THE DIFFERENTIAL THERMAL ANALYSIS OF A SERIES OF INTERMEDIATE CATALYSTS IN THE $xFePO_4\cdot yNi_3(PO_4)_2$ SYSTEM

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In this work, by means of a method of the differential thermal analysis (DTA), the behavior of a series of catalysts to K-1–K-7 which are implemented in the $xFePO_4\cdot yNi_3(PO_4)_2$ system is investigated temperature.

The corresponding series of catalysts to K-1–K-7 was synthesized according to the technique developed at department of physical and colloidal chemistry of the Uzhhorod national university [1, 2]. Processes of thermal dehydration of output hydrates of phosphates FePO$_4$ and Ni$_3$(PO$_4$)$_2$ are investigated in works [3, 4]. Relying on these results us transformations which happen when heating intermediate samples of catalytic $xFePO_4\cdot yNi_3(PO_4)_2$ system are described.

The K-1 catalyst on the structure the next to initial iron (III) orthophosphate crystalline hydrate (FePO$_4$•2H$_2$O) also has the most difficult enrollment of effects (fig. 1). The first of them and the only endothermic is observed at 139ºС. This effect corresponds to the beginning of loss of crystallizational water and destruction of structure of basic hydrate. All subsequent effects which are observed on the thermogram belong to exothermic. At a temperature 302ºС the most intensive is fixed
among them which answer the beginnings of crystallization of the dehydrated amorphous phase.

Fig. 1. Thermograms of a series of catalysts in the xFePO₄•yNi₃(PO₄)₂ system.

The following coupled effect at 528 and 560°C can be referred to partial transformation crystalline iron (III) phosphate. At 302°C and in the range 528–560°C crystallites of two kinds of waterless FePO₄ are formed: synthetic to a rodolicoite [5] and orthorhombic modification [6]. The effect at 637°C can be referred to recrystallization processes when the sample completely loses chemically bound water and already on the structure it is close to completely dehydrated FePO₄ [1]. The last from observed exoeffect at 858°C can be carried to α→β phase transition (high-temperature transformation) waterless iron (III) orthophosphate [7]. The contribution of other component of Ni₃(PO₄)₂ in the system has to be minimum as its share in the K-1 catalyst is insignificant.

Catalysts K-2 and K-3 have considerable similarity in thermograms to sample K-1. They are also characterized by one considerable by the size endoeffect at 145°C. Also we will note that at increase in a share of nickel upon transition from sample K-2 to K-3, the areas exoeffect significantly decrease. It demonstrates about
reduction of impact of iron containing structural modifications by the general physical and chemical characteristic of these catalysts.

Further increase in nickel in sample K-4 lowers temperature uniform to an a little endoeffect to 119°C. Also it is fixed exoeffect at 735°C which is already shown on the catalyst to K-3 (fig. 1). Possibly, at this temperature there is a formation of a quantity of nickel (II) pyrophosphate.

The thermograms of catalysts K-5–K-7 have considerable similarity. On all presented samples it is observed only endoeffect at 123ºС and one insignificant on the area exoeffect which can be carried to crystallization of Ni₂P₂O₇ [4].

The K-7 catalyst on the structure least of all differs from output hydrate of Ni₃(PO₄)₂. It agrees [4], up to the temperatures of 360–400°C octahydrate of nickel (II) phosphate loses the most part of crystallizational water – about 7 mol on one formular unit initial connections. At a temperature 420°C in a product contents (up to 7%) a pyro- and polyphosphates are observed low [4]. At 608°C it is observed only, but insignificant in size exoeffect that corresponds to partial crystallization of the dehydrated Ni₃(PO₄)₂. Further to 780°C leads heatings of a sample to increase in amount of orthophosphate and significant decrease in quantities of a pyro- and polyphosphates. At the same temperature, without visible changes on the thermogram, the catalyst becomes completely dehydrated.

References:


