Synthesis, purification and identification of some D-xylosides

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ABSTRACT

D-xylose was converted into new glycosides by treating it with ethylene glycol in acidic medium under kinetically controlled conditions so predominant product is 2-Hydroxyethyl-O-D-xylofuranoside (1). The hydroxyl groups of the glycosides then were converted into 2-acetyl ethyl 2,3,4 tri-O-acetyl-D-xylofuranoside (2) by treating with acetic anhydride in pyridine. The acetate group at C-3 was converted into O-tosyl ethyl -O-D-xylofuranoside (3) by treating with one equivalent of toluene sulfonyl chloride. Derivative (3) was converted to azido ethyl -O-D-xylofuranoside(4) by treating the tosyl with one equivalent of sodium azide in DMF. Reaction of the azido with p-Benzoquinone, maleic anhydride and cinnamyl alcohol was carried out to get the triazolines(5,6,7) via a 1,3-dipolar cycloaddition. The new glycosides and the triazoline compounds were identified by FT-IR, ¹H –NMR and ¹³C- NMR spectroscopy and C.H.N analyses .

Keywords: D- xylose, derivatives, glycosides, triazoline, furanoside, pyranose.

1. Introduction

The study of glycosides is one of the exciting fields of organic chemistry. Glycosides are important biological molecules that are involved in a vast number of biological processes (Nicotra et al., 2008) Many glycosides have therapeutic uses such as anti-cancer therapies, anti-inflammatory, enzyme inhibitors and antibiotics (Hanessian and Lou, 2000). They are very important due to their effective biological activity (Andry et al., 1982). It is also known that if an active nucleus or molecule is linked to another nucleus, the resulting molecule may possess
great potential biological activity (Tsuda and Haque, 1983). A number of fruitful and efficient methods for selective acylation have so far been developed and employed successfully (Ishii et al., 1980; Gupta et al., 1997). Most of these methods are based on the blocking-deblocking technique (Tsuda et al., 1983; Wagner et al., 1974). The seminal work of Koenigs and Knorr many glycosidation methods have been developed and the field has been extensively reviewed (Koenigs and Knorr, 2004). And also biological evaluation of the synthesized compounds was reviewed (Kabir et al., 2003, 2008). It was observed that the combination of two or more acyl substituents in a single molecular framework enhances the biological activity many fold than their parent nuclei, for example, some triazolins derivatives of D-xylopyranose and xylofuranose were found more active than those of the standard antibiotics (Kabir et al, 2009).

The objectives of this work is to synthesize and identify some new glycosides of D-xylose both in its furanoid and pyranoid forms and then converting them into new triazoline compounds by treatment of them with sodium azide. Both the approach and the products are newly emergent facets. Glycosides as nitrogen heterocyclic derivatives of sugars have gained increasing interest in biological and chemical activities for their uses in medication and pharmaceutical industries beside their many other benefits (Grek and Drak, 2009). Triazoles and triazolines of sugars are usually made via 1, 3-dipolar cycloaddition reactions accomplished by introducing an azide group on the sugar moiety followed by reaction with unsaturated organic molecules (Carmela, P Grunanger, 2009; Daiekh, 1983).

2. Material and Methods Experimental

All solvents and reagents were purchased from Sigma-Aldrich and Fluka. All solvents and chemical were purified before used and were kept in special bottles contain, KOH, NaOH and CaCl2 for dryness according to general methods. Evaporation was conducted under vacuum using Rotary Evaporator. FT-IR spectra were recorded in the (400 – 4000) cm-1 frequency range by using, FT-IR Spectrophotometer, Ministry of health, Sana'a, Yemen. NMR spectra(H1-C13) were recorded on a Varian spectrometer at ambient temperature in an appropriate due treated solvent using CDCl3 as the internal standard at ambient temperature. In King Abdul Aziz University, Jeddah - Saudi Arabia.

