Modern technology of sulphide-bearing wastewater sanitation

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Abstract. The industry that forms sulphide-bearing wastewater imposes a big man-made burden on the ambient environment. Existing treatment methods of sulphide-bearing wastewater intend for insignificant decrease of sulphide concentration, water purification without further use, and storing of sediments at polygons. Transportation of sulphide-bearing wastewater causes heavy corrosion of pipe system. The developed modern technology allows carrying out quality purification of wastewater, obtaining circulating water for process purposes as well as commodity products used in the main technology. The relevant is development of optimal techniques for sanitation of sulphide-bearing wastewater from the position of using the pollutant as a finished reagent solution. Research on the processes of forming sulphide-bearing wastewater was carried out, selection of purification method by treatment using ferrous sulphate was conducted, conditions for efficient use of this method with the purpose to develop waste-free technology of purification and disposal of purification products were determined. Environmental and economic characteristics of the developed technology for sanitation of sulphide-bearing wastewater were determined.

1. Introduction

Wastewater, containing sulphides, comes from leather, oil-extracting and oil-processing industries. At that, concentration of sulphides can amount from 1 g/dm³ to 15 g/dm³.

Sulphides are toxic compounds, which impart aggressiveness and foul smell to wastewater. Presence of sulphides in water also favours formation of hydrogen sulphide which, in turn, can form sulphur dioxide under oxidation. These compounds can penetrate into concrete’s interstices and cause its corrosion, which results in destruction of container structures at water discharge networks and stations [1]. Moreover, sulphides impede activity of organisms participating in the process of biological water purification, which can disrupt operation of wastewater treatment plants. Maximum allowable concentration of sulphides before water transmission for biological purification amounts 1 mg/dm³ [2]. The effect of sulphides on human body in the form of hydrogen sulphide leads to blocking of respiratory ferment, to decreased ability of hemoglobin to transport oxygen, and to excessive stimulation of nervous system.

Complexity of purification of sulphide-bearing wastewater is conditioned by high concentration of runoffs, and by toxicity and alkaline condition of spent solutions. All these characteristics impact the choice of flow diagram at water treatment plants. The existing flow diagrams for sanitation of such runoffs are targeted at partial decrease of concentration of pollutant substances, and at water discharge...
into a receiving water body, provided the absence of appropriate control, or provided that paying penalties for water discharge turns out to be more profitable from the position of economy.

In this regard, overload of wastewater treatment plants occur, as well as their destruction, disturbance of oxygen regimen in water receiver, accumulation of sediments with high humidity, disposal of which in big amount is unreasonable, and for which it is also necessary to organize sites for industrial waste disposal. Therefore, the increasing man-made burden on the environment causes decrease of ecological state in the country and around the world. Purification degree of strong wastewater in the majority of enterprises amounts about 40-70%.

Getting into a water body, sulphides get exposed to dissociation with formation of hydrosulphide ions. Within the first minutes, oxidation to sulphite, thiosulphate and sulphate ions takes place. Complete oxidation takes place during a day. It is exactly the rapid bonding of oxygen dissolved in water that has a negative effect on the regimen in the water body. Therefore, sulphides must be totally removed from wastewater.

At that, it should be noted that formation of sulphides takes place in municipal sewage systems as well. Natural conditions for reduction of sulphur to sulphides and hydrogen sulphide get created. The formation is caused by long sections of pressure in the network, by too-low oxygen content in wastewater, which leads to formation of hydrogen sulphide in the network, by insufficient self-cleaning ability of networks, resulting in rotting of accumulated sediment, and by runoff disposal into municipal sewage system, conducted by industries that produce sulphide-bearing water.

Conditions for sulphides formation get created in piping network of municipal sewage system, therefore disposals of water bearing even small concentration of these pollutants into the network must be eliminated.

The most challenging situation regarding wastewater formation is observed in leather industry, which produces runoffs with high concentration of sulphides, runoffs with high concentration of chrome, runoffs with high concentrations of nitrogen-containing compounds, ammonium included, and runoffs with high rates of biochemical and chemical consumption of oxygen, and high concentration of suspended substances.

Transfer of such runoffs into water bodies can lead to complete destruction of their ecosystem and hydrobiological regimen.

Wastewater of leather plants can be conditionally divided into the following groups [3, 4, 5]:
1) Wastewater formed after industrial process of leather production. Content in the total runoff is 70-80%.
2) Wastewater formed due to cooling of equipment, refrigerator and compressor devices. Content in the total runoff is 6-15%.
3) Household wastewater from shower rooms and toilets. Content in the total runoff is about 5%.
4) Stormwater after atmospheric precipitation or territory washing. Content in the total runoff is about 3%.

