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ADSORPTION METHODS FOR PURIFICATION OF PETROCHEMICAL INDUSTRIAL GAS EMISSIONS FROM SULFUR COMPOUNDS USING ADSORBENTS FROM INDUSTRIAL AND AGRICULTURAL WASTE. A SHORT REVIEW

A. A. Filimonova*, A. Yu. Vlasova, R. F. Kamalieva

Kazan State Power Engineering University, Kazan, Russia

A B S T R A C T

The adsorption process has established itself as one of the best technologies for cleaning process liquids and gases worldwide, and activated carbon, zeolites, silica gel, etc. are undoubtedly considered universal adsorbents for removing various types of pollutants. However, the extensive use of industrial adsorbents is sometimes constrained by their high cost. Attempts have been made to develop inexpensive adsorbents using numerous agro-industrial and municipal wastes. The use of waste as inexpensive adsorbents is attractive due to their contribution to reducing waste disposal costs, thereby promoting environmental protection. This review presents the main stages of gas emission formation at a petrochemical plant and their possible composition, and analyzes the negative impact of sulfur compounds on thermal power equipment. Based on the literature review, an extensive list of low-cost adsorbents (obtained using various types of waste) from the world literature was analyzed and compiled and their adsorption capacities for sulfur compounds were presented. It is evident from the literature review that various low-cost adsorbents have shown good potential for the removal of sulfur compounds from petrochemical industrial gas emissions. However, there are several problems associated with the use of low-cost adsorbents, which were discussed in this paper. Based on the literature review, several adsorption compositions prepared from the sludge of water treatment plants of thermal power plants were developed and presented for use as an adsorbent of sulfur compounds from gas emissions of petrochemical industries. The developed compositions had sulfur capacity similar to the literature data.

Keywords: Adsorbents; industrial waste; gas emissions; hydrogen sulfide; petroleum refinery.

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1. Introduction

The environmental crisis and the lack of fossil fuels are generating keen interest to the agricultural and industrial waste using as fuel for both traditional, widely used energy installations – boilers, gas turbines, and those that are only planned for widespread industrial use, for example, high-temperature fuel cells. Industrial and agricultural waste have a number of undeniable advantages, the main of which are the absence of fees and the environmental load reduction on the region.

At the same time, gaseous fuel from waste has a number of potentially dangerous effects for equipment: the problem of gas waste low calorific value, high water content in some types of waste, unsaturated and higher hydrocarbons in the composition, which at high temperatures decompose with the formation of carbon deposits, carbon oxides and sulfur compounds corrosion of equipment, poisoning of catalysts with sulfur. Low fuel calorific value, high water content, and carbon formation and precipitation («coking») are fuel composition problems that can be addressed by preconditioning the fuel gas through reforming.

To increase the calorific value of gaseous fuel from waste, as well as reduce negative effects for equipment and eliminate the effect of greenhouse gases (CO_2 and CH_4), various combined processes are used, for example, autothermal reforming with water vapor and oxygen [1], low-temperature steam reforming of methane, combined steam and dry methane reforming [2], the process of chemical cycling of low-temperature steam reforming [3], sorption-enhanced low-temperature steam reforming [4], as well as combined chemical cyclic low-temperature steam reforming of methane with improved sorption [5]. Each of these methods can produce synthesis gas with a different ratio of H_2 to CO, and the reaction conditions, such as temperature and pressure, determine the actual composition of the gas [6-7].

Based on the literature data analysis results and real industrial facilities, a table of potentially possible compositions of fuel gases for use in energy systems was generated [8-25]. Table 1 shows the types of industrial and agricultural gas wastes, their average composition, and calorie content.

Table 1 shows that fuel composition varies significantly.

