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STUDY OF THE INFLUENCE OF THE QUALITY OF THE FEEDSTOCK ON THE PROCESS OF CATALYTIC OXIDATION OF SO₂ TO SO₃ IN A CONTACT APPARATUS FOR THE PRODUCTION OF SULFURIC ACID

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ABSTRACT In the modern sulfuric acid industry, vanadium catalysts play an important role in the oxidation of sulfur dioxide (SO₂) to sulfur trioxide (SO₃). However, the efficiency of these catalysts can be significantly reduced by the presence of impurities such as calcium and iron. In this article, we will consider the effect of these impurities on the operation of the first layer of vanadium catalyst and try to identify the optimal conditions for ensuring stable operation of the process, starting from the feedstock and ending with the compounds formed as a result of combustion in furnaces. Special attention is paid to the requirements for the feedstock, because they can significantly affect first the heat transfer, and then the activity of the catalyst and its hydraulic resistance. It has been shown that factors such as the origin of sulfur, the method of its transportation and the combustion temperature can be the main sources of calcium impurities, while the temperature of the sulfur combustion process, corrosion products of metal parts of the furnace and boiler recycler are the main sources of iron impurities. In addition, attention should be paid to the influence of the physicochemical properties of the starting material on the process of formation of calcium and iron compounds. It is important to consider not only the composition of the raw material, but also the conditions of its extraction, transportation and storage, since this can significantly affect the quality of the final product and the efficiency of the production process. It should be noted that controlling and minimizing the level of calcium and iron impurities is important for ensuring the stability of sulfuric acid production and preventing possible technological failures. For this, it is necessary to develop effective methods for monitoring and regulating the content of these elements at different stages of production. Studies show that calcium and iron impurities can lead to the formation of precipitates on the catalyst surface, which complicates the accessibility of active reaction centers and reduces its activity. This negatively affects the conversion of SO₂ to SO₃ and, accordingly, the product yield. Understanding the mechanisms of action of these impurities will help optimize the composition of vanadium catalysts and develop methods to prevent possible negative consequences of their use.

Keywords: vanadium catalyst; combustion; sulfur; sulfuric acid; impurities; calcium compounds; iron compounds

ДОСЛІДЖЕННЯ ВПЛИВУ ЯКОСТІ ВИХІДНОЇ СИРОВИНИ НА ПРОЦЕС КАТАЛІТИЧНОГО ОКИСЛЕННЯ SO₂ ДО SO₃ В КОНТАКТНОМУ АПАРАТІ ДЛЯ ВИРОБНИЦТВА СІРЧАНОЇ КИСЛОТИ

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АНОТАЦІЯ У сучасній промисловості виробництва сірчаної кислоти важливу роль відіграють ванадієві каталізатори, що використовуються для окислення сірки діоксиду (SO₂) до триоксиду сірки (SO₃). Однак, ефективність роботи цих каталізаторів може значно знижуватися через наявність домішок, таких як кальцій та залізо. У цій статті ми розглянемо вплив цих домішок на експлуатацію першого шару каталізатора ванадієвого і постараємося виявити оптимальні умови для забезпечення стабільної роботи процесу, починаючи з вихідної сировини і закінчуючи сполуками, що утворюються в результаті горіння в печах. Особлива увага приділяється вимогам до вихідної сировини, тому що вони можуть суттєво впливати спочатку на тепловіддачу, а потім активність каталізатора та його гідравлічний опір. Було показано, що такі фактори, як походження сірки, спосіб її транспортування та температура горіння можуть бути основними джерелами утворення кальцієвих домішок, в той час як температура процесу спалювання сірки, продукти корозії металевих деталей печі та утилізатора котла є основними джерелами утворення залізних домішок. Крім того, слід звернути увагу на вплив фізико-хімічних властивостей вихідної сировини на процес утворення сполук кальцію та заліза. Важливо враховувати не лише склад сировини, а й умови її видобутку, транспортування та зберігання, оскільки це може суттєво вплинути на якість кінцевої продукції та ефективність виробничого процесу. Слід зазначити, що контроль та мінімізація рівня кальцієвих та залізних домішок мають важливе значення для забезпечення стабільності виробництва сульфатної кислоти та запобігання можливим технологічним збійм. Для цього необхідно розробити ефективні методи моніторингу та регулювання змісту даних елементів на різних етапах виробництва. Дослідження показують, що домішки кальцію та заліза можуть призводити до утворення опадів на поверхні каталізатора, що ускладнює доступність активних центрів реакції та зменшує його активність. Це негативно впливає на конверсію SO₂ в SO₃ і, відповідно, на вихід продукту. Розуміння механізмів дії цих домішок допоможе оптимізувати склад ванадієвих каталізаторів та розробити методи запобігання можливим негативним наслідкам їх використання.

