A new approach for recycling of spent activated sludge

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Abstract. The paper presents the basic methods of disposal and recycling of waste activated sludge. We propose a new technology of neutralization of waste activated sludge and subsequent disposal in the form of an adsorbent for cleaning oil-contaminated wastewater and production of complex fertilizers, allowing to intensify the process of phytoremediation of soils from oil.

Keywords: spent activated sludge, sorbents, fertilizers, sewage treatment.

1. Introduction

Chemical and petrochemical industry enterprises are the largest polluters of the environment, their activities produce a large number of polluted sewage. The main pollutants of sewage in chemical and petrochemical industry are heavy metal ions and oil products. Heavy metals are serious hazard in terms of their biological activity and toxic properties. Oil products getting into water affect ecological balance and make it impossible for biological systems to function normally.

In the world practice about 95% of chemical and petrochemical industries sewage is purified by biological methods, using activated sludge [1-13]. The main drawback of the classical scheme for cleaning sewage with activated sludge is the formation of a large amount of spent activated sludge as the result of the transformation of some of the initial contaminants into the active biomass. Utilization of spent activated sludge (SAS) is more labor-intensive than direct treatment of sewage, therefore, technologies that reduce the amount of this waste are economically viable and environmentally sound. There are a number of ways to recycle SAS: dropping into the seas and oceans, burning, burial in the soil, neutralization and use as organic fertilizers, as an additive in the preparation of various composts, etc.

Most often SAS is composted with household waste. Composting is a natural process of decomposition of organic waste under the influence of bacteria, insects, fungi and worms. The most widely this waste processing method is used in densely populated developed countries, where environmental problems are acute, and there is a shortage of natural resources. So, in the Netherlands 30-40% of household waste is processed for compost, in Austria and Belgium about 25%, in France 8%.
Studies have shown that adding sludge during to waste composting creates the conditions for the decomposition of the cellulosic components of the waste; in particular, it allows composting of waste containing large amounts of paper. At some composting plants in the United States addition of SAS allows to process containing up to 90% of paper waste to compost. In Germany semi-liquid precipitate with a moisture content of 92-96% (its share in the composted mass is 10-20%) and partly dewatered moisture 50-75% (its mass fraction is 14-34%) is used for this purpose.

Traditional way of field composting of waste in stacks under the open sky has not lost its importance. It is simple in technical terms, has low costs and high disinfecting effect. With this method compost is obtained from household waste and sewage sludge, which has a high agronomic value. There are two ways of composting: using so-called dynamic (with stirring of waste) and static (without stirring) stacks. Composting is carried out under the condition of forced aeration. Aerating improves the living conditions of microorganisms, thus significantly accelerating the process of waste rotting. Recycling of household waste mixed with SAS sediment by the method of field composting is organized at many specialized enterprises. For example, in the USA 180 of the 200 composting enterprises are recycle using the method of field composting.

In Poland about 4,000 tons of compost per year is produced by field composting. The waste is stacked in three rows (the width of each row is about 2 m) with a distance between them of 2.5 m. After faeces are added the bulldozer on both sides levels the waste and forms a pile with a height of about 1.5 m. In one stack there are about 700 m$^3$ of waste, and in total 16,000 m$^3$ of garbage are laid annually at the plant. Faeces are added in proportion of 3 m$^3$ per 5 m$^3$ of waste. At the same time, the initial humidity is 60-65%, which is considered optimal for the process of fermentation and obtaining ready compost with a moisture content of at least 30%.

The largest garbage processing plant in Europe, composting household waste and SAS was built in Flensburg, Germany. Its productivity is 400 tons of compost per day. The plant processes all garbage and AAS sewage from Flensburg, which was previously dumped into the Baltic Sea. The composition of nutrients produced by compost is close to manure, and the amount of lime is superior to the latter.

Italian cities (Bologna, Ferrara, Madena, Bari, and others) have centers for waste dumping and composting. Waste of meat and fish industry, oil production, winemaking and sewage sludge - SAS - are added to urban waste. Due to this nitrogen content in composts rises to 4%, phosphorus - up to 3%, potassium - up to 2%. When composting waste in stacks, bacteria are added in the calculation of 700 thousand live cells per 1 g of compostable mass, 10-20% of which are actinomycetes and streptomycetes [14].

