IJARSCT

International Journal of Advanced Research in Science, Communication and Technology

International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal

ISSN: 2581-9429

Volume 5, Issue 1, July 2025

Impact Factor: 7.67

Microwave Assisted Synthesis and Characterization of D- Mannose by Ditertiary Amyl Chromate in DMSO Solvent

Kriti kumari[1] and Hari Om Pandey[2]

Research scholar, Deptt. of Chemistry, Ranchi University, Morabadi Ranchi, Jharkhand, India[1]

Associate prof. Deptt. of Chemistry, Ranchi University, Morabadi Ranchi, Jharkhand, India[2]

kkriti51@gmail.com and hopandey01@gmail.com

Abstract: *The oxidation of D-Mannose with TAC is a convenient and very simple method which has been discussed. Chromium (VI) based oxidants such as TAC have been used for variety of synthetic works in organic chemistry. The effects of solvent DMSO have been studied under microwave irradiation as a source of energy. Studies were carried out to produce interesting result for determining the properties of obtained product. The product formed by the interaction of organic substrates like D-Mannose in different molar ratio with TAC may give valuable clue their mechanism of the reaction and the structure of the compounds formed were studied. The thermal loss pattern may be helpful for the formulation of complexes and assessing the strength of bonds. In the present work, we have carried out the oxidation of D-Mannose with TAC in DMSO solvent have been carried out under microwave irradiation conditions. The obtained products have undergone analysis using various techniques such as chemical analysis, spectroscopy, and thermogravimetry. The goal of these analyses was to identify patterns or make general observations about the properties or composition of the products.*

Keywords: Di tertiary amyl alcohol (TAC), Carbohydrates (Mannose), chromium trioxide tert. amyl alcohol (TAA), FTIR, TGA-DTA

I. INTRODUCTION

A large no. of CrO_3 (VI) based oxidants like chromium trioxide-3,5-dimethyl pyrazole complex[1], N-Methyl pyridinium chlorochromate[2], Dipyridine chromium oxide[3], 2,2'- Bipyridinium chlorochromate (BIPCC)[4],Collin's reagent[5], Tetramethyl ammonium fluorochromate (TMAFC)[6] ,TBC, TAC etc. have been developed. TBC has been in use since it was reported by Oppenaeur[7]. TAC which has also been the best versatile and efficient oxidant for organic substrates. Promoting the use of microwaves over conventional heating aligns with the 12 principles of green chemistry, providing a more environmentally friendly approach. The organic compound of D-Mannose (substrate) is an easily oxidizable. The expected oxidation products of D-Mannose are formic acid, Oxalic acid, Tartronic acid etc. and in the addition to Mesoxalic acid, glycolic acid etc. which may serve as ligands to give adducts or complexes of chromium in different oxidation states.

D- Mannose is a white crystalline solid compound which is soluble in water. It is monosaccharide belonging to the aldohexose series of carbohydrates and represents a structural isomer of glucose[8], differing specifically at the C-2 carbon position. In Fischer projection structure one aldehyde gr. Present in C-1 position and five hydroxyl gr. is present. It is used for an inherited disorder called carbohydrate- deficient glycoprotein. It acts as a preventive measure against certain types of bacteria adhering to the urinary tract also prevents walls, thereby reducing the likelihood of infection. 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. Biologically, mannose is important in several cellular pathways. It is involved in the mannose-6-phosphate pathway, which is vital for the targeting of enzymes to lysosomes [9]. Additionally, emerging

DOI: 10.48175/IJARSCT-28468

research suggests that mannose may influence cancer cell metabolism and immune responses, making it a candidate for cancer therapeutics[10].

II. MATERIALS AND METHODS

Chemical used: - D-Mannose, DMSO (Dimethyl sulphoxide), TAA (Tertiary amyl alcohol), acetone, CrO_3 (VI) oxide, $AgNO_3$ (silver nitrate), potassium persulphate, ammonium iron II sulphate - Mohr's salt, $K_2Cr_2O_7$ (potassium dichromate), barium diphenylamine-4-sulphonate.

