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TECHNOLOGY OF OXIDATIVE DESULPHURIZATION OF LIGNITE

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Abstract

Based upon the generalization of experimental research, the process flow scheme has been suggested and material and heat balances of the oxidative desulphurization process of Ukrainian high-sulphur lignite have been calculated. The use of such a technology as of the 1st stage of coal burning at heat power plant will make it possible to utilize more than 50% of lignite sulphur as concentrated hydrogen sulphide or marketable elemental sulphur and by this decrease the environment pollution with sulphur oxide at least by 53-56%. The thermal destruction product of the organic part of coal (resin), which is obtained in the process, comprises not more than 2% of sulphur mass and may be used as a component of fuel oil.

Keywords: lignite; oxidative desulphurization; pyrite; sulphur; hydrogen sulphide.

1. Introduction

In 2013 as a result of coal burning almost 30% of primary power was obtained and electric power – more than 40% [1]. On the other side, stationary objects of heat and electric power production, among which dominant positions are occupied by heat power plants (HPPs), are main environment pollutants of sulphur oxide (IV) [2]. Primarily, this is related with the fact that the main feedstock for HPPs is coal, that is characterized with high-sulphur content in comparison with the other types of hydrocarbon fuels (gas, oil fuel).

In 2013, 54,8% out of the proved world coal reserve is lignite [3], nevertheless this coal type is equal to only 9% out of the whole coal mining in 2013 [1]. This is stipulated mainly by the poor quality of lignite, i.e. by high sulphur content and moisture. For example, Ukrainian lignite is characterized with sulphur content 3.5-4.2 and moisture 50-60 % mass per a work sample [4]. If to solve the problem of SO_2 emission in lignite burning, one of the directions of possible coal industry development may become the lignite use rise for the production of heat and electric power.

The process of lignite oxidative desulphurization in a boiling layer, that is formed as a result of air supply or steam-air mixture (oxidant), is being developed at the Department of Chemical Engineering of Oil and Gas Technology of Lviv Polytechnic National University. The process is aimed at the decrease of sulphur oxide emission in coal burning at HPP [5-8].

In the previous researches [7-8], the boundaries of optimum values of the oxidative desulphurization process factors: oxidant linear rate (OLR) – 0.0125-0.01875 m/s, coal grain size – not more than 0,75 mm, temperature – 425-450°C, ratio between volumetric flow air-steam mixture and coal mass (OFR) – almost 0.6 m^3/(h per kg), the process time – 5-10 minutes. Except, it has been determined that to increase H_2S content in the desulphurization gases it is efficient to use as an oxidant steam-air mixture and not the air.

Under conditions indicated above, the transfer of lignite sulphur, mainly pyrite sulphur, to hydrogen sulphide, and partial gasification and oxidation of organic part of coal take place [5-8]. By this, desulphurization gases with the high concentration of hydrogen sulphide are obtained (4.7-7.4%vol.), what allows to concentrate or retreat them obtaining sulphur.

The goal of the paper was to generalize the previous researches dedicated to oxidative desulphurization of lignite and, basing on this:

- create a process flow scheme;

- calculate material and heat balances;
- suggest possible practical ways for using by-products.

2. Experimental

The sample of lignite for investigation was taken from the Morozivs'ke coal-field of Dnieper lignite basin. Sampling and some analytical preparation of lignite was made in compliance with [9]. The fraction of 0.1-0.25 mm was used for researches because it is the optimum size for coal burning at thermal power plant

The technical analysis was carried out [10-13] and different sulphur forms such as organic sulphur (S_o^d), pyritic sulphur (S_p^d) and sulphate sulphur ($S_{SO_4}^d$) were determined [14]. The results are given in Table 1.

Table 1 Characteristics of lignite

Fraction mm	Moisture W^{af}	Ash A^{d}	Volatility V^{daf}	Sulphur content, mass %			
	mass %			Total S_t^d	Pyritic S_p^d	Organic S_o^d	Sulphate $S_{SO_4}^d$
	[11]	[12]	[13]	[10]	[14]	[14]	[14]
0.1-0.25	13.96	9.42	63.78	4.28	2.10	2.06	0.11

The investigations were conducted on the laboratory plant based on fludized bed reactor that functioned in the mode close to isothermal. The scheme of the laboratory plant and its detailed description are presented in [6].

