcome of these results the PBI was used for its applications.

5.1. Fourier Transform Infrared Spectroscopy (FTIR)

The sample was subjected for Fourier Transform Infrared Spectroscopy (FTIR) spectrum ranging from 4000 cm^{-1} to 500 cm^{-1} to find the functional groups presents in it. The results of the study are given in the figure. It may be seen that the broad band at 2921 cm^{-1} is due to the stretching mode of the $\text{sp}^3\text{-C-H}$ groups in hydrocarbon. The peak at 2852 cm^{-1} is due to the stretching mode of $\text{sp}^2\text{-C-H}$ groups in hydrocarbon. The peak 1461 cm^{-1} denotes methylene group of C-H bending and the peak 1377 cm^{-1} denotes C-H bending of methyl group.

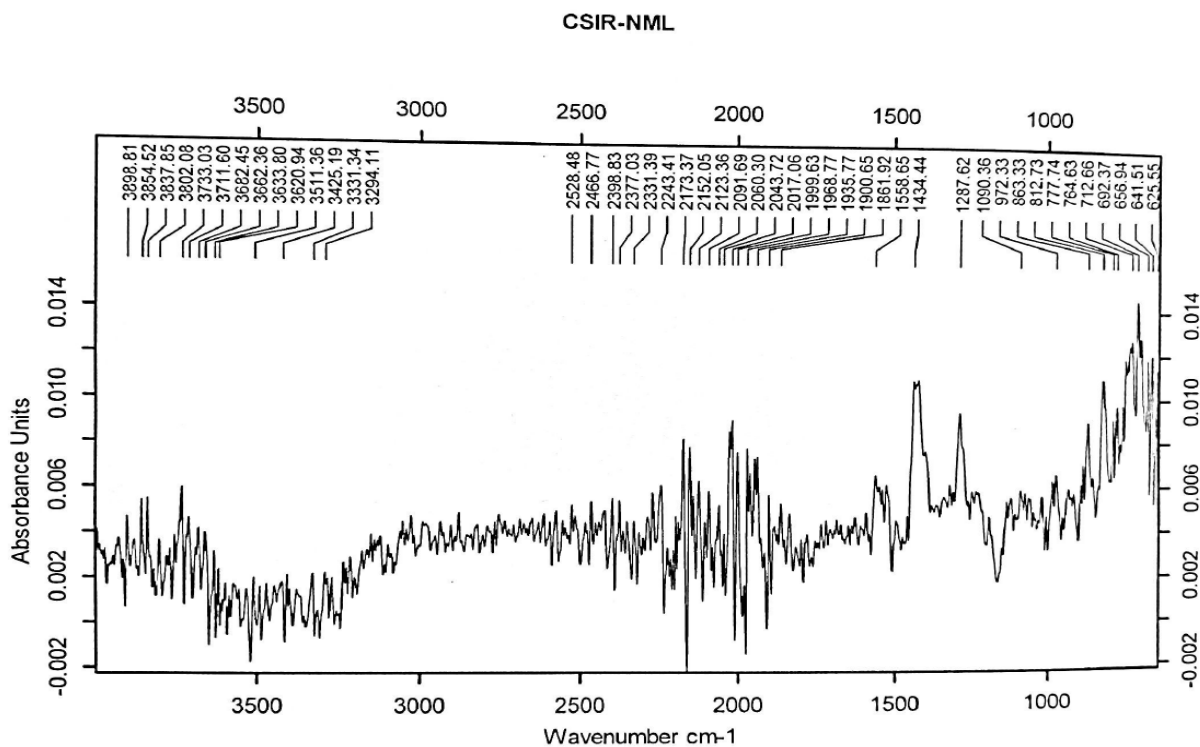


Fig:5.1. Spectra data of FTIR

The peaks at 1744 cm^{-1} denotes stretching mode of C=O in amino acids. The peak 1160 cm^{-1} denotes C-O stretching vibration. The peak 722 cm^{-1} denotes methylene group of CH₂ rocking vibration. These are the compound present in the 71 given oil. X-axis denotes the wavenumber (cm^{-1}) and Y-axis denotes the absorbance unit.

5.2. FT Raman Spectroscopy

Thermal analyses of the solution polymerized m-PBI and the commercially produced m-PBI were conducted via DSC and TGA. Modulated DSC was conducted from ambient temperature to 500 $^{\circ}\text{C}$ with 50 mL/min nitrogen flow and a heating rate of 5 $^{\circ}\text{C}/\text{min}$. The glass transition temperature (T_g) of the solution polymerized m-PBI was determined to be ~ 430 $^{\circ}\text{C}$ as indicated by a baseline shift and compared to the commercially produced m-PBI.

