J. Chemtracks 23 (1&2) 141-144, 2021

Abstracted / Indexed in
A division of the American Chemical Society
Chemical Abstract U.S.A.

ISSN 0973-239X
CODEN:JCOHF7
IMPACT FACTOR : 4.950

Microwave assisted synthesis and study of some chromium (III) complexes of Resorcinol with TBC

Chanda Kumari[1] and Hari Om Pandey[2]

[1]Department of Chemistry,C.I.T,Ranchi, Jharkhand

[2] Associate Professor, Department of Chemistry, Ranchi University, Ranchi-834008

Email : chandakumari007@gmail.com

Manuscript received online 20 April 2021, accepted on 29 July 2021

Abstract: Resorcinol dissolved in different solvents, namely tetrahydrofuran, I,4-dioxane etc. when oxidized with TBC in varying molar ratios e.g. 1:2,2:2,3:1, gave nine Cr(III) complexes which were analyzed for elemental composition and characterized by FTIR spectroscopy and thermogravimetric analysis(DTA TGA). The growing popularity of microwave technique is attributed to accelerated reaction rates, greater selectivity, milder reaction conditions and quicker synthesis. These reactions offer a novel methodology for oxidation of organic compounds.

(Key words : TBC,I,4-dioxane, FTIR, spectroscopy DTA, and TGA.)

Introduction

Resorcinol (m-dihydroxybenzene) is a phenolic chemical compound having chemical formula, $C_6H_6O_2$ with molar mass of 110.7g/mole. It is is used widely in industries for the production of pharmaceuticals, resins, plastics, medicine, cosmetics etc.[1-8] Resorcinol does not occur naturally in free state but it can be abstraced from argon soil.

Di-tertiarybutyl chromate(TBC) is a versatile oxidant. it is prepared by dissolving CrO_3 in tertiary butyl alcohol(TBA).The compound was prepared for the first time by Wienhaus but R.V Oppenauer. H.Oberrauch introduced it as a new oxidizing agent in 1949.

Chemical Used :

AR-Grade chemical, i .e., 1,4- Dioxane, tert- Butyl alcohol, Chromium trioxide, acetone etc has been used.

Experimental

The oxidation of resorcinol with TBC was carried out in different molar ratios in the solvent 1,4-dioxane in microwave oven at different wattage for different duration.

(a) Oxidant : Substrate (1:1) –R_1

1 gm of CrO_3 was dissolved in 10 ml of TBA to get the oxidant TBC. In another beaker, 1 gm of resorcinol was taken and dissolved in 10 ml of dioxane. The two solution were mixed well and heated in microwave oven at 160^0C for 90 sec. A brown coloured compound was obtained which was filtered and washed with acetone.

(b) Oxidant : substrate (1:2) –R_2

1 gm of CrO_3 was dissolved in 10 ml of TBA to get the oxidant TBC. In another beaker, 2.2 gm of resorcinol was taken and dissolved in 10 ml of dioxane. The two solution were mixed well and heated in microwave oven at 16^0C for 200 sec. The reddish brown colored product appeared which was filtered and washed with acetone and dried.

(c) Oxidant: Substrate (1:3)-R_3

1 gm of CrO_3 was dissolved in 10 ml TBA to get the oxidant TBC. 3.3 gm of resorcinol was dissolved in 10 ml of dioxane. These two solutions were mixed well and the mixture was heated in a microwave oven at 16^0C for 100 sec to get a dark brown solid compound. It was washed several times with acetone and collected.

Table – 1

FTIR Results (R_1)

PEAK	NATURE OF PEAK	GROUP ASSIGN
335.94	weak	O-H Stretching
1600.92	sharp	C=C stretching
1369.46	medium	O-C
1307.74	weak	O-H
1242.16	medium	C-O stretching
1172.72	weak	C-O stretching
1153.43	weak	C-O stretching
968.27	medium	C-O stretching
844.82	weak	O-H
771.53	weak	C-H
570.93	weak	M-O
536.21	weak	M-O stretchings

Table -2

FTIR results (R_2)

PEAK	NATURE OF PEAK	GROUP ASSIGN
3294.42	Weak	O-H
2893.22	Broad	O-H
1604.77	Sharp	C=O
1489.05	Sharp	C-O-O
1369.46	Medium	O-C
1300.2	Medium	O-C
1230.58	Weak	C-O
1149.57	Weak	C-O
1072.42	Weak	C-O
964.41	Medium	C-O
844.82	Weak	O-H
771.53	Weak	O-H
680.66	Medium	M-O
532.35	Weak	M-O
459	Weak	M-O

Table-3

FTIR Results (R_3)

