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# Features of Hydrogen Sulphide Elimination from Methane under Various Conditions in Barrier Discharge

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# Abstract

The process of elimination of hydrogen sulphide from methane in the plasma of barrier discharge in the presence of octane vapour is investigated. The use of octane vapour in the initial mixture with methane and hydrogen sulphide allows us to avoid the formation of deposit on the electrodes of the plasmachemical reactor and to achieve the high level of inhibition of the reactions leading to the formation of polymer-like compounds. The extent of hydrogen sulphide removal reaches 98 %, methane conversion does not exceed 10 %. Power consumption for the removal of hydrogen sulphide is 83 (kW  $\cdot$  h)/kg. The major gaseous products of the treatment in the barrier discharge are hydrogen (30 %) and ethane (30 %). Among sulphur-containing products, mainly mono- and dialkyl sulphides were detected. A possible mechanism of the process is proposed. The ways to manage the plasmachemical process are substantiated.

Keywords: methane, hydrogen sulphide, barrier discharge, removal of hydrogen sulphide, process mechanism

### INTRODUCTION

The attention of researchers to the use of the low-temperature plasma of barrier discharge (BD) for various purposes in the areas of the treatment of the materials, plasma catalysis, Plasma medicine, purification from organic pollutants has been growing during the recent years [1]. However, the degree of the introduction of processes involving the possibilities of low-temperature plasma chemistry remains low, in spite of their attractiveness, especially in petroleum chemistry, for the purification of hydrocarboncontaining gases from hydrogen sulphide (H<sub>s</sub>S). In part, this is due to the complicacy of processes that occur in low-temperature plasma, and the control of reactions comprising these processes. This mainly relates to plasmachemical processes participated by hydrocarbon molecules, which are often accompanied by tarring and profound destruction of reagents.

The use of BD plasma in petroleum chemistry for the removal of hydrogen sulphide from the flow of hydrocarbon gases has achieved substantial success [2–9], however, any data on the successful introduction of these processes are still absent from the literature sources. In this connection, accumulation of knowledge on the processes participated by hydrocarbon molecules in BD plasma remains an urgent problem; its solution is important for the efficient development of plasmachemical technologies of the treatment of hydrocarbon-containing raw materials.

A potential possibility of the efficient removal of hydrogen sulphide admixtures from methane  $(CH_4)$  with the formation of polymer-like organosulphur compounds under the action of BD was demonstrated previously in [10, 11]. Analysis of the possible mechanism of the process showed that the formation of polysulphide organic compounds proceeds on the surface of the electrodes in the reactor, while gas-phase reactions are completed mainly at the stage of the formation of alkyl sulphides and elemental sulphur ( $S_2$ ). The inhibition of chemical processes on the surface of electrodes in the reactor will allow one to govern the direction of reactions and to obtain the necessary composition of the products.

Subsequent investigations of the joint transformations of gaseous and liquid hydrocarbons and hydrogen sulphide, carried out within the Programme of fundamental research of the State Academies of Sciences for the years 2013-2020 (Project No. V.44.3.1), revealed the possibility in principle to govern the plasmachemical process and to obtain various organic sulphur-containing products, from mono-, di-, and polysulphide compounds to sulphones.

In the present work, we describe the results of the investigation of plasmachemical removal of hydrogen from methane in the BD in the presence of octane ( $C_8H_{18}$ ) vapour, and carry out a critical comparison with the results obtained by the authors previously.

## EXPERIMENTAL

Experiments aimed at the removal of hydrogen sulphide admixture from methane without additives were carried out in a metal single-barrier reactor. The initial concentration of H<sub>3</sub>S was 0.5-3.8 %, the active discharge power reached 15 W [10]. Experiments on the conversion of the mixtures of  $CH_4 - H_2S - CO_2/O_2$  (with the initial concentration of hydrogen sulphide fixed at 3 %,  $CO_2/O_2$  additives – up to 41 %) [11] and  $CH_4$  –  $H_{2}S - C_{8}H_{18}$  (the range of initial concentrations of hydrogen sulphide was 3-12 %) were carried out in a two-barrier glass reactor of coaxial type. The active discharge power in the case of the twobarrier reactor was 5-9 W, depending on the composition of the initial gas mixture [11], and was equal to ~8 W in the case of  $CH_4 - H_2S$  - $C_{8}H_{18}$  mixture (the initial concentration of hydrogen sulphide was 3 %).

