

# The investigation of iron oxyhydroxides precipitation process by potentiometric titration method

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**Abstract** – It was investigated the iron oxyhydroxide precipitation process by potentiometric titration method of such systems:  $Fe_2(SO_4)_3 \cdot X \cdot H_2O$ ,  $Fe(NO_3)_3 \cdot X \cdot H_2O$ ,  $FeCl_3 \cdot X \cdot H_2O$ , where  $X$  –  $NH_4OH$ ,  $Na_2CO_3$ ,  $CO(NH_2)_2$ . It was determined the optimum ratio (equivalent) between the amount of a precipitator and an initial salt solution on the base of the experimental data. It was assumed previous conditions of carrying out the precipitation process for this ratio.

Key words – metal salt, precipitation, iron oxyhydroxide, potentiometric, excess, optimum conditions.

## I. Introduction

Iron oxides (a group name for oxyhydroxides and oxides) have found important applications as pigments, catalysts, gas sensors, magnetic materials, etc. For that reason the syntheses and characterizations of iron oxides have been still extensively investigating [1]. In the most of the work done researchers are studying the iron oxides precipitation process from ferrous salts [1, 2].

The ferric salts preference are not random in this work because the pH value of the beginning and ending precipitation for iron (II) oxides are greater than that one for iron (III) oxides [3]. As a result, the precipitator amount required for the process is significantly reduced, when used iron (III) salts as an initial raw material. The selection of needed ratios precipitator: iron salt will enable to optimize the process by reducing material consumption.

It is proposed to use iron (III) sulphate, nitrate and chloride in order to get the most full and informative representation of this process. According to [4] the process should take place in the conditions of three-multiple precipitator excess (NaOH, KOH) to obtain iron (III) oxides from these solutions. At the same time alkali metal salts, those are difficult or not washed, are obtained indirectly. Therefore, it is proposed to use ammonia, sodium carbonate and hydrolyzed carbamide solutions to select and justify the choice of precipitator in the iron (III) oxide precipitation process.

## II. Experimental

For the experiment such reactants were used:  $Fe_2(SO_4)_3 \cdot 9H_2O$ ,  $Fe(NO_3)_3 \cdot 9H_2O$ ,  $FeCl_3 \cdot 6H_2O$ ,  $NH_4OH$ ,  $Na_2CO_3$  and  $CO(NH_2)_2$ . The solutions were prepared of 0,1N concentration. Hydrolyzed carbamide was obtained in conditions, described in [5].

The investigations were conducted by the direct potentiometric titration method by using of pH-meter

(pH-150MA) fitted with the measuring electrode EVL-1M3 and “Ezodo” – pH-meter fitted with the combination glass electrode and temperature compensator. Accuracy:  $\pm 0,01$  pH, permissible measuring range: 0-14 pH. The standard methodology was used [6, 7]. Equivalence point was determined by a differential method.

## III. Results and Discussion

The potentiometric titration curves for  $Fe_2(SO_4)_3 \cdot X \cdot H_2O$  system are presented on Fig.1.

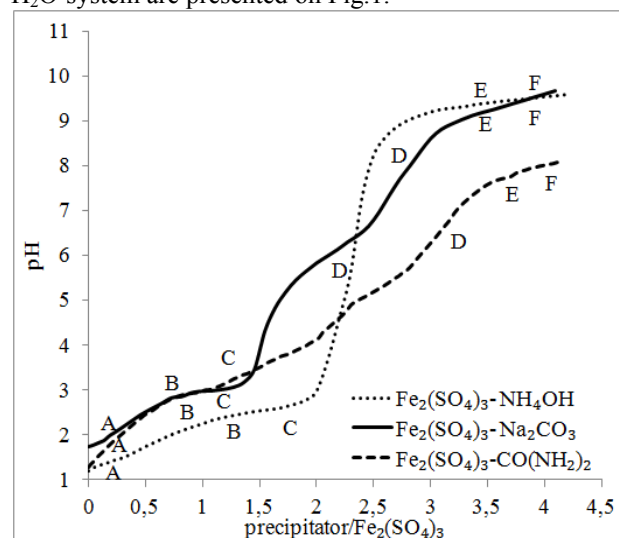


Fig.1 Potentiometric titration curves

It is seen from the presented curves next:

The initial pH of iron (III) sulphate solution is 1,21-1,74 (point A). In all cases it is clear that the precipitator adding raise the medium pH (AB line segment) to that time when the iron (III) ions precipitation pH is reached (2,3 – for the beginning of the precipitation process, ~9 – for the ending). To start the precipitation process the ratio precipitator: iron (III) sulphate has to be 0,6-1,2 (BC line segment) for different precipitators. The next more or less uniform pH increasing is caused by the following addition of the precipitator. When the ratio precipitator: iron (III) sulphate is 2,4-3,2 we can speak about the reaching of the inflection (equivalence) point (CD line segment). It is determined by the highest peak point on the differential curves. The addition of the precipitator with the precipitator: iron (III) sulphate ratio that exceeds the 3,4-3,9 value (point F) is not reasonable in this case.

The investigation results for  $Fe(NO_3)_3 \cdot X \cdot H_2O$ ,  $FeCl_3 \cdot X \cdot H_2O$  systems are presented in Table 1. The data obtained and calculations allow to choose the most optimum conditions for the carrying out of the iron (III) oxides precipitation process.

Based on calculations the precipitation processes were carried out. As an initial raw material iron (III) sulphate, nitrate and carbamide were used (precipitates obtained from chloride salts are not so stable [4]). The adoption of carbamide as a precipitator is caused by the necessity of more mild realization regime of the precipitation process that can significantly influences to the precipitate characterizations (ammonia and sodium carbonate solutions can form local supersaturation zones over the

solution volume, sodium carbonate – to form salts that are difficult to wash [8]).

TABLE 1

THE RESULTS OF IRON SALTS POTENTIOMETRIC TITRATION

Iron salt solution	Precipitator	Precipitation beginning		Point of inflection		Curve levels off	
		pH	ratio	pH	ratio	pH	ratio
nitrate	NH <sub>4</sub> OH	2,6	0,6	6,7	1,6	8,8	2,0
	Na <sub>2</sub> CO <sub>3</sub>	2,6	0,5	9,2	2,8	9,6	3,4
	CO(NH <sub>2</sub> ) <sub>2</sub>	2,7	0,2	5,4	1,3	8,1	2,0
chloride	NH <sub>4</sub> OH	2,1	0,2	8,1	1,7	8,9	2,6
	Na <sub>2</sub> CO <sub>3</sub>	2,7	0,4	8,4	2,8	9,7	4,2
	CO(NH <sub>2</sub> ) <sub>2</sub>	2,7	0,4	5,8	1,8	8,6	2,9

Carbamide was taken with excess: 1,6 – for iron (III) nitrate, 3 – for sulphate. The highest precipitation degree that was obtained for today is about 58% for the precipitation from nitrate. The filtration process of precipitates obtained was investigated. The laboratory setting working at constant pressure was used for our research. The resistance change of different age precipitates (2-15 days) was studied. In the experiments it was set: by the precipitates ageing process in mother precipitation liquor the resistivity is decreased. Next results were received (Table 2) [9]:

TABLE 2

THE RESISTIVITY OF IRON HYDROXIDE PRECIPITATE

Sample	Precipitate age, day	The precipitate resistivity, $r_m \cdot 10^{12}$ , m/kg
1	2	6,9
2	7	3,4
3	12	4,4
4	15	2,6

## Conclusion

Potentiometric titration in systems Fe<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>-X-H<sub>2</sub>O, Fe(NO<sub>3</sub>)<sub>3</sub>-X-H<sub>2</sub>O, FeCl<sub>3</sub>-X-H<sub>2</sub>O was carried out in order to study iron (III) oxides precipitation process, and namely to determine the most optimum ratios precipitator: salt solution. According to the results of experimental data calculations the precipitation processes from iron (III) sulphate and nitrate solutions with carbamide were conducted. The iron hydroxide precipitate FeOOH was obtained as a result of the precipitation process. By the ageing process studying it was set that the precipitate

resistivity decreasing is occurring. It indicates the crystal's growth of iron hydroxide obtained. To utilize mother precipitation liquor and sewage water biochemical method can be used. The precipitates obtained are highly dispersed and easily filtered.

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