Experimental process: - The oxidant TAC was synthesized by dissolving a precise amount of pure and dry CrO_3 in tertiary amyl alcohol (TAA) in the first beaker. This resulted in the formation of a clear brown solution. In a separate second beaker, a calculated quantity of D-mannose was dissolved in 10 ml of dimethyl sulfoxide (DMSO). The combination of the two solutions, a uniform reaction mixture was formed. This blend underwent continuous stirring for an extended period, aided by the use of a magnetic stirrer- more precisely, the REMI RS-12 Rotatory Shaker with a hot plate. Throughout this duration, careful attention was given to monitor and document any changes in the consistency of the mixture. The prepared mixture underwent heating in an LG MG-3937C 20-litre, 2450MHz, 650Watt Microwave Oven, with microwave irradiation set at 160W and a variable oxidation time as specified in the provided Table No. 1. Distinct solid products, varying in color and composition, were yielded in the case of all three compounds. These products underwent thorough washing with acetone until the washings were clear, followed by a drying process. The purified products were then collected and stored in an airtight glass bottle labelled as MDO12, MDO11, and MDO21, respectively. It appears that the water-soluble carbohydrate compound D-Mannose exhibited good solubility in dimethyl sulfoxide (DMSO). The oxidation of D-Mannose was carried out using TAC in three different scenarios with varying oxidant molar ratios: 1:2, 1:1, and 2:1.The initial and final temperatures of the reaction mixture were carefully noted to elucidate the exothermic or endothermic nature of the investigated reaction. The three compounds underwent further analysis and characterization. The percentage composition of carbon (C), hydrogen (H), and oxygen (O) was determined using the Euro EA Elemental Analyzer. Simultaneously, the chromium content in the samples was estimated volumetrically, employing $K_2S_2O_8$ in excess, 0.1 N $K_2Cr_2O_7$ solution, and 0.1 N Mohr's salt solution. Based on these findings, the empirical formula of the complexes was deduced. A summary of the recorded details has been presented in Table-1 and Table-2.

III.RESULTS & DISCUSSION

Table no. -1

Product code	Substrate	Solvent for the substrate	Oxidant	S:O ratio	Heating condition	Time	Yield
MDO12	D-Mannose	DMSO	TAC	1:2	160W	2 min. 20 sec.	2.51g
MDO11	D-Mannose	DMSO	TAC	1:1	160W	4 min. 30 sec	2.20g
MDO21	D-Mannose	DMSO	TAC	2:1	160W	7 min. 20 sec	1.38g

Table no. -2

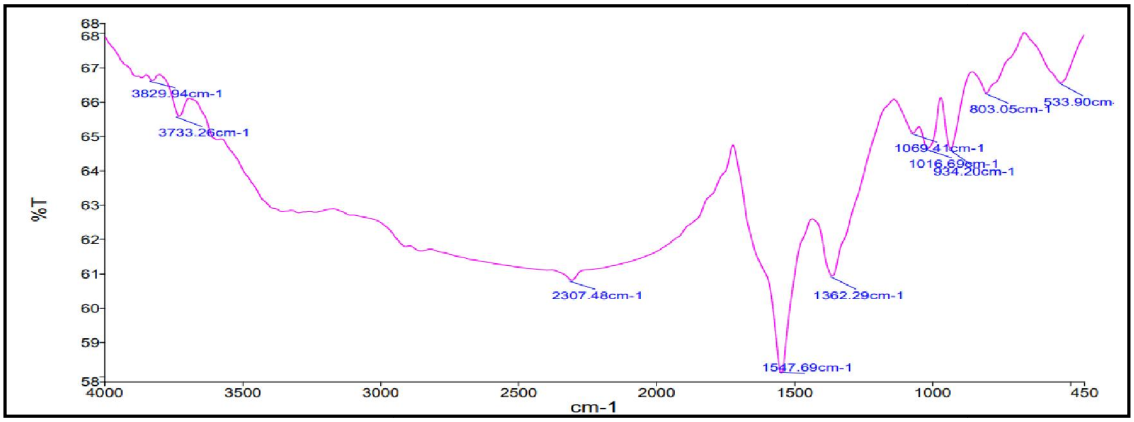
Product code	Colour	Solubility in water	Empirical formula	Proposed formulation of the product
MDO12	Greenish Brown	Sparingly soluble	$Cr_2C_5H_{12}O_{14}$	Cr_2O_3. $(HCOOH)_2$ $(C_3H_4O_5)$ $(H2O)_2$
MDO11	Light green	Sparingly soluble	$Cr_2C_6H_{14}O_{16}$	Cr_2O_3. $(HCOOH)_3$ $(C_3H_4O_5)$ $(H2O)_2$
MDO21	Dull green	Soluble	$Cr_2C_5H_{14}O_{14}$	2CrO. $(HCOOH)_2$ $(C_3H_4O_5)$ $(H2O)_3$

