

Spectroscopic and Thermogravimetric Characterization of Oxidation Products Obtained Using tert-Amyl Chromate as an Oxidant

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Abstract:

The development of efficient and selective oxidizing agents remains an important area of research in organic chemistry. Chromium(VI)-based oxidants have been widely employed for the oxidation of a variety of organic substrates under mild and non-aqueous conditions. However, many conventional chromium oxidants suffer from limitations such as instability, hygroscopic nature, requirement of excess reagent and safety concerns during handling. In the present study, a chromium-based oxidant, tertiary amyl chromate, was synthesized and evaluated for its oxidative behavior toward selected organic substrates. The oxidation products obtained were isolated and characterized using elemental analysis, FTIR spectroscopy and thermogravimetric (TG) analysis. Spectral data confirmed the formation of oxidation products, while thermal studies provided information regarding their stability and decomposition patterns. The results demonstrate that tertiary amyl chromate acts as an effective oxidizing agent under mild reaction conditions, producing the desired products in good yields. The combined spectroscopic and thermogravimetric investigations contribute to a better understanding of the nature and properties of the oxidation products and highlight the potential utility of tertiary amyl chromate in organic synthesis.

Keywords: Tertiary Amyl Chromate, Chromium(VI) Oxidant, FTIR Spectroscopy, Thermogravimetric Analysis, Acrylic acid.

Introduction:

Acrylic acid ($\text{CH}_2=\text{CHCOOH}$), also known as prop-2-enoic acid, is the simplest unsaturated carboxylic acid and an important industrial chemical. It is widely used in the manufacture of polymers, adhesives, coatings, and superabsorbent materials. Owing to the presence of both a carbon-carbon

double bond and a carboxylic acid group, acrylic acid undergoes various oxidation reactions. In the present study, acrylic acid was oxidized using tert-amyl chromate (TAC) to investigate its oxidative behavior and to characterize the resulting oxidation product(s) using suitable analytical techniques.

On the other hand, Chromium(VI)-based oxidizing reagents have been extensively developed and widely employed in organic synthesis due to their high efficiency, selectivity, and versatility in oxidation reactions.

In the present investigation, tertiary amyl chromate (TAC) was utilized as an oxidizing agent for the oxidation of acrylic acid in two solvent systems, namely dioxane and tertiary amyl alcohol. Acrylic acid served as the organic substrate and was allowed to react with TAC under varying substrate:oxidant molar ratios. The products obtained were systematically analyzed to elucidate the reaction mechanism and to assess the influence of experimental variables such as molar ratio, reaction time, and temperature on the course of the reaction.

Material and methods:

The chemicals were all A.R. grade. Freshly prepared solutions were used. The experimental method used in the present work consists of mainly:

- (i) Preparation of tertiary amyl chromate (Oxidant): The oxidant was prepared by dissolving accurately weighed amount of dry and powdered chromium trioxide in calculated volume of tertiary amyl alcohol at room temperature. Care was taken to avoid bigger pieces of CrO_3 as this may cause explosive reaction leading to fire.
- (ii) Preparation of solution of Acrylic acid (substrate solution): The calculated quantities of the substrate (Acrylic acid) were dissolved in approximately 10 ml of the solvent (Dioxane or Tert.amyl alcohol) to get solution of the substrate.
- (iii) Reaction between solution of Acrylic acid(substrate) and Tert.amyl chromate (Oxidant): The two solutions as prepared in (i) and (ii) were mixed carefully and stirred on a mechanical stirrer for several minutes before subjecting it to microwaveirradiation. The wattage, period of irradiating and

standing for different substrate: oxidant ratios are tabulated Table 1. The solid products of different colours and compositions were obtained which were washed several times with acetone to remove any impurities and collected in air tight bottles as samples AAA10, AAA11, ADA12.

- (iv) Characterization of the products: The products were characterized by elemental analysis, FTIR curves and TGA (thermogravimetric mass loss pattern). The solubility of the products in different solvents was also tested.

Figure 1: TG/TGA loss pattern of synthesized tertiary amyl chromate recorded at IIT Kharagpur.