The change of reactor type is due first of all to the lower active power, which is favourable from the viewpoint of energy saving because almost complete removal of hydrogen sulphide (95–98 %) from initial gas mixtures is achieved with lower energy consumption; second, the reactor had to be changed because of the use of octane, a liquid hydrocarbon, since a single-barrier reactor is not intended for working with liquid media. The discharge gap in the glass reactor is 1 mm. All experiments were carried out according to the same scheme which was described in detail in [10, 11]. Octane vapour obtained from liquid octane in the evaporator was supplied to the reactor with the gas flow. The concentration of octane vapour in the reactor was controlled by variations of the rate of liquid octane admission into the evaporator and was equal to 0.4 mL/min.

In all cases, BD was excited by the generator of high-voltage pulses lasting for a microsecond. The time of contact of the initial mixture with the discharge zone of the reactor was 13.5 s, reactor temperature was 20  $^{\circ}$ C, and the process was carried out at atmospheric pressure. The active power was kept constant.

The electric parameters of the BD were recorded in all cases using the same circuit (Fig. 1).

Typical oscillograms of high-voltage pulses and the volt-coulomb characteristic (VCC) are shown in Fig. 2.

High-voltage pulses of the voltage with alternating polarity formed by 1 are supplied to the high-voltage electrode of the reactor 2 (see Fig. 1). The amplitude of the pulses is up to 10 kV, the pulse-repetition frequency is 2 kHz), and the damped oscillations pulses appear as of approximately sinusoid shape (see Fig. 2, a). The damping decrement is determined mainly by the internal energy losses in the electric circuit of the generator: its output impedance exceeds the load impedance (reactor) substantially. The voltage oscillations damp practically completely during the intervals between pulses, and the major amount of energy is introduced into the discharge at the first period of voltage oscillations. The



Fig. 1. Schematic diagram for recording the electric parameters of BD: 1 – generator of high-voltage pulses; 2 – reactor; 3 – high-voltage divider; 4 – current/capacitive bypass resistor; CH1, CH2 – channels of the Tektronix oscillograph; n – switch key; R1 – resistance of the current bypass (0.1 Ohm); C – capacitor of the capacitive bypass (309 nF); C2, C3 – capacitors of the high-voltage divider (55 pF, 110 nF, respectively).



Fig. 2. Typical oscillograms of the pulse of BD voltage and current (a) and the volt-coulomb characteristic of BD (b).

voltage at the high-voltage electrode is recorded through a high-voltage capacitance divider 3 by the oscillograph through the CH1 channel, the charge carried between the electrodes, and discharge current – through the capacitive and current bypass resistors 4 of the oscillograph through the CH2 channel.

The active discharge power (W) is calculated using the equation

 $W = fE \tag{1}$ 

where f is the frequency of voltage pulse repetition, Hz; E is the energy per one voltage pulse, calculated relying on VCC, J (see Fig. 2, b).

Analysis of the gaseous products of the reaction was carried out with the help of an HP 6890 gas chromatograph (Hewlett Packard, USA) equipped with a thermal conductance detector, using a packed column (3 m long, 3 mm in diameter, with Porapak QS as a sorbent), in the isothermal mode. The column temperature was 70 °C, helium was used as a carrier gas. Hydrogen was determined with the help of a packed column (1 m long, 3 mm in diameter, a molecular sieve with pore diameter 5 Å was used as a sorbent). The column temperature was 40 °C, argon was used as a carrier gas. Gaseous products were identified by comparing retention times for individual substances. The composition of reaction products in the gas mixture after the reaction was calculated by means of normalizing taking into account the correction coefficients of detector sensitivity to the components of the mixture to be analyzed.

The products containing  $C_{5+}$  hydrocarbons were determined using a flame ionization detector and HP-PoraPLOT Q columns (for the gaseous part) and HP-1 (for the liquid part). The mixture of the formed  $C_{6+}$  hydrocarbons dissolved in water was analyzed using solid-phase microextraction [12].

Identification of the components of microextract was carried out by means of gas chromatography – mass spectrometry (GC-MS) using a DFS gas chromatograph – mass spectrometer (Thermo Fisher Scientific, USA) equipped with a Trace TR-50MS column.

The procedures to calculate the conversion of methane, hydrogen sulphide (to evaluate the total efficiency of the process) and the selectivity of product formation during the treatment of initial mixtures were described in [10].

The deposits formed on the surface of electrodes in the reactor in the experiments on the treatment of  $CH_4 - H_2S$  and  $CH_4 - H_2S - CO_2/O_2$  mixtures were analyzed by means of IR spectroscopy with the help of a Nicolet 5700 FTIR spectrometer (Thermo Electron, USA), the soluble components of the deposits were analyzed also by means of GC-MS with the DFS instrument.

