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## Synthesis and Characterization of Co-Polymer (Styrene / Allyl 2,3,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranoside) and Studying some of its thermal properties

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### Abstract:

In this research, a Co-polymer (Styrene / Allyl-2.3.4.6-tetra-O-acetyl- $\beta$ -D-glucopyranoside) was synthesized from glucose in four steps using Addition Polymerization according to the radical mechanism using Benzoyl Peroxide (BP) as initiator. Initially, Allyl-2.3.4.6-tetra-O-acetyl- $\beta$ -D-glucopyranoside monomer was prepared in three steps and the reaction was followed by (HPLC, FT-IR, TLC), in the fourth step the monomer was polymerized with Styrene and the structure was determined by FT-IR and NMR spectroscopy. The reaction conditions (temperature, reaction time, material ratios) were also studied to obtain the highest yield, the relative, specific and reduced viscosity of the prepared polymer was determined, from which the viscosity average molecular weight was calculated, which amounted to about (63,000 g/mol). Thermal properties of the polymer were studied by Differential Scanning Calorimeter (DSC) and determining the Oxidation Induction Time (OIT).

**Keywords:** Allyl bromide, Carbohydrates, Eco-friendly polymers, Glucose, Oxidation Induction Time, Polystyrene.

### Introduction:

Various carbohydrates of natural origin with all their simple and complex types are among the most widespread and abundant compounds in nature and serve as the main source of energy in living organisms.

Recently, the trend has increased towards organic synthesis using carbohydrates as building blocks of a renewable natural source to obtain monomers that can enter into polymerization reactions, also many sugar-based polymers have been discovered and used as biodegradable materials for drug delivery Systems within living organisms<sup>1-2</sup>.

Some polymers prepared from carbohydrates show anti-corrosion properties, which encouraged using them instead of toxic and high cost chemicals as they are biodegradable, chemically stable and eco-friendly materials at low cost. Bonds with the metal surface, and the inhibition activity of these polymers is due to their ability to form complexes with metal ions on the surface of the metals through the functional groups of those polymers and thus the polymer forms a cover on the metal surface due to the large specific surface area of the polymer

molecules and thus protect the metal from corrosion<sup>3</sup>.

In the last few years, using of plastic which is made of fossil materials has increased in several applications including food preservatives, canned foods, and others, which negatively affects the environment as a result of the non-decomposition of these consumables after they are used, Therefore, it was necessary to search for a better alternative. Polymers derived from glucose and galactose is one of the best alternatives because it is widely available, eco-friendly and non-toxic new polymers have been prepared starting from monomers in which glucose and galactose have a terminal alkene using addition polymerization with sugar ring opening. High molecular weights were obtained (reaching about 135 KDa) degradability of these polymers were studied in order to use them as an eco-friendly alternative instead of fossil polymers<sup>4</sup>.

Because of carbohydrates have eco-friendly and non-toxic properties and their high abundance in nature, many studies reported on introducing carbohydrates in preparing polycarbonates in order to improve the thermal properties of these polymers and the using of materials from natural renewable

sources, so both sugar d-mannose and carbon dioxide were used in preparing polycarbonate containing six-membered ring and comparing it with aliphatic polycarbonate. The cyclic polycarbonate showed higher glass transition temperatures than the aliphatic ones and possibility of modifying the polymer structure after the polymerization which leads to the possibility of using this type of polymers in multiple applications, especially in the biological and medical fields<sup>5</sup>.

Carbohydrate co-polymers that contain glucose in their structure showed special properties in the field of plastics and adhesives, where a co-polymer was prepared from monomers (6-acrylo-1,2,3,4-tetra-glucose acetate) and (n-butyl acrylate) to obtain a new polymer including environmentally friendly materials, the polymer possessed flexibility and rubbery structure which enables it to be used in making sustainable commodity products at low cost and less harmful to the environment. The mechanical properties and adhesive properties of the resulting polymer have also been studied to determine the areas of use<sup>6</sup>.

Therefore, based on the importance of carbohydrate polymers as environmentally friendly and biodegradable materials and the possibility of using them in various applications, we have seen that it is appropriate to synthesize this type of polymers, a glucose-Styrene Co-polymer was prepared in this research, characterized and studied some of its thermal properties.

