iJRASET
International Journal For Research in Applied Science and Engineering Technology



INTERNATIONAL JOURNAL FOR RESEARCH

IN APPLIED SCIENCE & ENGINEERING TECHNOLOGY

Volume: 6 **Issue:** I **Month of publication:** January 2018

DOI: http://doi.org/10.22214/ijraset.2018.1229

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International Journal for Research in Applied Science & Engineering Technology (IJRASET)
ISSN: 2321-9653; IC Value: 45.98; SJ Impact Factor :6.887
Volume 6 Issue I, January 2018- Available at www.ijraset.com

Synthesis and Characterization of Polyester by Using Starch, Maltose, PEG and Maleic Unhydride as Precursors.

Pankaj Meshram[1]
[1]*Department of chemistry, Shri Lemdeo Patil Mahavidyalaya Mandhal, Affiliated to RTM Nagpur University, Nagpur*

Abstract: Starting with starch, maltose, PEG (polyethylene glycol) and maleic anhydride, complex bio-degradable polymer called polyester is synthesized using HCl as catalyst. The molecular weight of synthesized polymer is found to be 1578 g/mol which determined by average viscosity method. This and other physicochemical properties of this polymer are presented. The ester group in the polymer is analyzed by IR and NMR spectral studies. From HLB ratio, it is assume that the synthesized polymer can be use in detergent formulation, paint, varnishes etc.
Keywords: Synthesis; Polymer; Polyester; Biodegradable; Detergent

I. INTRODUCTION

Useful novel eco-friendly polymers can be synthesized by using white dextrin, sorbitol, maleic anhydride [(1)]&starch, Glycerol, Maleic Anhydride [(2) (3)]with an adjustment in the order of addition, ratio of ingredients and temperature. By this, we can get polymers of desired molecular weight and H.L.B. Ratio. Unique detergent formulations have been prepared using Novel polymer and LABS. Acid slurry has been successively replaced by novel polymer from 1 to 100%. The fifty percent acid Slurry and fifty percentpolymers normally give excellent results. Carbohydrate based polymers have been synthesized dates back to as early as 1930's. Reppe in 1930 synthesized ether from glucose and fructose by alkali catalysed addition to protected sugar to acetylene. Biodegradable polymers (polyester) of low molecular weight, aliphatic esters and their delivery in protein delivery system have synthesized [(4)].These polymers tend to be used for injectable, or implantable, drug delivery systems [(5)]. Carbohydrate based polymers are useful in various field like pharmacological and biomedical application [(6)].There has been a worldwide realization that nature derived monosachharides, disachharides, oligosachharides and polysachharides can provide us a raw material needed for the production of numerous industrial consumer goods [(7)]. Functionalization of polymers has emerged as another important area of polymer science and technology. The esters of carbohydrates are completely soluble in water and found to be suitable in the detergent formulation.The detergents made out of acid slurry cause harm to aquatic flora and fauna. Acid slurry has a petroleum origin. The detergents of petroleum origin are responsible for river foaming and eutrophication. By using biodegradable polymers in detergent formulation the above-mentioned problems of water pollution can be minimized to a greater extent. Earlier work have done on polymer synthesis and its characterization using starch, glycerol and maleic anhydride as precursor [(8)] and its application in detergent formulation [(9)]. The present work encompasses the synthesis of polymer from carbohydrate starch, maltose, PEG and maleic anhydride using water as solvent and HCl as catalyst in order to see its application in detergent formulation.

II. EXPERIMENTAL

A glass reactor fitted with stirrer, heating mantle and condenser has been used in the synthesis of novel polymers. The temperature control of 2^0C can be achieved by using an efficient temperature regulator. A constant water supply through a condenser helps to control reactor temperature. Initially, stoichiometric quantity (Table 1) of starch PEG and maleic anhydride was added in the reactor. Hydrochloric acid was used as a catalyst. Now about 200 ml of water was added so that a free flowing homogenous paste was formed. The temperature was raised slowly and steadily in about 0.5 h to 120^0C. The reaction was continued for 3.5 h till the desired molecular weight was achieved. The consistency of the paste was maintained by adding additional water after 0.5 h. At the end of this period, the reaction was terminated and the prepared polymer was collected in a glass-stoppered bottle with least air gap. The final field of the product was measured. The molecular weight of the polymer was determined by viscosity average method. The acid value and the sap value and other physical constants were determined by standard methods. The polymer formed is believed to be an ester of carbohydrate, which has been corroborated by spectral studies.

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Table1.
Stoichiometric proportion of components in polymer synthesis

Sr. No.	Raw Materials	Concentrations (%)
1	Starch (400g)	30.76
2	Maltose (350g)	26.92
3	PEG (350g)	26.92
4	Maleic unhydride (200g)	15.38
5	Water as solvent	1000 ml (total)

Table 2.
Physico-chemical properties of polymer

Sr. No.	Polymer properties	Observations
1	Acid Value	50.8
2	P^H	2.00
3	Saponification value	154
4	Solid (%)	60
5	Solubility Maltose Starch PEG Maleic anhydride	 Soluble in water Soluble in hot water soluble in water Soluble in hot water
6	Solubility of polymer (g/mol) (i) In water (ii) In xylene (iii) In alcohol (iv) In dil. NaOH (v) In alcohol-water (1:1)	 Soluble Insoluble Insoluble Soluble Partially soluble
7	HLB value	11.95
8	Moleculer weight (g/mol)	1578

III. RESULT AND DISCUSSION

The synthesis of polymer from starch, maltose, PEG and maleic anhydride was performed in the presence of HCl as catalyst at 120^0C. The physiochemical properties and compositions of polymer are shown in Table 1 and table 2 respectively. Figs. 1 and 2 show the NMR and IR spectra, respectively, of the synthesized polymer.

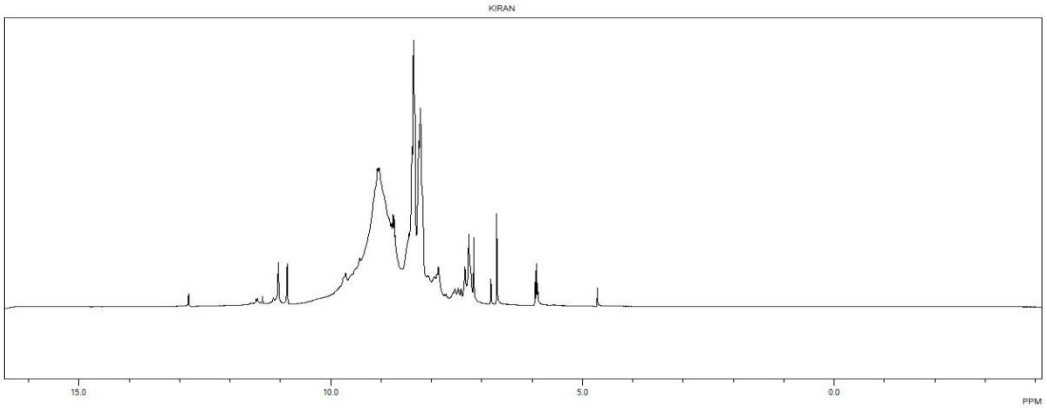


Fig.1 NMR spectra

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The IR spectra show a doublet near 1700 cm^{-1} (1729.80 and 1638.56 cm^{-1}), which is an indication of C=O stretching in the synthesized polymer. The peak at 1249.07 cm^{-1} shows C–O stretching. The IR spectra also show the various peaks between 1350 and 1100 cm^{-1}, which are due to maleic anhydride C–O stretch. This shows that all the molecules of maleic anhydride are not participating in the reaction. The peaks at 1729.80, 1638.56 and 1249.07 cm^{-1} collectively verify the presence of esteric group in the synthesized polymer. This is further verified by the NMR spectra showing peak around 4.50 ppm. The unreacted alcoholic groups are shown by a peak at 3414.83 cm^{-1} in the IR region. The reaction is specific with the reactive alcoholic groups in the glycerol and starch with maleic anhydride. The peak in the region of 4.50 ppm in NMRis an indication of the vinylic form (C=C– H) which is due to the maleic anhydride structure in the compound. The application of the synthesized polymer/resin with respect to detergent formulation is checked by HLB value. The HLB value of 11.95 is indicative of the use of synthesized polymer in detergent formulation and in some cases for paints, inks and emulsions, etc. The polymer is soluble in water and NaOH and insoluble in organic solvent xylene and alc`3ohol and is partially soluble in alcohol-water (1:1) mixture.