*E-mail: aachichirova@mail.ru

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Table 1

Compositions and calorific value of various types of gaseous fuels, including fuels from waste, mass %

Fuel type	Methane	Natural gas	Agricultural biogas	Wastewater biogas	Synthesis gas from dry coal	Synthesis gas from biomass	Oil refining waste
CH_4	100.0	96.4	59.0	60.6	4	4.7	21.3
H_2	0.0	0.0	1.0	0.0	27.4	20.0	3.8
CO_2	0.0	1.6	32.0	34.0	5	12.9	0.7
CO	0.0	0.0	0.0	0.0	56.9	15.3	0.0
H_2O	0.0	0.0	2.0	1.8	2.0	0.0	0.0
N_2	0.0	1.0	1.0	0.8	4.7	43.1	4.1
Other hydrocarbons	0.0	1.0	2	2	0.0	1	69.1
Oxygen	0.0	0.0	1	0.0	0.0	2	0.6
Sulfur compounds	0.0	<1.0	2	0.8	0.0	1	0.4
Lower calorific value (kJ/kg)	50.030	47.065	19.277	19.394	12.658	5.321	46

For example, the concentration of hydrogen can be from 0% to 30%, and carbon monoxide from 0% to 60%. The range of lower calorific value for fuel is from 5 to 50 kJ/kg.

The results show that hydrogen concentration is the most influential factor on the system power output [26]. It is followed in terms of system efficiency output by hydrocarbons and then carbon monoxide. According to research results, natural gas has the highest generation efficiency because it contains the largest amount of methane. By increasing the methane content, the concentration of hydrogen produced during fuel reforming and the amount of power output increase. Industrial gas wastes, such as oil refinery wastes, show a relatively good level of efficiency due to the high content of hydrogen, hydrocarbons and carbon monoxide [26].

The stages of the gas waste formation from oil production process are presented in table 2 and are characterized by the frequent use of hydrogenation processes and the formation of hydrocarbon-hydrogen containing gas waste (C1-C5).

According to the obtained results, various types of fuel can be used to generate electricity in power plants (high-temperature fuel cell, gas turbine, power boiler, etc.), but preference should be given to industrial waste and biowaste. Because renewable fuels produced from waste and residues, even at low power generation levels, are more attractive in power generation systems due to the attendant solution to environmental problems.

Most of the hydrocarbon fuels used, such as natural gas, synthesis gas obtained from coal, biogas, and petroleum gas,

Table 2

Stages of the waste gas production process at an oil refinery

Production plant	Gas emissions, %	Brief description of the process
Electrical dehydration and desalting with atmospheric-vacuum processing	5.1	Desalting and dehydration of crude oil followed by its separation into target oil fractions
Pressure swing adsorption	5.1	Extraction of high purity hydrogen from hydrogen containing gas
Production of base oils	0.3	Catalytic isodewaxing and hydrofinishing of raw materials, which results in base oils and deeply purified fractions of kerosene, diesel fuel, and naphtha
Light naphtha isomerization	1.3	Processing of pentane-hexane fraction purified from sulfur into a high-octane component of commercial gasoline
Flare gas recovery	4.9	Capture and purification of hydrocarbon gases from hydrogen sulfide with their subsequent return to the fuel network
Catalytic reforming with continuous catalyst regeneration	2.1	Processing of heavy hydrotreated naphtha
Hydrotreating of heavy coking gas oil	1.2	Hydrogenation of heavy gas oil from delayed coking
Naphtha hydrotreating	7.5	Reducing the content of sulfur and nitrogen compounds in naphtha
Kerosene hydrotreating	0.3	Hydrogenation of straight-run kerosene fraction
Diesel fuel hydrotreating	4.5	Hydrogenation of diesel fraction
Hydrotreating middle distillates	7.5	Hydrogenation of kerosene and diesel fractions
Hydrocracking of vacuum gas oil	6.0	Catalytic hydrocracking of heavy oil fractions
Delayed coking	46.2	Carrying out the process of converting tar into coke
Catalytic cracking	8.0	Processing of a mixture of gas oil and heavy gas oil in the presence of a catalyst
Isoparaffinization of diesel fuel	0.1	Catalytic isodewaxing to produce Arctic diesel fuel

contain a certain amount of sulfur. Biomass fuel contains less sulfur than fossil fuels. The hydrogen sulfide content in biosyngas can vary from 20 ppm to 200 ppm [27].

