e-ISSN:2321-6212
p-ISSN:2347-2278
RESEARCH AND REVIEWS: JOURNAL OF MATERIAL SCIENCES

A Novel Method for Preparation of In_2O_3, Bi_2O_3 and Sb_2O_3 Oxides Using Urea at Elevated Temperature.

Moamen S Refat[a,b,*] and Abdel Majid A Adam[a]

[a]Department of Chemistry, Faculty of Science, Taif University, Al-Haweiah, P.O. Box 888, Zip Code 21974, Taif, Saudi Arabia.
[b]Department of Chemistry, Faculty of Science, Port Said University, Port Said, Egypt

Short Communication

Received: 10/09/2013
Revised: 11/10/2013
Accepted: 22/10/2013

***For Correspondence**

Department of Chemistry, Faculty of Science, Taif University, Al-Haweiah, P.O. Box 888, Zip Code 21974, Taif, Saudi Arabia.

Keywords: In_2O_3, Bi_2O_3, Sb_2O_3 and urea.

ABSTRACT

The In_2O_3, Bi_2O_3 and Sb_2O_3 oxides were synthesized by a novel reaction of urea with $InCl_3$, $BiCl_3$ and $SbCl_3$ respectively, in an aqueous media at ~ 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 revels 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 [1-8]. 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 [7-14] 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(IV) 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 $SnOCl_2.2H_2O$ [7].

The aim of this publication is to report the synthesis and characterization study of the In_2O_3, Bi_2O_3 and Sb_2O_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 ~ 90 °C.

EXPERIMENTAL

All chemicals used throughout this study were Analar or extra pure grade. The white oxides, In_2O_3, Bi_2O_3 and Sb_2O_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_2O_3, Bi_2O_3 and Sb_2O_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:

$$2MCl_3+20NH_2CONH_2+23H_2O \xrightarrow{90^oC} M_2O_3(s)+6HCl(g)+20CO_2(g)+40NH_3(g)$$

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 $SbCl_3$.

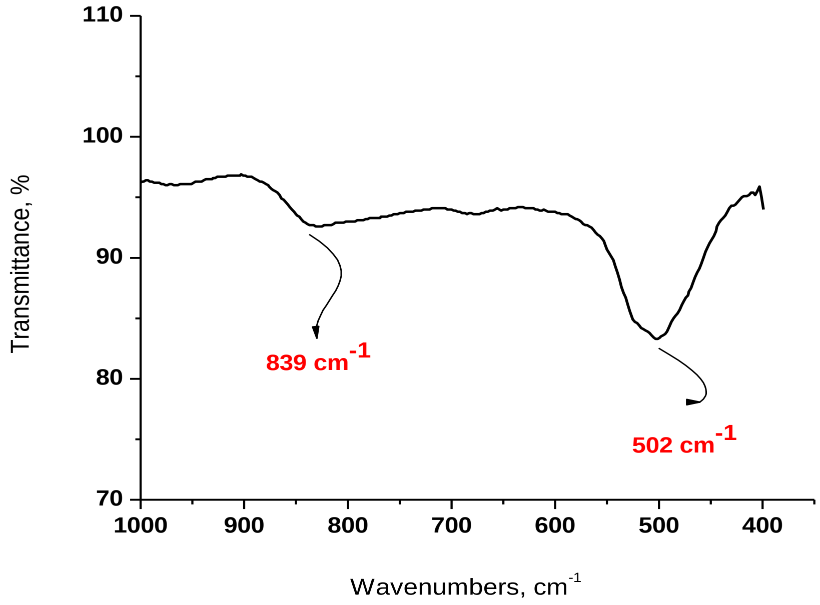

Figure 1a: Infrared spectrum of In_2O_3 oxide

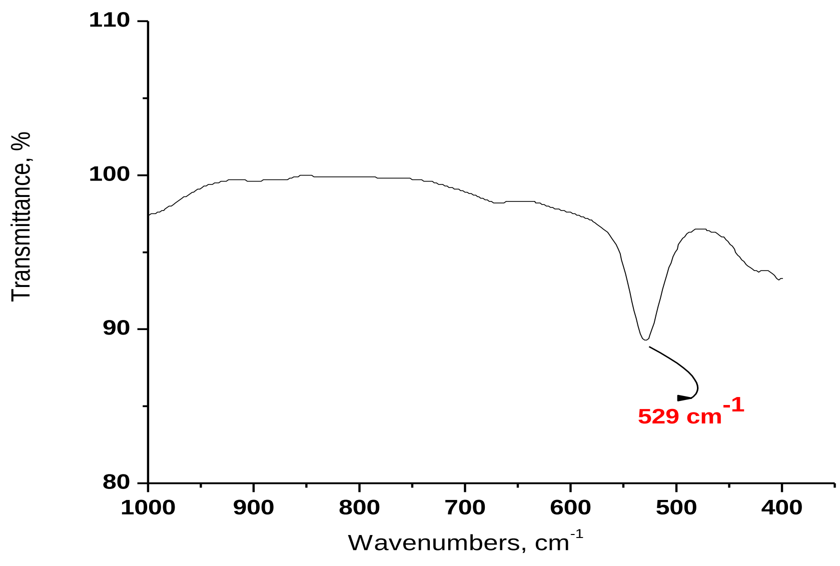

Figure 1b: Infrared spectrum of Bi_2O_3 oxide

e-ISSN:2321-6212
p-ISSN:2347-2278

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.