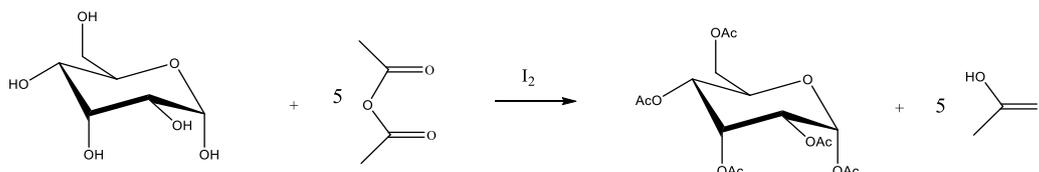
### Materials and Methods:

All reagents were commercially available. HPLC experiment were acquired using LC-Prominence from Shimadzu. IR spectra were acquired using potassium bromide within the 400-4000  $\text{cm}^{-1}$  range using an IR Affinity-1S spectrometer from Shimadzu. The  $^1\text{H-NMR}$  400 MHz spectra were run in  $\text{CDCl}_3$  using Bruker spectrometers.

### Experimental Section:

monomer preparation (allyl-2,3,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranoside):<sup>7</sup>

- First stage: Protection of the hydroxyl groups (Scheme 1):



### Glucose pentaacetate I

### Scheme 1. protection of the Hydroxyl groups in Glucose

Place 100 mL in a flask equipped with a magnetic stirrer, (0.105 mol) of acetic anhydride, then (0.0111 mol = 2.001 g) of pure d-glucose is added with stirring where a suspension is formed,

then the specified amount of iodine as catalyst is added on four experiments to study the effect of catalyst concentration on reaction time as mentioned in Table1:

Table 1. Effect of the catalyst amount on the reaction time

Catalyst amount (mmol)	0.2	0.4	0.8	1
Reaction time (min)	35	21	15	12

the end of the reaction was determined by two indicators, the first one is the complete reaction of glucose and transformation of the suspension into a transparent liquid with a reddish-brown colour, and the second one is the Thin Layer Chromatography test (TLC) with mobile phase hexane:ethyl acetate 1:1. After the reaction was finished, the product was purified by extraction with  $\text{CHCl}_3$  and washed

with sodium thiosulfate to get rid of iodine, separate the organic layer with separation funnel, dried with anhydrous sodium sulfate, the solvent was expelled by drying under vacuum to give white crystals with a pungent smell with yield 99%, mp 132  $^\circ\text{C}$ , TLC  $R_f$  0.75 silica gel, hexane:ethyl acetate 1:1. IR: ( $\text{cm}^{-1}$ ) 1750, 1242 as shown in Fig.1.

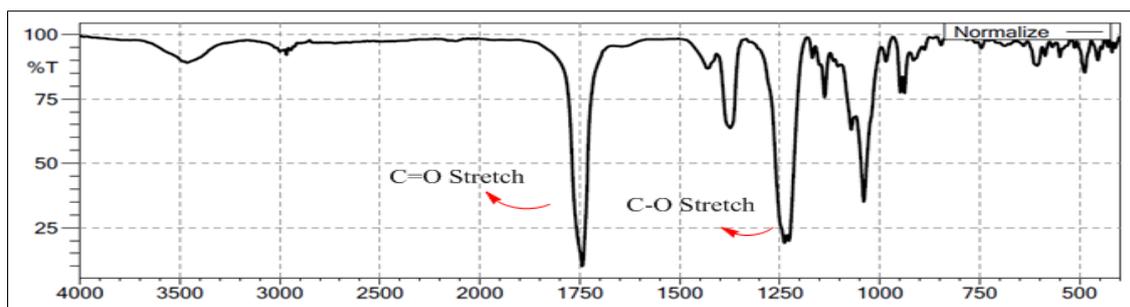


Figure 1. FT-IR Spectrum of I.

By comparing FT-IR with glucose as shown in Fig.2 we notice the disappearance of the absorption band at 3200-3500  $\text{cm}^{-1}$  belongs to the OH groups, and the appearance of a sharp and strong absorption

band at 1750  $\text{cm}^{-1}$  belongs to the stretch of the new carbonyl group in the product.