The reactions were monitored by T.L.C made or aluminum plates covered with 0.2 mm of silica –gel F254 made by March Company. Detection were achieved by Iodine vapor and by spraying with conc.H2SO4 in EtOH followed by heating in an oven. C.H.N Elemental analysis was carried out using an Elemental varrio EL analyzer Column chromatography was performed with silica gel G60 using Solvent system employed for TLC analyses which was ethyl acetate, ethyl methyl ketone, benzene and methanol.
Table 1 The Physical Properties of compounds 1-7.

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Table 2 C.H.N Elemental analysis of compounds 1-7

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2.1 Synthesis of 2-Hydroxy ethyl-O-D-xylofuranoside

D-xylose (10g, 0.68mmol) was dissolved in a (0.46%) Hydrogen chloride contains in ethylene glycol which was prepared by adding acetyl chloride (2ml) to ethylene glycol(228ml) and leaving the mixture for about (2h). The reaction mixture was placed in (500ml) round bottomed flask and was stirred magnetically for three hours at room temperature. The course of the reaction was monitored by T.L.C. using benzene-methanol (4:6v/v) as solvent. After all the sugar spot was disappeared, the reaction was stopped by neutralization with sodium hydroxide solution in ethylene glycol. The solvent was removed under reduced pressure, to give a thick syrup product (11g, Brown color, yield 89%, R. F 0.9). FT-IR : cm⁻¹ = 3250-3400 (-OH), 1150 (C-O-C), 2930 (-CH₂). 1H-NMR (600 MHz, CDCl₃) δ (ppm) = 5.8 (1H, d, CH) 0.7-0.8 (2H, t, CH₂) 2.1 (2H, t, CH₂) 1.5 (3H, s, OH sugar) 2.5 (1H, s, OH ethyl) 1.3-1.5 (1H, q, CH) 2.8 (2H, d, CH₂) 3.2 (1H, t, CH) 4.0 (1H, t, CH). 13 C- NMR (200MHz, CDCl₃) δ (ppm) = 110, 81.33, 77.10, 75.00, 66.70, 62.11, 61.00 Carbon atoms. Anal. Calcd. for C₇H₁₄O₆ (149.2): C 43.30, H 7.27 %; Found: C 43.12, H 7.60 %.

The product obtained from compound (1) 2-hydroxy ethyl-O-D-xylofuranoside syrup (11g, 0.45mmol) was dissolved in pyridine (88.8ml) and the solution was cooled in ice-water before acetic anhydride (66.64ml) was added drop wise. The reaction solution was left for three days at room temperature after which the brown solution was powered into water (250ml). 2-acetyl ethyl 2,3,4 tri-O-acetyl-D-xylofuranosides was extracted with chloroform (10x52) and the chloroform solution was dried over calcium chloride, filtered and evaporated under reduced pressure, which was examined by (TLC). The compound was purified using column chromatography Benzene : methanol 2:8. The solvent was removed under reduced pressure and the syrup product was examined. To give a thick syrup product (12.55g, Brown color, yield 85%, R. F 0.6). FT-IR : cm⁻¹ = 3250-3400 (-OH), 1150 (C-O-C), 2930 (-CH₂). 1H-NMR (600 MHz, CDCl₃) δ (ppm) = 2.3 (9H, s, 3CH₃) 0.9 (3H, s, CH₃) 3.2 (1H, s, OH sugar) 2.5 (1H, s, OH ethyl) 1.3 (1H, t, CH) 3.4 (2H, t, CH₂) 3.7-3.8 (2H, t, CH₂) 1.7 (1H, s, OH) 5.8 (1H, d, CH) 4.3 (1H, t, CH) 5.0 (1H, t, CH) 4.7-4.8 (1H, q, CH) 3.1 (2H, d, CH₂). 13C- NMR (200MHz, CDCl₃) δ (ppm) = 160.0, 160.03, 160.02, 120, 104.7, 103.2, 77.0, 76.00, 75.00, 68.3, 62.00, 23.00, 21.00, 20.00 Carbon atoms. Anal. Calcd. for C₁₃H₂₀O₉ (320.29): C 48.74, H 6.27 %; Found: C 43.12, H 7.60 %.