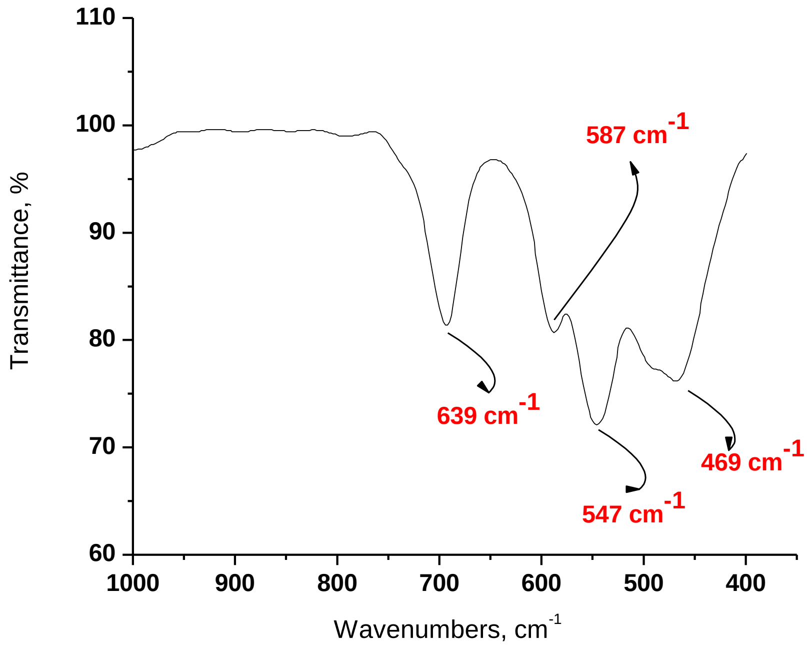

Figure 1c: Infrared spectrum of Sb_2O_3 oxide

REFERENCES

1. PC Srivastava, C Aravindaksahn. Complexes of urea with ruthenium(III), rhodium(III) and platinum(IV), Z. Phys. Chem. Leipzig. 1983;61:264.
2. F Berny, G Wipff. Interaction of M^{3+} lanthanide cations with amide, urea, thioamide and thiourea ligands: a quantum mechanical study. J Chem Soc Perkin Trans. 2001;2(1):73
3. Yu Ya, Kharitonov KS. Sulaimankulov NB, Khudaibergenova. IR absorption spectra of rare earth nitrate complexes with urea. Z Neorg Khim. 1990;35(8):2117.
4. CV Deshpande, VR Rao. Preparation and characterization of lanthanide complexes using substituted ureas and thioureas as ligands. J Ind Inst Science. 1989;69(5):329.
5. JP Barbier, R Hugel. New complexes of cobalt(II) and nickel(II) salts with N,N'-diethylurea. Inorg Chim Acta. 1976;18(3);253.
6. RB Penland, S Mizushima, C Curran, JV Quagliano. Infrared absorption spectra of inorganic coordination complexes. X. Studies of some metal-urea complexes. J Amer Chem Soc. 1957;79:1575.
7. SM Teleb, MS Refat, SA Sadeek. Spectroscopic studies of the reaction products of urea with Sn(IV) and Sn(II) chlorides at room and high temperature. Ukrainian Chem J. 2004;68(5):35.
8. SA Sadeek, MS Refat. Synthesis, Infrared spectra and thermal investigation of gold(III) and zinc(II) urea complexes. A new procedure for the synthesis of basic zinc carbonate. J Coord Chem. 2005;58(18):1727.
9. SA Sadeek, MS Refat , SM Teleb. Spectroscopic and thermal studies of $[Cr_2(NH_2)_2(H_2O)_2(SO_4)_2].2H_2O$, $[Cr(NCO)_3(H_2O)].3H_2O$ and $[FeO(OH)].0.5H_2O$ compounds formed by the reactions of urea with $Cr_2(SO_4)_3$, $Cr(CH_3COO)_3$ and $Fe_2(SO_4)_3$. J Korea Chem Soc. 2004;48(4):358.
10. MS Refat, SM Teleb, SA Sadeek. A novel method for preparation of cobalt(II) and lead(II) carbonates. Spectrochim Acta. 2004;A(60):2803.
11. MS Refat, SA Sadeek, SM Teleb. Reactions of urea with Mo(VI), V(V) and Se(IV) oxides at high temperature. J Argen Chem Soc. 2004;92(4-6):23.
12. MS Refat. A novel method for the synthesis of rare earth carbonates, Synth. React. Inorg Met Org Chem. 2004;34(9):1605-1613.

13. EM Nour, SM Teleb, NA Al-Khososy, MS Refat. A novel method for the synthesis of metal carbonates. I. synthesis and infrared spectrum of manganese carbonate, $MnCO_3.H_2O$, formed by the reaction of urea with manganese(II) salts, Synth. React. Inorg MetOrg Chem. 1997;27(4):505.
14. SA Sadeek/ Preparation, infrared spectrum and thermal studies of $[Zn_2(H_2O)_4(SO_4)_2]$ complex formed by the reaction of urea with zinc(II) sulphate. J Phys Chem Solids. 1993;54:919.
15. K Nakamoto. Infrared and Raman Spectra of Inorganic and Coordination Compounds, Wiley, New York, 1978.