Analysis of elemental sulphur formed during the treatment of initial mixtures of methane, hydrogen sulphide and octane vapour was carried out by means of photon correlation spectroscopy using a UNICOR-SP instrument (Russia).

Calculations of the parameters of BD plasma (the losses of electron energy for the excitation of molecules, the average energies of electrons) were carried out using the Bolsig+ software [13], the cross-sections of electron scattering by methane molecules were taken from [14]. Data on the cross-sections of the excitation of vibrational and electron states of hydrogen sulphide and octane are absent from the literature. So, these crosssections were obtained through a compilation of literature data on the full cross-sections of scattering, elastic collisions and ionization [15–19]:

$$\begin{split} \sigma_{\rm el}(\epsilon) &= \sigma_{\rm comp}(\epsilon) - \sigma_{\rm elast}(\epsilon) - \sigma_{\rm ion}(\epsilon) \eqno(2) \\ \mbox{where } \sigma \ensuremath{ \rm is \ electron \ energy; \ } \sigma_{\rm elast}(\epsilon), \ \sigma_{\rm total}(\epsilon), \ \sigma_{\rm el}(\epsilon), \\ \sigma_{\rm ion}(\epsilon) \ensuremath{ \rm are \ cross-sections \ of \ the \ excitation \ of \ electron \ states, \ complete \ electron \ states, \ elastic \ collisions \ and \ ionization, \ respectively. \end{split}$$

## **RESULTS AND DISCUSSION**

The dependence of hydrogen sulphide conversion in the  $CH_4 - H_2S - C_8H_{18}$  mixture on its initial concentration and similar data on the conversion of hydrogen sulphide in the  $CH_4 - H_2S$  mixture obtained previously [9] are shown in Fig. 3. One can see that the presence of octane in the initial mixture  $CH_4 - H_2S - C_8H_{18}$  leads to an increase in the conversion of hydrogen sulphide in comparison with pure methane. Hydrogen sulphide conversion within the whole range of its initial concentrations exceeded 92 % during the pass of the initial mixture through the reactor. With the removal of H<sub>o</sub>S from the mixtures of  $CH_4 = 3 \% H_2S = CO_2/O_2$ , the conversion of hydrogen sulphide was not less than 91 % [11] (not marked in Fig. 3). Methane conversion did not exceed 10 % in all cases [10, 11].

Energy consumption for the removal of hydrogen sulphide from the mixture of  $CH_4 - H_2S$ -  $C_8H_{18}$  was 83 (kW · h)/kg, which is somewhat higher than for the case of the mixture of  $CH_4 H_2S$  (68 (kW · h)/kg), and much lower than for the mixtures of  $CH_4 - 3 \% H_2S - CO_2/O_2$  (114– 127 (kW · h)/kg). Methane conversion was also not more than 10 %, octane conversion was not higher than 1.5 %.

For the mixtures under investigation, the major gaseous components of gaseous products are hydrogen and ethane (Fig. 4). The treatment of  $CH_4 - H_5S$  mixtures results in the formation of hydrogen at a level of 60-70 %, ethane 20-30 %, and in the case of  $CH_4 - H_2S - C_8H_{18}$  mixtures the percentage of hydrogen formed is only 30-40 %, while the selectivity of ethane formation remains the same. In the case of the treatment of  $CH_4$  –  $H_2S - CO_2/O_2$  mixtures, the percentage of hydrogen formed is 50-55 %, while ethane -30-40 %. The dependences of the selectivity of hydrogen and ethane formation on the initial concentration of  $H_3S$  in initial mixtures  $CH_4 - H_3S$ and  $CH_4 - H_2S - C_8H_{18}$  are generally similar: with an increase in H<sub>9</sub>S concentration, the selectivity



Fig. 3. Conversion of hydrogen sulphide in  $CH_4 - H_2S$  [10] and mixtures  $CH_4 - H_2S - C_8H_{18}$  depending on its initial concentration.



Fig. 4. Composition of gaseous products depending on the initial concentration of hydrogen sulphide in the mixtures:  $CH_4 - H_2S - C_8H_{18}$  (a)  $\mu$  CH<sub>4</sub> - H<sub>2</sub>S (b) [10].



