RESEARCH OF THE PROCESS OF PURIFICATION OF SPENT SULFURIC ACID PRODUCTION OF ACETYLENE (PVC1) AND CAUSTIC SODA (PVC1)

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Abstract. *Currently, there is no universal method for cleaning waste technical sulfuric acid. The technique directly depends on the impurities, volume, and final need, that is, the specific task: why purification is carried out, what needs to be obtained in the end. Also, the concentration of waste sulfuric acid complicates the process with the deposition of organic sediments on the inner surface of the pipes of expensive heaters with subsequent clogging of the pipes. This raises the problem of cleaning pipes from hardened sediments. In addition, organic impurities contribute to the decomposition of sulfuric acid with the formation of gas emissions of sulfur dioxide.*
Keywords: *spent sulfuric acid, caustic soda, neutralization, PVC, concentrate, fogging.*

As a result of modern physical and chemical methods of analyzing a sample of sulfuric acid from workshop 909 of Navoiazot JSC and its waste, the total content of organic compounds is 8.52%, and carbon – 5.65% (C-organics). Determining their individual content requires complex experimental studies, modern instruments and careful analysis of the data obtained.

The decomposition of sulfuric acid is accompanied by abundant foaming of the acid, which significantly complicates the concentration process. Therefore, when choosing a method for concentrating spent sulfuric acid, it is necessary to take into account the nature of the impurities present and strive to get rid of them or reduce their content to a minimum by careful settling or extraction from the acid before concentration [1].

The best option is considered to be the neutralization of waste technical sulfuric acid. The introduction of a neutralization department into commercial operation will ensure the utilization of technical sulfuric acid. To do this, we conducted an experimental study using the following method of neutralizing spent sulfuric acid from liquid and solid organic impurities using liquid ammonia. When selecting equipment and pumps for storing and pumping waste sulfuric acid from PVC-1 to PVC-2, it is necessary to know their rheological properties. Therefore, at the beginning of the study, rheological properties were determined in the temperature range 20–80°C using pycnometers and hydrometers. The data is shown in table 1.

Rheological indicators show that the density of sulfuric acid remains unchanged with increasing temperature, i.e. almost doesn't change. This is explained by the fact that the sample contains sufficient quantities of viscous sulfur-containing organic compounds.

Table 1

Rheological properties of sulfuric acid from PVC-1 to PVC-2

№	Workshop name	Density, g/cm3 Viscosity, cP				Density, g/cm3 Viscosity, cP			
		°C. at temperatures							
		20	40	60	80	20	40	60	80
1	Workshop 909	1,687	1,670	1,654	1,630	65,78	59,01	41,94	22,92

To determine the type of organic compound, the IR spectroscopic method of analysis was used (Fig. 1) [2].