General preparatory stages of leather production include soaking and liming processes, unhairing, deliming and softening. At that, the processes of liming and delimming produce highly concentrated sulphide runoff. Liming after soaking lies in leather working with a mixture that mainly contains calcium hydroxide, sodium sulphide and nonionic surfactant. After that, unhairing and epidermis removal take place. Deliming is intended for removal of alkali and sulphides out of the unhaired hide.

Wastewater after soaking usually is either neutral or weakly alkaline; suspended substances amount 10-20 g/dm³ after the first washing, and then remain within the limits of 0.5-8 g/dm³. This water also contains big amount of organic substances of protein origin [5 - 7].

Water after liming contains up to 18 g/dm³ suspended substances, which include lime, hair, hide shreds, etc. pH of such water ranges from 12 to 14, and the average content of sulphides amounts up to 2 g/dm³.

Water after deliming contains sulphides up to 1 g/dm³; reaction of the medium is from weakly acid to weakly alkaline, at that, there presents up to 1.5 g/dm³ of total nitrogen, the biggest part of which
composes ammonium nitrogen. At that, wastewater after the processes of liming and deliming amounts up to 25-30% from the total amount of wastewater. All process techniques require water treatment [8].

While analyzing data on sulphides content in wastewater of leather plants, a tendency of increasing concentration of pollutants was noted. This is connected with the fact that water treatment plants at the enterprises were designed long ago and intended for a lesser productivity, compared to what factories receive in the result of modernization and capacity enhancement. Moreover, pipelines for transporting sulphide-bearing wastewater get gradually destructed. As a result, efficiency of sulphide removal gradually decreases. The biggest difference is observed at the beginning of the plant’s work, when the used purification technique was prepared according to the project by its set qualitative and quantitative parameters [9].

With increase of the plant’s productivity, purification degree dropped down to 15-20%. At that, the decrease in concentration of sulphides in runoffs that were disposed for biological purification, regarding their initial concentration, was explained by the fact that aluminum chloride was used during sedimentation of coarse admixtures. Extensive cotton surface was sorbing some part of soluble substances, including sulphides.

As described in work [9], in a result of failure of pipelines for collecting sulphide-bearing wastewater, decrease of their concentration occurred due to dilution with other water.

Studying known methods of sulphide sanitation revealed the boundaries of their application. Methods of sulphide oxidation using chlorine and chlorine dioxide have downsides, such as formation of harmful chloroorganics, toxicity of reagents, and significant doses of reagents.

Atmospheric oxygen is more frequently used for oxidation of sulphides. At that, the reaction occurs in liquid phase under heating through 400°C. Efficiency of the method accounts for about 90%.

Carbon dioxide is also used for destruction of sulphides. Wastewater gets transferred into a column, to which water vapor and fume gas get delivered. As a result, sodium carbonate and hydrogen sulphide are formed and get carried away by fume gases for burning [6].

Oxidation of sulphides is also possible using ozone, though energy consumption required for producing 1 kg of ozone amounts 40-60 kWh. Ozone flowrate for sanitation will depend on many factors: reaction of the medium, concentration of substances and delivered ozone, oxidation method, and exposure time. It is necessary to take into account toxicity of ozone and the probability of explosion hazard.

Hydrogen peroxide allows selectively oxidize sulphides in wastewater due to its inertness to ammonium and organic substances. At that, it is very soluble in water, stable, and working within a wide range of pH; however, application of the reagent is held down by its cost and deficit.

Biological removal of sulphides is based on the effect of microorganisms which are capable of oxidizing sulphur-containing substances.

Research of the effect of biological removal of sulphides and hydrogen sulphide was carried out by many research institutes. Difficulty lied in determining the optimal conditions for water treatment on aerated filters, aerotanks and other similar facilities. This technology should take into account high sensitivity to the regime of wastewater delivery, dependency on pH, certain temperature range and the necessity to maintain it, requirements for concentrations of sulphur, nitrogen, phosphorus and biotoxins which should be removed, and constant supplementations for maintaining vital capacity of microorganisms in purification facilities.

Another known method of wastewater treatment allows eliminating sulphides using carbon-containing sorbent made of paddy straw. The sorbent is bundles of fibers obtained by alkaline processing, which are pulverized, washed with water and dried at room temperature. At that, claimed efficiency of sulphides removal amounts up to 90%.

Water disposal and purification of leather industry wastewater usually include preliminary removal of coarse admixtures, surfactants, phenols, sulphides and chrome. After that, wastewater gets transferred for biological purification. In this regard, several sewage networks get arranged at
industrial sites. Usually they include a network for industrial equalized wastes, for chrome-bearing wastes and for household wastes.