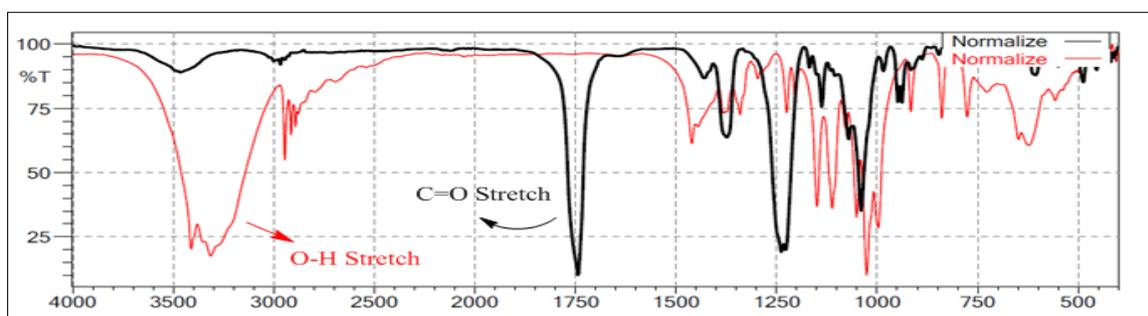
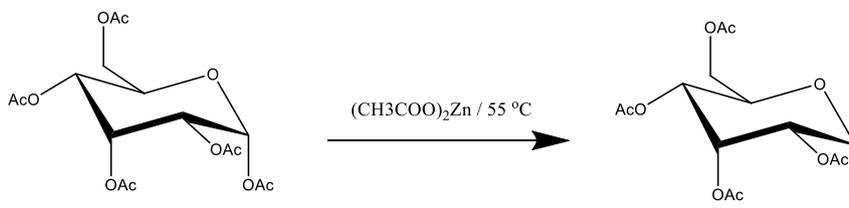


Figure 2. Comparison of (FT-IR) spectrums between Glucose pentaacetate and Glucose

- Second stage: selective deprotection of the Anomeric group (Scheme 2):



2,3,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranoside II  
Scheme 2. Anomeric group deprotection

In 250 mL flask equipped by a Heater with magnetic stirrer, and a glass condenser (10 mmol = 3.9093 g) of glucose pentaacetate was added with 100 mL of methanol, the mixture heated to 55  $^{\circ}\text{C}$  and then (1 mmol = 0.22 g) of zinc acetate added as catalyst, the reaction was followed with me by TLC. Stirring was continued until TLC Showed 3<sup>rd</sup> spot indicates to deprotection of another hydroxyl group which is undesirable product.

The reaction took 5 hours, after which the mixture was dried from the solvent under vacuum to get a white precipitate, extracted with  $\text{H}_2\text{O}:\text{CHCl}_3$  1:1, the organic layer was separated, the solvent was expelled, the product was purified by column chromatography to give yellowish-white precipitate, mp 118  $^{\circ}\text{C}$ , TLC and column chromatography (1m,  $\text{SiO}_2$  mesh 400)  $R_f$  0.527 silica gel, hexane:ethyl acetate 1:1 . IR: ( $\text{cm}^{-1}$ ) 3476, 1750, 1242 as shown in Fig.3.

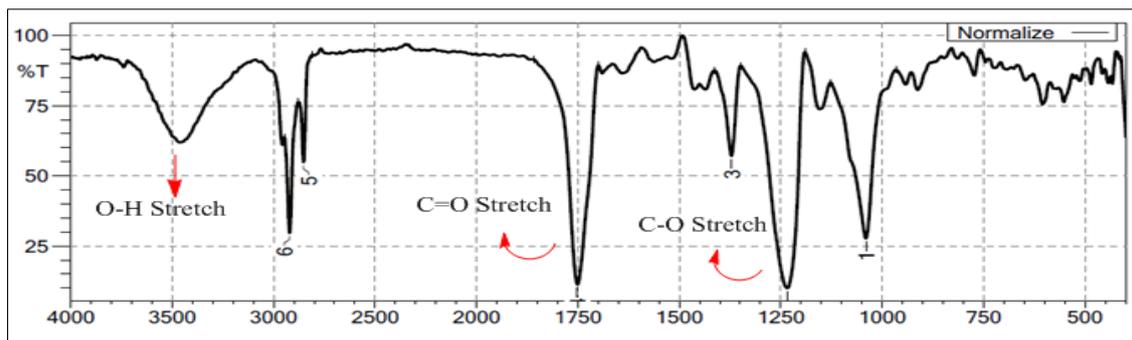


Figure 3. FT-IR Spectrum of II

By comparing FT-IR between the raw material and the product as shown in fig.4 we note reappearance of the absorption band at 3476 cm<sup>-1</sup> belongs to the hydroxyl group, while the sharp and strong

absorption band remained at 1750 cm<sup>-1</sup> due to the stretching of the carbonyl group in the remaining protective groups.

