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STUDY ON POLYMER CHARACTERIZATION

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ABSTRACT

In this review article, it contains information about the definition of polymer, types of polymer based on repetition or variety of monomers and how they are linked or joined and on the arrangements of the different chains that forms the polymer and on chemical classification. It also contains information about the polymer characterization techniques such as small angle x-ray scattering, nuclear magnetic resonance, x-ray diffraction, thermal characterization such as differential scanning calorimetry and thermogravimetric analysis, mechanical characterization such as tensile strength test and dynamic mechanical analysis, microscopic methods such as scanning electron microscopy and transmission electron microscopy.

KEYWORDS: Calorimetry and thermogravimetric.

INTRODUCTION

Polymer characterization is the analytical branch of polymer science. The characterization has a goal to improve the performance of a material. It should be ideal to the desirable properties of the material such as strength, impermeability, thermal stability and optical properties.[1]

Characterization techniques are typically used to determine the molecular mass, structure and its morphology, thermal and mechanical properties.

Molecular structure includes techniques such as UV spectroscopy, IR spectroscopy, Raman spectroscopy, Nuclear magnetic resonance spectroscopy, x-ray diffraction, mass spectrometry are used to identify the common functional groups in an unknown organic compounds.

Morphology includes microscopic techniques such as transmission electron microscopy, scanning electron microscopy, scanning transmission electron microscopy, atomic force microscopy is useful to know whether the polymer is crystalline or amorphous. Thermal properties such as thermogravimetric analysis, Differential thermal analysis, Differential scanning calorimeter are used to measure the crystallinity for semi crystalline polymers. Mechanical characterization involves tensile strength test and dynamic mechanical analysis to measure the strength, elasticity, visco elasticity, anisotropy of a polymer. Molecular weight can be determined by using Awagadro's number.

POLYMER

It is derived from greek word ["polus" meaning many, much and "meros" meaning parts]. It is a substance which has a molecular structure built up chiefly or completely from a large number of similar units bonded together. Some polymers exists naturally and others are produced in laboratories and factories.

These are studied in the fields of biophysics and macromolecular science and polymer science.[3]

TYPES OF POLYMERS

Depending on the repetition or variety of monomers, polymers are classified as

a) HOMOPOLYMERS: It is called when the polymer formed by the same monomer throughout its chain.

b) COPOLYMER: It is called when the polymer formed by at least 2 different monomers along the entire chain.

Depending on how they are linked (or) joined and on the arrangements of the different chains that forms the polymer. The resulting polymeric materials can be classified as:

a) Thermoplastics

Thermoplastic polymers are long-chain polymers in which inter-molecules forces (Van der Waal's forces) hold the polymer chains together. These polymers when heated are softened (thick fluid like) and hardened when they are allowed to cool down, forming a hard mass. They do not contain any cross bond and can easily be shaped by heating and using moulds. A common example is Polystyrene or PVC (which is used in making pipes).

b) Elastomers

Elastomers are rubber-like solid polymers, that are elastic in nature. When we say elastic, we basically mean that the polymer can be easily stretched by applying a little force. The most common example of this can be seen in rubber bands(or hair bands). Applying a little stress elongates the band. The polymer chains are held by the weakest intermolecular forces, hence allowing the polymer to be stretched. But as you notice removing that stress also results in the rubber band taking up its original form. This happens as we introduce crosslinks between the polymer chains which help it in retracting to its original position, and taking its original form. Our car tyres are made of Vulcanized rubber. This is when we introduce sulphur to cross bond the polymer chains.

c) Thermosets

Thermosetting plastics are polymers which are semi-fluid in nature with low molecular masses. When heated, they start cross-linking between polymer chairs, hence becoming hard and infusible. They form a three-dimensional structure on the application of heat. This reaction is irreversible in nature. The most common example of a thermosetting polymer is that of Bakelite, which is used in making electrical insulation.

