

Synthesis and characterization of compounds obtained by the interaction of D- mannose and TAC in distilled water as a solvent

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Abstract : Microwave-assisted organic synthesis has evolved into a potent tool for chemists, providing an effective and efficient method for conducting organic reactions. It has emerged as a convenient and valuable tool for chemists, optimized and enhancing various organic synthesis processes in the recent past. In the present work, the products obtained by the oxidation of D – Mannose in distilled water as a solvent by ditertiary amyl chromate (TAC) in varying molar ratios by microwave dielectric heating conditions are studied and the interesting result are formed. The compounds were analyzed using a combination of chemical and instrumental techniques, such as Elemental analysis, Fourier Transform Infrared Spectroscopy (FTIR), and Differential Thermal Analysis/Thermogravimetric Analysis (DTA/TGA) to assess mass loss patterns. Additionally, the chromium content was determined through volumetric analysis. The reaction clearly increases the advantages of Microwave Assisted Organic Synthesis (MAOS) and conventional synthetic methods with respect in reaction efficiency and product yield, less pollution and higher energy efficiency. It can lead to cleaner reactions, minimizing side products and impurities in the final product.

Keywords : Microwave, D–Mannose, Ditertiary amyl chromate (TAC), Distilled water (DW), Tertiary amyl alcohol (TAA) and FTIR spectroscopy, TGA-DTA.

Introduction

The initial identification of microwave dielectric technique as an energy source is credited to Percy Spencer, who made this discovery in 1947¹. R. Gedye et.al²⁻³ has the credit

in 1986 for introducing microwave dielectric heating in organic synthesis and now there are more than 2000 papers that describe the applications of this technique for the synthesis of new compounds. Microwave Assisted Organic Synthesis (MAOS) and Microwave Induced Organic Reaction Enhancement (MORE) chemistry have gained popularity as a non-conventional eco-friendly technique for rapid organic synthesis in conformity with 12 principles of green chemistry⁴. In comparison to conventional heating microwave irradiated organic reaction proceeds much faster, greater efficiency, better yield and less pollution with less energy consumption. P. Lidstrom et.al⁵ have reported a detailed survey of MAOS and MORE and also have a detailed review of the organic reactions carried out by the application of microwave. Keeping in view, the increasing importance of MAOS and MORE in organic synthesis, the author has undertaken the oxidation of D-Mannose in distilled water as a solvents with ditertiary amyl chromate by microwave heating. In many cases, microwave assisted organic techniques have proved to be more efficient than classical heating methods⁶⁻⁹.

Tertiary amyl chromate (TAC) has proven to be a potent and versatile oxidizing agent. TAC is used as an oxidant in organic synthesis is substantiated by a vast work carried out after its first use by R.V Oppenauer and H. Oberrauch¹⁰⁻¹³. The oxidation products of organic

substrates formed may serve as ligands to give adducts or complexes of chromium in different oxidation states¹⁴⁻¹⁵.

D- Mannose is an aldohexose series of carbohydrates. It is a sugar monomer and C-2 epimer of glucose¹⁶

The two different – sized rings are present in Mannose structure such as pyranose (six membered) form and the furanose (five membered) form. One aldehyde group and five hydroxyl group are present in structure. It occurs in plants and animals as a form of microbes. It is also found in many fruits such as apples, oranges and peaches in small amount. It is used as an antibiotic for preventing UTI (Urinary tract infectious)¹⁷. In this present paper, we have reported the synthesis and study of some complexes of chromium in lower oxidation states with D- Mannose by oxidizing with TAC to explore the versatility of oxidizing agent and expand the details of D- Mannose in the field of chemistry.

Materials & Methods

Chemical used:

The chemicals used were all A.R grade. Only freshly prepared solutions were used. In the present work the chemicals used were D- Mannose, DW (Distilled water), TAA (Tertiary amyl alcohol), acetone, Chromium (VI) oxide, silver nitrate, potassium persulphate, ammonium iron (II) sulphate (Mohr's salt), potassium dichromate, barium diphenylamine-4-sulphonate.

Procedure

1. Preparation of oxidant:-

Preparation of TAC: -In the present work the oxidant TAC which was prepared in situ by dissolving calculated quantity of pure and dry CrO_3 in tertiary amyl alcohol (TAA).

2. Preparation of substrate in different molar ratio: -

a. Sample code MDW12:

Substrate/oxidant ratio: 1.8g D- mannose was dissolved in 10ml of distilled water and 2gm of CrO_3 dissolved in 10 ml of tertiary amyl alcohol to prepare tertiary amyl chromate.

b. Sample code MDW11:

Substrate/oxidant ratio: 1.8g D- mannose was dissolved in 10ml of distilled water and 1gm of CrO_3 dissolved in 10 ml of tertiary amyl alcohol to prepare tertiary amyl chromate.

c. Sample code MDW21:

Substrate/oxidant ratio: 1.8g D- mannose was dissolved in 10ml of distilled water and 0.5gm of CrO_3 dissolved in 10 ml of tertiary amyl alcohol to prepare tertiary amyl chromate.