2.2 Synthesis of 2-acetyl ethyl 2,3,4 tri-O-acetyl-D-xylofuranoside

The product obtained from compound (1) 2-hydroxy ethyl-O-D-xylofuranoside syrup (11g, 0.45mmol) was dissolved in pyridine (88.8ml) and the solution was cooled in ice-water before acetic anhydride (66.64ml) was added drop wise. The reaction solution was left for three days at room temperature after which the brown solution was powered into water (250ml). 2-acetyl ethyl 2,3,4 tri-O-acetyl-D-xylofuranosides was extracted with chloroform (10x52) and the chloroform solution was dried over calcium chloride, filtered and evaporated under reduced pressure, which was examined by (TLC). The compound was purified using column chromatography Benzene : methanol 2:8. The solvent was removed under reduced pressure and the syrup product was examined. To give a thick syrup product (12.55g, Brown color, yield 85%, R. F 0.6). FT-IR : cm⁻¹ = 3250-3400 (-OH), 1150 (C-O-C), 2930 (-CH₂). 1H-NMR (600 MHz, CDCl₃) δ (ppm) = 2.3 (9H, s, 3CH₃) 0.9 (3H, s, CH₃) 3.2 (1H, s, OH sugar) 2.5 (1H, s, OH ethyl) 1.3 (1H, t, CH) 3.4 (2H, t, CH₂) 3.7-3.8 (2H, t, CH₂) 1.7 (1H, s, OH) 5.8 (1H, d, CH) 4.3 (1H, t, CH) 5.0 (1H, t, CH) 4.7-4.8 (1H, q, CH) 3.1 (2H, d, CH₂). 13C- NMR (200MHz, CDCl₃) δ (ppm) = 160.0, 160.03, 160.02, 120, 104.7, 103.2, 77.0, 76.00, 75.00, 68.3, 62.00, 23.00, 21.00, 20.00 Carbon atoms. Anal. Calcd. for C₁₃H₂₀O₉ (320.29): C 48.74, H 6.27 %; Found: C 43.12, H 7.60 %.

2.3 Synthesis of O-tosyl ethyl -O-D-xylofuranoside

The product obtained from compound (2) 2-acetyl ethyl 2,3,4 tri-O-acetyl-D-xylofuranoside syrup (10g) was dissolved in pyridine (80.2ml) at zero °C, then adding p-toluenesulfonyl chloride (4.5 g, 0.072mmol), after being dissolved in pyridine added drop wise by means of a separation funnel. The mixture reaction was stirred for (48h) hours at 0 °C. The course of the reaction was monitored by T.L.C. using benzene-methanol (2:8v/v) as solvent. When T.L.C shows completion of the reaction, the solution was powered into ice – water with stirring. The product was extracted with chloroform and dried with magnesium sulfate and chloroform was removed under reduced pressure. The compound was purified using column chromatography benzene: methanol 3:7 v/v to give (13.4g, Brown color, yield 82%, R. F 0.5). FT-IR cm⁻¹ = 1736 (R-COO-CH₃) 1140 (C-O-C) 2900 (-CH₂-) 1170-1180 (SO₂) 1000 (S-O) 1800-2000 substituted in Para aromatic 1500 C= C the aromatic group. 1H-NMR (600 MHz, CDCl₃) δ
(ppm) = 7.5-7.9 (4H, m, CH aromatic) 2.0 (9H, s, CH3) 3.7-3.9 (2H, t, CH2) 2.4-2.6 (4H, q, CH2) 5.0 (1H, d, CH) 4.5-4.6 (1H, t, CH).

13C-N.M.R (200MHz, CDCl3) δ (ppm) = 160.03, 160.3, 160.02, 138.0, 133.0, 129, 128.4, 104.7, 77.0, 76.0, 75.0, 69.9, 64.40, 62.50, 21.00, 20.07, 20.00 carbon atoms.

