A Novel Method for Preparation of In$_2$O$_3$, Bi$_2$O$_3$ and Sb$_2$O$_3$ Oxides Using Urea at Elevated Temperature.

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**ABSTRACT**

The In$_2$O$_3$, Bi$_2$O$_3$ and Sb$_2$O$_3$ oxides were synthesized by a novel reaction of urea with InCl$_3$, BiCl$_3$ and SbCl$_3$ respectively, in an aqueous media at $\sim$ 90 °C. The infrared spectra and microanalysis, CHN, of the solid products resulted indicate that the absence of the bands of urea, but appearing the characteristic bands of oxides. A general mechanism describing the formation of oxides and decomposition of urea are suggested.

**INTRODUCTION**

Urea and their derivatives are known to be the parent compound of a large and interesting class in both organic and inorganic compounds; it is used as a starting material for the synthesis of many applied compounds. The literature reveals that urea is forming coordinate bonds with many metal ions at room temperature in aqueous and non aqueous media through its oxygen or nitrogen atoms depending on the type of metal ion used [7-8]. From the chemical viewpoint, the reaction of metal salts with urea at high temperature has recently gained increasing interest [7-14]. The nature of the reaction products depend strongly on the type of metal ions and so the metal salt used. In our previously studies referenced by us concerning the reaction of urea with metals such as Co( III), pb(II), Sn(II), Cr(III), Fe(III), Au(III), Sn(IV), V(V) and Mo(VI) at high temperature demonstrate that the types of metal ions beside their anions have a pronounced effect on the nature of the reaction products. The published papers owned by us in this trend of the reaction of urea with different metal salts at elevated temperature lead to discovering a novel method for preparation pbCO$_3$ and CoCO$_3$ [10], lanthanide carbonates [12], limonite, FeO(OH) [9], 2ZnCO$_3$.3Zn(OH)$_2$ [8], and SnOCl$_2$.2H$_2$O [7].

The aim of this publication is to report the synthesis and characterization study of the In$_2$O$_3$, Bi$_2$O$_3$ and Sb$_2$O$_3$ oxides resulted from a novel oxidation reduction reaction between urea with InCl$_3$, BiCl$_3$ and SbCl$_3$, respectively, in an aqueous solution at $\sim$ 90 °C.

**EXPERIMENTAL**

All chemicals used throughout this study were Analar or extra pure grade. The white oxides, In$_2$O$_3$, Bi$_2$O$_3$ and Sb$_2$O$_3$ were prepared by mixing equal volumes of aqueous solutions of 0.1M of InCl$_3$, BiCl$_3$ and SbCl$_3$ and 1.0 M of urea. The mixtures were heated on a water bath to approximately 90 °C for about ~24 h. The white colored precipitate filtered off, washed several times with bi-distilled water and dried in vacuo over CaCl$_2$. The elemental analysis for the obtained products shows the absence of carbon, hydrogen and nitrogen elements. The percentages of indium, bismuth, and antimony were determined by using thermogravimetric method. The infrared spectra of the reactants and the solid products obtained were recorded from KBr discs using a Bruker FT-IR Spectrophotometer.
RESULTS AND DISCUSSION

The reaction of aqueous solutions of urea with InCl$_3$, BiCl$_3$ and SbCl$_3$, produces clear white colored oxides, In$_2$O$_3$, Bi$_2$O$_3$ and Sb$_2$O$_3$. The formation of these oxides upon the heating of an aqueous mixture of InCl$_3$, BiCl$_3$ and SbCl$_3$, respectively, with urea may be understood as follows:

$$2MCl_3 + 2\text{NH}_2\text{CONH}_2 + 2\text{H}_2\text{O} \quad \overset{90^\circ\text{C}}{\longrightarrow} \quad M_2\text{O}_3(s) + 6\text{HCl}(g) + 2\text{CO}_2(g) + 4\text{NH}_3(g)$$

Where (M= In$^{3+}$, Bi$^{3+}$ or Sb$^{3+}$)

Figure 1: Infrared spectra of the product resulted from the reaction of urea with InCl$_3$, BiCl$_3$ and SbCl$_3$.

![Figure 1a: Infrared spectrum of In$_2$O$_3$ oxide](image1a)

![Figure 1b: Infrared spectrum of Bi$_2$O$_3$ oxide](image1b)
For the reaction mechanisms an oxidation process for In(III), Bi(III) and Sb(III) occurs during the decomposition of urea into ammonia, carbon dioxide and hydrogen chloride gases. The infrared spectra of synthetic oxides products are shown in Fig. 1. The infrared spectra of the obtained products show no bands due to characteristic groups of urea (carbonyl and amide groups), but the bands associated to the oxides are observed [15]. Based on this observation, along with that obtained from elemental analysis data as well as determination of metals In(III), Bi(III) and Sb(III), the fact that infrared spectra of commercially obtained In(III), Bi(III) and Sb(III) are the same as that of the reaction products.

![Infrared spectrum of Sb$_2$O$_3$ oxide](image)

**Figure 1c**: Infrared spectrum of Sb$_2$O$_3$ oxide

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