Ключові слова: ванадієвий каталізатор; горіння; сірка; сульфатна кислота; домішки; сполуки кальцію; сполуки заліза

Introduction

One of the main problems in the operation of the first layer of vanadium catalyst for oxidation of SO₂ to

SO₃ in the production of sulfuric acid is the rapid growth of hydraulic resistance of the catalyst due to the influx of solid impurities (ash) in the form of dust [1-4]. This is

especially true after starting operations and after reloading the catalyst.

According to some authors [5,6], a component of this "ash" is a combination of calcium compounds in the form of CaO and iron in the form of sulfate salts such as FeSO_4 and $\text{Fe}_2(\text{SO}_4)_3$. The main way to remove such "ash" from the technology is the use of filters - settling tanks and liquid sulfur filters, which are installed in the supply of liquid sulfur at the entrance to the sulfur combustion furnace. But these methods only solve the consequences of the presence of undesirable impurities in production and do not solve the problem of their occurrence.

Purpose of work

The main purpose of this work was to address the origin of these calcium and iron compounds, so that in the future it was possible to take measures to eliminate them. Let us first consider the question of the nature of the occurrence of calcium compounds in reaction gases.

Presentation of the main material

Considering the possible sources of calcium during sulfur combustion, it can be assumed that it may arise either from sulfur itself, or from the destruction products of the lining of the sulfur combustion furnace and cement mortar used to bind this lining, or introduced into the furnace by some other means in the form of various impurities.

Sulfuric acid companies typically use lump sulfur grade 9950, which is a product of natural gas purification, and high-quality sulfur with a low carbon content of grades 9990 and 9998 as raw materials.

According to the technology, when sulfur enters the furnace through the nozzles, it must be liquid and free of ash and iron, not only to prevent clogging of the nozzles, but also, mainly to reduce dusting of the gas stream by solid particles that are deposited in the boiler. reducing its heat transfer parameters, and then planted on the catalyst, reduce its activity and increase hydraulic resistance.

Therefore, for liquid sulfur there are the following requirements:

- ash content not more than 0.003%;
- the content of organic impurities is not more than 0.06%.

When transporting lump sulfur, as a rule, open gondola cars are used, where the hatches are fixed with iron wire, the gaps are sealed with boards and plugged with paper. And all these impurities get into the sulfur, which, in turn, leads to difficulties in the operation of sulfur smelters and filters.

In addition to ash and organic impurities, the operation of the raw material preparation unit is complicated by corrosion of the equipment. One of the factors that can cause corrosion is the use of sulfur as a raw material, which is used in acid treatment, which takes place in some industries in other countries. The content of sulfuric acid in the sulfur obtained in this way is up to 0.1%, and the content of chloride ions up to 0.01 to 0.04%. An example is the use

of sulfur a few years ago at the Sumykhimprom plant, which was purified using AlCl_3 . When such sulfur got into the smelting furnace, which was made of ferrous metal, intense corrosion of the metal was observed.