2.4 Synthesis of azido ethyl-O-D-xylofuranoside

The product obtained from compound (3) O-tosyl ethyl-O-D-xylofuranoside (13.4g, 0.687mmol) was dissolved in dimethylformamide DMF(40ml) with an excess of sodium azide (2.2g, 0.06mmol). The mixture was heated under reflux conditions at (140-150 °C) for 24 hours. When T.L.C shows completion of the reaction after that mixture was cooled and powered into ice-water with stirring. The product was extracted with chloroform which was then removed under reduced pressure. The syrupy product was purified on silica gel column chromatography using benzene: methanol 2:8 to give 8.52gm, yield 80%, R.F(0.9). FT-IR cm⁻¹: 1730 cm⁻¹ (R-COO-CH₃) 1145 (C-O-C) 1000-1300 (C-N) 2900 (-CH₂) 2100-2160 (-N≡N) azide. 1H-N.M.R (600 MHz, CDCl₃) δ (ppm) = 2.0(9H, s, 3 CH₃) 3.7-3.8 (2H, t, O-CH₂) 2.7-2.9 (2H, t, CH₂-N) 3.1 (1H, d, CHbenzoquinon) 3.5 (1H, d, CHbenzoquinon) 5.8 (1H, d, CH) 4.6 (1H, t, CH) 4.2 (2H, d, CH₂) 5.3 (1H, t, CH) 5.0 (H, q, CH). 13C-NMR (200MHz, CDCl₃) δ (ppm) = 160.05, 160.03, 160.02, 104.7, 77.0, 76.0, 75.0, 64.40, 62.50, 51.00, 21.00, 20.22, 20.00 carbon atoms.

2.5 Synthesis of benzoquinone triazolin Propyl-O-D-xylofuranoside

The derivate obtained from(4) azido ethyl-O-D-xylofuranoside (1.43g, 0.012mmol) was dissolved in dioxane(64ml) with an excess of (P-benzoquinone 1.2g, 0.051mmol). The mixture was stirred and heated under reflux at (50-60 °C) for (92h) When T.L.C shows completion of the reaction, the mixture was cooled and powered into ice-water. The product was extracted with chloroform and dried with anhydrous magnesium sulfate and chloroform was removed under reduced pressure to give syrupy product. The syrupy product compound was purified on silica gel column chromatography using benzene: methanol 2:8v/v to give 3.2gm. Light-Yellow color, yield 79%, R.F 0.4, FT-IR cm⁻¹: 1710 (C=O) of benzoquinone 1300(C-O) 1500 (C=C) 1735-1740 (R-COO-CH₃) 1145 (C-O-C) 1670 (-N≡N) diazostrech. 1H-NMR (600 MHz, CDCl₃) δ (ppm) = 2.0 (9H, s, 3 CH₃) 3.7-3.8 (2H, t, O-CH₂) 2.7-2.9 (2H, t, CH₂-N) 3.1 (1H, d, CHbenzoquinon) 3.5 (1H, d, CHbenzoquinon) 5.8 (1H, d, CH) 4.6 (1H, t, CH) 4.2 (2H, d, CH₂) 5.3 (1H, t, CH) 5.0 (H, q, CH). 13C-NMR (200MHz, CDCl₃) δ (ppm) = 180.00, 179.00, 178.00, 160.4, 160.3, 160.22, 160, 137.03, 137.00, 104.7, 80.77, 77.00, 76.0, 75.00, 72.22, 64.40, 21.00, 21.88 carbon atoms.