The desulphurizative gases were analyzed by means of gas-adsorptive chromatography, using a chromatograph "LHM" (N 479). The analyzed gas (10.0 ml) is introduced by a metering device into the flow of gas carrier (helium). Gas is fed to the first column (diameter of 2.0 mm and length of 3.0 m) filled with a non-polar sorbent Polysorb-1 and then – to the second column (diameter of 2.0 mm and length of 4.5 m) filled with a polar adsorbent – zeolite of CaX type [5-8].

In the ash, which had been creating in the process of oxidative lignite desulphurization, the following indices were determined: kinematic viscosity [15]; mass sulphur content [16]; mass water content [17]; mass fraction of mechanical admixtures [18]. Conradson concarbon residue (CCR) [19]; ash [20]; temperature pour point [21]; ignition temperature in open crucible [22]; density [23]; content of water-soluble acids and alkalies [24].

The oxidant linear rate (OLR) is calculated as a ratio of oxidant volumetric flow rate (m^3/s) under the normal conditions) to sectional area of an empty reactor (m^2).

To characterize the oxidant consumption, the term "repetition factor of oxidant flow rate" (OFR) was used. OFR was calculated as the ratio between volumetric flow air-steam mixture (m^3/h) and coal mass (kg).

To characterize the relative rate of sulphuric compounds conversion, we used the term "sulphur conversion (SC)". This value indicated the amount of sulphur converted into gaseous sulphur-containing products that will not be in the atmosphere while further burning of desulphurized coal (the level of environmental pollution decrease). It is calculated in accordance with the formula (1), %:

$$SC = \frac{S_0^a \cdot 100 - S^a \cdot x_C}{S_0^a} \qquad (1)$$

where S_0^a – the content of sulphur in the initial coal relative to the analytical sample, mass %; S^a – the content of sulphur in the desulphurized coal relative to the analytical sample, mass %; x_C – the yield of desulphurized coal, mass %.

The sulphur content in the desulphurized coal depends on the ratio between the conversion rates of the coal and pyritic sulphur in it. Hence, the removal degree of sulphur (RDS) is calculated in accordance with the formula (2) and indicates the ratio between the rate of sulphur conversion followed by the production of gaseous products and the rate of organic matter reaction, i.e. process selectivity:

$$RDS = \frac{S_0^d - S^d}{S_0^d} \cdot 100 \qquad (2)$$

where S_0^d – the content of sulphur relative to the dry sample, mass %; S^d – the content of sulphur in the desulphurized coal relative to the dry sample, mass %.

The heat effect of the oxidative desulphurization process of lignite has been calculated by the formula, MJ:

$$Q_r = \frac{G_{H_2S} \cdot q_r^{H_2S} + G_{CO} \cdot q_r^{CO} + G_{CO_2} \cdot q_r^{CO_2} + G_{H_2O} \cdot q_r^{H_2O} + G_r \cdot q_r^r}{1000}, \qquad (3)$$

where $G_{H_2S}, G_{CO}, G_{CO_2}, G_{H_2O}, G_r$ – appropriately, quantity, kg, sulphur, transferred into hydrogen sulphide; carbon, burnt to CO; carbon, burnt to CO_2; carbon, burnt to H_2O; the quantity of resin decomposition. These values (except of G_{H_2O}) have been calculated on the base of material balances and contents of desulphurization gases; $q_r^{H_2S}, q_r^{CO}, q_r^{CO_2}, q_r^{H_2O}, q_r^r$ – heat effects of the appropriate reactions, kJ/kg.

The hydrogen quantity (H), transferred into H_2O during combustion of coal organic matter, calculated, taking the ratio C/H is equal in feedstock and desulphurized coal, by the following formula:

$$G_{H_2O} = (G_{CO} + G_{CO_2}) \cdot \frac{H}{C}, \qquad (4)$$

H = 6 i C = 65 % mass – the content of hydrogen and carbon in lignite, which have been accepted basing on [25].