The T_g s of these meta PBIs found using DSC were similar to the previously reported meta-PBI T_g of 427 $^{\circ}\text{C}$.³⁴ PBI type polymers are well known for their high thermal stabilities resulting from their fully aromatic structure and polymer chain rigidities.^{35–37} TGA was conducted from ambient temperature to 800 $^{\circ}\text{C}$ at a heating rate of 10 $^{\circ}\text{C}/\text{min}$ for both solution polymerized and commercially produced m-PBI. Both materials exhibited more than 90% weight retention at 600 $^{\circ}\text{C}$, which is typical behavior of aromatic PBIs.³⁸

The commercially produced m-PBI sample showed water loss between 100 and 200 $^{\circ}\text{C}$, as expected since PBI is very hygroscopic and has been shown to absorb and retain up to 15% (by weight) of water even during short handling times.³⁹ Although, the solution polymerized m-PBI was washed with water and dried under vacuum before testing, the product showed gradual weight loss between 200 and 500 $^{\circ}\text{C}$ which was attributed to residual DMAc within the polymer sample and to the degradation of end groups.

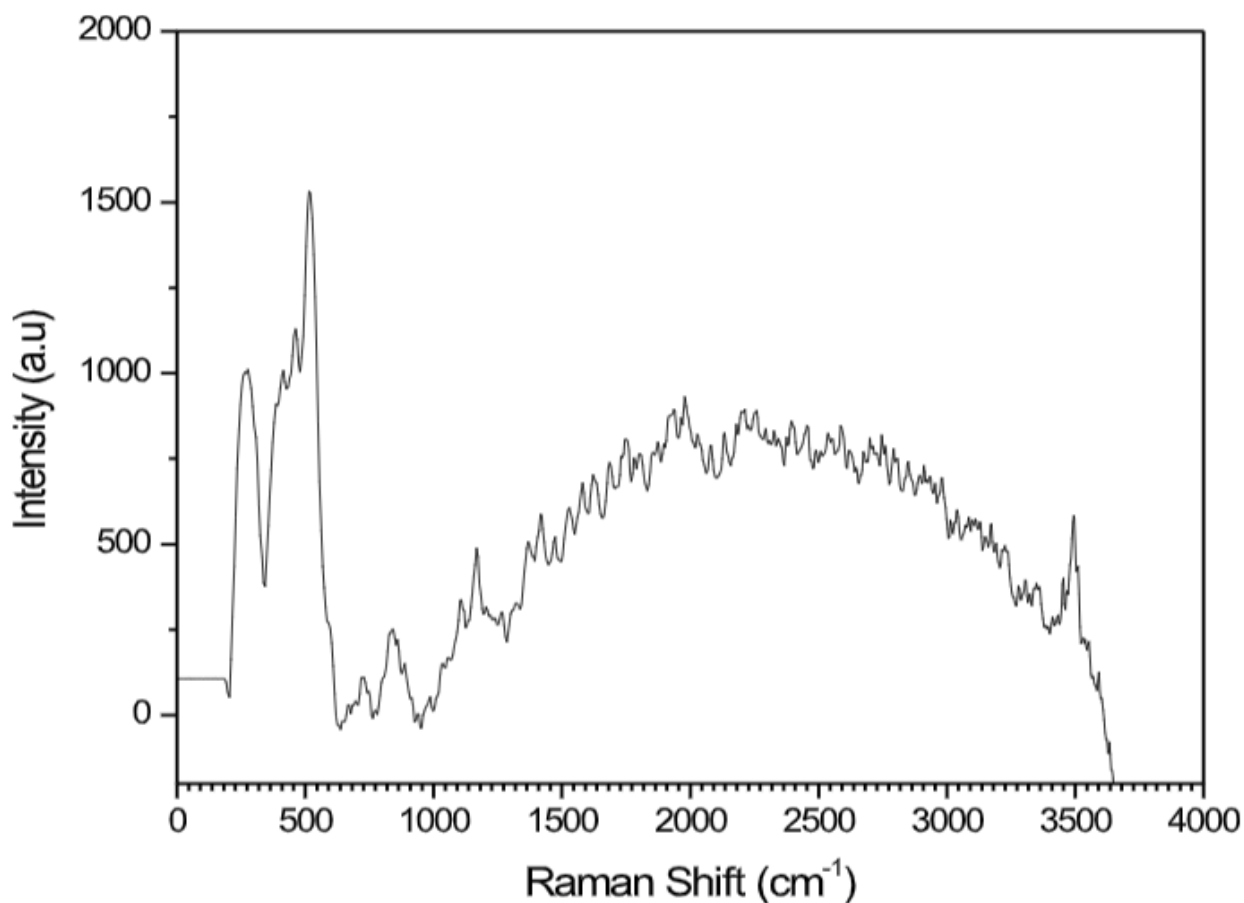


Fig:5.2. Spectral data of Raman Spectroscopy

5.3. Thermo Gravimetric Analysis

The peaks at 1744cm⁻¹ denotes stretching mode of C=O in amino acids. The peak 1160cm⁻¹ denotes C-O stretching vibration. The peak 722 cm⁻¹ denotes methylene group of CH₂ rocking vibration. These are the compound present in the 71 given oil. X-axis denotes the wavenumber (cm⁻¹) and Y-axis denotes the absorbance unit.