2.6 Synthesis of maleic anhydride triazolin-ethyl-O-D-xylofuranoside

The derivate obtained from(4) azido-ethyl-O-D-xylofuranoside (2.25gm, 0.084mmol) was dissolved in dioxane(64ml) and added to maleic anhydride (1.55gm). The mixture was stirred and heated under reflux at (50-60 °C) for (92h) When T.L.C shows completion of the reaction, the mixture was cooled and powered into iced-water. The product was extracted with chloroform(5x15ml) and dried with magnesium anhydrous sulfate, and chloroform was
removed under reduced pressure to give, a syrupy product; the syrupy product compound was purified on silica gel column chromatography using dichloromethane : benzene 3:8 v/v to give 1.8 gm, dark-yellow color, yield 79%, R.F 0.8. FT-IR(KBr) cm⁻¹: 1745 (-O- ether anhydride) 1710 (-CO-) 1300(-C=N-) 1500 (-N=N-), 2340 (diazostrech). 1H-NMR (600 MHz, CDCl₃) δ (ppm) = 1.7-1.8 (9H, s, 3 CH₃) 2.7 (1H, d, CH₃malic) 3.5 (1H, d, CH₃malic) 5.7 (1H, d, CH₃) 3.1 (2H, t, CH₂N) 4.7 (H, q, CH) 4.3 (2H, d, CH₂). 13C-NMR (200MHz, CDCl₃) δ (ppm) = 170.5, 170.22, 170.0, 167.3, 167.0, 104.7, 77.0, 76.0, 75.0, 69.0, 62.5, 60.5, 51.0, 21.8, 21.0, 20.0 carbon atoms.

Anal. Calcd. for C₁₇H₂₁N₃O₁₁ (443.3): C 46.05, H 4.74, N 9.48%; Found: C 46.02, H 4.71, N 9.41%.

2.7 Synthesis of cinamyl alcohol triazolin ethyl-D-xylofuranoside
The derivative obtained from compound (4) azido ethyl-D-xylofuranoside (2.00 gm, 0.054 mmol) was dissolved in dioxane (64 ml) and added to cinamyl alcohol (1.7 gm). The mixture was stirred and heated under reflux at (50-60 °C) for 9h. When T.L.C shows completion of the reaction, the mixture was cooled and powered into iced-water. The product was extracted with chloroform (5x15 ml) and dried with anhydrous magnesium sulfate, and chloroform was removed under reduced pressure to give, as syrupy product, the syrupy product compound was purified on silica gel column chromatography using dichloromethane : benzene 3:8 v/v to give 1.22 gm, dark-yellow color, yield 80%, R.F 0.3. FT-IR cm⁻¹: 1600-1650 (C=C aromatic) 3000-3500 (-OH) 1730-1740 (-COOCH₃) 1140 (-C=O-C) 1675 (N=N) 2335 (diazostrech). 1H-NMR (600 MHz, CDCl₃) δ (ppm) = 1.8 (9H, s, 3 CH₃) 4.2, 3.5 (2H, d, CH₂ heterocyclic) 2.8 (2H, t, CH₂N) 3.8-3.9 (2H, t, OCH₂) 4.5 (1H, t, CH) 3.3 (1H, t, CH) 6.2-6.4 (6H, m, phenal) 5.5 (H, s, OH) 2.4-2.5 (H, q, CH) 5.8 (H, d, CH) 2.1 (2H, d, CH₂). 13C-NMR (200MHz, CDCl₃) δ (ppm) = 170.55, 170.22, 170.0, 167.30, 167.00, 104.7, 77.00, 76.00, 75.00, 72.22, 64.40, 62.50, 60.55, 51.00, 21.88, 21.00, 20.00 carbon atoms. Anal. Calcd. for C₁₇H₂₁N₃O₁₁ (443.3): C 46.05, H 4.77, N 9.48%; Found: C 46.02, H 4.71, N 9.41%.