Fig. 5. Chromato-mass-spectrograms of the compounds obtained under the action of BD plasma on the mixtures:  $CH_4 - H_2S - C_8H_{18}$  (a),  $CH_4 - H_2S$  (b) [10],  $CH_4 - H_2S - CO_2$  (c),  $CH_4 - H_2S - O_2$  (d) [11].

of hydrogen formation increases, while the selectivity of ethane formation decreases [11]. This fact is one of the arguments in favour of the common mechanism of reactions with the participation of hydrocarbon molecules and hydrogen sulphide in the gas phase.

Comparing the results of GC-MS of sulphurcontaining products formed in the treatment of methane mixtures with hydrogen sulphide under different conditions (the treatment of the mixture without additives, with the addition of  $CO_2/O_2$ , in the presence of octane vapour, Fig. 5), one may see that in the case of the treatment of  $CH_4$  –  $H_2S$  mixture sulphur-containing products are represented mainly by organic polysulphides of linear structure with the terminal methyl groups

(for example, dimethyltrisulphide ~34 mass %), and the compounds with cyclic structure, with hexathiopane as the dominating component (~18 mass %) [10]. In the case of  $O_2$  addition to the CH<sub>4</sub> – H<sub>2</sub>S mixture, sulphones of different structures were detected. After the addition of CO, a decrease in the content of polysulphide compounds of cyclic structure in the soluble components of deposits is observed, while the addition of  $O_2$  results in a substantial decrease in the content of all polysulphide compounds with cyclic and linear structure [11].

The use of octane vapour in the initial mixture with methane and hydrogen sulphide allows is to obtain a high degree of inhibition of the reactions on electrode surface that are responsible for the formation of deposits, and achieve the dominant formation of mono- and dialkylsulphides. Their content in the products is less than 1 % and is to a high extent dependent on experimental conditions. With an increase in H<sub>o</sub>S concentration in the mixture, the fraction of disulphides remains practically unchanged and equal to ~16 % of the sum of all sulphides. Among the identified alkylsulphides, we observe the formation of several groups of substances with different hydrocarbon substituents. One group of compounds is characterized by the presence of alkyl substituents with the hydrocarbon framework of initial octane molecule (octylmercaptane C<sub>8</sub>S, methyloctylsulphide C<sub>9</sub>S, dioctylsulphide  $C_{16}S$ , dioctyldisulphide  $C_{16}S_{2}$ ). Another group is represented by sulphur-containing molecules with the regions of hydrocarbon framework composed of the fragments of the initial octane molecule (dipropylsulphide  $C_{s}S$ , propylbutylsulphide

 $C_7S$ , dipentylsulphide  $C_{10}S$ ). The third group of compounds is combined. The regions of hydrocarbon framework that occur in the compounds of this group include either initial octane molecule or its fragments (for example, propyloctylsulphide  $C_{11}S$  or butylactylsulphide  $C_{12}S$ ).

The presence of different alkyl substituents in the structure of the detected sulphur-containing molecules in the products of the treatment of initial mixtures of hydrogen sulphide with hydrocarbons suggests that plasmachemical processes are participated not only by methane as the major hydrocarbon component of initial mixtures but also by octane molecules subjected to destruction at the C-C bond as a result of electronmolecular interaction.

Indeed, analysis of the distribution of the losses of electron energies in the BD for the excitation of different states of the molecules in initial gas mixtures, aimed at evaluation of the composition of particles formed at the stage of dischargedriven initiation of the process and subsequent direction of their transformations (Table 1, the data concerning the  $CH_4 - H_2S - C_8H_{18}$  is absent from the Table because of the insufficient amount of data for calculations) clearly suggests that the largest fraction of the energy of electrons in the BD is consumed for the activation of methane and hydrogen sulphide molecules; their further interactions lead to the formation of final products according to reactions [10, 11]: 

$CH_4 + e \rightarrow$	$CH_{3} + I$	d + e (	(3)	

- $CH_4 + e \rightarrow CH_2 + H_2 + e \tag{4}$
- $\begin{array}{ll} \mathrm{CH}_{4}+e\rightarrow\mathrm{CH}+\mathrm{H}_{2}+\mathrm{H}+e & (5)\\ \mathrm{CH}_{4}+e\rightarrow\mathrm{C}+2\mathrm{H}_{2}+e & (6) \end{array}$

TABLE 1

Energy losses by BD electrons in different mixtures (reduced electric field strength (E/n) = 110 Td)