Preliminary treatment contains grids, hair catchers, primary and secondary sedimentation tanks, grease traps, flotation machines, in some cases – machines for sedimentation and regeneration of chrome-containing elements. Sediment, obtained at the preliminary treatment plants, gets sent for dehydramation and storage.

The method of leather plants’ wastewater purification is applied first by water acidification to pH 4-5, and then by alkalinization to 7.5-8.0 using lime, which allows removing chromium compounds, proteins and suspended matters; at that, double sediment detachment, low effect of grease removal (up to 85%) and processing complexity of plants are necessary.

According to [7], purification of total flow of the leather plant’s wastewater is proposed to be carried out using equalization, adding aluminum-containing coagulant and bringing pH to 10.3-10.7. Further goes using flotation and sludge removal, and additional treatment of circulating water with ultrasound. At that, multi-stage technology, which requires constant control and numerous personnel for maintenance, as well as using big amount of secondary reagents and devices, such as lime, aluminum-containing coagulant, flocculants, ethanolamine borate, aeration, ultrasonic units, etc., is necessary.

There are also proposals to perform purification of leather plants’ wastewater using reverse osmosis units, evaporation plants, adsorbers, etc. Application of such equipment leads to complex operation of such facilities and heavy expenses for electricity.

Process chart of leather plants' wastewater treatment has been developed; it assumes general purification of all types of sewage with obtaining flotation sludge from total runoffs, which can only be disposed as solid domestic wastes with preliminary treatment.

From the position of solving environmental problems, the most efficient technique of leather industry’s wastewater disposal is separation of soaking and liming wastewater from other wastes, which will allow carrying out their most efficient purification with less expenditures, as well as organize recycling water supply.

Therefore, it is necessary to implement process charts of local purification for each flows of wastewater, allowing for recovery of spent solutions after the main production processes. This solution will not just allow organizing purification of each runoff by the most efficient technology and implement recycling water supply, but also allow reducing consumption of reagents in main production. Consumption of reagents will only be conducted when adding them with the decrease of recovered solutions.

Table 1 describes activities proposed by us, necessary for reducing the effect of sulphide-bearing wastewater, discharged by enterprises, on the environment.

| №   | Description of activities                                                                 | Result of implementation                                                                 |
|-----|------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------|
| 1   | Sulphide-bearing wastewater, no matter where it is discharged or transferred to, must be exposed to efficient purification. | Reducing the load on ecological situation                                                  |
|     | Construction of a separate network for aggressive sulphide-bearing wastewater at production site. | Recirculation of solutions, reduced consumption of reagents                               |
| 2   | Introduction of technology allowing for a maximally efficient application of reagents and water in circulation. | Reduction of water consumption and of burden on water body and the environment             |
| 3   | Provided the impossibility to reuse reagents, it is necessary to organize obtainment of secondary raw material. | Supplementary raw material, reducing the load on the environment                           |
Introduction of the proposed activities will allow creating conditions for reconstruction of existing and design of new waste-free water carriage systems of leather industry enterprises.

2. Results
The authors carried out research for studying the nature of the sediment formed in the process of sanitation of sulphide-bearing runoff with ferrous sulphate (II), as well as research on intensification of sedimentation process with the use of various coagulants and flocculants.

First group of research is targeted at assessment of coagulants’ impact on the forming suspended matter. Commonly available aluminum sulphate (AS) and aluminum oxychloride (OXA) were chosen as coagulants. Also, an experiment was carried out for researching intensification of sedimentation process using aluminum oxychloride with a flocculant.

Second group of research was targeted at researching the process of sedimentation using aluminum sulphate with alkalinization in various doses. Simultaneously, an experiment with OXA was carried out in order to research and assess sedimentation kinetics. At that, various mixing modes were applied.

Third and fourth groups of research were intended for comparison of best samples from the previous groups, using various doses of coagulants. In the third one, low dose of coagulant was used, whereas the dose used in the fourth one was excessive.

For the research, a model of sulphide-bearing runoff containing natrium sulphide was simulated. Solution concentration equal 5g/l was chosen based on the worst conditions of incoming wastewater produced by leather industry.