3. Results and Discussion
The objectives of this work is to synthesize and identify some new glycosides of D-xylose in its furanoid forms and then converting them into new triazoline compound by treatment of them with sodium azide. Both the approach and the products are newly emergent facets. Glycosides as nitrogen heterocyclic derivatives of sugar has gained increasing interest in biological and chemical activities for their uses in medication and pharmaceutical industries beside their many other benefits. The target compounds; The derivative 2hydroxy ethyl – O – D – xylofuranosides (1) was synthesized by adaptation of abod method (Daiekh Abod, 1983), which treats D - xyloseose with ethylene glycol in the presence of acidic medium in the under kinetically controlled conditions to ensure the predominance of the furanoid ring. The compound was identified most likely as a mixture of α and β - anomers. The 2hydroxy ethyl – O – D – xylofuranosides (1), FT-IR spectrum, showed a new peak, as compared with D – xylose spectrum, at 3250 - 3400 cm⁻¹ due to OH group.
This peak is considered a characteristic one for the new glycosides as well as for the conversion into triazolinyl derivative to be eliminated by the cycloaddition reactions and showed peak the -CH2- group at 2930cm⁻¹. The peak at 1150cm⁻¹ is due to C-O-C bond of the etheric ring. More evident for the structure of the product was obtained from 1H-NMR spectrum gives further evident for the structure of the compound. There are eighth environment which is agreement with hydrogen environment of the compound. The anomic hydrogen gave signal at 5.8 ppm, which is usually resonate down field due to the anomeric effect and neighbor of etheric group. The signals at 0.7-0.8 ppm are due to 2H of CH2-O attached by hydroxyl. The signal at 2.1 ppm is due to 2H of O-CH2 group attached to the sugar ring. Signal at 1.7ppm are due to 3H of hydroxyl the sugar ring. The signal at 2.5 is due to 1H the hydroxyl of the ethyl. The signals at 1.3 -1.5, 3.2, 4.00, 2.8 ppm are due to 5H of the sugar ring. The 13C-NMR spectrum. There is with us seven carbon atoms resonances which is in agreement with molecular formula. The signal at 110 ppm is due to O-CH2 carbon atom. The signal at 81.33ppm is due to C-OH of the ethyl group. The signal at 77.01,75.,66.70,62.11,61 ppm are due to carbon atoms forming the sugar ring.

The derivative 2-acetyl ethyl 2,3,4 tri-O-acetyl-D-xylofuranoside (2) was synthesized by treating with pyridine and acetic anhydride and converted hydroxyl group to acetate group. The derivative was identified by the FT-IR spectrum showed disappearance absorbing band due to hydroxyl group and showed a strong Peak at 1730 cm⁻¹ for carbonyl of the ester groups, also showed a peak at 1130 cm⁻¹ indicating the present of etheric group of the ring. Peak at 2900 cm⁻¹ is of -CH2- attached by the ethyl group. The 1H-NMR gives further evident for the structure of the compound. Signals at 3.2-3.4ppm are due to 2H of the CH2-OAC of ethyl attached by acetate. Signals at 3.7-3.8ppm are due to 2H of the O-CH2 ethyl group. Signal at 2.3 ppm is due to 9H of the three ester group of the sugar ring. The signals at 0.9 ppm is due to 3H of the ester of the ethyl group. The signals at 5.8 ppm is due to anomic hydrogen, which is usually resonate down field due to the neighbor of etheric group. Signals at 4.3, 5, 3.4, 4.7-4.8, 3.1 ppm are due to 5H of the sugar ring. The 13C-NMR spectrum of the compound showed that, there are fifteen carbon atoms, which is in agreement with the molecular formula of the prepared compound. The signals at 160.33, 160.03, 160.02, 120 ppm are due to four the carbon atoms of the ester groups. Signal at 20, 21, 23 ppm are due to 4 O-CH3 groups. Signals at 104.7, 103.2 ppm are due to carbon atoms of the ethyl group. Signals at 77, 76, 75, 68, 62 ppm are due to carbon atoms of sugar ring. This is the feature expected to the furanoside ring (Daiekh Abod, 1983).