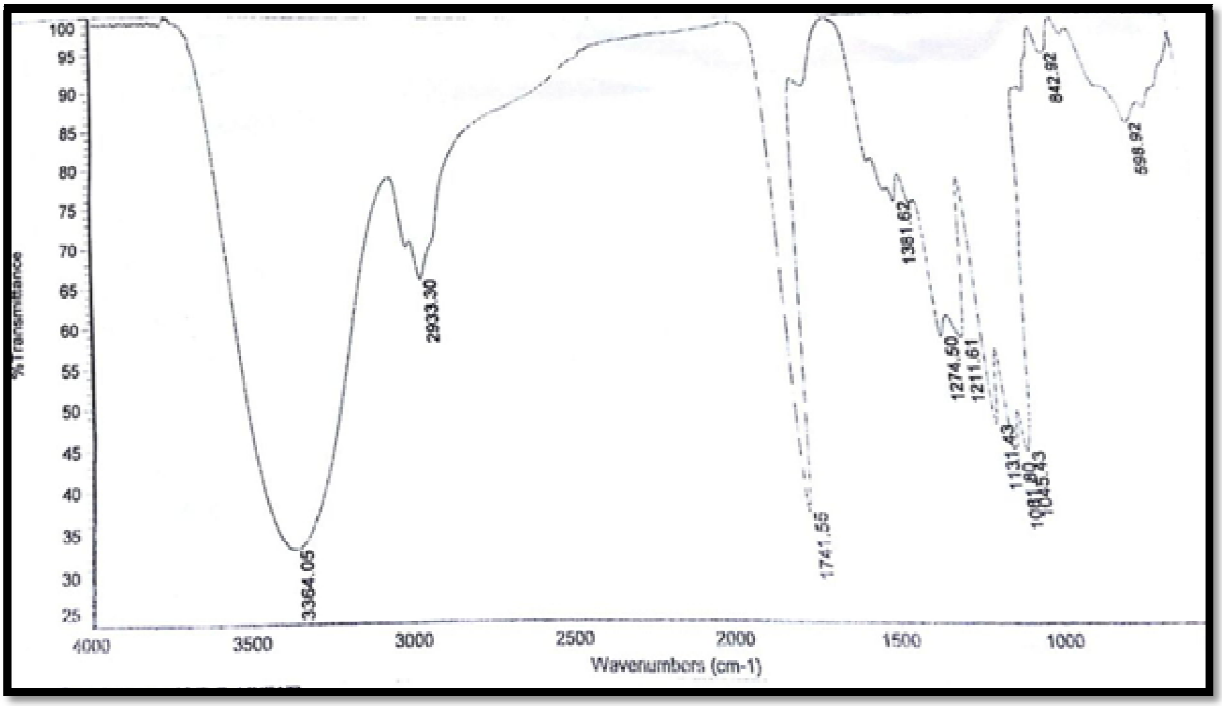


Fig.2 IR Spectra

The IR spectra show a doublet near 1700 cm^{-1} (1729.80 and 1638.56 cm^{-1}), which is an indication of C=O stretching in the synthesized polymer. The peak at 1249.07 cm^{-1} shows C–O stretching. The IR spectra also show the various peaks between 1350 and 1100 cm^{-1}, which are due to maleic anhydride C–O stretch. This shows that all the molecules of maleic anhydride are not participating in the reaction. The peaks at 1729.80, 1638.56 and 1249.07 cm^{-1} collectively verify the presence of esteric group in the synthesized polymer. This is further verified by the NMR spectra showing peak around 4.50 ppm. The unreacted alcoholic groups are shown by a peak at 3414.83 cm^{-1} in the IR region. The reaction is specific with the reactive alcoholic groups in the glycerol and starch with maleic anhydride. The peak in the region of 4.50 ppm in NMR is an indication of the vinylic form (C=C– H) which is due to the maleic anhydride structure in the compound. The application of the synthesized polymer/resin with respect to detergent formulation is checked by HLB value. The HLB value of 11.95 is indicative of the use of synthesized polymer in detergent formulation and in some cases for paints, inks and emulsions, etc. The polymer is soluble in water and NaOH and insoluble in organic solvent xylene and alcohol and is partially soluble in alcohol-water (1:1) mixture.

