Preparation and Characterization of Penta Allyl Sucrose

Patel Zalak J.[1*], Patel Mukesh C.[1], Chatrabhuji Parimal M.[1], Patel Viral A.[2] and Patel Dharmesh R.[2]
1. Department of Chemistry, P. S. Science and H D Patel Arts College, HNG University, Kadi, Gujarat, INDIA
2. Department of Chemistry, HVHP Institute of PG Studies and Research, KSV University, Kadi, INDIA
*zalak8466@gmail.com

Abstract

Allyl sucrose was prepared by condensation reaction among sucrose, allyl chloride, NaOH and DMSO which makes suspension. The employed method is new, faster, consumes less solvent and more convenient to isolate product than the existing methods. There is no need of column chromatography. The yield of synthesized product is 90%.

The prepared product is characterized by various spectral studies such as FT-IR (Fourier-Transform Infrared), NMR (Nuclear Magnetic Resonance) and Mass spectrometry. It is used in different industrial fields.

Keywords: Allyl chloride, allyl sucrose, cross-linking agent, FT-IR, NMR, Mass.

Introduction

Allyl sucrose can be in use as a cross linking agent in synthesis of co-polymers. The various cross linking agents are in work for the copolymerization of poly acrylic acids to optimize the thickening properties of the polymers. Allyl sucrose has become an important thickening agent for the preparation of the variety of polymer applied in many industrial fields, such as coating material, medicines, cosmetics, epoxy resins etc.[1-4] These compounds can be used as film- forming materials/plastic compositions which are on polymerization yield products that are highly resistant to the action of heat, solvents and other reagents. It is also used as an up grader of drying and semidrying oils[5-13]. Various carbohydrates are used in preparation of cross-linkers like starch, cellulose etc. described in various patents[14-18].

The conventional method for the preparation of allyl sucrose is reported by Tang et al[1] in which sucrose is treated with allyl chloride in the presence of caustic soda (NaOH) in medium di methyl sufoxide (DMSO). At the end of reaction there is no need of column chromatographic process to isolate allyl sucrose from the product mixture. However, in the synthesis of allyl sucrose described by Zief and Yanovsky[2], sucrose is reacted with an allyl halide in the presence of excess of a strong alkali. The allyl sucrose is prepared by reacting sucrose, allyl chloride, sodium hydroxide and of water[3]. The existing method employed in the present study is more advantageous than the available methods.

However, the existing methods have many limitations and drawbacks. The present research work comprises the improved process for synthesis of allyl sucrose and this revealed method has established few advantages over the reported methods.

Material and Methods

Materials: Sucrose, allyl chloride, dimethyl sulfoxide and sodium hydroxide are of synthetic grade and utilized after purification.

Synthesis of allyl sucrose: Weigh exactly 3.42 g of sucrose and transfer it quantitatively into 250 ml round bottom flask and then add 25 ml DMSO to dissolve the amount of sucrose. Weigh out 3.2 g of sodium hydroxide in 100 ml beaker and add 75 ml DMSO to make suspension. Now mix the contents of beaker into the round bottom flask and heat the reaction mixture on magnetic stirrer (90-100 rpm) at 80˚C for 1.5 h. After that add 6.12 g of allyl chloride and heat the contents with stirring for 2.5 h.

The crude product is light yellow colored and is kept overnight at room temperature. The product is diluted with water, shacked for 5 min and extracted with ethyl acetate. The isolated product is light yellow colored oily liquid and its yield is 90%. Whole reaction is summarized in figure 1.

Spectroscopic characterization: IR spectra are analyzed on a Bruker Alpha spectrophotometer. A Mass spectrum is recorded on TOF MS ES$^+$ 162. ^{1}H NMR and ^{13}C NMR spectra are determined in DMSO by Bruker Avance II 400 NMR at SAIF Punjab University Chandigarh.

Results and Discussion

IR Spectra: The spectral data (Table 1) of allyl sucrose observed sharp absorption peak at 3358 cm^{-1} to confirm the presence of hydroxy group. The group C=C-H gives stretching at 2924 cm^{-1} for titled compound. The peaks at 1648, 1557 cm^{-1} shows C=C stretching of allyl group. Bending vibration of >CH- adjacent to double bond gives the peak at 1437 cm^{-1}. For ether it shows the peak at 1053cm^{-1} of C-O stretching. A peak at 1008 cm^{-1} and 928 cm^{-1} in figure 2 helped to assign the presence of C=C-H bending of allyl group by Bruker FT-IR spectrophotometer (fig. 1).