Channel of losses	Mixture			
	$CH_4$	$CH_4 - O_2$	$CH_4 - CO_2$	
	Methane			
Vibrations	47.1	39.6	37.9	
Excitation of electron states	20.5	17.4	18.1	
Ionization	0.3	0.2	0.2	
	Hydrogen sulphide			
Vibrations + excitation of electron	31.4	29.6	28.1	
states				
Ionization	0.1	0.1	0.1	
Sticking	0.04	0.04	0.04	
Average energy of electrons, eV	3.9	3.8	3.9	

$$\begin{array}{ll} 2{\rm CH}_3 + {\rm M} \to {\rm C}_2 {\rm H}_6 + {\rm M} & (7) \\ {\rm CH}_4 + {\rm CH} \to {\rm C}_2 {\rm H}_4 + {\rm H} & (8) \\ {\rm C}_2 {\rm H}_6 + {\rm CH}_2 \to {\rm C}_3 {\rm H}_8 & (9) \\ {\rm H}_2 {\rm S} + e \to {\rm SH} + {\rm H} + {\rm e} & (10) \\ 2{\rm SH} \to {\rm S} + {\rm H}_2 {\rm S} & (11) \\ {\rm SH} + {\rm H} \to {\rm S} + {\rm H}_2 & (12) \\ 2{\rm S} \to {\rm S2} & (13) \end{array}$$

where e is BD electrons.

The presence of oxygen/carbon dioxide in the initial mixture causes only insignificant changes in the nature of the distribution of energy losses by BD electrons and serves the source of atomic oxygen in the system according to dissociation reactions:

$O_2 + e \rightarrow 2O(^{3}P) + e$	(14)
$CO_{1} + e \rightarrow O(^{3}P) + CO + e$	(15)

This leads to the appearance of additional channels of the transformations of radicals formed at the stage of discharge initiation with the formation of oxygen-containing compounds (sulphones, sulphoxides) [11].

In the case of the BD treatment of the  $CH_4$  –  $H_2S - C_8H_{18}$  mixture, it appears impossible to calculate plasma parameters because the values of cross-sections for electron-molecular reactions participated by octane are absent from the literature. Nevertheless, rough estimations show that octane vapour does not cause noticeable changes of BD plasma parameters in comparison with the parameters for the  $CH_4 - H_2S$  mixture, so its possible effect on the distribution of electron energy losses and the mechanism of the process may be neglected. However, a permanent presence of a film of liquid octane on reactor walls prevents further transformations of intermediate compounds with sulphur atom, in particular, inhibits their localization of the electrodes of the reactor, and leads to the formation of elemental sulphur according to the scheme [11]:

$RH + e \rightarrow R + H \text{ or } R' + R'' + e$	(16)
$R + S \rightarrow RS + R/S RSR/RSSR$	(17)
$R + SH \rightarrow RSH$	(18)

$$2S \rightarrow S_{2} + S_{2}$$
(19)

where R is an alkyl radical.

Alkylsulphides and thiols are formed as a result of reactions (17) and (18), while elemental sulphur is the product of reaction (19). The formation of elemental sulphur is confirmed by emission spectra, and particle size was determined by means of photon correlation spectroscopy: the maximum of the mass distribution of particles was ~10 nm [11].

So, the data obtained provide evidence of the possibility to govern the formation of deposits on the electrodes of the plasmachemical reaction during the purification of methane from  $H_sS$  in the BD with the conservation of a high degree of H<sub>s</sub>S removal. Varying process conditions (adding octane vapour to the initial gas mixture), one may achieve a high degree of inhibition of the reactions leading to the formation of deposits, while discharge parameters remain unchanged, and the degree of H<sub>3</sub>S removal within the studied concentration range is conserved at a level of 92-98 %. Changing the composition of the initial gas mixture by adding oxygen/carbon dioxide, octane vapour, one may govern the composition of sulphur-containing products and obtain different compounds: from cyclic and linear organic polysulphides (without additives), sulphones, sulphoxides (as a result of the addition of  $O_{a}/CO_{a}$ ) to alkylsulphides and elemental sulphur (in the presence of octane vapour). The concentrations of additives vary within a broad range, and the major stages of the mechanism - the interaction of BD electrons with methane and hydrogen sulphide molecules, followed by subsequent transformations of the formed radicals, - remain the same.

## CONCLUSION

A single-stage process of methane purification from hydrogen sulphide with the help of barrier discharge is developed. The method does not require the use of catalysts and chemical reagents, the process is carried out at atmospheric pressure and room temperature and it is promising for the preparation of natural and associated petroleum gas for further processing and application in alternative power engineering. The approaches to the control of chemical processes with the formation of valuable sulphur-containing products are elaborated. The obtained organosulphur compounds are promising for practical application: as additives to sulphur concrete, to surfactants, biologically active substances for agriculture, as extractants of rare and precious metals, floatation reagents in metallurgy.

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