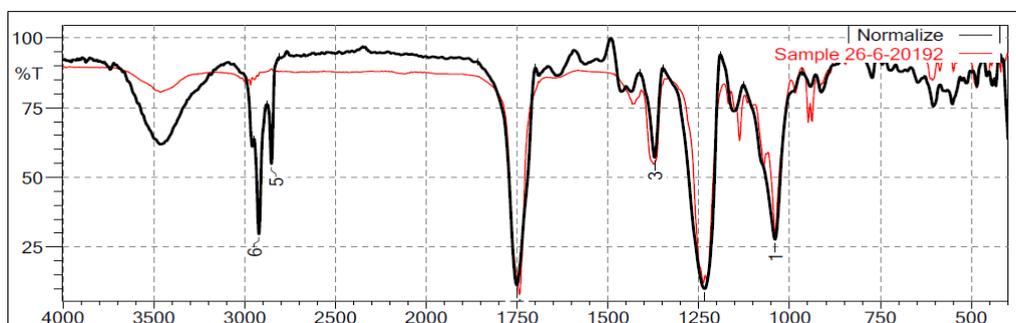
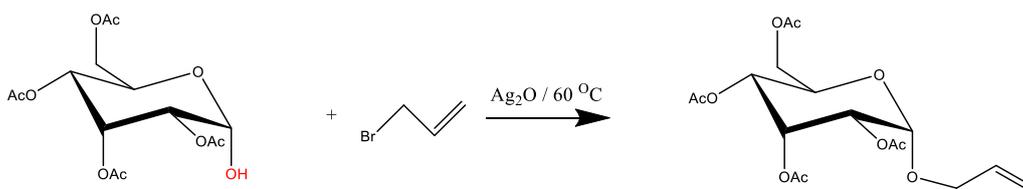


Figure 4. Comparison of FT-IR spectra of I and II

- Third stage: producing the double bond in the sugar ring (Scheme 3):



Allyl-2,3,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranoside (III)  
Scheme 3. Alkylation using Allyl Bromide

(1 mmol = 0.35 g) of 2,3,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranoside with 20 mL of acetonitrile (as solvent) added to a flask equipped with a heater, magnetic stirrer, and a glass condenser then (0.06 mmol = 14 mg) of silver oxide added to the flask as catalyst with stirring and heating up to 50 °C and then excess amount of Allyl bromide was added, the reaction was monitored by TLC. After 12 hours the reaction was stopped and the silver oxide was removed by simple filtration, the solvent was expelled, followed by extraction with water and CHCl<sub>3</sub>, dried the organic layer, the excess amount of the unreacted Allyl bromide was removed by heating to the boiling point of the Allyl, and its

disposal was ensured by using high-performance liquid chromatography HPLC, a standard of the Allyl and the reaction mixture prepared and injected into the HPLC, note the disappearance of the Allyl from the reaction mixture as shown in Fig.5. To separate the resulting material, thin-layer glass chromatography sheets were used with the same mobile phase, the product was obtained and extracted from silica gel with chloroform, evaporate solvent and dried to obtain white crystals 32.27%, mp 91.2 °C, TLC R<sub>f</sub> 0.704 silica gel, hexane:ethyl acetate 1:1 . IR: (cm<sup>-1</sup>) 3086, 1750, 1420, 1110.

FT-IR spectrum Fig.6 Showed disappearance of the band at 3450 cm<sup>-1</sup> belongs to the hydroxyl group,

while the Strong Sharp band remains at  $1750\text{ cm}^{-1}$  due to the stretching of the carbonyl groups, and shows new bands as a result of Allyl linkage: an absorption band at  $3086\text{ cm}^{-1}$  belongs to  $\text{C}_{\text{SP}^2}\text{-H}$

bond and at  $1110\text{ cm}^{-1}$  belongs to the new ether group and at  $1420\text{ cm}^{-1}$  belong to the new methylene group in the Allyl.

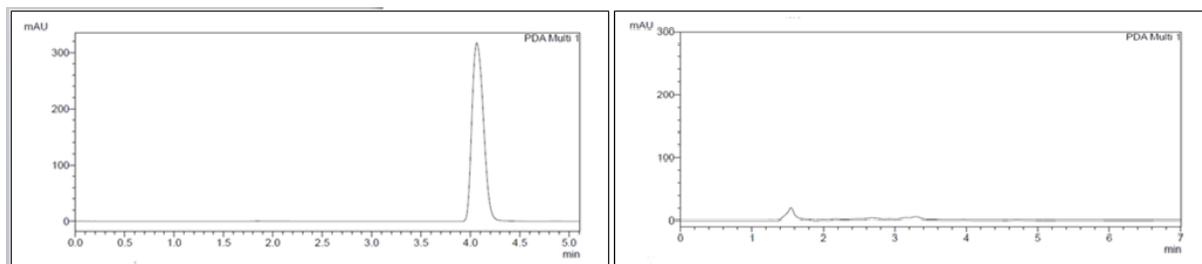


Figure 5. HPLC Chromatogram of the Allyl Bromide Standard and the reaction mixture