The sulfur contained in the fuel gas poisons the catalyst in the fuel cell [28-29]. Sulfur compounds block active reaction sites and impair the catalytic and electrochemical activity of the anode materials of fuel cells. Although the adsorption of sulfur on nickel is reversible at low concentrations (0.2 ppm), at higher concentrations, massive conversion of the anode material to nickel sulfide can occur and cause irreversible damage to the catalyst [30].

Without appropriate purification from sulfur compounds, it is impossible to use industrial or agricultural waste as fuel, and also to add fuel gas to natural gas, even in small quantities, taking into account fuel consumption and the requirements of the operating rules of power boilers. When the standard content of sulfur compounds increases, corrosion may occur and a negative impact of the fuel mixture on the technical condition of the heating surfaces of the boiler unit and burner devices of power equipment (turbines, boilers). In addition, with an increased content of sulfur compounds in the fuel, its coking properties increase. Furthermore, during the combustion of fuel containing hydrogen sulfide and mercaptans, sulfur oxides (SO_x) are formed, which pollute the environment.

Therefore, without proper pre-treatment of fuel from sulfur-containing substances, power plants will not be ready for the practical use of gaseous hydrocarbon waste. However, the preliminary desulfurization step increases the cost of the energy production process and adds complexity to the system. Cost and complexity depend on the specific desulfurization requirements, which determine the pressure and temperature required to reduce sulfur levels for safe operation of the energy system [31].

The aim of this study was to conduct a literature analysis and present the available and environmentally friendly adsorption materials developed by the authors of the article for cleaning gaseous hydrocarbon waste from sulfur compounds and further use as fuel for power plants.

The scientific significance of the study lies in the confirmation by literary data and our own developments of the efficiency of using inexpensive adsorbents from waste for cleaning petrochemical plants gas emissions from sulfur-containing compounds.

2. Traditional technologies used to purify gas from sulfur compounds

Selecting a process for purifying gases from sulfur compounds is a complex task that involves consideration of many factors, which include the availability of space for the construction of treatment facilities, the reliability of process equipment, restrictions on waste disposal, the desired final composition of the gas mixture, as well as capital and operating costs. Treating waste gases, both industrial and agricultural, to make them suitable for subsequent use requires physical, chemical and biological processes. A number of technologies are available to combat industrial gas pollution with varying degrees of success. Here are some of them: adsorption, absorption, membrane filtration, direct oxidation, biological purification methods, etc. [32]. An excellent overview of hydrocarbon gaseous fuel desulfurization options was presented by Aravind et al. [27]. However, most of them require significant financial investment and their use is limited due to cost factors that overshadow the importance of pollution control.

Among the various available technologies, the adsorption process is considered the best due to its convenience, ease of operation and simplicity of design [33]. In addition, a wide variety of different substances, including chemical, synthetic and natural materials, are available as adsorbents, allowing adsorption to become more widely used in gaseous fuel pollution control (table 3).

Today, the technologies for H_2S capture and removal have proven themselves quite well on an industrial scale. However, there is still much room for improvement and optimization of these processes in the pursuit of further reduction of process costs and increase of separation performance. Adsorption using zeolite or metal oxide based sorbents are currently the most successful technologies in commercial application for primary purification and fine removal of H_2S, respectively. While other materials (mesoporous adsorbents, metal-organic frameworks) require some modifications, mainly related to cost, to reach the industrial level. Research is ongoing to further improve the efficiency of H_2S separation by developing efficient and new materials and solvents using methods such as functionalization and molecular design/grafting. In this context, it should be emphasized that more environmentally friendly and cost-effective materials (clay, waste-derived adsorbents) are also key to improving the current state of the environment.

3. Inexpensive alternative adsorbents from waste

The use of waste for conducting the adsorption process has established itself as one of the technologies with the right to exist for purifying gaseous fuels from sulfur compounds. This is due to the fact that the widespread use of traditionally used adsorbents, such as carbon structures, zeolites, metal oxides, membranes, etc., is limited by higher costs. Attempts have been made to develop low-cost adsorbents using a variety of agro-industrial and municipal wastes. Low-cost adsorbents are attractive due to their contribution to reducing waste disposal costs, thereby contributing to environmental protection [34].

