Automated Control and Monitoring Systems for Technological Processes in Ammonium Nitrate Production

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Abstract – Two automated control and monitoring systems for the neutralization process in ammonium nitrate production have been elaborated. The methods are based on the dependence of measuring electrode potential on the concentration of nitric acid and ammonia in a neutralizer. The systems have been shown to be efficient in wide concentration and temperature ranges. In this paper, the monitoring system for the concentration of water in ammonium nitrate melt has been also presented. The proposed systems has been tested in industry and implemented into production.

Key words – automated control, redox potential, neutralizer, ammonium nitrate, concentration sensor, solidification, eco-friendly production.

I. Introduction

The most important challenges of the modern chemical technology are to improve productivity, to increase quality and to reduce environment pollution with current human labor levels. In certain cases, automatization, that is the use of control and monitoring systems, is an effective way to solve these problems. One of such processes is ammonium nitrate (AN) production.

AN is the second (after urea) major nitrogen fertilizer with respect to its high nutrient content as well as to its actual world production (about 16 million t/year) [1]. The AN production process comprises three main unit operations: neutralization, evaporation and solidification (prilling or granulation) [2].

The neutralization of diluted (55 – 65 %) nitric acid with gaseous ammonia by the reaction:

$$NH_3 + HNO_3 = NH_4NO_3 + Q \qquad (1)$$

is a highly exothermic process, which can be performed at atmospheric or at elevated pressure (up to about 4.5 bar). The neutralizers are designed to ensure that the most part of the reaction heat is used to evaporate as much as possible water from the obtained AN solution [3].

The most common way to control the neutralization step is to monitor HNO_3 and NH_3 stoichiometry using pH glass electrodes [4]. However, this method has a great response time, is dependent on a stable ammonia flow and is not useful at the start of the unit. Thus, the process steam, generated in the neutralizer and the evaporator, contains rather high concentrations of ammonia and AN. Emissions from neutralizers are very difficult to remove because the particles are very fine. Therefore, various approaches have been proposed and developed with the intention to control the process more accurately [5, 6].

II. Results and discussion

In this regard, we elaborated the automated control and monitoring system for the neutralization process, in which the HNO_3 and NH_3 stoichiometry is maintained on a potential difference between the working platinum electrode and the reference electrode. The dependence of the working electrode potential on the composition of the reaction mixture was found as S-shaped curve (Fig. 1).

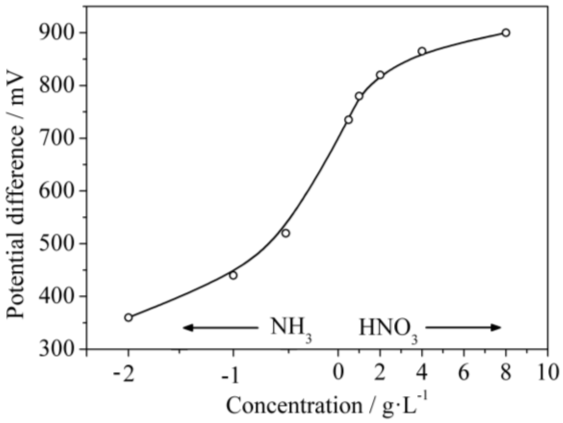

Fig. 1. Dependence of the potential difference between working and reference (E = 0,202 mV) electrodes on the concentration of reactants at T = 160 °C

Thus, the method allowed to maintain the set concentration value of ammonia or nitric acid. The system was tested in industrial environment and was proved to be effective in the temperature range 130 – 165 °C and AN concentration up to 80 %. Although established control and monitoring system was more reliable than the conventional one, it had a few disadvantages. Since the working electrode potential corresponded to the composition of the mixture at the point of its installation, efficient concentration control was impossible.

So, we have improved the design of this system and, herein, we present the automated control and monitoring system for the neutralization processes in AN production based on the dependence of redox potential of reactor walls on concentration of reactants. Using the metal walls of the reactor as a measuring electrode eliminates the concentration gradient effect on potential difference. Moreover, the system is also provided to control the concentration of excess HNO_3 or NH_3 at the neutralizer outlet, as well as at next stages of technological process. Measurement of the concentration at these points is based on the dependence of redox current on potential difference (Fig. 2). Working at different potential ranges allows to measure either concentration of ammonia (ΔE = 1200 – 1500 mV) or nitric acid (ΔE = -300 – 100 mV).

