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A Novel Method for Synthesis of Some Transition Metal Oxides: Fe_2O_3 and CuO

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Abstract: The Fe_2O_3 and CuO oxides were synthesized by a new reaction of methyl urea with $FeCl_3.6H_2O$, $Fe(NO_3)_3.9H_2O$, $Fe_2(SO_4)_3$ and $Cu(OAc)_2$, respectively, in an aqueous media at ~ 90 ^oC. The infrared spectra and microanalysis, CHN, of the solid products resulted indicate that the absence of the bands of methyl urea, but appearing the characteristic bands of oxides. A general mechanism describing the formation of oxides and decomposition of methyl urea are suggested.

Keywords: Fe_2O_3, CuO and methyl urea.

Introduction

Methyl 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 Fe_2O_3 and CuO oxides resulted from a novel oxidation reduction reaction between methyl urea with $FeCl_3.6H_2O$, $Fe(NO_3)_3.9H_2O$, $Fe_2(SO_4)_3$ and $Cu(OAc)_2$, respectively, in an aqueous solution at ~ 90 ^oC.

Experimental

All chemicals used throughout this study were Analar or extra pure grade. The colored oxides, Fe_2O_3 and CuO were prepared by mixing equal volumes of aqueous solutions of 0.1M of $FeCl_3.6H_2O$, $Fe(NO_3)_3.9H_2O$, $Fe_2(SO_4)_3$ and $Cu(OAc)_2$ and 1.0 M of methyl urea. The mixtures were heated on a water bath to approximately 90 ^oC for about ~10 h. The colored precipitate filtered off, washed several times with bi-distilled water and dried in vacuo over $CaCl_2$. The five oxides, Fe_2O_3 (brown-to-dark brown) and CuO (black) were prepared in a manner similar to that described above by the reaction of $FeCl_3.6H_2O$, $Fe(NO_3)_3.9H_2O$, $Fe_2(SO_4)_3$ and $Cu(OAc)_2$, respectively, with methyl urea. The elemental analysis for the obtained products shows the absence of carbon, hydrogen and nitrogen elements. The percentages of iron, cobalt, nickel, copper and silver were determined by using atomic absorption method. A spectrometer model PYE-UNICAM SP 1900 fitted with the corresponding lamp was used for this purposed. 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 methyl urea with $FeCl_3.6H_2O$, $Fe(NO_3)_3.9H_2O$, $Fe_2(SO_4)_3$ and $Cu(OAc)_2$, produces colored oxides, Fe_2O_3 and CuO. The formation of these oxides upon the heating of an aqueous mixture of $FeCl_3.6H_2O$, $Fe(NO_3)_3.9H_2O$, $Fe_2(SO_4)_3$ and $Cu(OAc)_2$, respectively, with methyl urea may be understood as follows:

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$$2FeCl_3+20CH_3NHCONH_2 \xrightarrow{90^oC} Fe_2O_3+6NH_4Cl+17CO+23CH_4+NH_4OH+2H_2+17N_2$$

$$2Fe(NO_3)_3+20CH_3NHCONH_2 \xrightarrow{90^oC} Fe_2O_3+6NH_4NO_3+17CO+23CH_4+NH_4OH+2H_2+17N_2$$

$$Fe_2(SO_4)_3+10CH_3NHCONH_2 \xrightarrow{90^oC} Fe_2O_3+3(NH_4)_2SO_4+7CO+CH_4+6C_2H_2+7N_2+10H_2$$

$$Cu(OAc)_2+10CH_3NHCONH_2 \xrightarrow{90^oC} CuO+2NH_4(OAc)+9CO+11CH_4+4NH_3+7.5N_2$$

For the four reactions an oxidation process for Fe(III) and Cu(II) occurs during the decomposition of methyl urea into ammonia, carbon mono-oxide and other fragment 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 methyl urea (methyl, 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 Fe(III) and Cu(II) and the fact that infrared spectra of commercially obtained Fe_2O_3 and CuO are the same as that of the reaction products.

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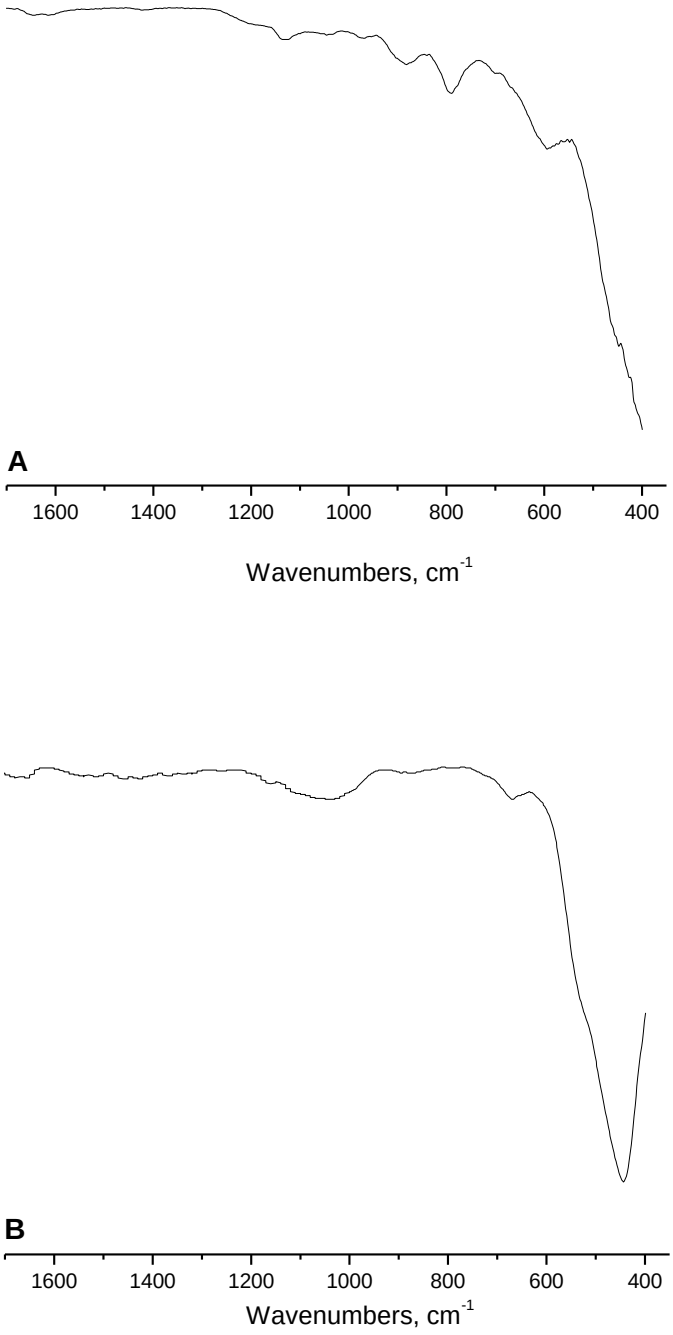

Fig. 1: Infrared spectra of the product resulted from the reaction of methyl urea with A: $Cu(OAc)_2$ and B: $FeCl_3$, respectively.