3.1. Agricultural waste as inexpensive adsorbents

Agricultural materials, especially those containing cellulose, have potential sorption capacity for various pollutants. The main components of agricultural waste include hemicellulose, lignin, lipids, proteins, simple sugars, water, hydrocarbons and starch containing various functional groups. Agricultural waste, being economical and environmentally friendly due to its unique chemical composition, availability in abundance, renewable nature and low cost, is a viable raw material option for the production of adsorbents. Agricultural waste is a rich source for the production of activated carbon due to its low ash content and acceptable hardness, so recycling agricultural waste into low-cost adsorbents is a promising alternative to address environmental concerns as well as reduce preparation costs.

Over the past few decades, various agricultural wastes have been investigated as low-cost adsorbents. Some of these include the shells and/or seeds of fruits such as nuts [35], peanuts [36], olive waste [37], almonds [38], apricot kernels [39], and cherries [40]; and waste generated from the production of

Table 3
Advantages and disadvantages of various types of adsorbents for adsorption desulfurization

Type of adsorbent	Advantage	Limitation	Reference
Metal oxide	Availability	Limited use for industrial fuel desulfurization	Liu D. et al. (2020)
	Structural stability		
	High selectivity	More research on regeneration is needed	
	Crystal structure		
Zeolite	Large surface area (150–500 m^2/g)	Zeolite micropores are poorly permeable to high molecular weight sulfur compounds	Ozekmekci M. et al. (2015)
	Inclusion of metal ions is allowed	Need to increase pore size	
	Good regeneration ability		
Mesoporous material	Due to the mesopores, high molecular weight sulfur compounds are easily adsorbed by diffusion	The inclusion of functional materials or metals causes a decrease in porosity and blocks the pores	Kailasa S. K. et al. (2020)
	Large pore volume and surface area	Poor hydrothermal stability	
Metal-organic framework	Large pore volume and surface area, variety of structure options, crystalline structure, adjustable pore size	Low regeneration capacity and reusability	Li Y. (2021)
	Possibility of modification with a functional material or metal that acts as active sites	Structural instability at high temperatures and poor mechanical strength	
Activated carbon	Easily available and low cost	Low mechanical and thermal stability	Georgiadis A. G. et al. (2020)
	Inclusions of surface oxygen functional groups and metals act as active inclusions	Large surface area and pore volume Wide pore size distribution causes mass transfer limitations and poor selectivity for industrial fuel desulfurization	
	Large surface area and pore volume	Difficult to regenerate thermally	
Clay	Low cost and high availability	More research required to improve productivity and reusability	Wal K. et al. (2021)
Industrial waste	Low cost	Information limited	Ahmad W. et al. (2021)

cereal crops such as rice [41], maize [42] and corn cob [43] as well as bagasse from sugarcane [44] and coconut kernels [45]. These agricultural wastes were used in their natural form or after some physical or chemical modification.

3.2. Industrial and municipal waste as inexpensive adsorbents

Widespread industrial activities generate huge amounts of solid waste as by-products. Some of this material is used, while others are not properly disposed of and are discarded elsewhere. Industrial waste is available almost free of charge and poses serious disposal problems. If solid waste could be used as low-cost adsorbents, it would provide a double benefit in environmental control. Firstly, the volume of waste can be partially reduced, and secondly, an inexpensive adsorbent, if developed, can reduce wastewater pollution and gas emissions at a reasonable cost. Due to the low cost of such adsorbents, there is no need to regenerate waste materials. Therefore, a range of industrial wastes, both purified and unpurified, have been investigated as adsorbents for the removal of pollutants from industrial discharges.

The main by-product of solid waste production at coal-fired thermal power plants is fly ash. The main uses of fly ash include road construction, brick making, cement making, etc. The high silica and alumina content of fly ash makes it a good candidate for use as a low-cost adsorbent for bulk use [46].

Various areas of metallurgy produce large amounts of sludge containing metal oxides capable of binding hydrogen sulfide. The steel industry produces a number of wastes such as blast furnace slag, dust and sludge, etc. An example is Zn-containing electric arc furnace dust, which is proposed to be used as a regenerable waste for the removal of sulfur compounds [47]. Red mud, a solid waste from the aluminum industry, consists mainly of aluminum, iron, silicon and sodium oxides. Hien et al. showed an efficiency of 80.2% for removing hydrogen sulfide from air using an adsorbent prepared from red mud [48].