Targeted dose of ferrous sulphate was determined according to stoichiometric coefficients by the chemical equation (1):

\[
\text{Na}_2\text{S}+\text{FeSO}_4 \rightarrow \text{Na}_2\text{SO}_4+\text{FeS}_\downarrow
\]

Therefore, a solution with concentration of 420 g/l expressed as 7-hydrate salt was prepared for ferrous sulphate dosage. Characteristics of obtained solutions are given in Table 2.

| Test solution | pH value | Concentration, g/l |
|---------------|----------|--------------------|
| FeSO₄         | 1,5      | 420                |
| Na₂S         | 12-13    | 5                   |

Targeted dose of ferrum-bearing reagent amounted 10 ml of solution with concentration of 420 g/l expressed as 7-hydrate salt of ferrous sulphate. Aside from studying sedimentation kinetics, experiments were carried out on the targeted dose with strongly diminished doses amounting 0,5 and 2 ml.

After dosing the ferrous sulphate, exchange reaction took place immediately, having filled the entire cylinder with black sediment of ferrous sulphide.

At that, the process of flocculation and sedimentation in the cylinders by known techniques were registered.

It was determined that formation of suspended matter and gravitational compaction occurs slowly, and clarification of supernatant layer practically does not happen; finely-dispersed suspended matter was floating in the depth of water. More extended wait that lasted 10 hours did not show positive results.

Based on known techniques, residual concentrations of sulphides, which amounted less than 0,25 mg/l, were determined. Thus, data obtained by the conducted experiment show that targeted dose allows lowering concentration of sulphides for more than 99,95%. At that, however, the processes of flocculation and sedimentation occur too slowly. As not only effect but also time of purification significantly influence the process of wastewater purification, methods for intensifying the process of ferrous sulphide sedimentation need to be researched.
In order to carry out intensification of ferrous sulphide sedimentation process, it was decided to perform coagulation of suspended matter with commonly available aluminum-containing coagulants: aluminum sulphate and aluminum oxychloride.

First group of research was targeted at assessment of coagulants’ impact on the forming finely-dispersed suspended matter.

After determining targeted dose of coagulant based on known techniques, the volume of forming sediment, the nature of flocules, the rate and efficiency of upper water layer purification, and thickening of sediment were determined.

The coagulant was added one minute after formation of suspended matter of ferrous sulphide; mixing was carried out through the entire volume of the cylinder with frequency of two turns per second.

Results of the research indicate the fact that operation of aluminum sulphate did not provide a significant effect in intensifying the process of sedimentation. Clarification of supernatant layer was insignificant.

In order to apply aluminum sulphate, water alkalinization with lime should be carried out. Addition of lime suspension will create necessary conditions for coagulation by aluminum sulphate, as well as additionally intensify the process of finely-dispersed suspended matter’s sedimentation.

An experiment of suspended matter coagulation was carried out in a similar way, using aluminum oxychloride. For that, 0.1% aluminum oxide coagulant solution was prepared.

It should be noted that within initial minutes after the reagent’s dosage, clarification of 2-3 mm of the upper layer occurred, flocules visibly got enlarged, and interflocule space became easy to look through.

As it was determined, it is within ten minutes that aluminum oxychloride forms the main amount of sediment, which in time starts thickening. At that, thickening with enhanced doses (105% and 110%) starts much earlier. It is necessary to note this important moment due to the fact that the choice of sedimentation facilities will depend on the time of formation of the main amount of sediment as well as its gravitational compaction. Nevertheless, though clarification effect is significantly more visible compared to aluminum sulphate, it still is unsatisfactory. The existing fine suspended matter of ferrous sulphide imparts strong coloring and turbidity of the wastewater under treatment. Therefore, in order to be sent for recycling water supply, such water requires additional treatment.

The conducted experiments showed that the best result was achieved with aluminum oxychloride, therefore it was decided to intensify this process by adding floacculant in doses of 0.1; 0.4 and 0.8 mg/l.

We can say that during this experiment the clarification effect significantly enhanced. Clarified upper layer of water was not colored but completely transparent, if not taking into account the “attached” flocules on the cylinder’s walls.

As it was determined by the research results, the whole sediment gets formed within the initial 5 – 10 minutes, and then it thickens, so the sedimentation process had passed more efficiently.

The next group of tests was targeted at researching the process of sedimentation using aluminum sulphate with alkalinization intended for improvement of conditions for sedimentation. At that, an experiment was conducted simultaneously using aluminum oxychloride for the experiment’s demonstrativeness.

The dose of aluminum sulphate coagulant with regard to aluminum oxide amounted 167 mg/l by calculation.

For lime dosage, a solution with CaO concentration of 76 mg/ml was prepared.

