

Preparation of Sucrose Complexes of Chromium and the Study of their Viability as Food Supplement for Chromium Deficiency

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Abstract: Chromium(III) is biologically active ingredient of food and is known to enhance the action of insulin, a hormone critical to metabolism and storage of carbohydrate, fat and protein in body. On the other hand, chromium(VI) is toxic substance in any form. Chromium deficiency may be cured by commercially available food supplements like chromium picolinate and chromium polynicotinate. In the present work, we report the method of preparation and characterization of products formed by the interaction of di-tertiary butyl chromate with sucrose in order to explore whether the products containing chromium(III) in combination with oxidised products of sucrose can be used as food supplement for chromium deficiency.

Keywords: Sucrose, Microwave, Diteritary butyl chromate, Chromium food supplement

Introduction

Toxic chromium(VI) can be reduced to beneficial chromium(III) by the reaction with organic substrates. Chromium picolinate¹, chromium polynicotinate², chromium propionate³ *etc* are commercially popular variants of Cr(III) which are used to cure chromium deficiency which causes malfunctioning of hormones like insulin². A large number of organic variants of chromium(VI) like Collin's reagent⁴ pyridinium chlorochromate⁵, pyridinium fluorochromate⁶ *etc*. have been reported as potent oxidants for different classes of organic substrates. The study of redox processes involving chromium(VI) variants and biologically active substrates like carbohydrates and amino acids *etc*. may give us a clue to understand the role of chromium(III) in biological processes as well as to get rid of toxic chromium(VI) from the body. Chromium(III) forms complexes with amino acids as ligand^{7,8}. Khade *et.al.*,⁹ has reported the preparation and stability of chromium complexes with drug salbutamol and amino acids. Luo *et al.*,¹⁰ have made a detailed study of relative sorption of organic complexes of Cr(III) like its glutamate, oxalate, citrate, pyruvate *etc*. The complexes of chromium with naturally occurring amino acid have been reported by Shahawi¹¹.

In order to explore the possibility of formation of Cr(III) complexes with biologically significant organic compounds, we have prepared a large number of products by the interaction of di-tertiary butyl chromate with carbohydrates and amino acids under microwave irradiation condition. Microwave assisted organic synthesis involving oxidation of organic substrates by di-tertiary butyl chromate has been reported¹². In this paper, we report the preparation and characterization of six products of different colour and composition formed by the interaction of di-tertiary butyl chromate (TBC) with sucrose in water and dimethylformamide as solvents.

Experimental

All the chemicals used in this work were of A.R grade.

Preparation of TBC

The oxidant TBC was prepared by dissolving accurately weighed amount of dry and powdered chromium trioxide in 10 mL of *t*-butyl alcohol. The clear brown solution obtained after decantation and filtration.

Preparation of solution of sucrose

Accurately weighed amounts of pure sucrose required for different substrate- oxidant molar ratio were dissolved in 10 mL of water and the same procedure was repeated in DMF as solvent. The substrate-oxidant molar ratio (S:O) taken in water as well as DMF were 1:1, 2:1 and 3:1. For 1:1 molar ratio the amount of sucrose and CrO₃ taken were 3.42 g and 1 g respectively. Similarly for 2:1 molar ratio, the amounts were 6.84 g and 1 g and for 3:1 the amount of sucrose and CrO₃ were 10.26 g and 1 g respectively.

Reaction between substrate solution and TBC

The two solutions were mixed, stirred, heated and then subjected to microwave irradiation before allowing to stand for the period specified in Table 1. The solid product obtained after washing with acetone till colourless washing and filtration was collected in air tight bottles. The three samples in water as solvent were SW-11, SW-21 and SW-31 whereas in DMF the samples were SD-11, SD-21 and SD-31.

Table 1. Time taken in various processes for different substrate-oxidant molar ratio (S:O)

Sample Code	Solvent for Substrate	S:O ratio	Stirring, min	Irradiation	Left
SW-11	Water	1:1	21	3 min (180W)	1 day
SW-21	Water	2:1	30	1.5 min (180W)+ 1.5 min (360W)	1 day
SW-31	Water	3:1	30	3 min (180W)	7 days
SD-11	DMF	1:1	10	3 min (180W)	7 days
SD-21	DMF	2:1	10	3 min (180W)	10 days
SD-31	DMF	3:1	5	3 min (180W)	10 days

Estimation and characterization of the samples

The analysis of the samples was done instrumentally and titrimetrically. Chromium was estimated volumetrically using K₂S₂O₈ (excess), 0.1 N K₂Cr₂O₇ solution and 0.1 N Mohr's salt solution. Percentage composition of carbon and hydrogen were estimated using Elemental analyser- Heraeus Vario EL III Carlo Erba-I108. The IR spectrum of the products

were recorded on a Fourier Transform Infra Red Spectrophotometer- Shimadzu 8201 PC. The TGA/DTA curves of the samples SW-11, SW-31, SD-11 and SD-31 were plotted between temperature range 25 to 700 °C at the rate of 10° C/min on Perkin Elmer Diamond TG/DTA-Thermal Analyser System. The formulations possible on the basis of these studies are given in Table 2.