The proposed control and monitoring system was also tested in industrial environment and was shown to be effective in the temperature range 115 – 195 °C, concentration ranges 0.1 – 1 $g \cdot L^{-1}$ (NH_3), 0.5 – 20 $g \cdot L^{-1}$ (HNO_3) and pressure up to 6 bar.

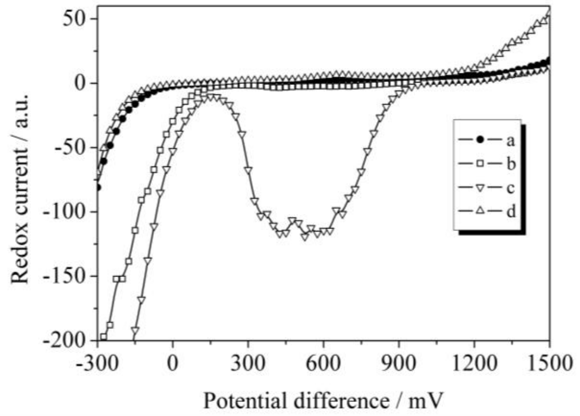

Fig. 2. Dependence of the redox current on the potential difference at neutral medium (**a**), $C(HNO_3) = 5\ g \cdot L^{-1}$ (**b**), $C(HNO_3) = 10\ g \cdot L^{-1}$ (**c**), $C(NH_3) = 0.4\ g \cdot L^{-1}$ (**d**)

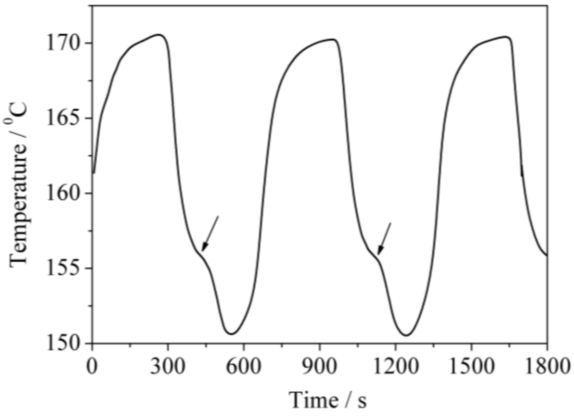

Fig. 4. Dependence of the temperature on the sensor time showing the determination of the AN melt crystallization point. The needed temperature is marked by an arrow

The system has been implemented in the AN production in a number of chemical plants in Ukraine and abroad. It has been found that the method allows to reduce the consumption of ammonia and nitric acid as well as to make the process more environmentally friendly (Fig. 3). It has been estimated that the economic impact of the introduction of the system into production is 700 thousand dollars per year.

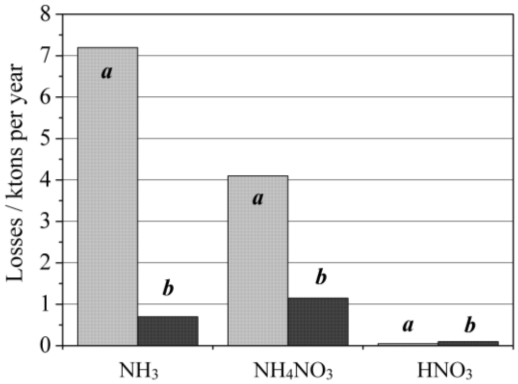

Fig. 3. Technological losses before (**a**) and after (**b**) the implementation of the control and monitoring system at "Severodonetsk Azot"

The last stage in AN production is solidification, that is making the melt into the small beads. There are a lot of quality indicators of the final product, such as concentration of water, of nitrogen oxides, *etc*. Herein, we also present the system for continuous monitoring of the mass fraction of water in the AN molten solution. It is based on the dependence of the crystallization temperature of AN melt on the water content. The concentration sensor consists of a thermocouple and the crystallization vessel cooled by the process air. The working cycle of the sensor includes the steps of cooling below the solidification temperature and heating above (Fig. 4). The inflection point on the graph corresponds to the crystallization.

The system for continuous monitoring of water content is also put into the production of AN and allows to measure water concentration in the range of 0.1 – 10.0 %.

Conclusions

Proposed automated control and monitoring systems for technological processes in AN production have several advantages in comparison with conventional systems used in chemical plants. It is shown that our methods provide non-inertial measurement process, determination of the concentrations with high accuracy, possibility of working at all stages of the process (including start of the unit). The economic effect of the introduction of the systems into production is achieved by reducing losses of ammonia and AN with process steam as well as energy consumption for evaporation of the melt, reduction of corrosion destruction of the equipment and increasing the quality of the finished product.

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