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Microwave assisted synthesis and study of some chromium (III) complexes of p-cresol with TBC

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Abstract- p-cresol dissolved in tetrahydrofuran and I,4 dioxane, when oxidized with TBC in varying molar ratios 1:1, 1:2, 3:1, gave six Cr (III) complexes which were analyzed for elementary composition and characterized by FTIR spectroscopy. The Process of oxidation was carried out in micro-oven for different time interval and at different wattage.

Key words: p-cresol, TBC, FTIR spectroscopy,

INTRODUCTION

p-Cresol is a phenolic chemical compound having chemical formula C_7H_8O with molar mass of 108.14g/mole which is used widely in industries like pharmaceuticals, leather and cosmetics industries.[1,2] p-Cresol has been used for over past 120 years in the production of resins, plastic, medicines, dyes and other materials.[3-6] TBC is a versatile oxidant.[7,8] It is prepared by dissolving CrO_3 in tertiary butyl alcohol.[9,10] The compound was prepared for the first time by Wienhaus but R. V. Oppenauer and H. Oberrauch introduced it as a new oxidizing agent.

MATERIALS & METHODS

CHEMICALS USED

1,4 Dioxane, tertiary butyl alcohol, chromium trioxide, acetone.

EXPERIMENTAL

(a) Oxidant : Substrate (1:1) –PC101

1 gm p-cresol was added to oxidant, TBC prepared by dissolving 1 gm of CrO_3 in 10 mL of tertiary butyl alcohol. The mixture was stirred vigourously and heated in microwave oven at 16°C for 90 seconds. The brown-coloured product was washed with acetone and collected as sample R1.

(b) Oxidant: substrate (1:2) –PC112

2.2 gm p-cresol was added to oxidant, TBC prepared by dissolving 1 gm of CrO_3 in 10 mL tertiary butyl alcohol. The mixture was stirred vigourously and heated in microwave oven at 16°C for 200 seconds. The brown coloured product was washed with acetone and collected as sample R2.

(c) Oxidant: Substrate (1:3) –PC113

3.3 gm p-cresol was added to oxidant, TBC prepared by dissolving 1 gm of CrO_3 in 10 mL tertiary butyl alcohol. The mixture was stirred vigourously and heated in microwave oven at 16°C for 100 seconds. The brown coloured product was washed with acetone and collected as sample R3.

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Table 1- FTIR Results (sample no.01)

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 (sample no.02)

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 (sample no.03)

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 (sample no.01)

Temperature	Formulation sequence showing the change	Percentage Theoretical	Loss percentage
31.4 -150^{0}C	$Cr_2(CH_3CO_2)_4\ (H_2O)_2$ |-2H_2O	18	18.32
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		

Table 5- Thermogravimetric results (sample no.02)

Temperature	Formulation sequence showing the change	Percentage Theoretical	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

RESULT

The FTIR curves and DTA TGA analysis support the following formulation for the sample PC1, PC2 and PC3.

PC1—$Cr_2(CH_3CO_2)_4\ (H_2O)_2$

PC2—2CrO.2H_2O.$CH_2COOH.CH_2(COOH)_2$

PC3—$Cr_2O.CH_3COOH.CH_3COCOOH.2H_2O$

The FTIR Curves (table-1) of sample R1 contain almost all the peaks which are expected for the formulation.

Similarly, the proposed formulation of sample R2, on the basis of empirical formulation, is strongly supported by the FTIR curves as well as TGA-DTA analysis $Cr_2O_3.2(COOH)_2.4H_2O$.

The expected broad peak for O-H stretching (H-bonded) at 3402.3 cm, bidendate carboxylic acid functioning as ligand at 1562.3 cm, C-O stretching at 1280.0 cm, C-C stretching at 950.7 cm^{-1} and M-O stretching at 622.0 cm^{-1} are present in the curve. Similarly, the loss pattern is just what we expected for the formulation as shown. We observed that the FTIR curves (table -2) of sample no.02 contain almost all the peaks which are expected for its formulation as shown. We observe that the FTIR curves contains almost all the peaks which are expected for its formulation 2CrO.2H_2O.$CH_3COOH.CH_2$ $(COOH)_2$. It is supported by the peaks at 33766.8cm (O-

H stretching), 2370.2cm (C-H stretching) 1565.4 cm (bidentate carbxylate ligand), 1434 cm (-COOH co-ordinated) 809.8 cm (C-C stretching) etc. Also the loss pattern in TGA-DTA curve supports the proposed formulation.

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