Table 2. Characterisation of the products (Products: Sucrose + TBC)

Sample Code	Colour	Solubility in water	Emperical Formula	Formulation
SW-11	Grey	Partially soluble	$\text{CrC}_3\text{H}_{14}\text{O}_{10}$ ($\text{Cr}_2\text{C}_6\text{H}_{28}\text{O}_{20}$)	$\text{Cr}_2\text{O}_3 \cdot \text{HOOC}(\text{CHOH})_4\text{COOH} \cdot 9\text{H}_2\text{O}$ ($\text{Cr}_2\text{C}_6\text{H}_{28}\text{O}_{20}$)
SW-21	Yellowish green	Partially soluble	$\text{Cr}_2\text{C}_7\text{H}_{23}\text{O}_{18}$	$\text{Cr}_2\text{O}_3 \cdot \text{HOOC}(\text{CHOH})_4\text{COOH} \cdot \text{HCOOH} \cdot 5\text{H}_2\text{O}$. ($\text{Cr}_2\text{C}_7\text{H}_{22}\text{O}_{18}$)
SW-31	Dark grey	Partially soluble	$\text{CrC}_6\text{H}_{20}\text{O}_{10}$ ($\text{Cr}_2\text{C}_{12}\text{H}_{40}\text{O}_{20}$)	$\text{Cr}_2\text{O} \cdot \text{OHC}(\text{CHOH})_4\text{CH}_2\text{OH} \cdot \text{HC}(\text{CHOH})_2\text{CH}_2\text{OH} \cdot \text{CH}_2\text{OHCOOH} \cdot 6\text{H}_2\text{O}$. ($\text{Cr}_2\text{C}_{12}\text{H}_{36}\text{O}_{20}$)
SD-11	Bottle green	Insoluble	$\text{CrC}_6\text{H}_{24}\text{O}_{10}$	$\text{CrO} \cdot \text{OHC}(\text{CHOH})_3\text{CH}_2\text{OH} \cdot \text{HC}(\text{CHOH})_2\text{CH}_2\text{OH} \cdot 3\text{H}_2\text{O}$ ($\text{CrC}_6\text{H}_{18}\text{O}_{10}$)
SD-21	Light green	Partially soluble in hot water	$\text{CrC}_{10}\text{H}_{22}\text{O}_{11}$ ($\text{Cr}_2\text{C}_{20}\text{H}_{44}\text{O}_{22}$)	$2\text{CrO} \cdot 2\text{OHC}(\text{CHOH})_4\text{CH}_2\text{OH} \cdot 2\text{OHC}(\text{CHOH})_2\text{CH}_2\text{OH}$. ($\text{Cr}_2\text{C}_{20}\text{H}_{40}\text{O}_{22}$)
SD-31	Light grey	Insoluble	$\text{CrC}_{25}\text{H}_{53}\text{O}_{25}$	$\text{CrO} \cdot 4\text{OHC}(\text{CHOH})_4\text{CH}_2\text{OH}$ ($\text{CrC}_{24}\text{H}_{48}\text{O}_{25}$)

Results and Discussion

The observations of methods of preparation, colour and formulations of the products lead to many generalization such as

- Microwave irradiation may be helpful in carrying out synthetic processes which are otherwise very difficult. The yield of products formed by the oxidation of carbohydrates and amino acid under ordinary heating conditions have not been satisfactory⁷.
- The products are formed more efficiently when the solvent for sucrose is water rather than when it is DMF.
- As expected, the formation of product takes more time when the substrate-oxidant ratio is more (Table 1).
- The more stable Cr(III) state in the products is observed in case of water as solvent for substrate. In case of DMF as solvent, chromium is reduced to lower oxidation state *i.e.* II state.
- The products prepared by using DMF as solvent for substrate are not suitable for human consumption as the oxidation state is not III in them as per formulation.
- Out of SW-11, SW-21 and SW-31, only the sample SW-21 may be more suitable as food supplement as the III oxidation state SW-21 is also supported by its colour yellowish green as compared to SW-11 and SW-31 which are grey and black respectively.
- The extent of oxidation in case of water as solvent increases as the substrate-oxidant ration decrease. SW-11 contains glucaric acid whereas SW-31 contains the less oxidised glucose.
- The extent of oxidation is less in case of DMF as solvent for substrate as substantiated by the absence of acidic groups in the products SD-11, SD-21 as well as SD-31.

- The samples SW-21 and SW-31 were found to be partially soluble in water. Chromium was found to be present in rice grains kept in the aqueous solution of the two samples for 48 hours.

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