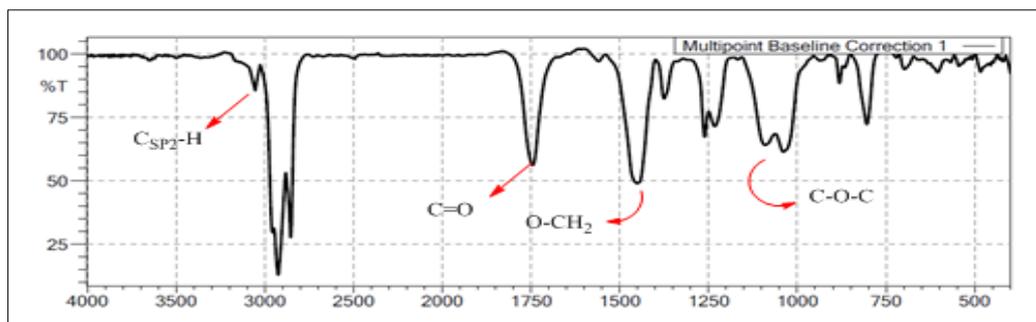
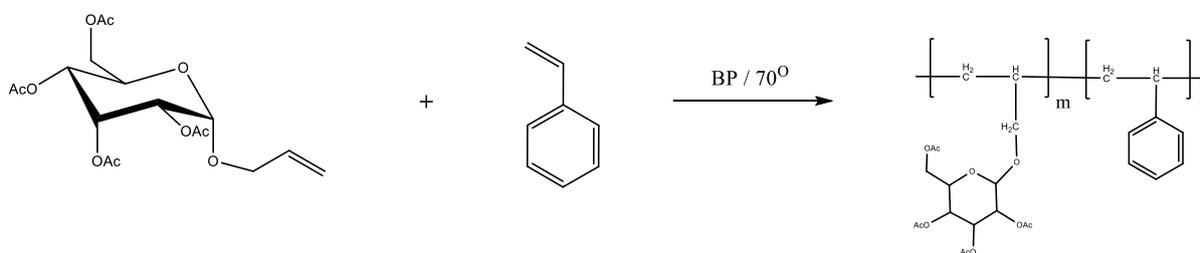


Figure 6. FT-IR Spectrum of III

### 3-2- Polymerization (Scheme 4):



The Co-Polymer IV  
Scheme 4. Polymerization with Styrene

After (10 mmol = 3.888 g) of Allyl-2,3,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranoside with (0.02 mmol = 48.3 mg) of Benzoyl Peroxide as initiator and 20 mL of acetonitrile (as a solvent) have been added to a Two-neck round bottom flask equipped with a magnetic stirrer and vacuumed by passing Nitrogen gas, the reaction mixture was heated to  $70\text{ }^{\circ}\text{C}$ , then (10 mmol = 1.039 g) of Styrene was added. The reaction was followed by TLC with the same conditions. The viscosity of the mixture increased

after 8 hours, where the reaction took 9.5 hours, after which the polymer was purified by extraction with water and  $\text{CHCl}_3$ , taking the organic layer, the solvent was expelled, re-extracted with  $\text{CHCl}_3$ , evaporate solvent to get a solid, plastic texture with a very light orange colour, mp  $144.2\text{-}146.8$ , IR: ( $\text{cm}^{-1}$ ), Fig.7: 3028, 2920, 1753, 1226, 758-700.  $^1\text{H-NMR}$   $\text{CDCl}_3$ , Fig.8:  $\delta$  0.86 (t,  $J=8\text{Hz}$ ), 1.04-1.15 (t,  $J=11\text{Hz}$ ), 1.23 s, 1.39 s, 1.99-2.03 (t,  $J=8\text{Hz}$ ), 2.07 (d,  $J=7.2\text{Hz}$ ), 3.95-4.68 (m,  $\text{H}_{\text{gxluc}}$ ), 7.11 (m,  $\text{H}_{\text{arom}}$ ).