The derivative O-tosyl ethyl -O-D-xylofuranoside(3) was synthesized by treating with p-toluenesulfonyl chloride, after being dissolved in pyridine added drop wise by means of a separation funnel. Tosyliton is taken place selectively an acetyl group of the glycoside probably due to anomic effect. The derivative was identified by the FT-IR spectrum this is showed peaks at 1170-1180cm⁻¹ are due to SO2 sulfonic group. The peak at 1500cm⁻¹ is of the aromatic group. The peak at 1736 cm⁻¹ is due to C=O bond the etheric of the glycosidic moiety. The peak at 2900cm⁻¹ is due to -CH2-terminal. 1H-NMR spectrum showed signal at 7.5-7.9 ppm belong to the 4H of the aromatic group. Signal at 2.0 ppm is due to 9H of the 3O-CH3 group. The signals at 3.7-3.9ppm are due to 2H of the -CH2-OTS group. Signals at 3.2-3.4ppm are due to 2H of the O-CH2-group. The signal at 5.9 ppm is due to anomic hydrogen.
The signals at 4.3-4.4, 5.00, 4.5-4.6, 4.1ppm are due to 5H of the sugars ring. The 13C-NMR spectrum showed that appear twenty carbon atoms which is in agreement with the molecular formula of thy synthesized compound. Signals at 160.30, 160.3, 160.02 ppm are due to carbon atoms of the ester carbonyl groups. Signals at 20, 20.7, 21 ppm are due to carbon atoms of the O-CH3 group. Signal at 138,133,129,128.44 ppm are due to carbon atoms of the aromatic group. Signals at 104.76, 77.00 ppm are due to carbon atoms of the ethyl group. The Signal at 76, 75, 69.99, 64.40, 62.50 are due to carbon atoms of the sugar ring. The derivative azido ethyl-O-D-xylofuranoside(4) conversion of OTS group into azido group was carried out in DMF in presence of NaN3 . The FT-IR spectrum of the derivative Proved the conversion of the OTs group by showing a peak absorption at 1000-1300cm-1is due to C-N bond and showing a peak absorption at 2100-2160cm-1is due to-N3 bond of a zide. The ester group showed a peak at 1730 cm-1, the peak at 1145cm-1is due to C-O-C etheric group. The1H-NMR spectrum of the compound gives good evident for the structure of the compound. There are eight environment which is in agreement with hydrogen environment of the compound. The signals at 2 ppm is due to 9H of the 3O-CH3groups of the ester. Signals at 1.5-1.6, 3.3-3.4 ppm are due to 4H of the ethyl groups. The signals at 5.8ppm are due to anomic hydrogen, which resonate down field because of the anomic effect. Signals at 3.7-3.8, 5.0, 4.5-4.6, 4.1ppm are due to 5H of the sugar ring. The 13C-NMR spectrum of the compound showed thirteen signals the carbon atoms which validated the molecular formula. The signals at160.05, 160.03, 160.02 ppm are due to carbon atoms the carbonyl of the ester. The signals at 20, 20.22, 21.00 ppm are due to carbon atoms of the O-CH3 of the ester group. Signals at 104.7, 77.00 ppm are due to carbon atoms of ethyl group. Signals at76, 75, 64, 62.5ppm are due to carbon atoms of sugar ring.