The tests were carried out using the following samples: № 1 – source runoff treated with ferrous sulphate (for comparison), № 2 – runoff containing ferrous sulphide, treated with aluminum sulphate (for comparison with samples 4 and 5), № 3 – runoff containing ferrous sulphide, treated with aluminum sulphate and flocculant (for comparison with samples 4 and 5), № 4 – runoff with ferrous sulphide, treated with aluminum sulphate featuring alkalinization, № 5 – runoff with ferrous sulphide, treated with aluminum sulphate featuring alkalinization and a flocculant, № 6 – runoff with ferrous...
sulphide, treated with aluminum oxychloride, № 7 – runoff with ferrous sulphide, treated with aluminum oxychloride and a flocculant. It was determined that alkalinization of the runoff and the use of flocculant significantly intensified sedimentation process of fine suspended matter of ferrous sulphide by aluminum sulphate. Third group of research on application of reduced coagulant doses showed the lack of efficiency in this action. Fourth group of research on application of enhanced coagulant doses showed that clarification effect only gets obtained with the use of aluminum oxychloride. Therefore, increase of the dose of coagulants will be unjustified. It was determined that working with target coagulant dose of aluminum sulphate with joint use of a flocculant forms the least amount of sediment. Within the initial half an hour, they are 18-20% of original volume of the runoff under treatment. After two-hour precipitation, the volume of sediment formed due to gravitational compaction amounted 15-16% from the original volume. Obtained sediments were treated with flocculant solutions in order to improve their filtration abilities. It was determined that the volume of sediment reduced by half within 2 hours as a result of gravitational compaction. Thus, preliminary treatment of sediments with flocculants and two-hours-long gravitational compaction should be considered as preparation for mechanical dewatering intended for further disposal [10, 11]. In order to find out the required operation time for sedimentation facilities, which are necessary in the process of sulphide-bearing wastewater deisinfecion, hydraulic size of particles excluding addition of intensifying reagents as well as including ones was determined. While researching the formed suspended matter in the course of laboratory testing, it was noted that ferrous sulphide was forming small sphere-shaped forms, at that it is necessary to take into account that the real forms are far from the “perfect conditions”.

As precipitation process takes place in a polydisperse aggregatively-unstable system with size range of particles which can agglomerate or change their form or density in the process of sedimentation, which results in alteration of their sedimentation speed. This served as a foundation for selection of sedimentation facilities: radial flow sedimentation tank, vertical flow sedimentation tank with ascending and descending flow, and a vertical flow sedimentation tank with thin-layered units. Operation factors and required depth of the flow section were determined for each type of sedimentation facilities [12, 13].

Results of the laboratory research served as a foundation for developing a process chart of sulphide-bearing wastewater sanitation.

After preliminary preparation (nominally a basic filter), runoffs get into the mixer-regulator, where qualitative and quantitative parameters of incoming sulphide-bearing wastewater get leveled. Then water gets supplied into a reactor, to which ferrous sulphate in amount equivalent to sulphides is transferred from a chemical feed plant. After a minute, lime (when using aluminum sulphate), coagulant (either sulphate or aluminum oxychloride) and flocculant get added into the water. After that, the runoff gets supplied to a sedimentation tank for water clarification and for gravitational compaction of sediment. Sediment from the sedimentation tank gets disposed to a thickener using a drainage pump, and then it gets disposed to dewatering equipment. Filtrate and clarified water from the sedimentation tank get transferred for biological purification. Purified water returns to the process cycle of production. After dewatering, sediment gets sent for disposal. After sediment filtration, clarified water gets disposed to the mixer-regulator for the necessary dilution of incoming wastewater and then undergoes retreatment.

By analyzing literature data on modern methods of industrial wastewater sanitation [14 - 21], it can be concluded that the use of belt filter press is the most optimal variant. At that, only anionic flocculant needs to be added into the forming sediment of ferrous sulphide. Application of such pattern helps reducing given expenses for sediment dewatering for 20-35%, compared to existing patterns.
Capital and operational expenditures for water treatment plants of various performance were determined taking into account preparation and dewatering of sulphide-containing sediments, which allowed for a more thorough analysis of economic indicators of local water purification plants.

The most optimal performance is from 30 m³/h to 200 m³/h. Payback period for plants within this range takes from 1.81 to 2.36 years. Payback period for plants with performance from 5 m³/h to 30 m³/h is from 3.78 to 4.32 years.

3. Conclusion

The obtained evaluation data allows concluding that introduction and application of this technology for sulphide-bearing wastewater sanitation is a profitable, fast-payback and environmentally reasonable way to ensure complete removal of sulphides from wastewater and obtain secondary product that contains sulphides subject for treatment and disposal.

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Acknowledgments
The work was supported by Act 211 Government of the Russian Federation, contract No. 02.A03.21.0011.