The paper industry also produces a number of by-products in large quantities, which pose serious disposal problems and degrade the environment. Experimental results showed that paper sludge from deinking leads to the formation of mesoporous materials, while highly porous adsorbents are formed from pulp mill sludge [49].

Various industries produce as by-products a sludge consisting of a mineral-like phase of catalytically active metals such as iron, zinc and copper [50], vanadium, iron, calcium, copper, nickel [51], mesoporous materials [52], porous carbon [53], which have been investigated as adsorbents.

The mechanism of H_2S adsorption is a complex process with the simultaneous influence of several important factors, especially: porosity and specific surface area [54-56], pH [54-55, 57-58], humidity [54, 58-60], metal inclusions [56, 59-64].

Table 4 presents various substrates used as materials

Table 4

Adsorbents from waste and their characteristics

Substrate	Primary treatment	Activation method	Working gas	Result, mg/g	Reference
Rice husk	Pyrolysis 400 °C	Drying 48 hours	Humidified model H_2S	382.7	Shang et al. (2013)
Camphor Bamboo Biochar Perilla Korean Oak Japanese oak Soy	Pyrolysis 700 °C		Biogas	109.3 - 336.7	Sethupathi et al. (2017)
Potato peelings	Carbonization 500 °C		H_2S/air	53	Sun et al. (2017)
Sewage sludge	Pyrolysis 500 °C	Drying	H_2S/air	59.6	Xu et al. (2014)
Pig manure		Drying	H_2S/air	65.5	Xu et al. (2014)
Sewage sludge		Drying, hydration	H_2S/air	18	Ros et al. (2006)
Sewage sludge	Gasification at 700 °C with nitrogen/air	Drying	H_2S/air	62	Ros et al. (2006)
Sewage sludge	Gasification at 700 °C with nitrogen/air	Drying, hydration	H_2S/air	131	Ros et al. (2006)
Activated carbon			Biogas	4.42	Ortiz et al. (2014)
Sewage sludge	Pyrolysis and calcination at 500-800 °C		Biogas	8.63	Ortiz et al. (2014)
Dried fish	Pyrolysis 950 °C	Drying	Humidified H_2S/air	22	Wallace et al. (2014)
Sewage sludge and dried fish	Pyrolysis 950 °C	Drying	Humidified H_2S/air	87.1	Wallace et al. (2014)
Drinking water sludge		Drying	Biogas	75.64	Polruang et al. (2017)
	Chemical activation with NaOH	Drying	Biogas	87.93	Polruang et al. (2017)
	Addition of iron ions Chemical activation with NaOH	Drying	Biogas	105.22	Polruang et al. (2017)
Coffee cake	Pyrolysis 500 °C		H_2S/air	6.2	Nowicki et al. (2014)
Coffee cake	Pyrolysis 500 °C	Hydration	H_2S/air	97.3	Nowicki et al. (2014)
Coffee cake	Pyrolysis 500 °C	Hydration, activation with carbon dioxide at 800 °C	H_2S/air	281.5	Nowicki et al. (2014)
Ash		Drying	H_2S	10.4	Wu et al. (2018)
Ash		Drying	Dry biogas	14.5	Fontseré Obis et al. (2017)
Ash		Drying	Humidified biogas	56.3	Fontseré Obis et al. (2017)
Commercial iron oxide			Humidified H_2S	260	Skerman et al. (2017)
Granulated steel Furnace slag			Humidified H_2S	1.1	Skerman et al. (2017)
Granular activated carbon			Humidified H_2S	6	Skerman et al. (2017)
Food waste			Humidified H_2S	3.4	Skerman et al. (2017)
Biomass			Humidified H_2S	10.4	Skerman et al. (2017)
Red clay			Humidified H_2S	18.8	Skerman et al. (2017)
Fertilizer from sewage sludge	Pyrolysis 400-950 °C	Impregnation with zinc chloride Flushing with hydrochloric acid	Humidified air	52	Bagreev et al. (2001)
Sewage sludge	Pyrolysis 500-700-950 °C	Drying	Wet biogas with air	460.8	Hien et al. (2024)

for creating absorbents from industrial and agricultural wastes, designed specifically for purification from sulfur compounds, as well as methods for their modification.