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The process of sulfur combustion has been sufficiently studied [7-9] and we should only note that at high temperatures the rate of the chemical reaction of sulfur oxidation is greater than the rate of physical processes occurring in the furnace. Therefore, the speed of the sulfur combustion process will be limited by the processes of mass and heat transfer, ie it takes place in the diffusion mode.

In the study of burning sulfur droplets using kinograms and oscillograms, it was confirmed together with the authors [8,9] that the process has an "explosive" nature, and the evaporation time of sulfur droplets is less than their time in the combustion chamber. In addition, it was found that the process of combustion in the flare mode is much faster than from the surface of the droplets, due to the lack of products of interaction of impurities with sulfur in the reaction gases.

In the course of research, the quality of granulated sulfur of Kazakh origin, obtained from oil and gas raw materials, was analyzed for the presence of calcium and iron compounds in sulfur.

According to the results of research it can be concluded that a significant amount of calcium and iron in the samples of granular sulfur was not found, their content was less than 0.015% of the mass.

It is known from the literature [2,8,10] that solid and organic impurities are separated from the molten sulfur in the process of its settling, and, in addition, before feeding into the furnace, the melt is filtered. Lime (0.15 kg / t of sulfur) is sometimes added to sulfur to neutralize acidic impurities and prevent corrosion, and this may be one of the sources of calcium introduced into the study.

Another object of study was the lining of the sulfur combustion furnace. An analysis of the literature on the theory of the use of refractory materials for thermal insulation of thermal technological units revealed that there are no special materials for lining in the sulfuric acid industry, namely sulfur combustion furnaces.

All proposals in this regard are based only on theoretical considerations about the critical combustion temperature of sulfur 1200 - 1300 °C and the provisions that at temperatures above 1300 °C the lining loses performance [11,12]. There are almost no research results on the processes that occur in the materials used for

lining, taking into account their physicochemical properties. It is not clear the nature of the processes occurring in the thermal insulation material in the conditions of operation of the furnace for burning sulfur, due to which processes is the destruction of the lining.

It can be assumed that for the metallurgical (steel, blast furnace), cement, construction, glass industry, as well as coke production, it is not particularly important, but for the chemical industry (production of sulfuric and nitric acids, etc.), where there are catalysts that react to the presence of foreign substances and catalytic poisons, it is very important.

It should also be noted that the specified temperature of 1300 °C is not critical for refractory materials, in the above-mentioned metallurgical processes, materials are widely used at operating temperatures of 1500 - 1850 °C. As a rule, refractory products are used for lining for stabilizing chambers of gas burners of the SHA and MLSG-62 brands, in which only the content of Al₂O₃ (62% by weight) and Fe₂O₃ (not more than 1.5% by weight) is normalized.

On the other hand, the clay used for the installation of refractory fireclay bricks contains:

- silicon oxide SiO₂ 60 - 80%;
- alumina Al₂O₃ 5 - 20%;
- iron oxide Fe₂O₃ and FeO - 10%;
- calcium oxide CaO 0 - 25%;
- magnesium oxide MgO 0 - 3%.

The presence of chalk particles (CaCO₃) with a size of 1 - 2 mm leads to the formation of CaO during firing.

Studies of MSh (marl-chamotte) bricks and chrome-magnesia bricks of the Slavic plant and cement, which were used in the lining of the sulfur combustion furnace at RPE "Zarya" Ltd. in May 2021, showed that the CaO content is insignificant.

The studies were performed by X-ray diffraction analysis of the phase composition of the samples on an X-ray diffractometer. X-ray structural studies of the phase composition of the samples showed that the samples have the following phases:

- cement: quartz (SiO₂), kaolinite (Al₂O₃ • 2SiO₂ • H₂O), calcite (CaCO₃), feldspars, mica;
- marl-fireclay brick: mullite (3 Al₂O₃ • 2SiO₂), quartz (SiO₂), tridymite (SiO₂), cristobalite (SiO₂);
- chrome-magnesia brick: periclase (MgO), magnesia-chrome spinel (MgO • Cr₂O₃).