DOI: 10.48175/IJARSCT-28468

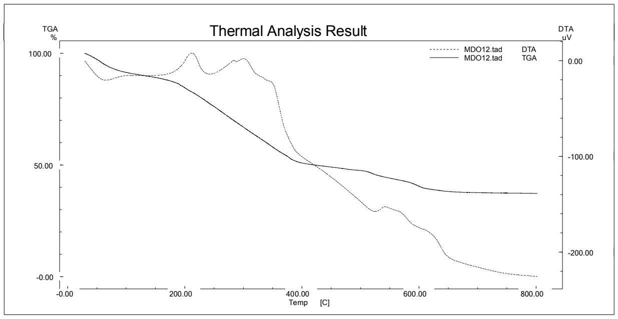
1. The three complexes derived from the oxidation of D-Mannose with TAC in DMSO solvents exhibited distinct physical characteristics, including variations in color and solubility.
2. The reaction performed in DMSO were exothermic. Comparing the reaction time and corresponding yield of different substrate: oxidant molar ratio of products, it can be concluded that the sample MDO12 was most efficiently prepared sample of D- Mannose with low time of formation and good yield.
3. The substrate oxidant ratio also affects the nature and characteristics of the products formed. With increase in molar concentration of the oxidant for each reaction set intensification of the colour of the products was performed and this may be indicative of the increase percentage of chromium in the sample. This was confirmed from the elemental analysis of the sample as evident from the empirical formula of the compound. It may be due to different oxidation states of chromium in different products.
4. The colours observed in the products obtained through the oxidation of D-Mannose by TAC suggest the formation of complexes in different oxidation states.
5. The predominant observation of a green color in the synthesized products suggests that it is likely a result of charge transfer phenomena rather than electronic transitions.
6. The higher the ratio of the oxidant, the more pronounced the oxidation of the substrate, and conversely, a decrease in the oxidant ratio corresponds to a reduction in the extent of substrate oxidation.
7. When we increase the proportion of oxidant, it will take less time to form product and the yield of the product will be high and it will be shown in the sample no. is MDO12.
8. When we decrease, the proportion of oxidant, it will take more time to form product and the yield of the product will be less and it will be shown in the sample no. is MDO21.
9. For sample MDO12, where the oxidant ratio is at its maximum, the most stable oxidation state observed is III, represented by the formation of Cr_2O_3. In contrast, for sample MDO21, the oxidation state of chromium is II, in the form of CrO.
10. The MDO12, MDO11 products are characterized by limited solubility, indicating that it dissolves only to a small extent in a given solvent. On the other hand, the MDO21 product is soluble, indicating that it readily dissolves in the solvent under the experimental conditions.

FTIR & Thermogravimetric Analysis

MDO12



DOI: 10.48175/IJARSCT-28468

Peaks	Group Assignments	TGA-DTA Graph
3829.94	O-H Stretching vibration	
3733.26	O-H Stretching (free)	
2307.48	C-O Stretching	
1547.69	COOH Coordinated	
1362.29	C-H bending	
1069.41	Vibration of C-O in alcohol	
1016.69	C-C stretching	
934.20	Coordinated Water	
803.05	O-H Stretching for water	
533.90	Cr-O stretching	**MDO12**

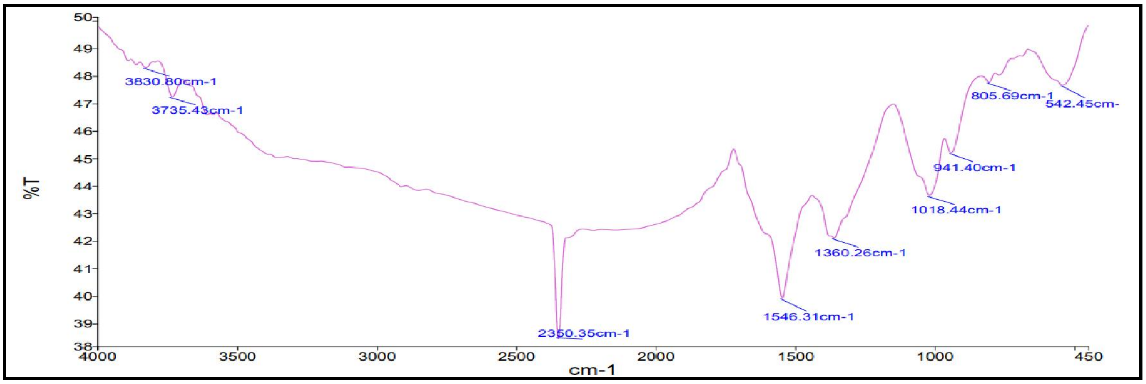
The FTIR spectra exhibit nearly all the expected peaks corresponding to the formulation and is further supported by thermogravimetric analysis, providing strong support for its chemical composition and structural characteristics.