The heat effects of reactions were calculated and accepted accordingly [26]. By this, it has been accepted that the transfer of lignite pyrite into hydrogen sulphide takes place by the following reaction

$$FeS_2 + H_2 \rightarrow H_2S + FeS \qquad (5)$$

For drawing up the heat balance of the process of oxidative coal desulphurization, the heat quantity, that is giving off as a result of burning desulphurization gases after H_2S removal, has been taken into consideration. The useful heat of desulphurized gas burning, MJ, has been calculated by the standard methods [27]:

$$Q_k = \frac{\eta \cdot (G_{gas} - G_{H_2S})}{\rho_{gas}} \cdot (0.358 \cdot CH_4 + 0.637 \cdot C_2H_6 + 0.912 \cdot C_3H_8 + 0.591 \cdot C_2H_4 + 0.108 \cdot H_2 + 0.126 \cdot CO), \qquad (6)$$

where G_{gas}; G_{H_2S} – the quantity of desulphurized gases and hydrogen sulphide, kg; ρ_{gas} – the density of desulphurized gases, kg/m^3; $\eta = 0.8$ – the furnace efficiency; CH_4, C_2H_6, C_3H_8, C_2H_4, H_2, CO – the content of appropriate components in coal, % vol.

3. Results and discussion

Described researches in [7-8] underlie the development of technological scheme of oxidetive desulphurization of lignite. The oxidative desulphurization technology can be implemented into production if the following stages are solved correctly:

- heating of oxidant and feedstock coal up to the process temperature;
- providing reliable contact of oxidant with coal in the reaction area;
- heat regeneration of desulphurization gases;
- separation of desulphurization gases and resin decomposition;
- providing the capability to remove hot desulphurized coal into the boiler of the 2nd burning degree (in case of implementation of the process at HPS) or cooled desulphurized coal (in case of the necessity of transportation to customers).

Taking into consideration all above, the technological scheme of setting the facility for oxidative desulphurization of lignite has been suggested (Fig. 1).

The coal from the store is transferred into the feedstock bin (1), next with the batcher – into the mill (2), where it is comminuted to the size 0,1-0,75 mm. The comminuted coal is classified with a sieve (3) and via the screw (4) is transferred into the bin (bunker/hopper) (5). From the bunker the comminuted coal is transferred into the middle part of the upper section of the heat utilizer (6), where it is heated up to 220-300°C through cooling desulphurization gases. Small quantities of flue gases are transferred from the waste-heat boiler/furnace (14) into the utilizer (6). The gases contact with the coal directly, preventing adhesion of coal particles and facilitating its movement down by utilizer section (6).