Mass Spectra: The Mass spectrum of the synthesized compound was run using TOF detector ES+ 162 (from SAIF) and is presented in figure 3. The spectrum exhibits m/z -541.75 [M+1] peak which confirms the molecular weight of allyl sucrose. The remaining peaks also support structure of allyl sucrose.

^{1}H NMR Spectra: In ^{1}H NMR spectra (in deutarated DMSO), the doublet to triplet peak at 5 to 5.9 ppm [10 H, J=15, J=8.0, J=5-5.9Hz] helped to prove the presence of

alkene hydrogen (–C=CH_2–) group in titled derivative. The protons of –O – CH_2 – CH in compound are obtained between δ values 3 to 4.9 ppm [8 H, J=3.99 Hz]. The presence of methoxy group –O-CH is in figure 4 confirmed by a sharp doublet peak at δ = 2.3 to 3 ppm [5 H, J=2.54 Hz] of the titled compound (fig. 3).

^{13}C NMR Spectra: In ^{13}C NMR spectra, the δ values were obtained between 30 to 140 ppm which has confirmed the formation of the final compound. The – CH_2 terminal group and the presence of –O – CH_2 = CH were identified by the sharp singlet at 138 and 113 ppm in the spectrum. The presence of –O-CH_2 group in compound is confirmed the peak in the range of 61.7 ppm in figure 5. The FTIR, NMR and Mass spectroscopic study accepted and confirmed the structure of allyl sucrose. Mass spectrum of the synthesized cross-linking agent shows the connection of five allyl groups to sucrose. The present synthetic method is completed within 4.5 h using the extraction of product with ethyl acetate while the existing methods required overnight period to separate the product. Therefore, the present synthetic method has successfully arranged allyl sucrose with allyl chloride and the method is faster and has ease of isolation than the available methods[2,3]. The prepared product may be further used for cross-linking agent in polymerization process.

