Study of Thermal Decomposition and Instrumental Analysis of Synthesised *Polyvinyl Alcohol Polymer*

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Abstract

Polyvinyl alcohol has various applications in the food industries as a binding and coating agent. It is a film coating agent specially in applications where moisture barrier protection properties are required. Polyvinyl alcohol protects the active ingredients from moisture, oxygen and other environmental components, while simultaneously masking their taste and odor. The obtained films were transparent and homogenous with enhanced mechanical properties and thermal stability[1-4]. In recent years water-soluble polymers are gaining more and more interest in different applications. One of the most widely used water-soluble polymers is polyvinyl alcohol polymer (PVA). For more than 70 years it is applied in a variety of applications in medicine, pharmacy, cosmetics and industrial production[5-10]. PVA binds to polar molecules exceptionally well.

The physico-chemical characterization and compatibility of Water-soluble synthetic polymers were analyzed by means of FT-IR spectroscopy, TGA, HPLC and UV visible spectrophotometer.

Key words: polyvinyl alcohol polymer, water-soluble, FT-IR spectroscopy, TGA, HPLC.

Introduction

Polyvinyl alcohol is an efficient binder for solid pigments, ceramic products, plastic, cement, fibers, non-woven fabrics, catalyst pellets, cork compositions etc. Also, PVA has gained increasing attention in the biomedical field due to its bioinertness[11-16]. The word polymer designates an unspecified number of monomer units. When the number of monomers is very large, the compound is sometimes called a high polymer. Polymers are not restricted to monomers of the same chemical composition or molecular weight and

structure. Some natural polymers are composed of one kind of monomer. Most natural and synthetic polymers, however, are made up of two or more different types of monomers. Such polymers are known as copolymers. Organic polymers play a crucial role in living things, providing basic structural materials and participating in vital life processes. Synthetic polymers are produced in different types of reactions. Many simple hydrocarbons, such as ethylene and propylene, can be transformed into polymers by adding one monomer after another to the growing chain. Analysis of the absorption spectra in the lower energy part gives information about atomic vibrations, while the higher energy part of the spectrum gives knowledge about electronic state in the atoms. The thermogravimetry analysis and differential thermal analyses curves provide some information about the various unexpected observations during heat treatment. The combination of these experimental techniques allows us to gain more complete understanding of the effect of composition on the structure and thermal stability.

In the present work the structural, IR-spectra, HPLC, optical and thermal properties of PVA were studied and discussed.

Experimental

PVA BDH chemical with appropriate weight were dissolved in distillated water, Complete dissolution was obtained using a magnetic stirrer at room temperature for 8hr. The stirred solution was cast on to glass plate for overnight, polymer layers was formed as thin films with a appropriate thickness (0.05 mm). Chromatographic analysis were carried out using UFLC SHIMADZU HPLC column C18, to study retention time and purity, The infra red spectral analysis was carried out using BRUKER FTIR spectrophotometer over the range 400 to 4000 cm^{-1}., The UV/VIS absorption spectra were measured in the wavelength 200 to 800 nm using PERKIN ELMER, LAMBDA 35- double beam spectrophotometer and The thermal analysis (TGA) was carried out using PERKIN ELMIER 4000. The heating rate used for samples under investigation was 10^0C/min from room temperature to 400^0C under nitrogen atmosphere with flow of 30 ml/min.

Result and Discussion

High Performance Liquid Chromatography:

HPLC is a separation technique that involves, injection of a small volume of liquid sample into a column packed with tiny particles (3 to 5 micron (μm) in diameter called the stationary phase where individual components of the sample are moved down through column with a liquid mobile phase, forced through the column by high pressure delivered by a pump. The speed, efficiency, sensitivity and ease of operation of HPLC is vary superior. The components are separated from one another by the column packing that involves various chemical and physical interactions between their molecules and the packing particles. These separated components are detected at the exit of this column by a flow-through device detector that measures their amount. An output from this detector is called a "liquid chroma-togram". Solvent system is acetonitrile water (60:40), Column-C18. The sample is dissolved in HPLC grade water and 5 micron solution was injected with the help of syringe through injector. The peak at 2.840 (Fig. 1) shows retention.
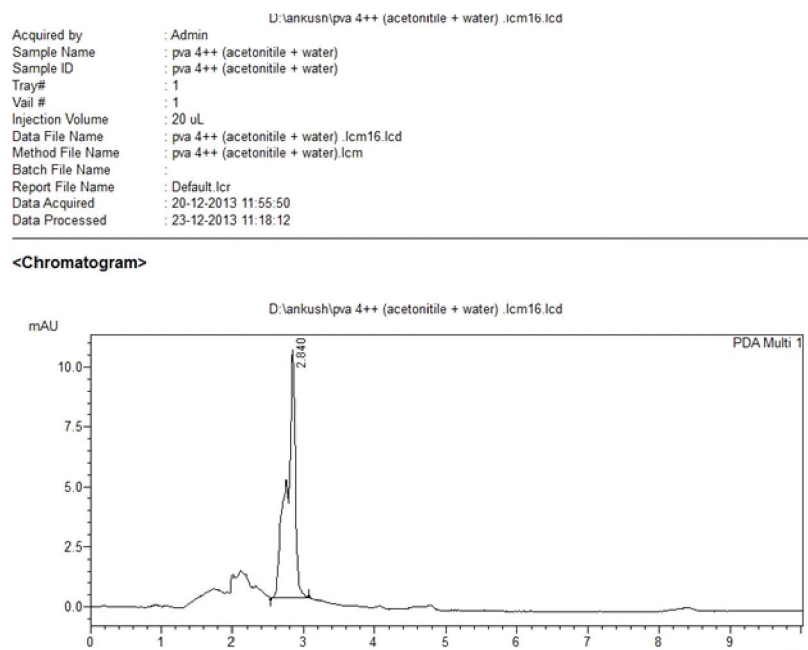
D:\ankush\pva 4++ (acetonitile + water) .lcm16.lcd