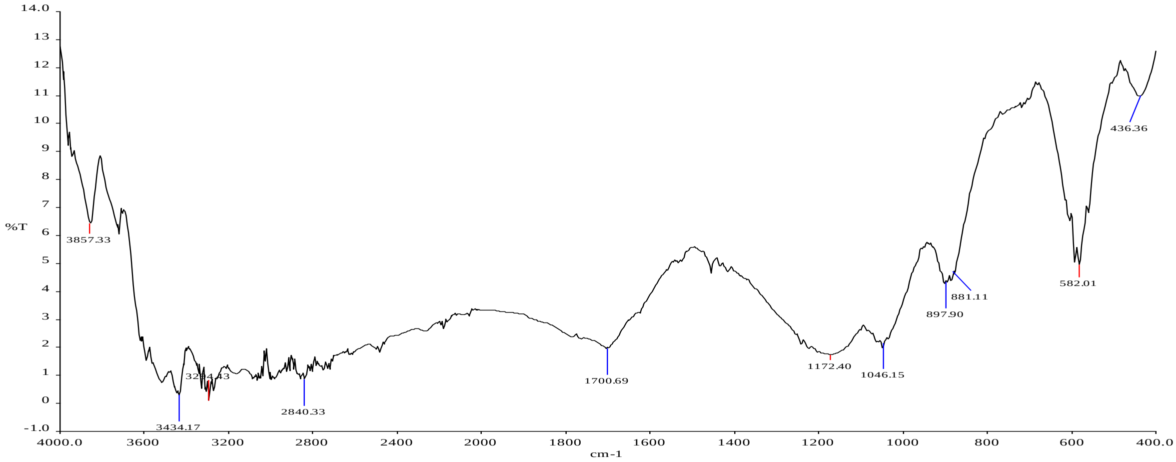

Fig. 1 IR spectra of a sample of the initial sulfuric acid waste from workshop 909

As the IR spectrum curve of the samples of the initial sulfuric acid waste and its analysis (Table 2) shows, the third sample is complex, i.e. the limiting and unsaturated range of classes of organic compounds: from simple alcohol, ketones, carboxylic acids to simple and esters.

Table 2

IR spectroscopic analysis of a sample of sulfuric acid waste from workshop 909

Characteristic vibrational frequencies of organic compounds /70/		Sample of workshop 909
Groups and types of vibrations	Frequency range (cm-1), intensity of absorption bands	
Simple covalent C-H bonds		
	1470–1430 (average)	
Csp3 –H deformation	1380–1370 (strong)	
	3040–3010 (average)	
Csp2 –H		
valence (=CH–)		
Simple covalent X–H bonds		
O-H free valence deformation		
O–H, H-bonded valent alcohols, phenols, carbohydrates		
Simple X–Y covalent bonds		
C–O valence primary alcohols alcohols secondary alcohols tertiary phenols	1075–1000 (strong)	

Ethers dialkyl (–CH2–O–CH2–)	1150–1060 (very strong)	
C–S	710–570 (weak)	
		690
Double covalent bonds X=Y		
C = O valent saturated aldehydes, ketones, carboxylic acids, esters	1750–1700 (strong)	
Unsaturated and aromatic aldehydes and ketones	1705–1660 (strong)	
		6 5 2

As studies have shown, the sulfuric acid sample from workshop 909 cannot be concentrated by evaporation. The high corrosive chemical activity of concentrated sulfuric acid did not allow us to determine the quality and quantity of the organic component directly from the acids. Therefore, in order to reduce the activity of the acid and (separation of) fractionation of the organic phase, as well as to propose an optimal option for its processing, the process of neutralizing the sulfuric acid waste from the workshop with ammonia was studied.

During direct ammoniation, strong heating of the reaction mass is observed due to the release of a large amount of heat [3]. Therefore, the sample was diluted with water at a ratio of H2SO4:H2O = 1:1 and ammoniated to pH 1.2 and 7. When ammoniated to pH = 2, the process proceeds quite slowly, and after pH = 2, in a short time, the pH quickly rose to 7 or more. The obtained data are shown in Table 3.

Table 3

Study of the process of ammoniation of waste sulfuric acid from workshop 909

№	pH	The proportion of sediment adhering to the wall relative to массы H_2SO_4, %	Black precipitate relative to H2SO4 mass, %	Liquid phase relative to the initial mass of the mixture, %	Proportion of salt relative to the mass of H2SO4, %
1.	1	0,4892	5,29	98,75	-
2.	2	2,84	1,68	69,044	35
3.	7	2,98	3,882	69,4	53,33

From Table 3 it can be seen that the organic mass is distributed into three parts:

- the first part sticks to the walls of the reactor;

-the second part crumples on the surface of the reaction mass;

-the third part remains in the liquid phase of the reaction mass.

Neutralization of sulfuric acid (84.78%) with liquid ammonia is accompanied by the release of heat and ammonium sulfate in the form of crystals. The pH values of acidity were adjusted from 1 to 7. In the selected measurements and conditions, solutions with ammonium sulfate crystals with a total mass of sulfuric acid of 53.3% were obtained.

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