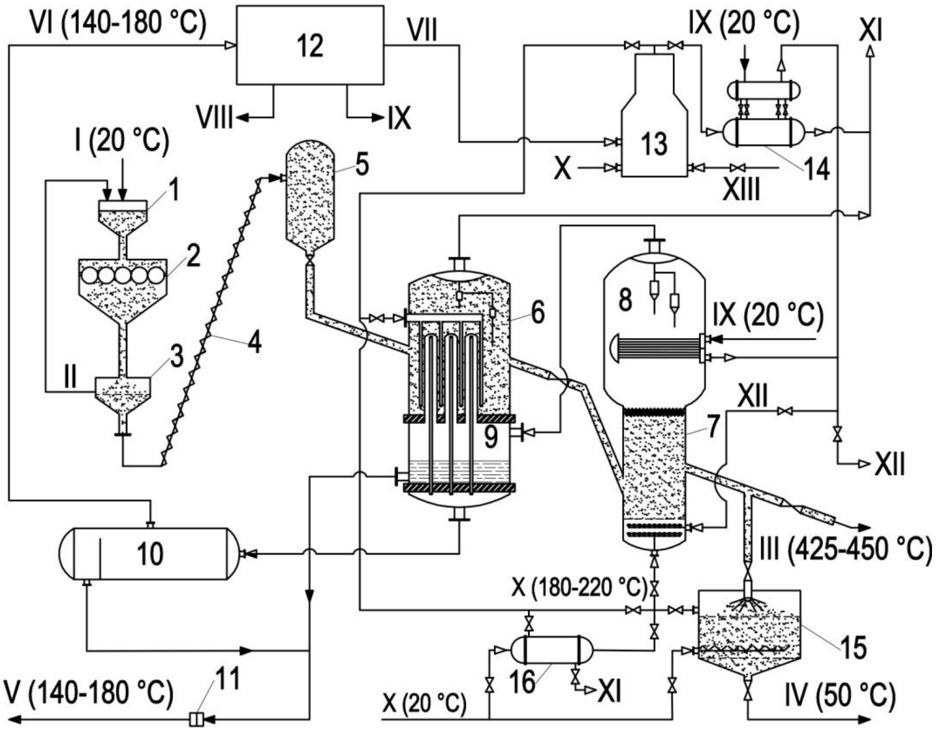

Fig. 1 Principal technological scheme of installation for oxidative desulphurization of lignite

1 – feedstock bin; 2 - mill; 3 - sieves; 4 - screw; 5 – bin (bunker); 6 – upper section of heat utilizer; 7 – reaction area of desulphurization reactor; 8 – separation area of desulphurization reactor; 9 – bottom section of heat utilizer; 10 - separator; 11 - filter; 12 – processing unit of desulphurization gases; 13 – postcombustion furnace; 14 – waste-heat boiler; 15, 16 – heat-exchange unit;

I – feedstock (lignite); II – fraction lignite > 0,75 mm.; III – desulphurization of uncooled coal; IV – desulphurized cooled coal; V – resin of decomposition; VI – gases of desul-phurization and water steam for treatment; VII – cleaned gases of desulphurization from

H_2S for burning up; VIII – sulphur; IX – combustion gas; X – air; XI – cooled combustion gases; XII – water steam; XIII – fuel gas

The heated coal poured into the reactor, that consists of two sections, i.e. the bottom reaction (7) and the top separation (8) ones. In the reaction area at 425-450°C the coal contacts with the oxidant in the apparatus of the fluidized bed (7). The necessary-for-reaction quantity of previously heated water steam in the section (8) and in the boiler (15) is transferred under the distribution grid of the low area (7). In the area of desulphurization (7) via the contact of the oxidant with the coal, the processes of sulphur conversion into H_2S occur. H_2S is derived from the reaction area along with the other gases and steams. To remove the excess heat into the coiler, which is mounted in the reaction area of separation (8), water is sprayed.

Water steam, desulphurization gases and resin decomposed from the reaction area (7) are transferred into the separation section (8), where coal dust is separated, next is the bottom (distribution) section of heat utilizer (9) and then – its upper section (6), where all these are condensed and cooled to the temperature 140-180°C. The cooled flue gases, water steam and resin of decomposition get into the separator (10). From the bottom distribution section of the utilizer (9) and the separator (10), the condensed resin of distribution gets into the system of filters (11) for separating mechanical impurities and is withdrawn from the facility.

Desulphurized coal from the reaction area (7) gets directly into the boilers of HPP. In case the coal desulphurisation facility is independent, the desulphurised coal is precooled by the air in the remote heat-exchange unit (15) and then transported to its destination. The air entering the remote heat-exchange unit (15) is heated up to 180-220°C and gets into the reaction area of the reactor (7). If the coal does not enter the heat-exchange unit (15), the air is heated up to the necessary temperature in the heat- exchange unit (16) via the heat of the combustion gases.

The gases of lignite desulphurization from the top separator (10), after drying up, are processed to give off sulfur. The gases of desulphurization, after extraction of H_2S, get into the postcombustion furnace (13), next – to the waste-heat boiler (14), where water steam is obtained as a result of cooling. The cooled flue gases emit into the atmosphere.

Based on the created process flow scheme and researches conducted under optimum conditions [5-6], material and heat balances of the processes, which are presented in Table 2, have been calculated.