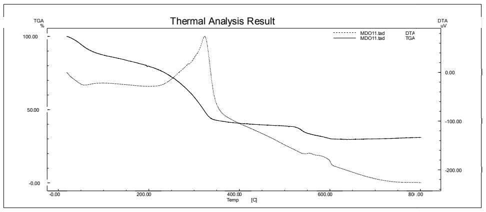
(1). ***Thermogravimetric loss pattern of MDO12***

Temperature	**Formulation Sequence showing changes**	**Percentage Loss**	
		Experimental	**Theoretical**
	$Cr_2O_3(HCOOH)_2(C_3H_4O_5)$ (H2O)$_2$		
0-180°C	↓ Loss of HCOOH, H_2O	17.14	16.05
	Cr_2O_3(HCOOH)(C3H4O5) (H2O)		
180-360°C	↓ Loss of $C_3H_4O_5$, H_20	35.71	34.65
	Cr_2O_3(HCOOH)		
360-620°C	↓ Loss of HCOOH	12.85	11.55
	Cr_2O_3 Chromium and its oxides		
		34.30	37.75

DOI: 10.48175/IJARSCT-28468

MDO11



Peaks	Group assignment	TGA-DTA Graph
3830.80	O-H stretching vibration	
3735.43	O-H stretching free (alcohol)	
2350.35	O-H Stretching (Carboxylic acid)	
1546.31	COOH Coordinated	
1360.26	C-H Bending	
1018.44	C-C stretching	
941.40	Coordinated Water	
805.69	O-H Stretching	
542.45	Cr-O stretching	**MDO11**

The FTIR spectra contain almost all the peaks which are expected and this formulation is strongly supported by thermogravimetric analysis also.

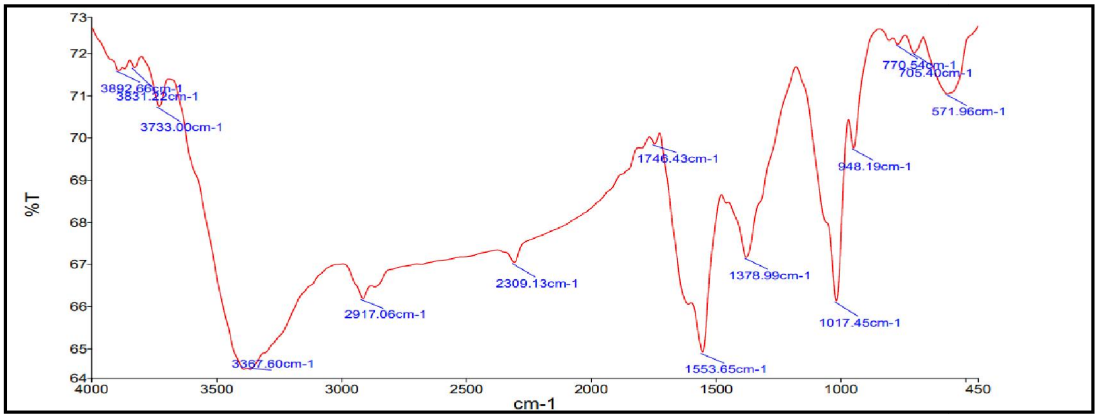
(2) ***Thermogravimetric loss pattern of MDO11***

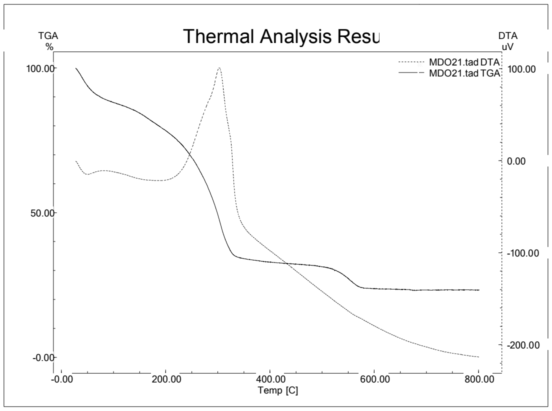
Temperature	**Formulation Sequence showing changes**	**Percentage Loss**	
		Experimental	**Theoretical**
0-80°C	$Cr_2O_3(HCOOH)_3(C_3H_4O_5)\ (H2O)_2$ ↓ Loss of HCOOH $Cr_2O_3(HCOOH)_2(C_3H_4O_5)(H_2O)_2$	11.42	10.31
80-320°C	↓ Loss of $C_3H_4O_5$, HCOOH, H_20 $Cr_2O_3(HCOOH)\ (H_2O)$	42.85	41.24
		5.71	4.03