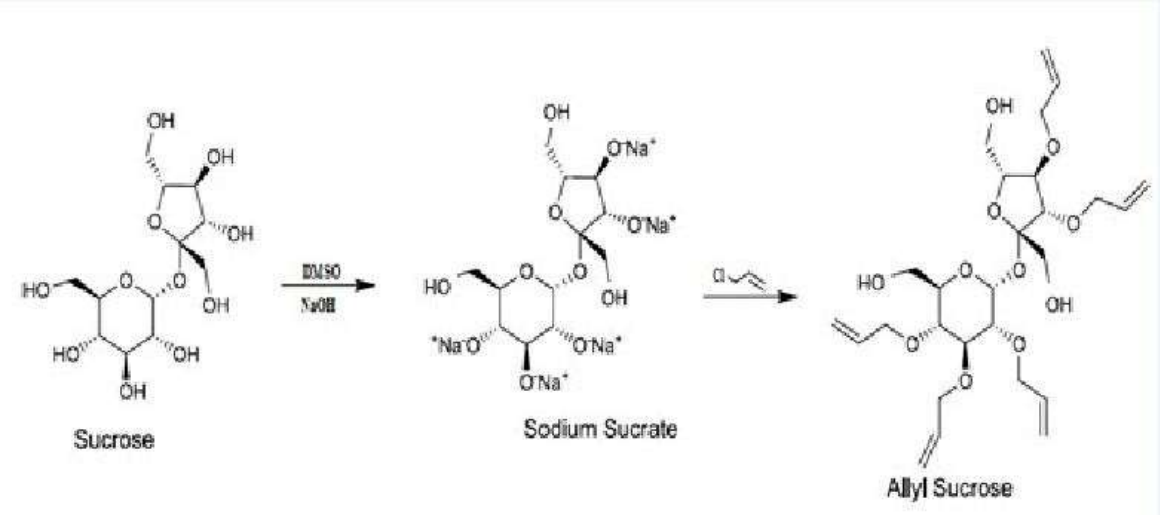

Figure 1: Reaction scheme of allyl sucrose

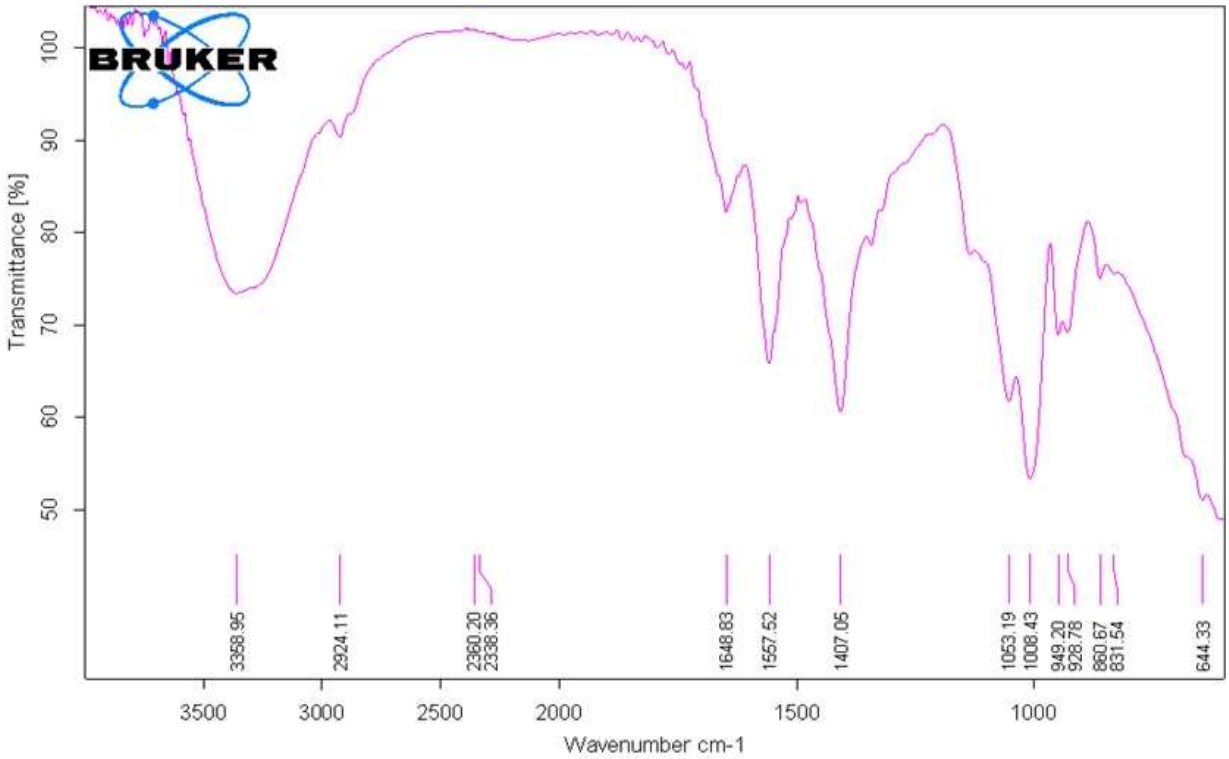

Figure 2: FT-IR spectrum of allyl sucrose

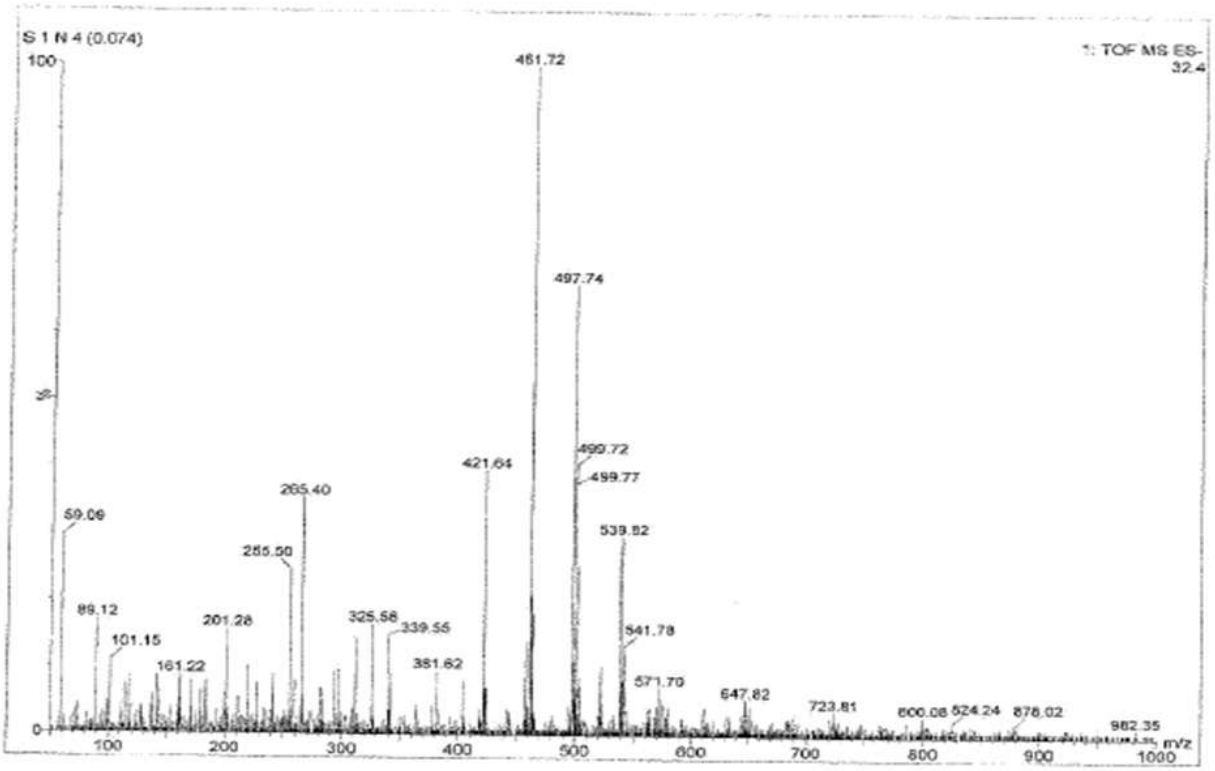

Figure 3: Mass spectrum of allyl sucrose

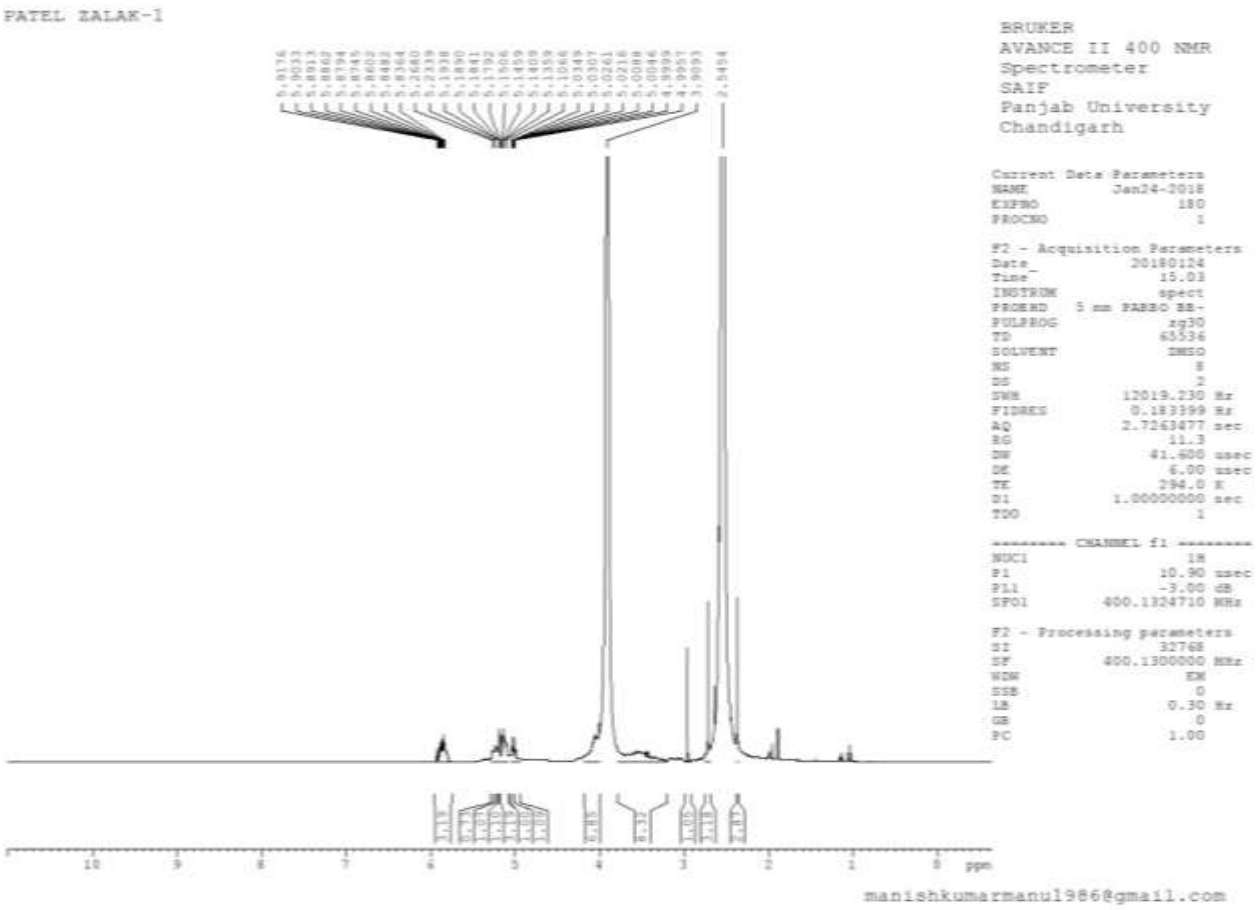

Figure 4: ^{1}H NMR spectrum of allyl sucrose

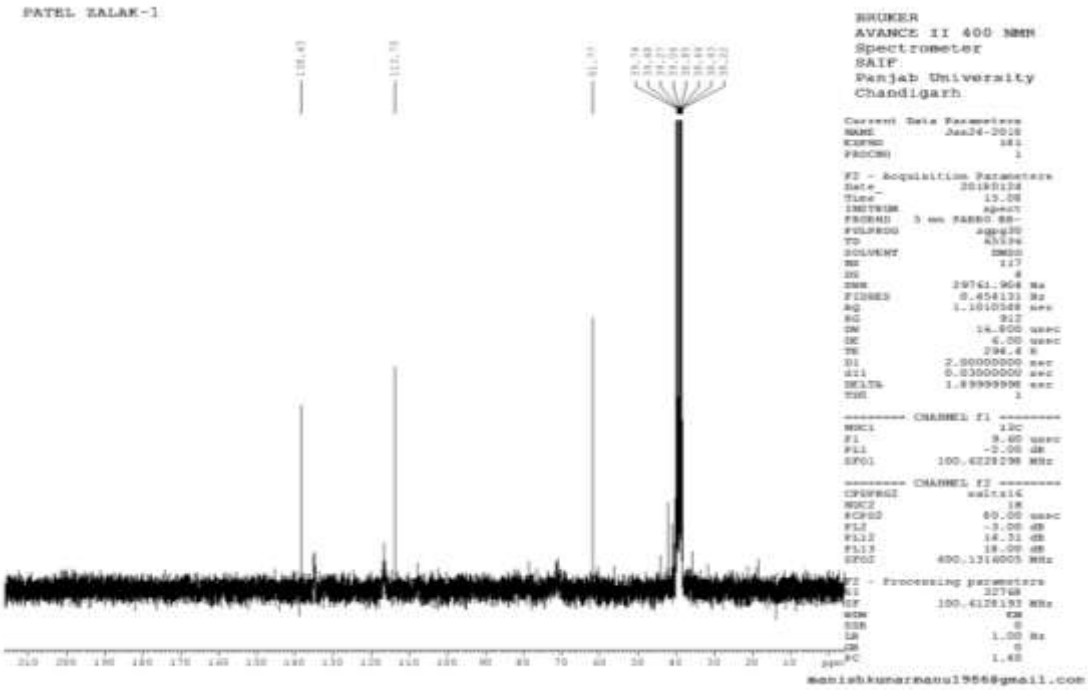

Figure 5: ^{13}C NMR spectrum of allyl sucrose

Table 1
Spectral data of allyl sucrose

S.N.	Absorption Range [cm^{-1}]	Type of Vibration
1	3358	O-H Stretching
2	2924	C = C-H Stretching
3	1648, 1557	C = C Stretching of allyl group
4	1053	C-O Stretching
5	1008	C = C-H Bending of allyl
6	928	C = C-H Bending of allyl

Conclusion

From the resultant data of mass spectra it is clear that five allyl groups are attached to a sucrose molecule. This method is more rapid, economically efficient and has no difficulty of isolation than the available methods. The present method is also used on large scale industrial preparation.

Acknowledgement

We acknowledge thanks to Research Laboratory, Pramukh Swami Science and H D Patel Arts College, Kadi for providing research facilities.

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(Received 28th August 2019, accepted 22nd October 2019)