The derivative benzoquinone triazolin Propyl -O-D-xylofuranoside (5), the FT-IR spectrum of the derivative. The peak at1710 cm-1 is due to -CO- of the carbonyl group in benzoquinone, the peak at1300cm-1is due to C-N. The peak at 1500cm-1is due to C=C of the benzoquinone. The ester group appeared peak at 1735-1740cm-1. The peak at 2344cm-1 is due to diazo group stretch. The Peak at 1145cm-1 is due to C-O-C bond glycoside. The 1H-NMR spectrum gives good evident the structure of the derivative. The signal at 2 ppm is due to 9H of the O-CH3 group. Signals at 2.7-2.9, 3.7-3.8 ppm are due to 4H of the ethyl group. Signals at 3.1,3.5 ppm are due to 4H of the benzoquinone ring. Signal at 5.8ppm is due to anomeric hydrogen. Signals at 4.4-4.5, 4.2, 5.3, 5 are due to 5H of the sugar group. The 13C-NMR spectrum of the derivative showed nineteen signals of carbon atoms which is in agreement with the molecular formula. The signals at 180,179,178 ppm are due to carbon atoms the carbonyl of the ester group. Signals at 20,21, 21.88 ppm are due to three carbon atoms in O-CH3 group. The Signals at 160.44, 160.33, 160.22,160 ppm are due to carbon atoms of the carbonyl group in benzoquinone ring, signals at 104.7, 80.77 ppm are due to carbon atoms of ethyl group. Signals at 77, 76, 75, 72.22, 64.40 ppm are due to carbon atom of the sugar ring. The derivative maleic anhydride triazolinyl ethyl -O-D-xylofuranoside(6), FT-IR spectrum of the derivative. Which showed peak absorption at 1745cm-1is due to C-O-C ether anhydride the hetero cyclic ring. The peak at 1710cm-1of the -CO-carbonyl group in the hetero cyclic ring. The peak at 1300cm-1 is of the C-N bond. The peak at 1730-1735cm-1 is due to ester groups. As well as showed peak at 1140cm-1is due c-o-c ether group of the sugar ring. The peak at 1650cm-1 of the –N=N- bond. The peak at 2340 cm-1 is due to diazo Stretch. The 1H-NMR
spectrum of the derivative, which showed twelve invieroment of hydrogen atoms, which is in agreement with hydrogen invieroment of compound. The signals at 1.7-1.8 are due to 9H of the three O-CH3 groups. The signals at 2.7, 3.5 ppm are due to 2H of the -CH-CH- bond of the hetero cyclic ring. Signal at 5.7ppm is due to anomeric hydrogen, which is usually resonate down field due to the anomeric effect. Signals at 4.9, 3.8-3.9, 4.5-4.7, 5.3, 4.2 ppm are due to 5H of the sugar hydrogen's. Signals at 3.0 - 3.1, 5.5 ppm are due to 4H of the ethyl group. The 13C-NMR spectrum showed seventeen carbon atoms which is in agreement with molecular formula of the synthesized compound. Showed signals at 170.55, 170.22, 170 ppm are due to carbon atom of the ester group signals at 21.88, 21.0, 20.07 ppm are due to carbon atoms of the three O-CH3 groups. Signals at 167.30, 167.009 ppm are due to -CO- of the hetero cyclic. Signals at 76, 75ppm are due to carbon atoms of the ethyl group. Signals at 72.22, 64.40, 62.50, 60.55, 51.00 ppm are due to carbon atoms of the sugar ring.

The derivative cinamyl alcohol triazolinyl ethyl-O-D-xylofuranoside(7), FT-IR spectrum. The peak at 1600-1650cm-1 is due to C=C group, indicating to insertion the aromatic group to the compound. The weak peak at 3000-3500cm-1 is due to OH. The peak at 1730-1740cm-1 of the carbonyl of ester groups. The peak at 1140 cm-1 is due to -o-c group and of the -N=N- group at 1675 cm-1 and showed peak at 2335cm-1 is due to diazo stretch group. The 1H-NMR spectrum of the compound Proves the structure of the derivative. The signal at 1.8ppm is due to 9H of the O-CH3. The signals at 4.2, 3.5ppm are due to 2H of the ethyl of the hetero cyclic. The signals at 2.8 ppm is due to 1H of the CH2-N, 3.8-3.9 ppm are to 1H of O-CH2. The signal at 5.8 ppm is due toanomeric hydrogen. The signals at 6.1-6.3 ppm are due to 5H of phenyl. The signal at 5.5ppm is due to 1H of hydroxyl in hetero cyclic ring. The signals at 4.3, 3.3, 2.4-2.4, 3.1 ppm are due to 5H of the sugar ring. The 13C-NMR spectrum, showed that there are twenty one carbon atoms which is in agreement with molecular formula of the synthesized compound. Showed signals at 170.55, 170.33, 170 ppm are due to carbon atoms of the ester. Signals at 21.77, 21.0, 20.077 are due to carbon atoms of the 3O-CH3. Signals at 140, 129, 128.88, 127, 126.33, 104.7 ppm are due to carbon atoms of the phenyl. Signals at 99.5-77.88 ppm are due to ethyl in the hetero cyclic. Signals at 76, 75 ppm are due to ethyl attached by glycoside bond. Signals at 69, 62.50, 61, 60, 48.22 ppm are due to carbon atom of the sugar ring.
Fig 1 Synthetic route for compound 4
Fig. 2 Synthesized of Compounds 5, 6, 7 from derivative 4
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