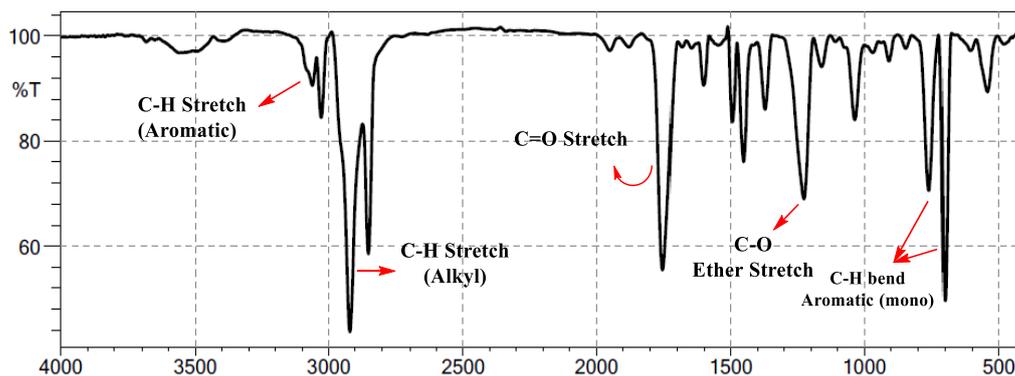


Figure 7. FT-IR Spectrum of the Co-Polymer

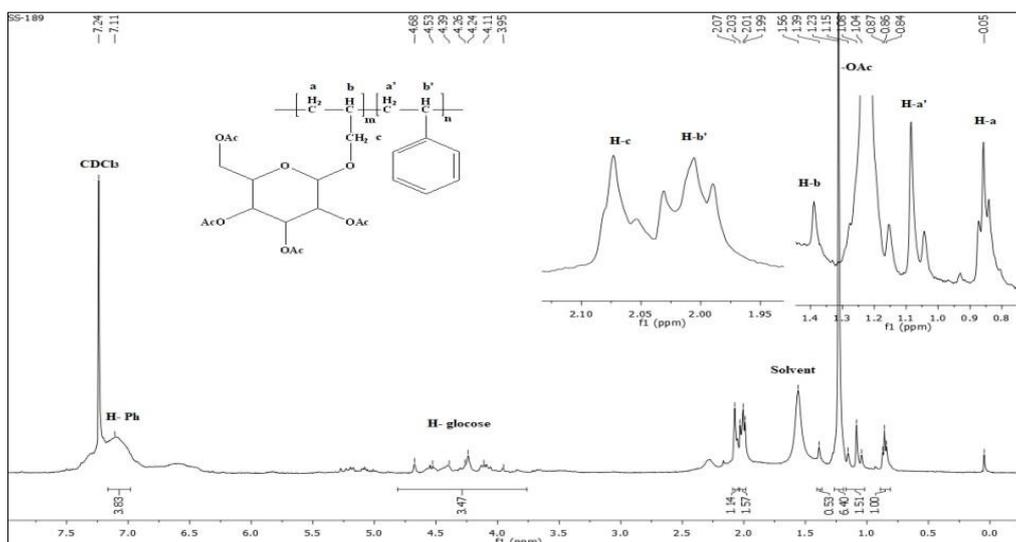


Figure 8. <sup>1</sup>H-NMR Spectrum of the Co-Polymer

By taking the integration ratio of the two signals (a) at 0.86 and (a') at 1.04 as shown in Eq.1 we can find the ratio of each monomer within the co-polymer structure:

$$\frac{a}{a'} = \frac{1}{1.51} \gg \frac{m}{n} = \frac{1}{1.51} = \frac{2}{3.02}$$

1

Therefore, the co-polymer consists of 60.15% Styrene and 39.85% of Allyl-2,3,4,6-tetra-O-acetyl-β-D-glucopyranoside.

### 3-3- Viscosity Average Molecular Weight calculation $M_v$ :

Viscosity is usually measured using the Ostwald scale U form by measuring the time required for the solvent and the polymer solutions with known concentrations through the tube and recording them ( $t_0$ ,  $t$ ) respectively, then calculating the relative viscosity ( $\eta_{rel}$ ) from the law  $\eta_{rel} = \frac{t_{solution}}{t_{solvent}}$  and the specific viscosity ( $\eta_{sp}$ ) from the Eq. 2:

$$\eta_{sp} = \frac{t_{solution} - t_{solvent}}{t_{solvent}}$$

2

Then the reduced viscosity ( $\eta_{red}$ ) was determined ( $\eta_{red} = \frac{\eta_{sp}}{C}$ ) where  $C$  is the concentration of the polymer solution in g/mL as shown in Table 2, and to determine the rate of molecular weight we measure the flow time for standard solutions of the polymer and draw a graph expresses the reduced viscosity changes ( $\eta_{red}$ ) in terms of concentration as shown in Fig.9, then we extend the Line to  $C = 0$  to get the real viscosity  $[\eta]$ , the average molecular weight can be calculated using the following Mark-Houwink equation :