PEAK	NATURE OF PEAK	GROUP ASSIGN
3298.28	Weak	O-H
2889.37	Broad	O-H
1604.77	Sharp	C=O
1489.05	Sharp	C-O-H
1369.46	Medium	O-C
1300.02	Medium	O-C
1222.87	Weak	C-O
1149.57	Weak	C-O
1072.42	Weak	C-O
964.41	Medium	C-O
844.82	Weak	C-O
771.53	Weak	O-H
686.66	Medium	M-O
540.07	Weak	M-O

Table-4

Thermogravimetric results (R_1)

Temperature	Formulation sequence showing the change	Percentage Theoritical	Loss percentage
31.4 -150^{0}C	$Cr_2(CH_3CO_2)_4(H_2O)_2$ |-$2H_2O$	16.68	16.54
150 -460^{0}C	$Cr_2(CH_3CO_2)_4$ |-$2(CH_3CO_2)_4$	47.55	47.73
460 -600^{0}C	Mixed oxides of Cr		

Thermogravimetric results (R_2)

Temperature	Formulation sequence showing the change	Percentage Theoritical	Loss percentage
31.98 -140^{0}C	$2CrO.2H_2O.CH_2COOH.CH_2(COOH)_2$ |CH_2COOH	17.85	17.50
140 -360^{0}C	$2CrO.2H_2O.CH_2(COOH)_2$ |$2H_2O$	10.55	12.73
360 -700^{0}C	$2CrO.CH_2(COOH)_2$ |$CH_2(COOH)_2$2CrO and other oxides	30.87	29.34

Results and Discussion

The FTIR curves and DTA TGA analysis support the following formulation for the sample R_1, R_2 and R_3.

R_1—$Cr_2(CH_3CO_2)_4(H_2O)_2$

R_2—$2CrO.2H_2O.CH_2COOH.CH_2(COOH)_2$

R_3—$Cr_2O.CH_3COOH.CH_3COCOOH.2H_2O$

The FTIR Curve (table-01)of sample R1 contain almost all the peaks which are expected for the formulation $Cr_2(CH_3CO_2)_4$ $(H_2O)_2$.The expected peak for O-H stretching at 33.594cm^{-1},C-H stretching at 771.53 cm^{-1},O-C stretching at 1369.46 cm^{-1} and M-O stretching at 536.21cm^{-1} are all present in the curve.

Similarly, the proposed formulation of sample R_2,on the basis of empirical formulation, is strongly supported by the FTIR curves as well as TGA-DTA analysis $2CrO.2H_2O.$ $CH_2COOH.$ $CH_2(COOH)_2$

The expected broad peak for O-H stretching (H-bonded) at 3294.94 cm^{-1}, bidendate carboxylic acid functioning as ligand at 1489.05.cm,C-O stretching at 1230.58 cm^{-1},O-C stretching at 1369.46 cm^{-1} and M-O stretching at 680.66 $cm^{-1}$are present in the curve.Similarly, the loss pattern is just what we expect for the formulation as shown.we observe that the FTIR curves (table -3) of sample R_3 contain almost all the peaks which are expected for its formulation. We observe that the FTIR curves contains almost all the peaks which are expected for its Formulation $Cr_2O.CH_3COOH.CH_3COCOOH.2H_2O$ .It is supported by the peaks at 3298.28cm^{-1} (O-H stretching),964.41cm (C-O-H stretching) 1489.05 cm^{-1} (bidentate carbxylate ligand),844.82 cm^{-1} (-COOH co–ordinated), 964.41.cm^{-1}(C-O stretching) etc.Also the loss pattern in TGA-DTA curve supports the proposed formulation.The First stage loss between the temperature range 31.98-140^0 C corresponds to the fragmentation of CH_3COOH. The second stage loss of lattice water. The substrate might have been co-ordinated through two COOH groups,as the loss is taking at higher temperature.

Acknowledgements

The authers are thankful to the ISM Dhanbad department of Chemistry for FTIR spectroscopic measurements and BIT Mesra for TGA/DTA Analysis

References

1. D Adam, Nature, 421, 571, 2005
2. C.O Kappe, Angew. *Chem.Int. Ed,* **43**, 6250 (2006)
3. G.D.Mishra,H.O.Pandey and Reshma Parveen, *J.Chemtrack*,**10** (1&2),61-66(2008)
4. Neeraj, Anil kumar Pandey and GD. Mishra, *J. Chemtracks* **11**(2), 2009
5. Sreerupa Roy, H.O.pandey,International Reaserch Journal of Engineering and Tecnology,Vol 03, Issue 02, Feb (2016)
6. G.H. Jeffery, J.Bassett,J.Mendhan and R.C Denny, 5th ed, ELBS, 377,(1989).
7. A.Barik.K.I Priyadarsini,Hari Mohan,Radiation physics and Chemistry 70,pp.687 (20004)
8. G.D Mishra,H.O pandey and Reshma Parveen, *J.Chemtracks*,**10**(1&2), 61-66, 2008