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FORMATION OF COBALTITE BY THERMAL DECOMPOSITION OF NICKEL-COBALT TARTARATE

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ABSTRACT The solutions of nickel sulphate hexahydrate and cobalt sulphate heptahydrate were well stirred. Then 10% sodium tartarate was added at appropriate pH to get nickel-cobalt tartarate monohydrate. The metal analysis of this tartarate compound was carried out by atomic absorption spectroscopy (AAS). The elemental analysis of carbon and hydrogen was done by the microanalytical technique. The thermo gravimetric analysis (TGA), derivative thermogravimetry (DTG) and differential thermal analysis (DTA) of tartarate compound were done under a static air atmosphere. The intermediate compounds were analyzed. The TGA, DTG and DTA measurements of nickel-cobalt tartarate monohydrate show first the dehydration and then decomposition of carbonate yields nickel cobaltite.

KEYWORDS :

Experimental:

A mixture of nickel sulphate hexahydrate (6.068 gm in 100 ml distilled water) and cobalt sulphate heptahydrate (12.299 gm in 100 ml distilled water) was placed in 500 ml beaker. The pH was adjusted between 5 to 6 by addition of dilute H_2SO_4 so that the hydroxide precipitate did not form. The solution was stirred vigorously with a magnetic stirrer. The temperature of the solution was maintained at 50^0C then 10% sodium tartarate was added slowly with constant stirring till a permanent precipitate obtained. Equal amount of distilled acetone was added to get the homogenous co-precipitate. The precipitate was filtered after stirring it for 30 min. the precipitate was washed with cold distilled water and then with acetone to speed up drying. It was dried. Thus nickel-cobalt tartarate monohydrate was prepared.

The metal analysis of nickel-cobalt tartarate monohydrate was carried out by using the Perlein Elmer Model 3100 Atomic Absorption Spectrophotometer (AAS) employing an air acetylene flame and a hollow cathode lamp as the light source. The elemental analysis of carbon and hydrogen for tartarate compound was done by micro-analytical technique. The results are summarized in Table-1.

Thermal decomposition of nickel-cobalt tartarate monohydrate was recorded on Sieko instruments. TGA, DTG and DTA were done under static air atmosphere. All the experiments were carried out under the identical conditions as : Sample weight :- 5 mg, Sample holder :- Platinum Crucible for TGA and DTA, Temperature :- 30-700^{0}C, Reference material : α Alumina, Heating rate :- 10^0C min^{-1}. DTG curve was obtained from TGA curve.

Results and Discussion:

The estimation of nickel and cobalt by AAS and elemental analysis were carried out. The analytical data of tartarate compound is presented in Table-1.

Table-1: Analytical data of Nickel-cobalt tartarate monohydrate

Compound	Formula	Formula weight	Elemental analysis in wt.%, 0.5							
			C		H		Co		Ni	
			Cal	Obs	Cal	Obs	Cal	Obs	Cal	Obs
Nickel-cobalt tartarate mono-hydrate	NiCo2(C4H4O6)3.H2O	638.58	22.5	22.16	2.19	2.33	17.00	17.50	8.47	9.51

It is in good agreement with calculated values. The presence of water of crystallization for tartarate compound was confirmed on the basis of thermal analysis curves. The TGA, DTG and DTA curves of tartarate compound are shown in fig. 1.

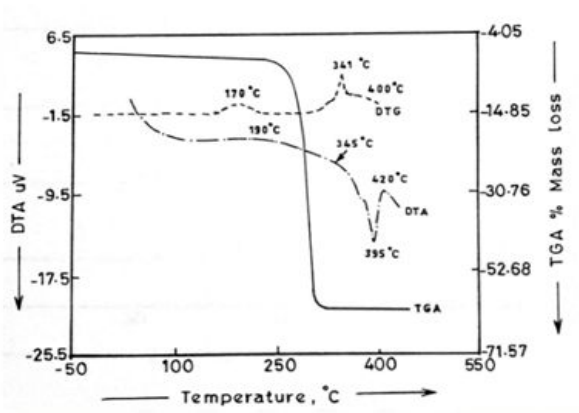

Fig. (1) – TGA, DTG, DTA curve for Nickel-cobalt tartarate

The complete data for the observed and calculated mass losses for different stages of decomposition of tartarate compound and the corresponding ranges are shown in Table-2.

Table-2: TGA – DTG – DTA data of tartarate under oxygen atmosphere

Compound	TGA			DTG peak temp. (^{0}C)	DTA peak temp. (^{0}C)
	%mass loss		Temp. range (^{0}C)		
	Obs.	Cal.			
Nickel-cobalt tartarate mono-hydrate	2.91	2.82	35 – 210	170	190
	36.81	37.18	210 – 320	341	345
	39.03	38.28	320 – 434	400	420

The dehydration takes place in single stage in the nickel-cobalt tartarate monohydrate compound. The DTG and DTA curves are produced broad peaks as the dehydration peak in the particular temperature range (fig. 1). However, the TGA curves for tartarate compound show continuous mass-losses at 35 – 210^{0}C.

The decomposition of tartarate compound takes place in two stages with very strong exothermic peaks on DTA curve and a peak of DTG curve at same temperature (fig. 1). The exothermic peak was attributed to the oxidation of Co^{2+} to Co^{3+} in oxygen atmosphere. The mass-losses are nearly the same as theoretical values (Table-2). Isothermal heating at 360^{0}C produces nickel-cobalt carbonate with the evolution of ethylene, CO_2 and Co gases.

The intermediate carbonate decomposes in between 320^0 to 434^0 C forming nickel cobaltite. The observed mass losses are in good agreement with calculated values. The change occurs in DTA at about 420^0 C in nickel cobalt tartarate.

Carbon dioxide was detected by precipitations $CaCO_3$ from the

solution of $Ca(OH)_2$ while CO was detected by reduction of Iodine-pentoxide to Iodine. Ethylene gas was detected by the decolourization of bromine water (2% Br_2 in CCl_4) or $KMnO_4$ solution (0.5%).

The results of the present investigation indicate the following reactions for decomposition of nickel-cobalt tartarate monohydrate.

$$NiCo_2(C_4H_4O_6)_3.H_2O \xrightarrow{35^0-210^0C} NiCo_2(C_4H_4O_6)_3 + H_2O$$

Hydrated tartarate — Anhydrous tartarate

$$NiCo_2(C_4H_4O_6)_3 \xrightarrow{210^0-320^0C} NiCo_2(CO_3)_3 + 2C_2H_4\uparrow + 2H_2O\uparrow$$

Anhydrous tartarate — Carbonate

$$NiCo_2(CO_3)_3 \xrightarrow{320^0-434^0C} NiCo_2O_4 + 2CO_2\uparrow + CO\uparrow$$

Carbonate — Nickel Cobaltite

Thus, TGA, DTA and DTG measurements of nickel-cobalt tartarate monohydrate show first dehydration and then decomposition of carbonate yields to cobaltite.

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