Thus, it is obvious that inexpensive waste-derived adsorbents can be effectively used to remove H_2S from gas mixtures.

Based on a review of literature data, several adsorption compositions prepared from sludge from water treatment plants of thermal power plants for use as an absorber of sulfur compounds from gas emissions from petrochemical industries were developed and presented in table 5. Hydrogen sulfide was used as a gas model. The residual concentration of sulfur compounds in the absorption solution after the adsorber was determined according to GOST 22387.2-2021 using a Shimadzu UV-1800 UV spectrophotometer.

The obtained results show that the use of water treatment plant sludge can be an effective solution for creating sulfur compound adsorbents. The sludge consists mainly of oxides and hydroxides of calcium, magnesium, iron and silicon. Various methods of primary treatment and subsequent activation of the sludge allow capturing high concentrations of hydrogen sulfide, similar to literature data.

Component composition and characteristics of the developed adsorbents — **Table 5**

Substrate	Primary treatment	Activation method	Result, mg/g
Sludge 30%/red clay 40%/ Na metasilicate 30%	Drying at 105 °C	Rinsing with 4% NaOH solution	16.7
Sludge 50%/$Ca(OH)_2$ 10%/ Na metasilicate 30%	Drying at 105 °C	Rinsing with 4% NaOH solution	16.9
Sludge	Pyrolysis 1000 °C 2 hours	Dry NaOH 4% / activated sludge 96%	333
Sludge	Pyrolysis 1000 °C 2 hours	Dry NaOH 4% /activated sludge 95% / zinc oxide 1%	377
Sludge	Pyrolysis 1000 °C 2 hours	Dry NaOH 1% / activated sludge 50% / non-activated sludge 20% / Na metasilicate 29%	29
Sludge	Pyrolysis 1000 °C 2 hours	Dry NaOH 2%/activated sludge 48%/sawdust 50%	225
Sludge	Pyrolysis 1000 °C 2 hours	Dry NaOH 2%/activated sludge 48%/starch solution 50%	36.5

Conclusions

The use of waste materials as low-cost adsorbents for the removal of various pollutants from gaseous fuel mixtures has many attractive properties, especially their contribution to reducing waste disposal costs, thereby helping to protect the environment. Although the available literature on the use of low-cost adsorbents is growing at a tremendous rate, there are still several gaps that need to be filled, namely:

1. Selection and identification of a suitable low-cost substrate is one of the key issues to achieve maximum capture of pollutants depending on the characteristics of the adsorbent-adsorbate.
2. The economic factor is decisive in industrial operating conditions. Low cost with higher adsorption efficiency will make the process economical and effective.
3. To improve the economic feasibility of the process, the ability to regenerate the adsorbent containing pollutants to recover both the adsorbate and the adsorbent is important.
4. It is necessary to evaluate the potential of low-cost adsorbents for multi-component pollutants and their competitive adsorption. This will have a significant impact on the commercialization of low-cost adsorbents in industrial systems.
5. Also relevant is the task of developing indicator sorbents for timely replacement due to the uneven content of sulfur compounds in gas emissions and the need to control the cleaning process.
6. The purification efficiency depends not only on the properties of the adsorbent and adsorbate, but also on various environmental conditions and variables used for the adsorption process, such as pH, ionic strength, temperature, the presence of competing organic or inorganic compounds in the gas, the initial concentration of the adsorbate/adsorbent, contact time and reaction rate, adsorbent particle size, surface modification, etc. These conditions can be optimized to improve the purification process.
7. Environmentally friendly disposal of adsorbents containing contaminants is another important topic that should not be overlooked.

If the project is successful, developed adsorbents with all of the above-mentioned characteristics could offer significant advantages over currently available expensive industrial treatment methods and further contribute to the overall waste minimization strategy.

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