DOI: 10.48175/IJARSCT-28468

320-540°C	↓ Loss of H_2O Cr_2O_3 (HCOOH)	10.0	10.31
540-620°C	↓ Loss of H_2O Cr_2O_3 Chromium and its oxides	30.02	34.11

MDO21



Peaks	Group assignment	TGA-DTA Graph
3733.00	O-H stretching intermolecular bonded	
3367.60	O-H stretching carboxylic acid	
2917.06	C-H Stretching for alkane	
2309.13	C-O Stretching	
1746.43	C=O stretching (aldehyde)	
1553.65	COOH Coordinated	
1378.99	C-H Bending	
1017.45	C-C Stretching	
948.19	Coordinated water	
770.54	Cr-O Stretching	
705.40	Cr-O Stretching	
571.96	Lattice Water	**MDO21**

The FTIR spectra exhibit nearly all the expected peaks corresponding to the formulation and is further supported by thermogravimetric analysis, providing strong support for its chemical composition and structural characteristics.

3*) Thermogravimetric loss pattern of MDO21*

Temperature	**Formulation Sequence showing changes**	**Percentage Loss**	
		Experimental	**Theoretical**
	2CrO $(HCOOH)_2(C_3H_4O_5)$ $(H2O)_3$		
0-160°C	$\downarrow$ Loss of HCOOH	12.85	16.05
	2CrO (HCOOH)$(C_3H_4O_5)$ $(H2O)_3$		
160-320°C	$\downarrow$ Loss of $C_3H_4O_5$, HCOOH, H_20	47.14	45.76
	Cr_2O_3(HCOOH)		
320-580°C	$\downarrow$ Loss of H_2O, H_2O	10.00	9.17
	2CrO Chromium and its oxides		
		30.01	33.63

IV. CONCLUSION

In conclusion, the microwave-assisted synthesis and characterization of D-Mannose using ditertiary amyl chromate (TAC) in DMSO (dimethyl sulfoxide) solvent have resulted in a successful and efficient method for obtaining the desired compound. The utilization of microwave dielectric heating has proven to be a highly efficient and rapid method for the synthesis of D-Mannose, demonstrating advantages in terms of reaction time and overall yield. The characterization of the synthesized product involved a comprehensive analysis, including elemental analysis and FTIR. These analytical techniques have not only confirmed the successful synthesis of D-Mannose but also provided valuable information about its elemental composition and functional groups.

Furthermore, the DTA-TGA data obtained has contributed insights into the thermal behaviour and stability of the synthesized D-Mannose. This challenging approach to characterization enhances our understanding of the chemical and physical properties of the product. This work contributes to the expanding field of microwave-assisted organic synthesis and provides a foundation for further exploration and applications in carbohydrate chemistry.

REFERENCES

[1]. Corey E. J. and Fleet, G.W.J (1973): Chromium trioxide- 3,5- Dimethyl pyrazole Complex as a reagent for oxidation of Alcohols to Carbonyl compounds, Tetrahedron Lett. , **4499.**

[2]. Tajbakhsh, M; Heravi, M. M.; Mohanzadeh, F.; Sarabi S; Ginassemzadeh, M. M.: Methyl piperidinium chlorochromate absorbed on Alumina: A New Deoximation Reagent. Montash Chem. **(2001)**, 132, 1229 – 1231.

[3]. Collins, J.C. and Frank, F.J. **(1986)**: Dipyridine Chromium (VI) oxide: Oxidation of alcohol in Dichloromethane, Tetrahedron Lett. 3363.

[4]. Guizice, F.S; Luzzio, F.A: The oxidation of Alcohols using 2,2'- Bipyridinium Chlorochromate, Synthesis **(1980).**69

[5]. *Collins*, J.C.; Hess, W.W.; Frank, F.J. Tetrahedron Lett.3363, **1968**

[6]. Kassaee, M. K.; Mahjoub, A. R.; Ghammami, S. Tetrahedron Lett. 44, 4555-4557, **2003**.

[7]. Oppenaeur R.V, Oberrauch H, (Univ. Innsbruck, Austria), Anaus Assoc. Guin-Argentina **1949**, 37, pp 246-62.

[8]. http://en.wikipedia.org/wiki/Mannose

[9]. Freeze, H. H. (2006). Genetic defects in the human glycome. **Nature Reviews Genetics**, 7(7), 537–551. https://doi.org/10.1038/nrg1894.

[10]. Gonzalez, P. S., O'Prey, J., Cardaci, S., et al. (2018). Mannose impairs tumour growth and enhances chemotherapy. **Nature**, 563(7733), 719–723. https://doi.org/10.1038/s41586-018-0729-3

DOI: 10.48175/IJARSCT-28468