Depending on chemical classification polymers can be

a) Inorganic
b) Organic.[4]

CHARACTERIZATION OF POLYMERS

The structural characterization to determine the conformation of polymers such as polysaccharides is based on understanding the characteristic energy release for specific types of linkages which is measured by the angle of rotation about the linkage. It is particularly effective for compounds such as polysaccharides and oligosaccharides. A random conformation is also assumed if a polysaccharides shows independent rotations at each monosaccharide linkage it also helps to determined the crystalline or amorphous nature of a polymer.[5]

Polymer characterization is the analytical branch of polymer science. Molecular characterization uses common method to evaluate the polymer solutions. The chemical structure of the polymer materials is determined by the spectroscopic methods such as UV visible absorption spectroscopy, IR spectroscopy, Nuclear Magnetic resonance (NMR) and

Mass spectroscopy polymer characterization is useful to give detail information about structure and properties of polymer. Polymer molecular weight is necessary to determine physical properties.[6]

Polymer characterization spans many techniques for determining the chemical composition, molecular weight distribution, and physical properties. The common techniques include the following:

- Size-exclusion chromatography(also called gel permeation chromatography), sometimes coupled with static light scattering, can used to determine the number-average molecular weight, weight- average molecular weight and dispersity.
- Scattering techniques, such as static light scattering and small-angle neutron-scattering, are used to determine the dimensions (radius of gyration) of macro molecules in solution or in the melt. These techniques are also used to characterize the three-dimensional structure of micro phase-separated block polymers, polymeric micelles, and other materials wide angle x-ray scattering (also called wide angle x-ray diffraction) is used to determine the crystalline structure of polymers (or lack thereof).
- Spectroscopy techniques, including Fourier-Transform Infrared Spectroscopy [FTIR], Raman Spectroscopy and Nuclear Magnetic Resonance Spectroscopy [NMR], can be used to determine the chemical composition of a polymer.
- Differential scanning calorimetry is used to characterize the thermal properties of polymers, such as the glass transition temperature, crystallization temperature and melting temperature. The glass transition temperature can also be determined by dynamic mechanical analysis.
- Thermogravimetry is useful technique to evaluate the thermal stability of polymer.
- Rheology (viscosity) is used to characterize the flow and deformation behavior. It can be used to determine the viscosity, modules and other rheological properties. Rheology is also often used to determine the molecular architecture (molecular weight, molecular weight distribution, branching) and to understand how the polymer can be processed.[6]

Small angle x-ray scattering [SAXS] is an x-ray based method that is characterized by a small angle and can produce rapid analysis of polymers such as protiens and polysaccharides in solution. It is based on the principles of reciprocal law which relates the distance [r] in a real space with the scattering vector [q] in a scattering space also known as the fourier space.

Small angle x-ray scattering provides rapid but low resolution structural characterization of polymers it is also used in combination of other methods.

Nuclear Magnetic Resonance [NMR] is the assignment of specific protons and carbons to specific linkages and determination of conformation of their linkages provides more detail information about a material. It is a non invasive spectroscopic method used for the structural analysis and conformational analysis of polymers. It consists of a magnet, a radio frequency transmitter [oscillator] and radiofrequency detector. A sample placed between magnets is subjected to a radio frequency (RF) known as frequency. The material absorbs the radio frequency (RF) and detector picks up the absorption of the radio frequency (RF) at a particular frequency and the magnetic field strength. The absorption of radio frequency (RF) is called resonance. This is used to obtain data on the conformation, stereo-regularity, primary and secondary structure of proteins, polysaccharides and synthetic polymers in liquid, solid and gel forms. When compared to other methods such as x-ray scattering, Nuclear magnetic resonance has a better sensitivity to microscopic structure within a short range order. It does not accurately determine the special portion of atomic groups. It takes the considerable amount of time to run compared to other more rapid methods such as small angle x-ray scattering. Nuclear magnetic resonance (NMR) is used for determining relaxation times and primary structures of carbohydrates and sugars in solution, solid state high resolution. It is applied to determine the structure of polymers in viscous solution, gel and solid forms while 2-D and 3-D. Nuclear magnetic resonance technique provide information on the primary and secondary structures and conformation of polymers. It depends on the nature of polymer to be analyzed and the information required. This technique is based on the chemical shifts and relaxation times recorded from a Nuclear Magnetic Resonance [NMR] spectrometer.

