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Phase Diagram for the Ternary System $Ni(NO_3)_2$-$Al(NO_3)_3$-H_2O between 20 and 40°C

Mohamed Jouaiti*, Bouchra Mekkaoui, Rachid Lbibb, Latifa Laallam, Ahmed Jouaiti

Sustainable Development Laboratory, FST, USMS Beni Mellal, Morocco

ABSTRACT

The solid-liquid equilibrium of the ternary system of H_2O-Al $(NO_3)_3$-Ni $(NO_3)_2$ were studied using a synthesis process based on conductivity measurements. Three isotherms included between 20 and 40°C were determined and the stoichiometric phases that appear are $Al(NO_3)_3.9H_2O$ and $Ni(NO_3)_2.6H_2O$, it was found that there is one eutectic point and two crystallization regions corresponding to aluminium nitrate and Nickel nitrate. The system belongs to a simple co-saturated type, and neither double salts nor solid solution was found.

Keywords: Ternary systems, Solubility isotherms, Nitrate, Aluminium, Nickel

INTRODUCTION

A phase diagram is a thermodynamic tool, allowing after its analysis to give us a deep description about what happens at the microscopic level in the studied system. The liquid-solid phases equilibrium diagrams based on water and metal nitrates were the subject of many previous studies due to their properties, which give them opportunities for interesting industrial applications, especially for the synthesis of bi- or tri-metallic oxides [1]. Indeed, the wide application of metal oxide catalysts as precursors or oxidation catalysts, gives them much importance and attention in materiel chemistry field. The synthesis of the transition alumina is one of the most important processes from the point of view of organizing new material technologies. A way to obtain activated alumina is the thermal decomposition of metallic nitrates that lead to solids with a spinel structure ($NiAl_2O_4$, $CuAl_2O_4$, $ZnAl_2O_4$) [2-4].

A good knowledge of equilibrium between phases allows us an optimization of the mixed oxides production from metal salts, furthermore it also can be useful for other applications, such as the extraction of salts from minerals [5], purification of salts [6]. This investigation represents a study of the $Al(NO_3)_3.Ni(NO_3)_2.H_2O$ ternary system from 20-40°C.

EXPERIMENTAL SECTION

The solubility curves are determined by a synthetic method based on conductivity measurements [7-9]. In practice, it consists, to gradually add small amounts of water to a saturated solution to toggle it of its equilibrium state. After each water addition, once the new equilibrium state is restored, we measure the solution conductivity at thermodynamic equilibrium, and we represent its evolution in terms of the quantity of added water [10-12]. Then, we follow for a set of poly-phase mixtures (liquid+ solid) of the appropriate composition, the variation of the liquid phase conductivity according to the dilution of the initial mixture. The electric conductivity-composition curve of solution introduces a discontinuity at each phase change. When the identification of the solid phase was too difficult with the previous method, the components were subjected to the dosage. Initially, salts are the Nickel nitrate with six water molecules and the aluminium nitrate with nine water molecules. Water has been used bi-distilled.

The Nickel nitrate and the aluminium nitrate identification was led by the wet residues and the "ensembles" methods [13-15]. The composition of the solid phases was determined by a dosage of the Ni and Al ions in various nitrates. The Ni ions have been measured by spectrometric method, the Al ions by volumetry using the EDTA method. We calculated the weight composition coordinates of considered mixture points by using the following expressions:

$$W\ (Al(NO_3)_3 = 100 \times \frac{\quad}{\quad} \qquad (1)$$

$$W\ (Ni(NO_3)_2 = 100 \times \frac{\quad}{\quad} \qquad (2)$$

$$mt = (Al(NO_3)_3 + (Ni(NO_3)_2 + m(H_20) \qquad (3)$$

RESULTS AND DISCUSSION

An isothermal representation of this ternary system was established. The experimental results that have been obtained are graphically outlined in the classical representation, in which the reference mark is the equilateral triangle as shown in Figures 1, 2 and 3. The compositions are expressed in terms of mass fraction. (i) Liquid field, (II) The solid phases in equilibrium with the saturated solution is $Ni(NO_3)_2.6H_2O$, (III) The saturated solution is in equilibrium with a stable $Al(NO_3)_3.9H_2O$ solid phase (IV) The solid phases in equilibrium with their saturated solution are $Al(NO_3)_3.9H_2O$ and $Ni(NO_3)_2.6H_2O$. Otherwise, only one invariant isotherm and isobar is found at 20, 30 and 40°C with the following weight composition coordinate (Table 1):

Table 1: Weight composition coordinate of eutectic points for the ternary system Al $(NO_3)_3$-Ni $(NO_3)_2$-H_2O at 20°C, 30°C and 40°C

T (°C)	W ($Al(NO_3)_3$)%	W ($Ni(NO_3)_2$)%	Domains limits
20	11.2	36.22	AL_9+Ni_6+Liquids
30	11.47	38.98	------
40	7.075	47.69	------

The phases which appear at theses temperatures are: $Al(NO_3)_3.9H_2O$: $Ni(NO_3)_2.6H_2O$

The 20°C isotherms of $Al(NO_3)_3$-$Ni(NO_3)_2$-H_2O ternary system

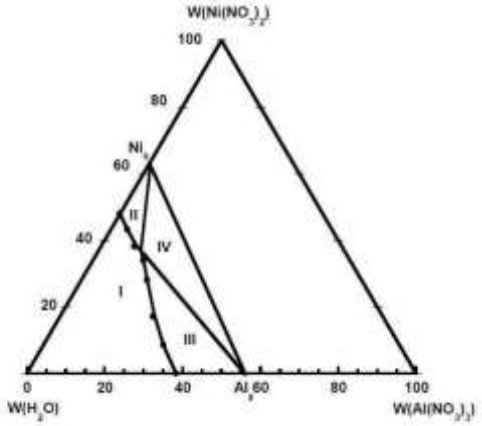

Figure 1: The 20°C solubility isotherm of $Al(NO_3)_3$-$Ni(NO_3)_2$-H_2O ternary

system The measured solubility data of the ternary system $Al(NO_3)_3$-$Ni(NO_3)_2$-H_2O at 20°C are presented in Table 2.