IV. CONCLUSION

The polymer with MW 1578 g/mol was synthesized by direct condensation using HCl as catalyst. The polymer is as ester based on starch, maltose, PEG with maleic anhydride. The polymer can be used in the detergent formulation, paints, inks, emulsions, etc. The polymer is soluble in water and NaOH.

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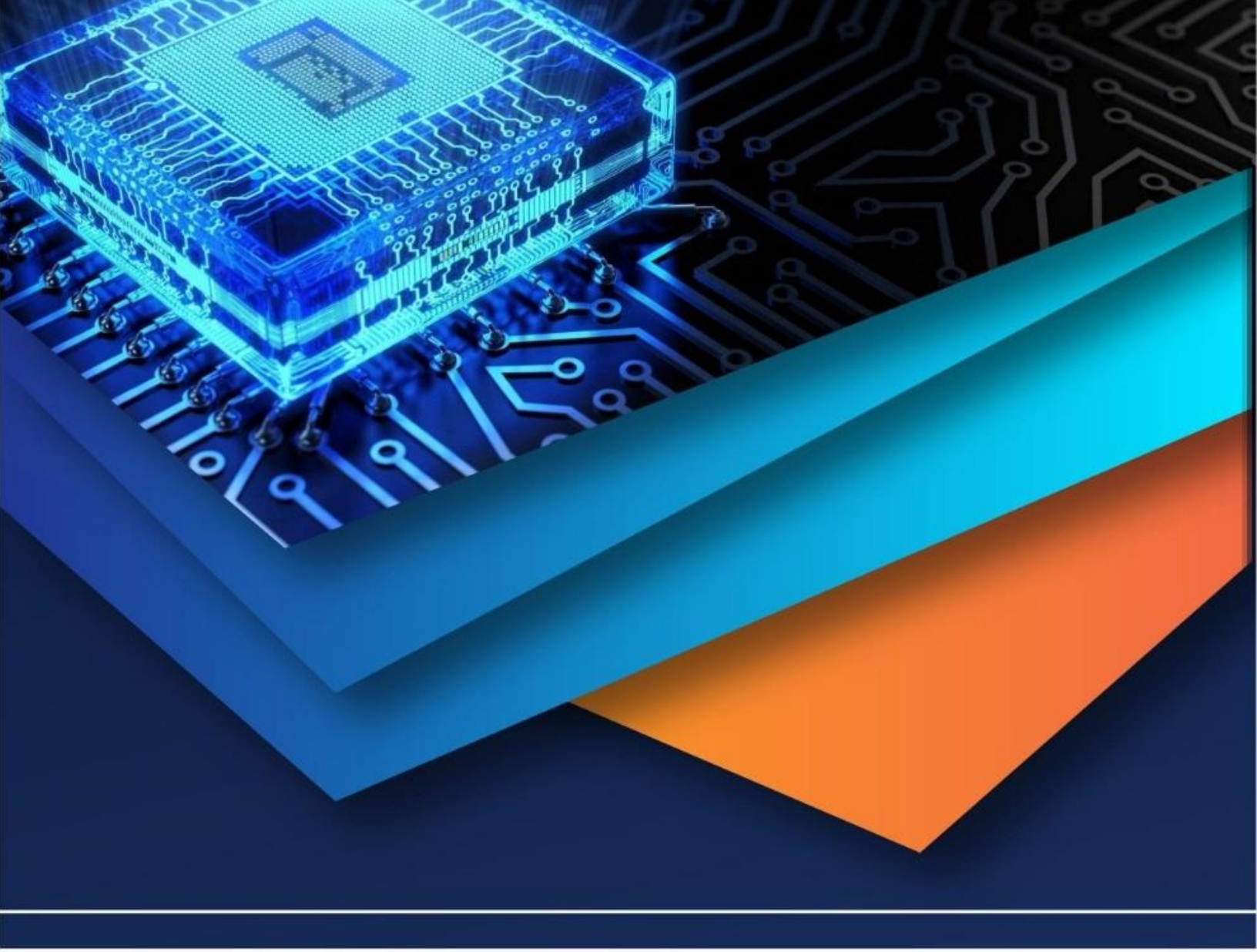
V. ACKNOWLEDGEMENTS

The author is thankful to Director IICT, Hyderabad for recording IR and NMR spectra.

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