One of the most common methods of utilization of SAS is its use as an organomineral fertilizer (OMF), thus solving simultaneously a number of tasks: the need for storage (burial) is eliminated, soil fertility and crop yields are increased and surrounding environment is not polluted.

In the former USSR, the total annual volume of SAS for 1986 was 4-4.7 million tons of dry matter. By 1990 it had to increase to 9-10 million [15,16]. Unfortunately, at the present stage the level of SAS use in agriculture of CIS countries is still low. No more than 4-6% of sewage sludge is deposited into the soil from sewage treatment facilities in large cities. Most of the waste is taken to landfills that create dangerous foci of environmental pollution. At the same time, the useful components contained in the SAS are irretrievably lost.

At the beginning of the 21st century the problem of using SAS as fertilizers for soils attracted great attention. EU countries, using SAS in agriculture, agreed that the content of heavy metals in dry matter should not exceed the following values, mg/kg: 100 Ni, 100 Pb, 300 Cr, 600 Cu, 1500 Zn, 1 Hg, etc. In Russia usage of SAS in agriculture should comply with the norms of SanPiN 2.1.753-96. According to these standards the content of HM in dry matter should be as follows, mg/kg, not more than 1000 Pb, 20 As, 15 Hg, 30 Cd, 400 Ni, 1200 Cr, 2000 Mn, 4000 Zn, 1500 Cu. For Russia there is a clear excess of the content of metals in soils3-4 times in terms of MACs and 10 times more than in the EU countries.

Many Russian and international scientists at the moment are developing technologies that allow the use of SAS as secondary raw material.
The authors of the patented technology [17] offer to process SAS containing heavy metals into fertilizers. For this purpose after thickening SAS is mixed with material containing poorly soluble calcium salts (CaCO₃, CaSO₄ · nH₂O) in the form of natural minerals, at a ratio of 5-15 parts of the material per 100 parts of sludge, stirred for 3-6 hours at 6-300 °C, dividing solid and aqueous phase containing heavy metals. The mixing and stirring stages can be carried out prior to the thickening. HM are isolated from the aqueous phase by reagent precipitation, methods of ion exchange or adsorption.

To obtain fertilizer from SAS authors [18] proposed preliminary dehydration of SAS; mixing with sand from grit chambers; disinfection by means of a reagent and production of a recycling product by further neutralizing the sediments with a reagent. A complex powder is used as reagent: clay 40.0-60.0% by weight, lime 5.0-40.0% by weight, cement 5.0-40.0% by weight, complexing agent (mixture of metal oxides, ash, crushed slag, dolomite flour), ground limestone 5.0-10.0% by weight. The amount of reagent is 10.0-30.0% by weight of the mixture. The proposed invention provides neutralization of sewage sludge from various eco toxicants (heavy metals and their toxic complexes) and utilization of crushed and crushed waste, after mechanical cleaning.

Many scientists believe that in order to reduce soil contamination while using SAS as fertilizer it is essential to apply it in strictly controlled conditions, as soils react differently to its application. Its use should be excluded on clayey and compacted soils with very low or excessively high water resistance, and also in bare and poorly drained soils where periodic excessive moistening of the upper 50cm layer is possible, since soil pH has a significant effect on the degree of mobility of HM in it, increasing or reducing their absorption by plants. On acidic soils with pH less than 5.5 SAS should not be used at all. Soils with pH 5.5-6.5 should be preliminarily limed to a pH value exceeding 6.5.

Considering dry matter, SAS contains 37-52% of proteins, 20-35% of amino acids, fats, carbohydrates, as well as B vitamins. SAS is rich in nitrogen, phosphorus, copper, molybdenum and zinc. After special processing it can be used as a protein vitamin product for feeding animals, fish and birds. The protein is considered to be the most valuable raw material, millions of tons of which are practically not used. The processing mechanism is deep hydrolysis of SAS biomass into amino acids, followed by the synthesis of amino acid esters.