$$[\eta] = k * \bar{M}_v^a \gg \bar{M}_v = e^{\frac{\ln[\eta] - \ln k}{a}}$$

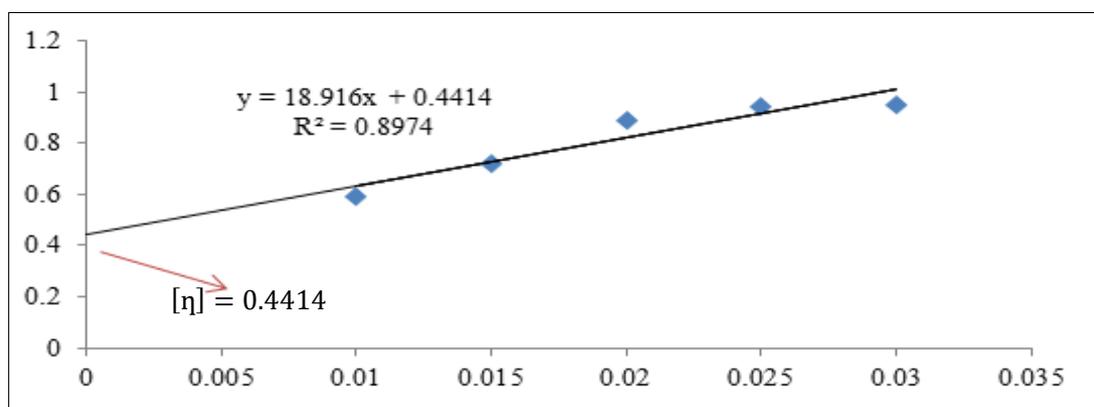
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Where:  $K$ ,  $a$  are constants that depends on the type of polymer, the solution, and temperature of the solution and they are reference values.

$[\eta]$ : real viscosity ,  $\bar{M}_v$ : average molecular weight

**Table 2. viscosity values:**

Solvent flow time (sec)	concentration (g/mL)	Flow Time (sec)	Relative viscosity	Specific viscosity	reduced viscosity
10.15	0.01	10.21	1.0059113	0.00591	0.591133
	0.015	10.26	1.0108374	0.01084	0.722496
	0.02	10.33	1.017734	0.01773	0.8867
	0.025	10.39	1.0236453	0.02365	0.945813
	0.03	10.44	1.0285714	0.02857	0.952381



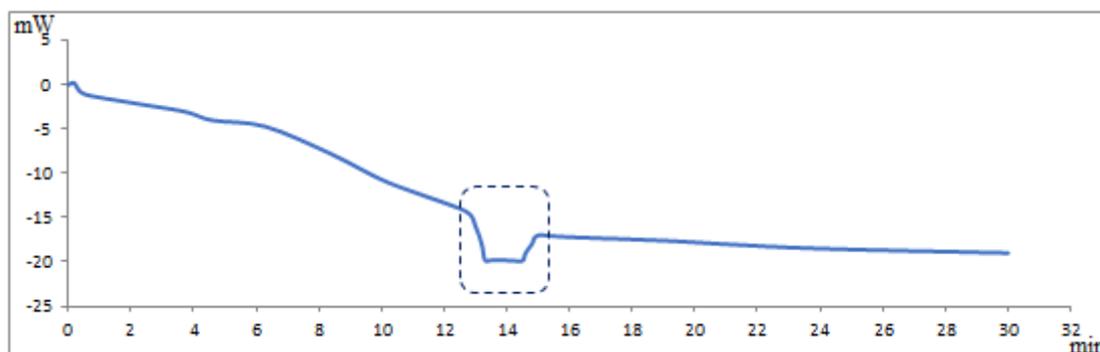
**Figure 9. reduced viscosity changes in terms of concentration for the Co-polymer**

From the study of changes, we find the value of the real viscosity  $[\eta] = 0.4414$  and by applying the Mark-Houwink equation where  $k = 0.000123$  and  $a = 0.74$ :

$$\bar{M}_v = 63673.05 \text{ g/mol}$$

Differential Scanning Calorimetry (DSC):

The DSC spectrum was recorded Fig.10 and the Oxidation Induction Time (O.I.T) was determined which amounted 1.3 min, also the (O.I.T) of PolyStyrene prepared at the same conditions used in the preparation of the Co-polymer was determined in Fig.11 which amounted 4.3 min.



