

Проведено парову конверсію вугілля в умовах технології, що має назву «аерозольний нанокаталіз». Процес проведено в реакторі нового типу – із шаром каталітичної системи, що обертається. Збільшення температури підвищує вихід водню та монооксиду вуглецю. Показано збільшення співвідношення CO:H₂ від температури. Результати потрібні для розробки основ технології синтез-газу з покращеними техніко-економічними показниками

Ключові слова: аерозольний нанокаталіз, механохімічна активація, синтез-газ, парова конверсія, каталітична система

Проведена паровая конверсия угля в условиях технологии, имеющей название «аэрозольный нанокатализ». Процесс проведен в реакторе нового типа – с вращающимся слоем каталитической системы. Увеличение температуры повышает выход монооксида углерода и водорода. Показано увеличение соотношения CO:H₂ от температуры. Результаты необходимы для разработки основ технологии синтез-газа с улучшенными технико-экономическими показателями

Ключевые слова: аэрозольный нанокатализ, механохимическая активация, синтез-газ, паровая конверсия, каталитическая система

OBTAINING SYNTHESIS-GAS BY THE STONE COAL STEAM CONVERSION USING TECHNOLOGY OF AEROSOL NANOCATALYSIS

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1. Introduction

There are several technologies for obtaining synthesis-gas in industry at present. The most commonly applied are such as steam conversion of methane, partial methane oxidation, plasma gasification of waste and raw materials, coal gasification. The synthesis gas is composed of a mixture of CO and H₂ and is a multi-purpose substance. However, it can be a raw material for most chemical reactions [1].

Any carbon-containing compound can serve as a raw material for the production of synthesis-gas. The most common are methane or natural gas, liquid fractions of oil and coal. The basic reaction of steam conversion of hydrocarbons occurs on the catalyst Ni/Al₂O₃. The reaction is strongly exothermic with the equilibrium shifting towards products of reaction at increasing temperature. That is why the process is conducted at 800–900 °C and with excess of water vapor. In this case, there is a parallel exothermic reaction of carbon oxide conversion, which at an increase in temperature shifts equilibrium to the starting reagents while the excess of water vapor increases CO₂ in the products of reaction [2].

At a refinery, the required source is hydrogen. It is obtained by the separation of mixture of hydrogen/hydrocarbon fractions, which form after a reforming unit. If a given method cannot produce enough hydrogen, a unit for steam conversion of methane is additionally created [3].

Conversion of coal into a mixture of gases relates to the process of coal gasification. The following gasifying agents

are used for this process: air, oxygen, water vapor, carbon dioxide and hydrogen, as well as mixtures of these compounds. As a result, the following products are obtained: carbon oxide and dioxide, hydrogen, a certain amount of methane, water vapor and nitrogen (air gasification). Depending on the technique for obtaining synthesis-gas, ratio CO:H₂ ranges from 1:1 to 1:3 [4].

Creating an installation for the processing of coal into a product required by the chemical industry is promising for the development of industry in the country. One of such processes is the steam conversion of coal. There exists the possibility of implementing a process of steam coal conversion using the technology of aerosol nanocatalysis in a rotating reactor.

The principles of aerosol nanocatalysis technology (AnC) are described in [5]. The main feature is using only a catalytically-active substance in the aerosol state without a carrier. Application of mechanical action in order to maintain active state of the catalytic surface. Mechanical activation increases the speed of reaction and reduces deactivation of the catalyst.

Due to mechanical impacts the catalyst particles are destroyed in a collision with a solid surface (free impact effect) [6]. Substances in the finely-dispersed state are obtained in the shredding devices. In this case, there are defects of structure in the volume and active states on the surface of a particle, which increase reactivity [7].

Chemical progress and continuous motion of the dispersing material in a reactor lead to the mechanochemical acti-

vation of the catalyst. Permanent shocks and friction lead to a change in the thermodynamic potentials of the activated substance; to the emergence of surface structure defects, its activation and grinding to the nano-dimensional state [8].

The relevance of present work lies in the fact that the new technique for obtaining synthesis-gas is proposed, which is better than those applied in the industry at present. Synthesis-gas, in turn, is a valuable, necessary product for the country's chemical industry. The raw material, which is proposed to utilize in a given process, is an affordable resource with stocks in the country. Implementation of the process of steam coal conversion using the technology of aerosol nanocatalysis in a rotating reactor opens up new prospects for the chemical industry of Ukraine.

2. Literature review and problem statement

In paper [9], authors investigated a catalytic effect on the process of interaction between carbon and water steam. The use of a catalyst is examined; however, the possibility of increasing the catalytic action was not addressed.

Authors of [10] designed gasification reactors for a steam-gas unit of high power. They tackled issues of creation and development of the estimation model of a current reactor for thermal-chemical conversion of coal dust. They considered aerodynamic mechanisms that influence the operation of generators. However, the paper failed to outline shortcomings and advantages of different aerodynamic mechanisms of influence.

Paper [11] reports a thermodynamic analysis of coal gasification. By using a method of chemical thermodynamics, the authors estimated equilibrium composition of gasification products in a boiling layer of coal. The study, however, did not consider effect of blowing coefficient on the composition of products of gasification of organic mass of coal and the way it changes the ratio of $\text{CO}:\text{H}_2$.