Table 2: The measured solubility data of the ternary system $Al(NO_3)_3$-$Ni(NO_3)_2$-H_2O at 20°C

W ($Al(NO_3)_3$)%	W ($Ni(NO_3)_2$)%	Limit domains
0	47.98	Liquids+Ni_6
4.33	43.18	--
8.6	38.1	--
11.2	36.22	AL_9+Ni_6+Liquids
13.11	33.84	Liquids+Al_9
16.9	28.1	--
23.9	17.1	--
30.9	8.5	--
38.37	0	--

The 30°C isotherms of $Al(NO_3)_3$-$Ni(NO_3)_2$-H_2O ternary system

The experimental results of the solubility isotherm at 30°C are given in Figure 2.

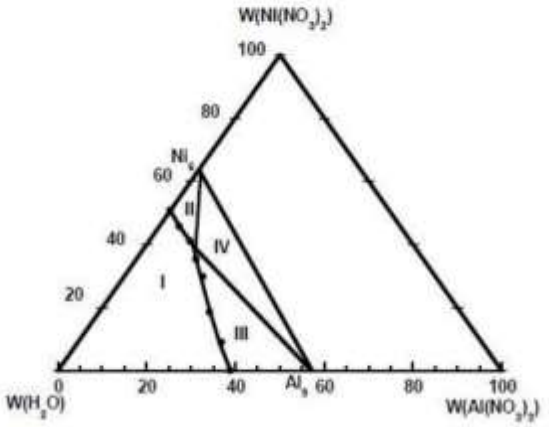

Figure 2: The 30°C solubility isotherm of Al $(NO_3)_3$-Ni $(NO_3)_2$-H_2O ternary system

The measured solubility data of the ternary system $Al(NO_3)_3$-$Ni(NO_3)_2.H_2O$ at 30°C are presented in Table 3.

Table 3: The measured solubility data of the ternary system $Al(NO_3)_3$-$Ni(NO_3)_2$-H_2O at 30°C

W ($Al(NO_3)_3$) (%)	W ($Ni(NO_3)_2$)%	Domain limits
0	50.69	Liquids+Ni_6
4.58	45.62	--
9.46	40.88	--
11.47	38.98	AL_9 + Ni_6 + Liquids
13.63	35.20	Liquids+Al_9
17.88	29.69	--
25.05	18.48	--
32.45	8.98	--
38.9	0	--

The 40°C isotherms of $Al(NO_3)_3$-$Ni(NO_3)_2$-H_2O ternary system

The experimental results of the solubility isotherm obtained at 40°C are given in Figure 3.

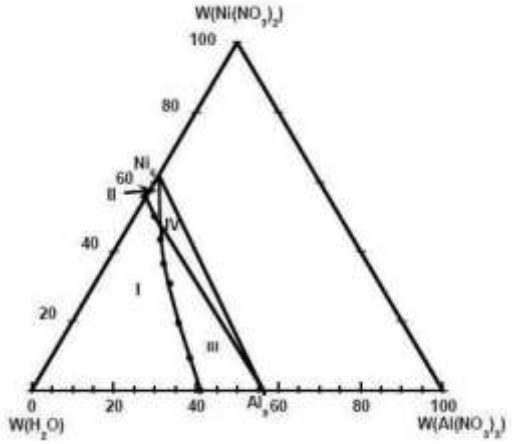

Figure 3: The 40°C solubility isotherm of $Al(NO_3)_3$-$Ni(NO_3)_2$-H_2O ternary system

The measured solubility data of the ternary system $Al(NO_3)_3$-$Ni(NO_3)_2$-H_2O at 40°C are organized in Table 4.

Table 4: The measured solubility data of the ternary system $Al(NO_3)_3$-$Ni(NO_3)_2$-H_2O at 40°C

W($Al(NO_3)_3$),%	W($Ni(NO_3)_2$),%	Limit domains
0	55.62	Liquids+Ni_6
5.02	50.06	--
7.075	47.69	AL_9+Ni_6+Liquids
9.79	43.35	Liquids+Al_9
14.08	36.36	--
18.47	30.66	--
26.21	19.34	--
33.90	9.38	--
41.15	0	--

CONCLUSION

The phase equilibrium diagrams can be easily determined using only a synthetic method based on electrical conductometry. The conductivity measurements permit, in general, to define the equilibrium curves between solid-liquid as well as liquid-liquid phases. In this work, we have investigated the phase diagram of the H_2O-$Ni(NO_3)_2$- $Al(NO_3)_3$ ternary system at 20, 30 and 40°C by using this technique. 20, 30 and 40°C isotherms of H_2O-$Al(NO_3)_3$-$Ni(NO_3)_2$ system are composed by two curves corresponding to the liquids of the $Ni(NO_3)_2.6H_2O$ and $Al(NO_3)_3.9H_2O$ solid phases. One equilibrium invariant isotherms isobars is found: Liquid $Ni(NO_3)_2.6H_2O$+ $Al(NO_3)_3.9H_2O$

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