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Acquired by        : Admin
Sample Name        : pva 4++ (acetonitile + water)
Sample ID          : pva 4++ (acetonitile + water)
Tray#              : 1
Vail #             : 1
Injection Volume   : 20 uL
Data File Name     : pva 4++ (acetonitile + water) .lcm16.lcd
Method File Name   : pva 4++ (acetonitile + water).lcm
Batch File Name    :
Report File Name   : Default.lcr
Data Acquired      : 20-12-2013 11:55:50
Data Processed     : 23-12-2013 11:18:12
```

<Chromatogram>

Fig. 1 Retention time peak of PVA polymer

Impurity : Not Detected.
Peak purity index : 0.999995
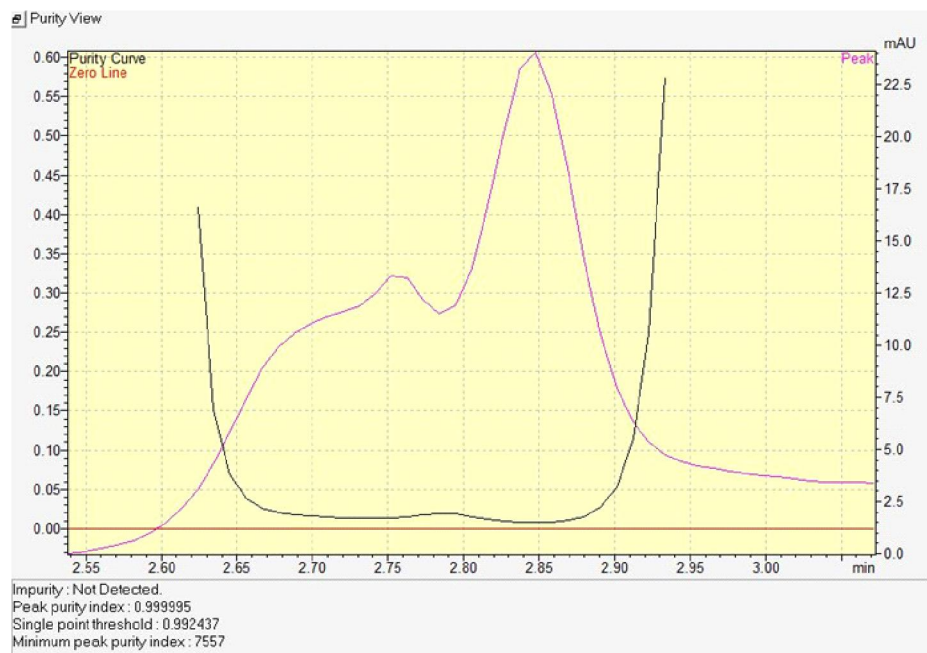Single point threshold : 0.992437
Minimum peak purity index : 7557

Fig. 2 PVA polymer Purity graph

Time of polymer and (Fig 2) shows purity index 0.999995 of the sample.

FTIR

When the atoms interact with the IR light the radiation creates the Stretching and Bending vibration in the atoms of molecule. This stretching and vibration modes of every atoms is unique for all the functional groups of organic compounds.
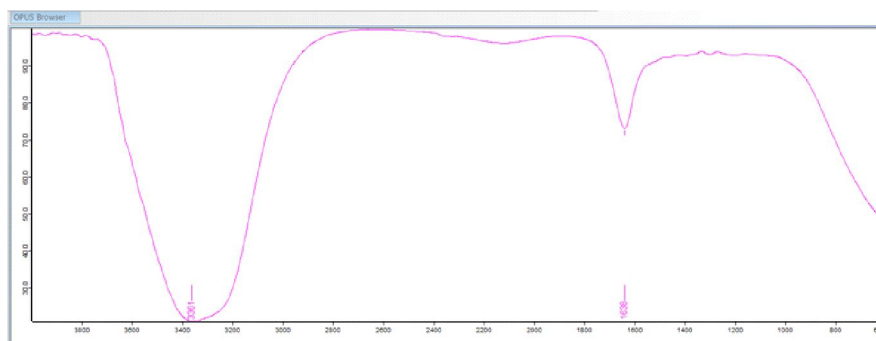

Fig. 3 IR spectra of PVA polymer

Broad Peak at 3361 cm⁻ (Fig. 3) shows exchangeable Hydrogen present in –OH group of Polyvinyl alcohol and second peak at 1638 cm^{-1} is because of H-O-H bonding present in sample, this peak indicates the presence of water molecule in the polymer.

UV- visible spectra :

When sample is placed for analysis the light passes from the sample and some light gets absorbed, while some of it is reflected, the absorbed light intensity is directly proportional to the solution present in the sample. The study of optical absorption, particularly the absorption edge has proved to be very useful for elucidation of the optical properties and optical constants of crystalline and non-crystalline materials. Fig. 4 shows the absorption spectra of PVA in the range between 200 and 800 nm. The spectra of pure PVA at 200.12 nm, which may be due to electronic transition of n-π^* of lone pairs of oxygen atom PVA molecule has large crust near to 200.12nm may be because of remaining bonding electrons of the polymer.

Thermogravimetric analysis :