Various technological schemes for the production of a protein-vitamin product ("Belvitamil"), the production of a mixture of fodder yeast with silt and the production of technological vitamin B12 for the feed industry have been developed. The preparation of vitamin B12 consists of several stages of SAS processing: it is compacted to a moisture content of 95-96%, acidified with sulfuric acid to pH = 3 and sent to the reactor where it is heated up to 110 °C with steam. After cooling, solid particles are separated from the sludge in the centrifuge, which are then dried, crushed and used as a fertilizer, and the filtrate is treated with alkali in the coagulator to pH = 5. The mass is left to stand for 1 to 6 hours, the precipitate is separated in a centrifuge and processed into a fertilizer, and the filtrate is evaporated, then treated with alkali, dried and crushed and packaged.

To improve the quality of yeast, enriching them with the necessary amount of vitamin B12, yeast mixtures with SAS is prepared in a ratio of 10:1. The feed mixture is made by mixing condensed components of yeast with silt. SAS with the humidity of 96.5% from the secondary settling tanks is sent to the tank, from where it is fed into the mixer with concentrated yeast. The resulting mixture is dried.

Microbial protein can be obtained from SAS. To produce protein by extraction concentrated SAS is treated with hydrochloric acid so that its concentration after mixing is equal to the decinormal concentration. Under these conditions, the mixture is held for 24 hours with intermittent stirring. After separating the water the sludge is treated with 0.1 N NaOH solutions and held for 24 hours. After that the alkaline liquid fraction is separated from the sludge and heated to 80-900 °C and then cooled to 250 °C and the protein is precipitated by adding 10% hydrochloric acid to pH 4.6-4.7. The protein is dried and packaged.

According to another technology microbial protein can be extracted by alkaline or acid hydrolysis of cell membranes, transfer of intracellular proteins to the solution and by precipitating them at isoelectric point. The alkaline extraction is most effective at a temperature of 80-100 °C, followed by filtration and precipitation with acid at pH 4-6. At the same time 30-40% of the protein is extracted from the sludge.
To fulfill the requirement of reliable sterilization of the product, the temperature of the hydrolysis and extraction process was selected in the range of 100-135 °C. Heat treatment time is 20-30 min. During such a period cell membranes are sufficiently deeply destroyed, but intracellular proteins do not have time to hydrolyze into amino acids and polypeptides, not settling at the isoelectric point [19, 20].

The authors [21] proposed production of artificial ground mixtures on the basis of SAS after cleaning of household and industrial wastewater, for reclamation of slurry storage. The technical result is in the transformation of sludge-sediment into soil, characterized by ecological safety and low costs of production. The mixture for reclamation of slurry tanks contains OAI 100 parts by weight (w.p.), fly ash 15-20 w.p., fine-grained filler 15-30 w.p. (Clay or loam, aluminosilicate rock 15-30 w.p.), and may additionally contain lime or Portland cement in the amount of 2-5 w.p. Mixture provides SAS cementing without its preliminary dehydration, which provides energy and labor saving, removal of sludge due to chemisorption of heavy metals contained in it, sterilization of the mixture - destruction of microorganisms, helminth eggs, etc.

The authors [22] propose technology for production of molded (briquetted, extruded or granulated) solid fuel from SAS. Molded fuel based on the dried mixture of crushed solid fuel and condensed SAS, containing, by weight: SAS with water content of 5-14% – 13-38, crushed solid fuel – the rest. The method for producing molded fuel comprises of mixing SAS and ground solid fuel, molding the mixture and subsequently drying the moldings. The mixture is formed at a pressure of 0.1-25 MPa. The moldings are dried at 50-180 °C for 1.5-0.4 hours or at an ambient temperature for 5-30 hours. Molded fuel possesses high mechanical strength, which allows reducing the cost of its storage, loading, unloading and transportation.

In Japan since 1981 there are about 500 units of final processing of waste in operation, while the amount of obtained SAS is about 24x10 m³. SAS is burned and material is obtained, which consists of 80% of the dehydrated briquette and 11% of ash (resulting from combustion after dehydration) and other waste (dry or aggregated sludge) in an amount of 9%. These wastes (42%) are buried in the ground, dumped into the sea (36%), in the amount of 15% are effectively used as fertilizers for grassland and agricultural lands.