X-ray diffraction is the extent of crystallinity, crystalline microstructure occurrence of amorphous structures and the phases present in a polymeric material can be determined using x-ray diffraction. Based in the diffraction and the interference on x-ray beams as they leave a crystal. It also provide other information about a material such as orientation of the filler within the polymer or the orientation of the polymer itself. The crystallinity of the polymer sample can also be measured in-situ while the process is ongoing which can allow for controlling parameters during processing.

Thermal characterization of polymers to determine the behavior of the polymer under different temperature conditions is usually carried out using Differential scanning calorimetry (DSC) and thermo gravimetric analysis (TGA) to measure the crystallinity for semi crystalline polymers. Thermo gravimetric analysis involves the monitoring the changes in the mass of a substance with respect to temperature over a given time under controlled atmospheric conditions. The change in mass over the duration of heat is indicative of the degradation property of the material at different temperature.

Mechanical characterization involves tensile strength test and dynamic mechanical analysis to measure the strength, elasticity, visco elasticity and anisotropy of a polymer. The tensile test is most commonly applied to polymer materials to establish the amount of work input required to cause the material to yield or fail. Property such as stress at break, elongation at break, young's modulus and work of failure can be obtained from a static strength test on a polymer material. The standard procedure for natural polymer materials is the same for other synthetic materials.

Dynamic mechanical analysis refers to the study of materials behavior under sinusoidal applied force at a frequency of Hz and an angular frequency of ω. A lag phase δ and usually exists between the stress and strain of a visco elastic body.

Most commonly used microscopy method for characterization of natural polymers is scanning electron microscopy and transmission electron microscopy. Scanning electron microscope is often used to obtain microphotographs of composites in the micro nanoscale in order to study the morphology of material. Scanning electron microscope gives good information about the dispersion of the fiber within the composite and compatibility between the polymers used as fillers, fibers or matrix in films and composites.

Transmission electron microscopy used for the analysis of polymer morphology Fourier Transform Infra-red (FTIR) spectrometry is commonly used to analyze polymers with the aim of identifying the chemical bonds which exists within a sample. It is also a measure of compatibility with in the polymers. It is used to determine the chemical components of a sample.[7]

Small molecules and many bio polymers are mono disperse all molecules of a given pure compound have the same molecular weight and which can be calculated by using Awagadro's number. The macromolecules are hetero disperse that is they have different chain lengths and a range of molecular weight and its distribution.

Molecular weight can be determined from a solution viscosity. The reduced viscosity of a polymer is proportional to its molecular weight. Reduced viscosity is specific viscosity divided by concentration. The reduced viscosity is replaced by the intrinsic viscosity or limiting viscosity number which is the reduced viscosity extrapolated to infinite dilution. The viscosity measurements do not afford absolute molecular weight determinations like osmometry, light scattering or ultra centrifugation. Because of the simplicity of the equipment, speed and accuracy of the measurements viscosity determination in the method most widely and routinely used to determine polymer molecular weight.

Preparing of polymer solutions is to caused dispersal of the powders and avoid lumping, the initial solvent temperatures should be conductive to limited wetting and poor dissolution. Water soluble polymers such as poly vinyl alcohol and sodium carboxy methyl cellulose are more soluble in hot than in cold water. Some polymers are used in mixed solvents.[8]

Polymer characterization studies mainly concentrate on the molecular structure and morphology of polymer. The thermodynamic and mechanical property depends upon their chemical or biological composition. Gravimetric methods can also be used to measure vapor solid interactions for polymer characterization. Organic solvents also interacting with polymer permeating the molar structure and solvating it to degradative products mechanical stability.[9]

Thermal analysis and physical property testing are placed an important role to understanding polymers and their characterization. To characterize the thermal property of polymer including the glass transition, melt temperature, heat of fusion, heat capacity, weight loss, thermal stability. Impact analytical provides: thermal testing-differential scanning calorimetry for such information as glass transition, melt temperature, oxidative induction time and thermogravimetric analysis for weight loss, thermal stability inorganic content. Physical testing – density and hardness. Rheological testing – capillary and rotational. Optical testing – color, haze and gloss.[10]