In thermo gravimetric analysis substance is decomposed in the presence of heat which causes bonds within the molecule to be broken. TGA plays an important role in determining thermal stability of the material. TGA curves and their derivatives of PVA Fig. 5, show that the thermal stability of the samples lies in between those of the two individual homopolymers and the degradation is completed in three steps. These steps are distinguishable in the diagram of mass loss (TGA%) during heating as well as –more clearly-in the diagram of derivative mass loss (DTG). The sample shows a small (2.799 %) mass loss for the first decomposition step and more significant (2.53 %) for the second step, and (0.682%) in third step. Sample was taken
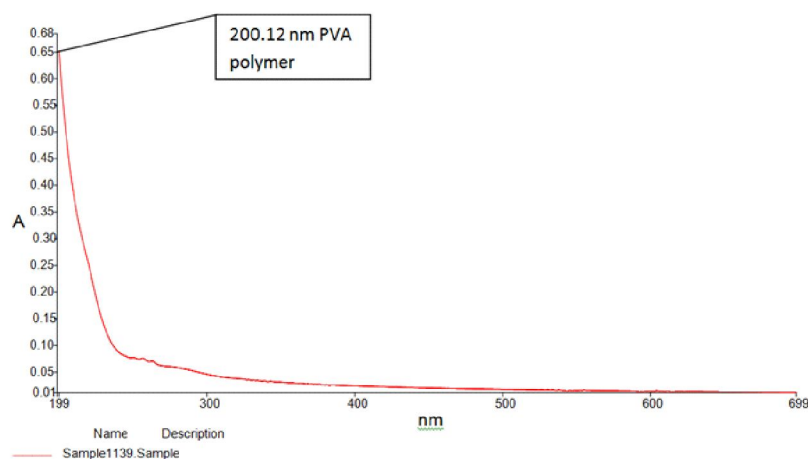
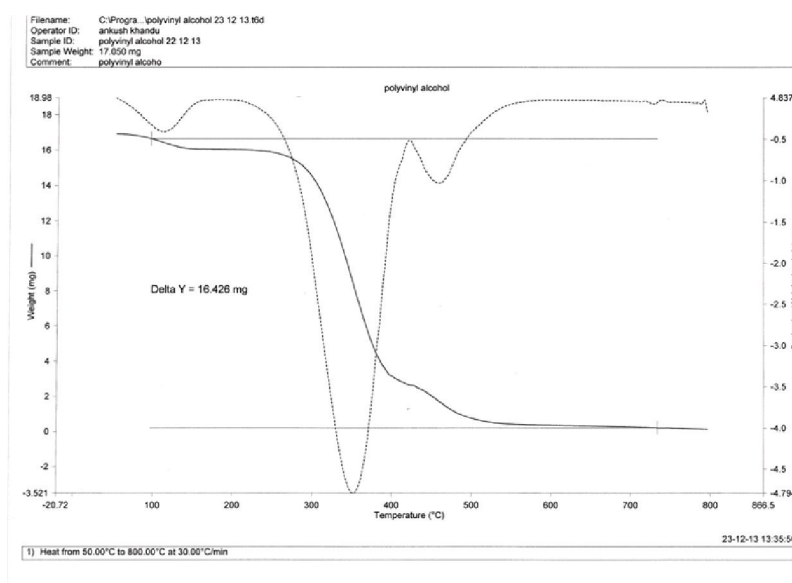Fig. 4 Absorbance spectra of PVA polymer

Fig. 5 TGA of PVA polymer

17.050mg difference in weight was found to be 16.426mg, 14.85 mg and 0.624mg ash was remained in the pane. The polymer is stable upto300ºc it starts degradation at 300⁰c The rate of degradation is highest at temp 350ºc as shown by the graph. The lower values of percentage weight loss in the first decomposition step are attributed to the volatilization of small molecules and\or evaporation of residual absorbed water. The percentage weight loss in the second decomposition step, which included both melting point and the degradation temperature of PVA, is attributed to splitting of monomers and bond scission in

the polymeric backbone. The major percentage weight loss in the third decomposition step is attributed to fragmentation of the macromolecular structure of PVA polymer.

Conclusion

The HPLC retention time 2.8min confirms that the sample synthesized contains the polyvinyl alcohol polymer. The polymer thin films were synthesized economically, conveniently and quickly, The IR Spectra at 1638 and 3361 cm^{-1} shows the spectra of polyvinyl alcohol. The optical absorption studies conforms the formation PVA polymer. The absorption studies have led to a variety of interesting thin film optical phenomena which have thrown considerable light on the band structure of solids and phonic states. The thermal stabilities of samples were confirmed by the corresponding difference in their activation energies. The thermal stability of PVA detected by TGA found to be at 300ºC and the rapid decomposition of polymer occurs at 350ºC.

Acknowledgement

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