It should also be noted another possible source of calcium in the system. Since Soviet times, it has been recommended to place a so-called substrate on the lattice of the catalyst bed, which usually consisted of pieces of quartz, which was necessary to maintain the heat capacity of the catalyst.

Thus, in [12] it is noted that this quartz was not specially made for the production of sulfuric acid, there were no regulations for its production and the characteristics to which it must meet were not regulated. All this led to the fact that there were cases when sulfate plants were supplied with quartz not only different in size, but also with different chemical composition, including different amounts of

calcium salts. Sometimes instead of ground quartz used the so-called fireclay crumb, which was obtained by crushing fireclay bricks and which may also contain calcium salts.

Some instructions recommended laying quartz on the surface of the catalyst bed. It is obvious that quartz can be used only after a special wash, which would eliminate the formation of dust and dirt on the quartz itself.

In modern Western technologies, such as BASF systems, it is recommended to use as the first layer special "support ceramic balls" type C167, article number BASF 54641770, which are inert ceramic balls with a diameter of about 20 - 25 mm and have a high degree of thermal resistance. and mechanical impact. The operating temperature is above 980 °C, and the low porosity and hygroscopicity do not lead to interaction with the reaction gases. These beads have the following chemical composition, % wt.: 64 - 75 SiO₂; 19-26 Al₂O₃; <1.5 TiO₂; <0.5 CaO; <1.5 Fe₂O₃.

Let us now consider the problem of iron compounds entering the first catalyst bed. It is known that at relatively low temperatures in the presence of potassium and iron can form iron-potassium alum [13], which can cause a change in the chemical composition of the active substance of the oxidation catalyst SO₂ to SO₃ by reducing the potassium content in the mass. To confirm this, an X-ray phase analysis of spent vanadium catalysts was performed, which resulted in the presence of KFe(SO₄) and [K(Fe, Al)SO₄]₂ salts, both on the surface and in the depth of the granules.

One of the possible sources of iron may be pyrite FeS₂ [13], which can be formed at the initial stage of melt sulfur, as well as in stagnant areas of the kiln due to contact of sulfur with metal parts of the equipment. Also, the corrosion products of metal parts of the equipment of the kiln and the boiler-utilizer can be a source of iron compounds, which through the pipeline is on the catalyst layer.

In [14] it is shown that per 1 m² of the surface of the first layer of the catalyst is sometimes delayed about 250 - 300 g of iron oxides, if we assume that the mass of 1 cm² of iron oxides is 0.28 g. It can be argued that under such conditions the layer will be damaged by about 3% of the surface of the catalyst layer.

The causes of corrosion can be a violation of the temperature regime in the furnace, because it is known that at temperatures of 350 - 400 °C the formation of SO₃ (up to 35%), and at temperatures of 1200 °C and above nitrogen oxides are formed [2,8,11].

Conclusions

To reduce the impact of contamination of the layers of vanadium catalysts, especially I and IV, in the DK / DA system, we propose to take the following measures:

- to organize storage of lump sulfur in warehouses, excluding its pollution by dust, lime, corrosion products;
- to improve the filtration stage of liquid sulfur, excluding impurities that may contain lime;
- maintain the norms of the technological mode of operation of the sulfur combustion furnace, excluding the operation of the furnace at temperatures below 500 °C and

above 1200 °C, understanding that at such temperatures can be reproduced acid-forming oxides SO₃ and NO₂, which can lead to equipment corrosion;

- before each start-up of the system to carry out thorough cleaning of the inner surface of the furnace from dust, corrosion products and dirt;

- install filters on the gas path of the reaction gases after the boiler-utilizer;

- carefully follow the provisions for laying and starting vanadium catalysts, especially the first layer;

- load as the first layer of the upper part of the catalyst or an annular tubular catalyst with a diameter of 10 - 15 mm, or ceramic refractory balls on a sample from BASF;

- to develop refractory elements (bricks and cement) exclusively for the sulfuric acid industry for lining, which would not contain calcium and iron compounds.

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