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Differential Scanning Calorimetry(DSC) and Differential Thermal Analysis (DTA), Thermo Gravimetric Analysis (TGA), Thermo Gravimetry (TG), Thermo Mechanical Analysis (TMA), Thermal Optical Analysis (TOA) are used to determine the nature of the polymers. Non equilibrium nature of the polymers can be determined by the melting and crystallization behavior of flexible chain polymer, glass transition of amorphous and phase transition in liquid crystalline polymers. Thermal analysis gives information about the morphology of crystalline polymers.[12] Differential scanning calorimetry and differential thermal analysis are useful for the determination of the physical and chemical properties of the polymers. the techniques used to elucidate the endothermic and exothermic processes at temperatures ranging from very low to high Differential scanning calorimetry (600 degree centigrade) and Differential thermal analysis (1800 degree centigrade). Physical properties of the polymer can be measured by thermal analytical methods such as melting transitions, vaporization, sublimation, solid-solid transitions, thermal conductivity and the glass transition temperature. Chemical polymers can be determined by chemisorptions, solid state reactions.[26] Thermal analysis is the important tool for determining the polymer by using the mechanism of improvement of thermal properties such as Differential Scanning Calorimetry (DSC) Modulated Temperature Differential scanning calorimetry (MT-DSC) Dynamic Mechanical Thermal Analysis (DMA), Thermal Mechanical Analysis (TMA), Thermo Gravimetry (TG).[31] Differential scanning calorimetry and differential thermal analysis or analytical tools to characterize melting crystallization and mesomorphic transitions and to determine the enthalpy and entropy changes of a polymer. The advantage of differential scanning calorimetry and differential thermal analysis when compared to other calorimetric methods are constant heating and cooling rates.[33]

Higher spectroscopy is one of the method is useful for the characterization and to study the chemical and physical structure of compounds. It also provides qualitative and quantitative information about the chemical and physical structure of polymer.

1. Chemical nature of polymers: type and degree of branchings nature of end groups, impurities.
2. Stearic order: Cis–Trans isomerism, stereo regularity.
3. Conformational order: physical arrangement of polymer chain, planar zigzag conformation, helix conformation.
4. Crystallinity: number of chains per unit cell, intermolecular forces.[13]

Vibrational spectroscopy consists of two techniques IR absorption and Raman scattering is useful for the determining the poly atomic molecules. IR spectroscopy is used to identify polymers and to determine crystallinity, conformation, end groups, branching, cross linking, hydrogen bonding and fold structure of polymers and composition and sequence distribution of copolymers.[14] Vibrational spectroscopy is used for the characterization of polymer structure. It gives information about the molecular and crystalline structure of high polymers.[20]

Mass spectrometry is used to measure molecular masses of the polymer. [MALDI] Matrix – Assisted Layer Desorption or Ionisation and Electron Spray Ionisation is useful for the characterization of synthetic polymers. Electron Spray Ionisation is a method for transforming ions that once present in solutions the gas phase for mass spectral analysis.[15]

Fourier transform infrared is used to study both the chemical composition and morphology of optically thick polymer samples. It is a convenient, non contact, non destructive method of characterizing both qualitatively and quantitatively the chemical and physical properties of thick polymer samples is obtaining structural and morphological data on polymeric materials.[16] The advantages of Fourier Transform Infra - red spectroscopy (FTIR) over conventional dispersive instrument such as increased signal to noise ratio, higher energy to put is for the polymers.[34] IR spectroscopy of polymers depends on a change in permanent dipole moment of the polymer where as Raman spectroscopy depends on the change in the induced dipole moment or polarization to produce Raman scattering. Hence these two vibrational techniques are used to study the polymers.[35]

Solid state characterization is useful to understand the properties of formulation and formulation components. Analytical techniques are useful to measure the responses to perturbation of the objects under examination. The perturbation may be of thermal, mechanical, light, radio frequencies electro magnetic forces or a combination thereof.[17]