Perm State Technical University scientists developed technology for thermal energy neutralization of refineries SAS. According to this technology SAS is mixed with calcium oxide (1.0 to 1.5% dose), dehydrated in centrifuges (up to 40% moisture) with the removal of the fugate and dried (to 10% moisture), then fed to a continuous pyrolysis furnace. Pyrolysis gas at the outlet from the furnace enters the condenser, where the liquid phase of hydrocarbons is separated with a boiling point up to 400 °C. The liquid phase can be used as fuel or returned to the technological cycle of refining oil products. The primary heating of the furnace to the operating temperature is made by burning liquid fuel (fuel oil), after removing the pyrolysis furnace to the operating mode, its heating is carried out by burning pyrolysis gases. The fugate containing HM with a concentration of not more than 500 mg / l can be returned to the biochemical sewage treatment stage or processed for isolation of HM in the form of sparingly soluble compounds. Dyakov M.S., Glushankova I.S. used obtained organomineral composition after thermoenergetic neutralization of SAS as bio sorbents in oil and oil product spills on soils of podzolic soil [23-25].

2. Materials and Methods
Despite the development of numerous methods for utilizing spent activated sludge, this problem has not been completely solved. In industrial plants where sewage contains toxic elements, it is unacceptable to use spent activated sludge as a secondary raw material without preliminary neutralization. Therefore, work aimed at developing the technology for neutralizing spent activated sludge for the purpose of its further re-usage and solving environmental problems is very relevant and of great scientific and practical importance.

The authors of this article developed technology for neutralizing spent activated sludge with subsequent utilization as an adsorbent for cleaning oil-contaminated wastewaters and obtaining complex fertilizers that make it possible to intensify the processes of phytoremediation of soils from oil products.
Research was carried out on SAS, produced at sludge sites of JSC Saratov Oil Refinery (Saratov Refinery, Russia) after biological treatment of sewage waters of the enterprise.

3. Result and Discussion

Physicochemical studies of spent activated sludge from the sludge sites of "Saratov Refinery" were carried out on the X-ray fluorescence spectrometer "Spectroscan" and the gross composition of the elements was determined. It is shown that the spent activated sludge is characterized by high toxicity due to the presence of heavy metal impurities (Table 1).

| Element | Concentration, mg / kg | Element | Concentration, mg / kg |
|---------|------------------------|---------|------------------------|
| Al      | 1.29                   | Cr      | 0.14                   |
| Si      | 2.74                   | Mn      | 3.97                   |
| P       | 3.46                   | Fe      | 27.80                  |
| S       | 4.58                   | Ni      | 0.14                   |
| Cl      | 0.70                   | Cu      | 1.04                   |
| K       | 1.09                   | Zn      | 1.01                   |
| Ca      | 8.97                   | Sr      | 0.34                   |
| Ti      | 0.40                   | B       | 0.57                   |

The thermogravimetric analysis of the spent activated sludge on the derivatograph, at a heating rate of 10 deg/min, showed that during the combustion in the furnace the decomposition process proceeds in three stages: in the first stage (20-1500 °C), water and volatile organic substances are removed; at the second stage (150-500 °C) decomposition of organic substances proceeds; at the third stage, at temperatures above 500 °C, uncontrolled combustion of samples and the formation of a coke residue are observed.

Obtained physicochemical results showed the need to neutralize the spent activated sludge for secondary use, therefore it is necessary to carry out detoxification and thermal destruction of the spent activated sludge. Thermal destruction without access to oxygen makes it possible to obtain material containing amorphous carbon with adsorption properties and to use it as an effective adsorbent for extracting oil products from sewage. Detoxification from heavy metal ions from the spent activated sludge was carried out by treating the sludge with calcium-containing reagents (calcium hydroxide, calcium oxide, calcium carbonate, calcium sulfate, calcium chloride) in the amount of 1.0; 2.5 and 5.0% of the total mass of spent activated sludge. During the interaction of calcium-containing components with spent activated sludge, the ion exchange of heavy metal ions takes place on Ca$^{2+}$ forming insoluble compounds of heavy metals that precipitate and are removed.