Table 2 Material and heat balances of the unit of oxidative desulphurization of lignite

Articles	t, ºC	Phase state	Enthalpy, kJ/kg	Quantity, G_i, kg	Heat quantity, MJ
4.5 % vol. of water steam in oxidants					
Entered:					
1. Lignite	20	solid	29.00	100.00	2.90
2. Air	20	gas	20.10	12.35	0.25
3. Water	20	P	83.80	0.36	0.03
4. Reaction heat	-	-	-	-	81.71
5. Useful heat of desulphurization gas combustion after H_2S extraction	-	-	-	-	42.06
Total:	-	-	-	-	126.95
Obtained:					
1. Desulphurized lignite	50	solid	70.00	55.41	3.88
2. Resin of decomposition	150	liquid	298.81	18.85	5.63
3. Water steam	150	steam	2778.35	12.06**	33.50
4. Desulphurization gases	150	gas	158.35	19.52	3.09
5. Excess heat	-	-	-	**-**	**76.76**
6. Losses	-	-	-	-	4.09*
Total:	-	-	-	-	126.95
50 % vol. of water steam in oxidants					
Entered:					
1. Lignite	20	solid	29,00	100,00	2,90
2. Air	20	gas	20,10	6,47	0,13
3. Water	20	liquid	83,80	4,02	0,34
4. Reaction heat	-	-	-	-	53,11
5. Useful heat of desulphurization gas combustion after H_2S extraction	-	-	-	-	37,60
Total:	-	-	-	-	94,08
Obtained:					
1. Desulphurized lignite	50	solid	70,00	61,06	4,27
2. Resin of decomposition	150	liquid	298,81	20,65	6,17
3. Water steam	150	steam	2778,35	14,86**	41,29
4. Desulphurization gases	150	gas	161,37	12,38	2,00
5. Excess heat	-	-	-	-	**35,64**
6. Losses	-	-	-	-	4,70*
Total:	-	-	-	-	94,08

**Losses into environment have been accepted as 5 % out of the reaction heat. **The quantity of water steam, that was entered with oxidant and was created as a result of coal moisture decrease, has been taken into consideration.*

Temperatures of the products have been accepted on the basis of process scheme for the facility (Fig. 1) and the calculation of heat balances of separate devices for the case of cooling desulphurized coal to 50 C. The values of entered and spent quantities of in-the-process heat (in MJ) is assigned to 100 kg feedstock carbon.

According to the above calculations, the heat quantity, which released as a result of reactions occurring during the process and of desulphurized gas post-combustion, is enough for heating coal up to the reaction temperature and oxidant preparation. The excess heat (in quantity 0,36-0,77 MJ per 1 kg of coal) can be used to obtain water steam.

The material balance results prove that the following is obtained in the process of lignite desulphurization:

- 55.41-61.06% by mass per desulphurized lignite feedstock;
- 18.85-20.65% by mass per feedstock of decomposition resin;
- 12.38-19.52% by mass per feedstock of desulphurization gases.

The characteristics of desulphurized coal and basic technological parameters of the process are presented in Table 3 and 4.

Table 3 Characteristics of desulphurized lignite

Moisture, , W^{af}, mass %	Ash, A^d, mass %	Volatility, , V^{daf}, mass %	Sulphur content, mass %			
			total, S_t^d	pyritic, S_p^d	organic, S_o^d	sulphate, $S_{SO_4}^d$
[11]	[12]	[13]	[10]	[14]	[14]	[14]
4,5 % vol. of water steam in oxidants						
2.29	14.40	44.31	3.02	0.49	0.29	2.24
50 % vol. of water steam in oxidants						
2.98	13.42	46.88	2.92	0.46	0.31	2.15

Table 4 Degree of sulphur distraction and conversion of lignite

Sample of desulphurized coal	Removal degree of sulphur, RDS, %,		Sulphur conversion, SC, %,	
	total	pyritic	total	pyritic
4,5 % vol. water steam in oxidant	29.44	76.67	56.04	85.46
50 % vol. water steam in oxidant	31.78	78.10	53.46	85.06

In compliance with the data above, in the process of the lignite desulphurization it is possible to reduce total sulphur content on average only to 2.92-3.02% mass (Table. 3). Accordingly, the total sulphur removal degree is 29.44-31.78% (Table 4). The reason is abnormally high content of organic sulphur in the resulting coal, which quantity stays practically the same during the process, and low desulphurized coal caused by the high moisture content in the feedstock and high reactive capability of the organic part of lignite. However, if we assume that all the hydrogen sulphide contained in the desulphurization gases will be removed or converted into sulphur, the degree of conversion of total sulphur will be equal to the degree of environmental improvement. Therefore, we can state that the suggested method makes it possible to reduce emissions of sulphur compounds into the air during lignite combustion on average by 53.46-56.04%.