To study the actual behavior of a polymer during its transformation the thermal dependence of the thermo physical properties is necessary. In semi crystalline polymers the crystallization phenomenon plays an important role. Thermal characterization of semi crystalline powders can be performed by thermal conductivity which can be measured by using hot wire method. The physical structure of the polymer is responsible for the thermal and mechanical characteristic. Differential scanning calorimetry (DSC) was used to measure the enthalpy changes in a sample.[18]

The use of **photon calibration spectroscopy** is to study the dynamic concentration and fluctuations in polymer solutions. The theory of dynamic light scattering from pure liquids can be applied to polymers density and optical anisotropy of polymers can be studied by photon correlation spectroscopy.[19]

Wide angle x-ray scattering and small angle x-ray scattering and small angle neutron scattering are used to determine crystalline structure of polymer. Gel permeation chromatography is used to average molecular weight and poly dispersity, Fourier transformed infrared spectroscopy (FTIR), Raman and Nuclear magnetic resonance (NMR) can be used to determine composition. Thermal properties such as the glass transition temperature and melting point can be determined by differential scanning calorimetry and dynamic mechanical analysis. Thermo gravimetry is a technique to determine the thermal stability of a polymer. Rheological properties are used to determine the molecular architecture (molecular weight, molecular weight distribution and branching).[21]

Atomic force microscopy can also be done for that polymers to characterize whether they are macro molecules or micro molecules and to study the lamellar and microphage morphology. It is also useful for the application of Kelvin Force Microscopy (KFM) dC/dZ are useful for the heterogeneous polymers in which polar components are present.[22]

IR spectroscopy is used in the analysis and characterization of polymers. Polymer molecules varies in composition and configuration and other components. The benefit of the resolving

polymer discrete identifiable entities by combining chromatographic separation. It is also useful to determine the mass distribution of discrete components across the chromatographic distribution of sample.[23]

The propagation of low intensity ultra sound in polymers acting as a high frequency dynamic mechanical deformation to monitor the changes in the modulus of polymers which are associated with glass transition, crystallization etc., Ultrasonic Dynamic Mechanical Analysis(UDMA) is useful to evaluate visco elastic moduli of the polymers as a function of time or temperature.[24]

Mass (molecular weight) is one of the property of the polymer. It may be polydispersity or mono dispersity. Monodispersity is the rare event of the polymer having about the same molecular weight. Size (radius of gyration) is the another property of the polymer which can be determined by the microscopic techniques.[27]

Spectroscopy techniques are used to obtain information whether there is any physic chemical change in the polymer. It is a non destructive techniques and provide information at a molecular level electron spectroscopy for chemical analysis is used to analyze surface properties of a polymer which a new technique.[28]

Polymer characterization studies mainly concentrate on the molecular structure and morphology of polymer. The thermodynamic and mechanical properties depends upon their chemical or biological composition. Gravimetric methods can also be used to measure vapor solid interactions for polymer characterization. Organic solvents also interacting with polymer permeating the molar structure and solvating it to degradative products mechanical stability.[29]

A review of recent trends in polymer characterization using non destructive vibrational spectroscopic models and chemical imaging. Vibrational spectroscopy and chemical imaging are used to characterize polymers in diverse forms, their behavior and transient phenomenon.[30]

Solid dispersion is molecular dispersion of drug in polymer matrix which leads to improved stability and hence better availability. The formulations where characterized by advanced techniques like optical microscopy, differential scanning calorimetry hot stage microscopy,

dynamic vapor sorption and x – ray diffraction. Amorphization of drug increases the stability of drug because of increased surface area and better ability of the solvent to wet the drug.[32]

CONCLUSION

Polymers play a vital role in the drug delivery. So, the selection of polymer plays an important role in drug manufacturing. The best solution calls for meaningful characterization, based on the selection, from all possible molecular and physical parameters, of those whose determination will insure the desired performance within the limits of current knowledge. We explained all possible parameters of polymer characterization.

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