In order to remove pathogenic microorganisms, organic contaminants and active carbon formations, after detoxification and dehydration thermal destruction of spent activated sludge was carried out, at t = 105 °C. Conducted studies on the choice of heat treatment parameters: temperature (t = 350÷600 °C, step 50 °C) and time (τ = 20 · ÷ · 60 min, step 10 min) showed that the most rational is t = 500 °C and τ = 30 minutes (Figure 1). Under these conditions are formed materials that ensure the highest purification efficiency (75%).
Figure 1. Relation between the efficiency of wastewater treatment from oil products with adsorbent neutralized spent activated sludge and temperature (°C) and time of destruction (min).

X-ray spectral microanalysis (Figure 2) of spent activated sludge after detoxification and thermal destruction showed the presence of reflection angles (0.330) characteristic of amorphous carbon. That allows us to recommend this material as an adsorbent.

The adsorbent obtained from the spent activated sludge was tested for the ability to extract oil products from the Saratov Refinery sewage water. Adsorption was carried out for 1 hour under static conditions in a ratio of 10 g of sorbent per 1 liter of wastewater with an initial concentration of NP - 32.6 mg/l. Then the adsorbent was extracted and the final concentration of oil products was determined on the AN-1 photometer in the accredited laboratory of Saratov Refinery (Table 2). Test results show that optimum ratio of calcium-containing reagents was 1% of the total mass of spent activated sludge.

| Detoxification reagent | Content of the reagent, % Wt. | Efficiency, % |
|------------------------|-------------------------------|---------------|
| Calcium carbonate, CaCO₃ | 1.0                           | 65.0          |
| + (T = 500 °C, τ = 30 min) | 2.5                           | 63.9          |
|                         | 5.0                           | 53.4          |
| Calcium sulphate, CaSO₄ | 1.0                           | 61.6          |
| + (T = 500 °C, τ = 30 min) | 2.5                           | 56.9          |
|                         | 5.0                           | 50.1          |
| Chlorine lime: 30% Ca(OCl)₂ + 35% CaCl₂ + 35% | 1.0                           | 75.0          |
| Ca(OH)₂ + (t = 500 °C, τ = 30 min) | 2.5                           | 69.2          |
|                         | 5.0                           | 61.4          |
| Calcium hydroxide, Ca(OH)₂ | 1.0                           | 74.0          |
| + (T = 500 °C, τ = 30 min) | 2.5                           | 71.6          |
Calcium oxide, CaO + (t = 500 °C, τ = 30 min)

It has been established that the highest efficiency of treating sewage from oil products was shown by materials pretreated with chloric lime (E = 75%) and calcium hydroxide (E = 74%), taken in the amount of 1% of the total mass of spent activated sludge. The concentration of ions of heavy metals in the treatment of sludge with calcium-containing reagents in an amount of 1% is reduced significantly (Table 3).

Table 3. The content of heavy metal ions in the sludge mass before and after detoxification of spent activated sludge by various reagents.

| Calcium-containing reagent, 1% | Concentration of metals, mg / kg |
|--------------------------------|---------------------------------|
|                                | Fe^{2+}, Cr^{2+}, Cu^{2+}, Zn^{2+}, Ni^{2+} |
| Initial OAI                   | 27.80, 0.14, 1.04, 1.01, 0.14 |
| Calcium carbonate (CaCO_3)     | 19.53, 0.09, 0.83, 0.93, 0.09 |
| Calcium sulphate (CaSO_4)      | 21.21, 0.11, 0.95, 0.92, 0.10 |
| Chlorine lime (30% Ca(OCl)_2 + 35% CaCl_2) | 18.11, 0.07, 0.64, 0.68, 0.06 |
| Calcium hydroxide, Ca(OH)_2    | 12.35, 0.05, 0.42, 0.45, 0.08 |
| Calcium oxide, CaO             | 14.65, 0.06, 0.55, 0.65, 0.09 |

A technological scheme (Figure 3) for manufacturing and using an adsorbent based on spent activated sludge is proposed. Thermal processing of spent activated sludge produces flue gases of CO_2, SO_2, N_2, O_2 composition that must be to be trapped in order to prevent air pollution. Pyrolysis forms pyrolysis gases that can condense into the C11-C17 hydrocarbon fraction (M.S. Dyakov) with a boiling point up to 400 °C, which are recommended to be captured and used as a liquid fuel to maintain the temperature in the furnace. Separated precipitate of insoluble heavy metals after additional processing can be used as pigment-fillers for paint.