**Figure 10. DSC spectrum for the Polymer**

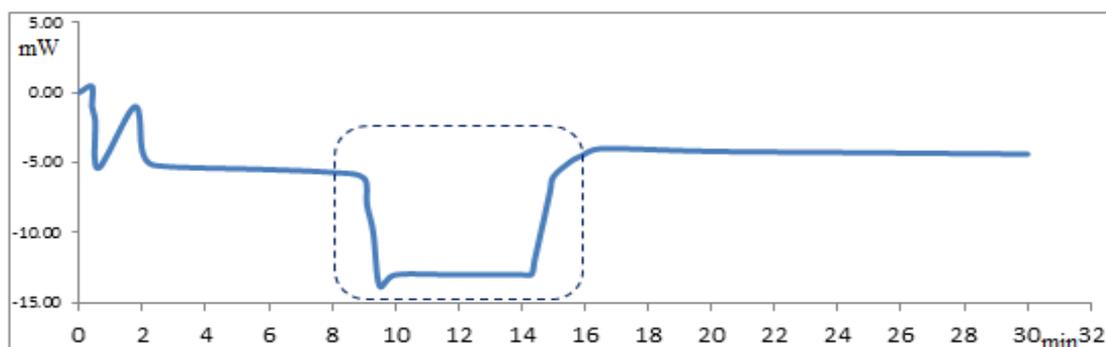


Figure 11. DSC spectrum for the PolyStyrene.

By comparing the (O.I.T) values of the polymers, we note that the Co-polymer can decompose by heat more than Polystyrene, which makes it more biodegradable and environmentally friendly than polystyrene with the same physical structure. Thus, it is possible to study the use of the new polymer in industries as an alternative to Polystyrene such as plastic.

### Conclusion:

The monomer (Allyl-2,3,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranoside) was synthesized from glucose in three stages, then polymerized with Styrene according to the radical addition mechanism. The structure of the Co-polymer was confirmed by using FT-IR and  $^1\text{H-NMR}$ . The average molecular weight was calculated by using Mark-Houwink equation. (DSC) spectrum of the polymer was recorded and the Oxidation Induction Time O.I.T was determined and compared with PolyStyrene prepared with the same conditions, it was found that the new polymer is more degradable than Polystyrene which makes it more eco-friendly with the same physical structure.

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### Authors' declaration:

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Besides, the Figures and images, which are not ours, have been given the permission for re-publication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in University of Al-Baath.

### Author's Contribution Statement:

G. R Conceived the idea. W. Y developed the theory and performed the computations and reactions. K. M designed and revised the MS. All authors discussed and analysed the results and contributed to the final manuscript.

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## تخليق وتشخيص بوليمير مشترك (ستايرين/أليل 2,3,4,6-رباعي-O-أسيتيل-β-D-غلوكوپيرانوزيد) ودراسة بعض خصائصه الحرارية

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### الخلاصة:

تم في هذا البحث اصطناع بوليمير مشترك مكون من أحاديي حد (الستايرين / α-أليل-2,3,4,6-رباعي خلات الغلوكوز) من خلال إجراء عملية البلمرة بالإضافة وفق الآلية الجذرية باستخدام محرض من البيروكسيد بنزويل (BP) على أربع مراحل، أولاً تم تحضير أحادي الحد α-أليل-2,3,4,6-رباعي خلات الغلوكوز على ثلاث مراحل وتم تتبع سير التفاعل باستخدام التقنيات (HPLC, FT-IR, T.L.C) وفي المرحلة الرابعة تم إجراء عملية البلمرة مع الستايرين وإثبات بنية البوليمير الناتج باستخدام مطيافية الطنين النووي المغناطيسي البروتوني ومطيافية الأشعة تحت الحمراء. كما تمت دراسة شروط التفاعل للمراحل (درجة الحرارة، زمن التفاعل، نسب المواد المتفاعلة) للحصول على أعلى مردود، ومن ثم عُينت اللزوجة النسبية والنوعية والمخفضة للبوليمير المحضر و تم حساب معدل الوزن الجزيئي اللزوجي له والذي بلغ حوالي 63000g/mol. دُرست بعض الخصائص الحرارية للبوليمير عن طريق تسجيل منحنى المسح الحراري التفاضلي وتحديد زمن التحريض التأكسدي (O.I.T).

**الكلمات المفتاحية:** أليل البروم، كربوهيدرات، بوليميرات صديقة للبيئة، غلوكوز، زمن التحريض التأكسدي، بولي ستايرين.