Sample: SWETHA / NSP-2
Size: 4.9400 mg
Method: Ramp
Comment: CHY, SATHYABAMA INST, N2 ATM

TGA

File: C:\220215-TGA-N2-SWETHA-NSP-2.001
Operator: TA
Run Date: 15-Feb-2022 12:59
Instrument: TGA Q500 V20.10 Build 36

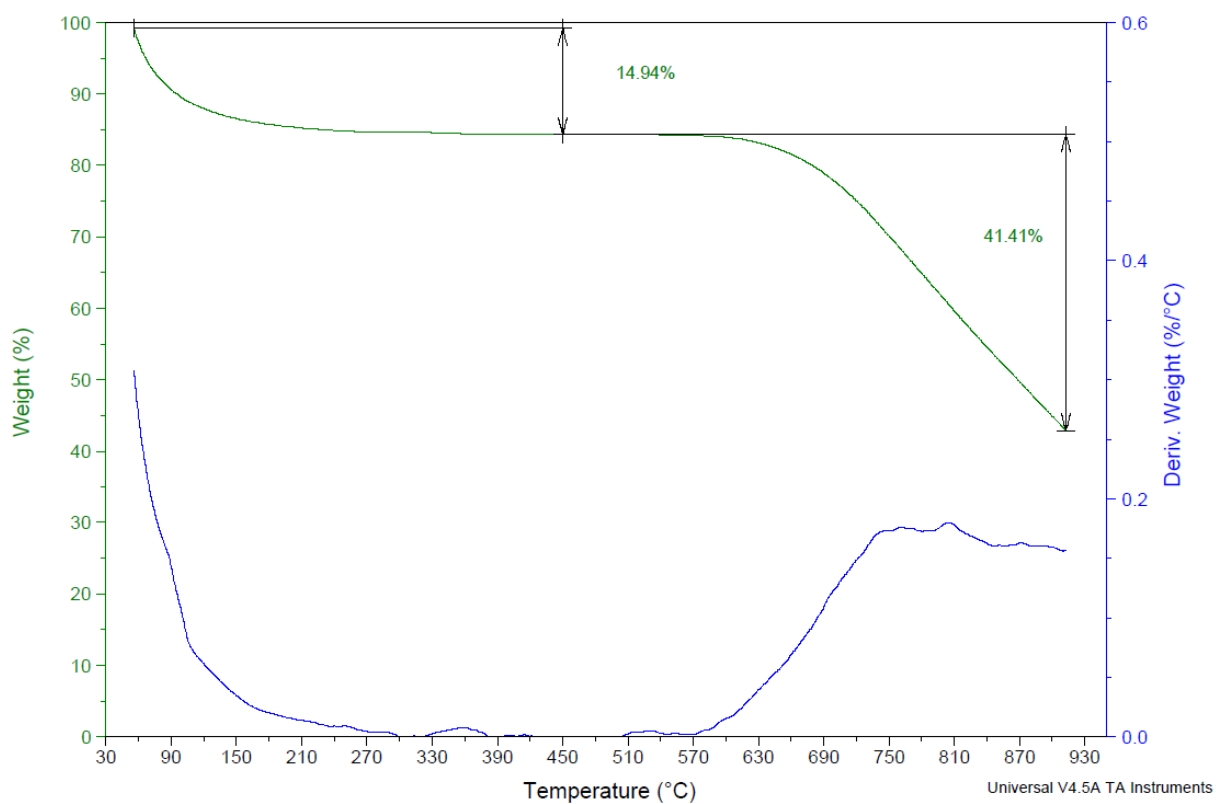


Fig:5.3. Spectral data of TGA

CHAPTER 6

CONCLUSION

- Polybenzimidazoles (PBIs) and benzimidazole-connected polymers (BILPs) highlight incredibly high warm soundness and magnificent synthetic protection from be utilized under unforgiving circumstances.
- Viable conventions, for example, projecting, sol-gel process, stage reversal, turning (utilizing stage reversal) and interfacial polymerization, were created for manufacturing films, along with or followed by medicines including adjustment, cross-connecting, corrosive doping, polymer-mixing and intensity therapy.
- The PBI based films have been broadly read up for gas division (additionally BILP layers), natural dissolvable nanofiltration, water treatment, pervaporation and high-temperature proton trade.
- The exhibitions of some PBI films are practically identical with or surpass benchmark ones.
- PBI based layers for H₂ partition and proton trade at raised temperatures for sanitizing and changing clean energy, where conventional films were not qualifiedBe that as it may, the business PBI layers are as yet utilized in a limited scale.
- Reproducibility, cost, execution and mechanical strength are the potential viewpoints requiring further endeavors
- . The presence of BILPs will positively rejuvenate the exploration of PBI layers toward gas division, evaporation and natural dissolvable nanofiltration.
- Presumably, the practicality of IP for handling these insoluble polymers will save the expense on the grounds that fundamentally less solvents and monomers will be utilized in film creation, advancing the improvement of green compound designing.
- Also, screening more feasible monomers, cross-linkers and solvents ought to be continued.

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