The adsorption capacity of the adsorbent is determined primarily by the amount and size of pores of the material. Specific adsorption capacity (E, %) for iodine was 22.86%, indicating the presence of micropores with d = 1 nm, "E" for methylene blue was 115 mg / g, which is typical for mesopores with d = 1.5-1.7 nm. Since micro- and mesopores are present in the resulting material, it belongs to polyporous systems, which is confirmed by microstructural studies (Figure 4). It is shown that the dewatered spent activated sludge at a temperature of 105 °C has a microporous surface (Figure 4, a),
while the adsorbent based on sludge after detoxification and thermal degradation has larger pores of tubular shape (Figure 4, b)

![Figure 4. Microstructure of spent activated sludge surface: a) dehydrated at t = 105 °C; b) after detoxification, dehydration and thermal degradation at 500 °C.](image)

The permissible mass ratio of the adsorbent to the volume of sewage is 10 g / l. To determine the adsorption equilibrium time and the adsorption capacity, the adsorption of oil products from aqueous solutions by adsorbent (neutralized spent activated sludge) was carried out under static conditions with stirring and thermostating of the solution (t = 22 ± 2 °C).

The spent adsorbent is proposed to be used as a burning additive in the production of ceramics and as a filler for construction, road construction, foam glass, asphalt and paving tiles.

The possibility of utilizing spent activated sludge as a complex fertilizer has also been studied. After detoxification with calcium-containing reagents (1% of the spent activated sludge mass) and thermal destruction at t = 350 °C, for 30 min. sludge was mixed with sawdust in various proportions (30:70, 50:50, 70:30%).

The resulting mixture was added into oil-contaminated soil and wheat was planted (Triticum). It was found that the use of a complex fertilizer on the basis of a mixture of neutralized activated sludge and sawdust in a ratio of 30:70% accelerates phytoremediation processes by 28% compared to soil purification technology without the application of fertilizers (Figure 5). The use of neutralized spent activated sludge as fertilizers will significantly reduce the permissible content of oil products in soils (MACs in soils are not established), solve the problem of its utilization and expand the production of organic fertilizers, that are of a very high demand.

![Figure 5. Concentration of oil products in the soils of the sludge collector after the phytoremediation process (wheat (Triticum)): 1 - without phyto-mediation processes; 2 - without the use of fertilizer; 3 - using fertilizers (70% of SAS: 30% of sawdust); 4 - using fertilizers (50% SAS: 50% of sawdust); 5 - using fertilizers (30% of SAS: 70% of sawdust); 6 - permissible level of oil products content in soil.](image)

The calculation of ecological and economic indicators of production of adsorbent from SAS in the amount of 12 tons per year showed that its cost was 44.9 rubles per kg with the selling price of 56 rubles.
per kg, thus capital costs will pay off in 1.6 years. The calculated value of the prevented ecological and economic damage of Saratov Refinery water resources contaminated with oil products by using an adsorbent based on spent activated sludge is 2.3 million rubles.

4. Conclusion
1. Main technologies for obtaining secondary raw materials from SAS and ways of using it (composting, use as fertilizer, raw materials for protein and vitamin supplementation for animals and birds, amino acids, technical vitamin B12 for mixed fodders, etc.) were considered.
2. Physico-chemical studies of spent activated sludge of "Saratov Refinery" have been carried out, the technology of its neutralization with rational parameters of sludge neutralization processes: detoxification (1% of calcium-containing reagent - chloric lime) and thermal destruction (temperature T = 500 °C and time t = 30 min) was offered.
3. The possibility of using an adsorbent from spent activated sludge obtained with the established parameters for effective purification of oily wastewater (E = 70-75%) has been offered for the first time, and methods and directions for utilization of spent adsorbents have been proposed.
4. The technology of complex fertilizer production based on a mixture of spent activated sludge and sawdust was developed. It has been established that the application of complex fertilizer (spent activated sludge and sawdust in a ratio of 30:70%) to oil-contaminated soils accelerates the processes of phytoremediation of soils from petroleum products by 28%.

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