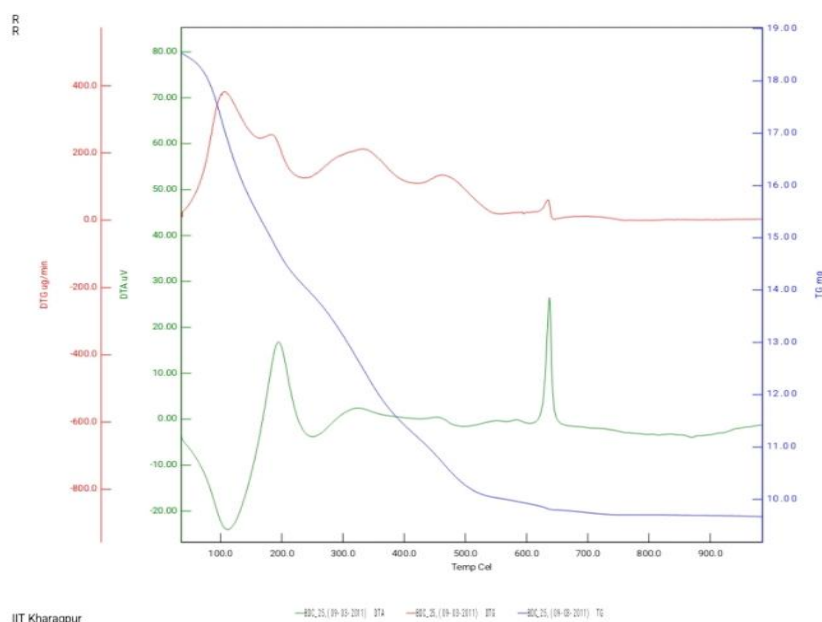
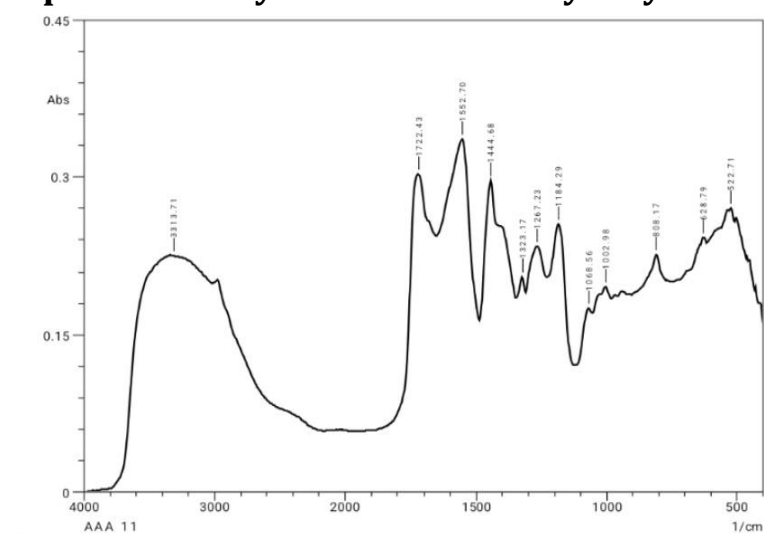


Figure 2. FTIR spectrum of synthesized tertiary amyl chromate.



Oxidation of Acrylic Acid with TAC :**Table – 1**

Solvent	S:O	Product code	Colour	Empirical formula	Formulation
Tert. amyl alcohol	1:1	AAA10	Dark brown	$C_4H_{10}O_5Cr_2$	$Cr_2O \cdot CHO \cdot CHO \cdot 2HCHO$
Tert.amyl alcohol	3:2	AAA11	Light Brown	$C_6H_{12}O_{10}Cr$	$CrO \cdot OHC \cdot COOH \cdot CHO \cdot CO \cdot COOH \cdot HCHO \cdot H_2O$
Dioxane	2:1	ADA 12	Mud	$C_2H_6O_4Cr$	$CrO \cdot 2HCHO \cdot H_2O$

While this paper introduces three different compounds, it specifically focuses on the in-depth analysis of one selected compound i.e. compound name: AAA – 11.

AAA – 11

Color – Light Brown

Composition of the sample:

Table – 2

Element	Observed %	Calculated %
Carbon	24.28	27.3
Hydrogen	4.33	4.05
Oxygen	54.06	54.5
Chromium	17.33	17.5

Empirical formula of the compound – $C_6H_{12}O_{16}Cr$

Proposed formulation: $CrO \cdot OHC \cdot COOH \cdot OHC \cdot CO \cdot COOH \cdot HCHO \cdot H_2O$

FTIR Results:

Table – 3

Peaks	Nature of Peaks	Group Assignment
3313.71	Strong	O – H stretching (H-bonding)
1722.43	Medium	C=O stretching
1552.70	Sharp	COOH coordinated
1444.68	Medium	C=O stretching
1323.17	Weak	C – H stretching

Results and discussion:

The compound was found to undergo degradative oxidation in almost all the cases to form OHC-CHO, CHO-CO-COOH, OHC-COOH, HCOOH & HCHO. The colour of the complex in most of the cases was brown which might be due to the charge transfer rather than electronic transition.

The reaction time was found to increase in higher ratio of the substrate in almost all the cases. The product could not be isolated without waiting overnight in spite of heating and stirring for a long time as observed in cases of tert. amyl alcohol and dioxane as solvents for the substrate (acrylic acid).

The colour of the products ranges from dark brown to light brown which indicates the presence of chromium in different oxidation states depending on the conditions of the reaction.

The nature of the complexes formed by the oxidation of Acrylic acid by ditertiary amyl chromate (TAC) provides valuable insights into the reaction mechanism. The large difference in the reaction duration required to yield products when dioxane and TAA are used as solvents for the substrate emphasizes the crucial role the solvent plays during the process.

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