3. Experimental Process

D-Mannose, a water-soluble carbohydrate compound, exhibited excellent solubility in distilled water, readily dissolving to form a homogeneous solution. Oxidation of this substrate with TAC was performed in three different substrates: oxidant molar ratio 1:2, 1:1, 2:1. The solution of the substrate was prepared in distilled water. The solution of the oxidant, i.e., TAC was prepared separately in situ from chromium trioxide and tertiary amyl alcohol. The two solutions were then mixed together to prepare a homogenous reaction mixture, stirred continuously for a considerable time using a magnetic stirrer [REMI RS-12 Rotatory Shaker with hot plate] and any change in consistency was noted. The mixture was then heated for the periods in an LG MG-3937C 20 litre, 2450MHz 750Watt Microwave Oven (MW irradiation 160W, variable oxidation time) as mentioned in given table no. 1.

The solid products of different colour and composition were obtained in all the three compounds which were washed with acetone until clear washing, dried and collected in labelled as MDW12, MDW11, MDW21. Initial and final temperature was recorded of the reaction mixture to specify the exothermic/ endothermic nature of the reaction under investigation.

4. Estimation and characterization

The three compounds were used for further analysis and characterization. Percentage composition of C, H and O was determined using Euro EA Elemental Analyzer while the chromium

content in this sample was estimated volumetrically using $K_2S_2O_8$ (excess), 0.1 N $K_2Cr_2O_7$ solution and 0.1 N Mohr's salt solution.

Empirical formula of these complexes was then deduced. The recorded details have been summarized in Table -1& Table -2.

Characateristics of the products of D – Mannose with TAC

Table -1 (Reactant)

Product code	Substrate	Solvent for the substrate	Oxidant	S:O ratio	Heating condition	Time	Yield
MDW12	D-Mannose	DW	TAC	1:2	160W	3 min. 40 sec.	6.05g
MDW11	D-Mannose	DW	TAC	1:1	160W	7 min. 30 sec	4.72g
MDW21	D-Mannose	DW	TAC	2:1	160W	10min. 30 sec	3.12g

Table -2 (Product)

Product code	Colour	Solubility in water	Empirical formula	Proposed formulation of the product
MDW12	Deep Brown	Sparingly soluble	$Cr_2C_3H_8O_{11}$	$Cr_2O_3 \cdot (HCOOH)(C_2H_2O_4) (H_2O)_2$
MDW11	Brown	Insoluble	$CrC_3H_{10}O_{12}$	$CrO \cdot (HCOOH)_2(C_3H_2O_5) (H_2O)_2$
MDW21	Light brown	Insoluble	$CrC_4H_{12}O_{12}$	$CrO_2 \cdot (HCOOH)(C_3H_4O_5) (H_2O)_3$

Results and Discussion

1. Effect of Substrate/Oxidant Ratio:

a) The substrate oxidant molar ratio effects the characteristics and nature of the compounds /complexes formed. When the molar concentration of the oxidant for each reaction set increased the colour of compound was intensified which indicated the increased percentage of chromium in the product formed by using distilled water as a solvents. Further the elemental analysis of the sample was also evident from empirical formulae.

b) When we increase the proportion of oxidant, it will take less time to form product and the yield of the product will be high.

c) When we decrease, the proportion of oxidant, it will take more time to form product and the yield of the product will be less.

2. Effect of solvent:

The reaction performed in Distilled water was found endothermic in nature. Comparing the reaction time and corresponding yield of the products, it can be concluded that the most efficient sample MDW12 was the most efficiently prepared complex of D-Mannose, with low time of formation and a good yield. The product formed using more oxidant have more yield are their colours is more intensified.

3. Characteristics of complex/compound:

- a) The colour of the products changes from light brown to brown or deep brown on increasing the ratio of oxidant which may be due to different oxidation states of chromium in different products.
- b) The oxidation products of D-Mannose with TAC such a MDW12 is sparingly soluble, & MDW11, MDW21 are insoluble in water.
- c) The extent of oxidation of the substrate increases as the ratio of the oxidant increases and vice versa.
- d) The ratio of oxidant is maximum in the sample no. MDW12, it will take less time to form

product and the yield of product will be high and the most stable oxidation state (III) is shown in the form of Cr_2O_3 . The ratio of oxidant is minimum in the sample no. MDW21, it will take more time to form product and the yield of product will be less and the oxidation states of chromium is (II) in the form of CrO .

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