During oxidative lignite desulphurization, volatile organic products, which in the condensation process form the pasty or semiliquid brown substance (decomposition resin), are obtained. The resin obtained during the lignite desulphurization is similar to boiler fuels that is why its quality research was carried out from the perspective of its use as fuel components. In the Table 5 the characteristics of decomposition resin of lignite and its comparison with the requirements to the petcoke of the mark "100" are presented. Based on the data obtained, it can be stated that lignite resin under research mostly meet the requirements for the fuel oil of the mark "100". So, the resin obtained as a result oxidative lignite desulphurisation can be used as a boiler fuel component.

Table 5 Characteristics of decomposition resin of lignite and requirements to the petcoke of the mark «100»

Index	method	Values	
		Lignite resin of decomposition	Requirements to the fuel oil of the mark 100 [28]
Kinematic viscosity, cSt (mm²/s)	[15]		
at 50 °C, ν_{50}		44.19	–
at 80 °C, ν_{80}		12.38	not more than 118.0
at 100 °C, ν_{100}		7.09	–
Mass fraction, % mass			
sulphur,	[16]	1.95	not more than 2.0
water,	[17]	0.70	not more than 1.0
mechanical admixtures,	[18]	0.84	not more than 1.0
Conradson concarbon residue, CCR, % mass	[19]	5.47	–
Ash, % mass	[20]	0.08	not more than 0.14
Temperature, °C			
pour point,	[21]	42	not more than 42
ignition in open crucible	[22]	114	not less than 110
Density at 20 °C, kg/m^3	[23]	934	–
Content of water-soluble acids and alkalies, % mass	[24]	0.02	–

To determine the possible directions of desulphurization gas utilization, the analysis of the gases has been conducted by the chromatographic methods (Table 6).

Table 6 The content of desulphurization gases of lignite

Desulphurisation gases	Content, % vol.	
	4.5 % vol. water steam in oxidant	50 % vol. water steam in oxidant
H_2S	8.01	12.49
H_2	0.79	1.34
CH_4	2.68	3.28
C_2H_4	0.68	0.98
C_2H_6	0.93	1.56
C_3	1.23	1.97
CO	5.51	6.45
CO_2	23.54	24.99
O_2	1.23	0.65
N_2	54.76	45.76
Ar	0.64	0.53

The hydrogen sulphide obtained by oxidative lignite desulphurization, which content in desulphurization gases is equal to 8.01-12.49 % vol. (Table 6), can be concentrated according to chemisorptive or absorption methods [29-30]. Concentrated H_2S is typically used for the production of sulfuric acid or sulphur by the "direct" Claus method [31]. However, when the content of hydrogen sulphide in the gas is above 5% vol., it is advisable to process hydrogen sulphide directly into sulphur by the known methods [29-30].

4. Conclusions

The process flow scheme of oxidative desulphurization of lignite has been suggested, and on its base the material and heat balances of the processes have been calculated. It has been shown that in the process of implementation at the very heat power plants, the technology will make possible the emission reduction of sulphur compounds into the air during combustion of lignite on average by 53.5-56.0%.

In the desulphurization process, the decomposition resin in the quantity 18.85-20% mass per feedstock, which by most indices meets the requirements for the petcoke of the mark "100", is obtained. It should be noted that the boiler fuel is much more expensive than lignite.

In the process of lignite desulphurization via oxidative methods, the hydrogen sulphide, which content in desulphurization gases is 8,01-12,49% vol., is advisable to concentrate further or reprocess aiming at obtaining sulphur. After removing hydrogen sulphide, the desulphurization gases should be burnt to obtain extra heat quantity, and then cooled flue gases can be thrown into the atmosphere.

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