In paper [12], authors examined a problem of excessive water vapor in the process of coal steam gasification. A conclusion was drawn regarding the ratio of water vapor to carbon, which was 2:1. The effect of excessive carbon was not studied.

In [13], a problem of chemical performance efficiency of the process is addressed. The authors argue that while conducting the air gasification of solid fuels, temperature at the output of the layer turns out to be insufficient to achieve the maximum values of chemical performance efficiency of the process of gasification process. A conclusion was made on the necessity of supplying additional amount of heat to the reaction zone. The technology of aerosol nanocatalysis solves this problem through mechanical treatment of coal and its additional grinding in a rotating reactor.

Authors of [14] detected a problem on applying pressure in the process of coal gasification at Gukovo-Gryaznovski deposit. A coal layer gasification process was investigated at atmospheric and elevated pressure. Elevated pressure is a disadvantage. When employing the aerosol nanocatalysis technology, it is proposed to use atmospheric pressure.

Paper [15] examined systems of coal gasification in chemical industry and energy generation. A conclusion was drawn about the effectiveness of operation and the extent of commercial use of these systems was considered. When compared with the examined systems of gasification, coal conversion using the technology of aerosol nanocatalysis

demonstrates technological advantages and prospects of industrial implementation.

Authors of paper [16] identified a problem of the inappropriate use of coal through burning it in the form of a water-coal suspension, which is carried out in the boiler units of small capacity. The study, however, did not compare a given technique for using coal with alternative methods. The authors drew examples of experimental-industrial implementation of technologies for flame-drip burning of water-coal mixture in furnaces over a layer of burning coal and a vortex furnace chamber. Compared with those techniques, coal conversion using the technology of aerosol nanocatalysis has a better perspective from the economic point of view.

Conducting a process of stone coal steam conversion employing the technology of aerosol nanocatalysis in a rotating reactor makes it possible to solve the problems considered in our analysis of the scientific literature. Examining conversion of stone coal under conditions of the technology of aerosol nanocatalysis leads to a decrease in the temperature of the process. The degree of conversion of raw materials improves as well; the amount of catalytically-active substance in the reactor decreases by up to 10^6 times. All these factors make it possible to eliminate the constraints (mechanical and thermal) associated with the use of the carrier.

3. The aim and objectives of the study

The aim of present study is to determine the effect of temperature on the course of the process of converting stone coal under conditions of the technology of aerosol nanocatalysis in a rotating reactor. This will make it possible to develop the base of technology for converting coal into synthesis-gas with improved technical and economic indicators.

To accomplish the aim, the following tasks have been set:

- to modernize the circuit of a laboratory installation (to improve the node of water vapor supply and the node of gas mixture selection) and to devise a procedure for research;
- to conduct experiments on steam conversion of stone coal and to analyze the results obtained;
- to study changes in the yield of reaction products caused by temperature.

4. Preparation to the study into a process of obtaining synthesis-gas by steam conversion of stone coal

4.1. Schematic of the laboratory installation

A circuit of the laboratory installation with a rotating reactor was prepared for studying a process of coal steam conversion. Specifically, we improved the node of water vapor supply and the node for selection of the resulting gas mixture. Figure 1 shows schematic of the laboratory installation for studying the process of stone coal steam conversion.

Water (flow A) via a syringe dispenser (1) is fed to a water supply unit (2), and then to a furnace (4). Inside the furnace is a cylindrical rotating reactor (5), which contains coal, as well as the catalytic system. Air-tight design is ensured by numerous seals using graflex and two sealing nodes. Heating is conducted by means of electric heating spiral (12), onto which ceramic rings are mounted. Reaction chamber is rotated by electric motor (9) and the transfer mechanism (3). The transfer mechanism consists of a host and slave pulleys

and a flexible item – the belt. Voltage that is fed to power the electric motor and electric heating spiral is controlled by means of laboratory auto-transformers (7, 10). The change of voltage makes it possible to adjust the speed of reactor rotation and the temperature in the reactor. The furnace has a pocket for temperature measurement using a thermocouple (13). To minimize heat loss to the environment, on top of the furnace is a layer of heat insulating material (11) [17].

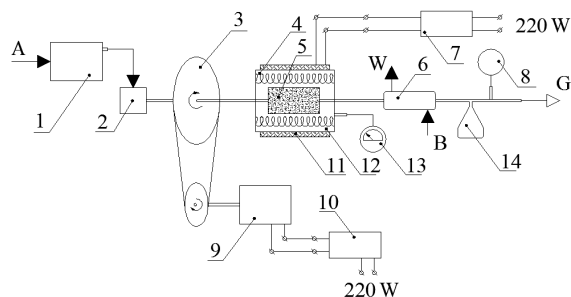


Fig. 1. Schematic of the laboratory installation:

1 – dispenser; 2 – water supply unit; 3 – transfer mechanism; 4 – furnace; 5 – reactor; 6 – heat exchanger; 7, 10 – controlled laboratory auto-transformers (CLAT); 8 – sample-taker; 9 – electric motor; 11 – thermal insulation; 12 – electric heating spiral; 13 – thermocouple; 14 – condensate collector. Flows: A – water (raw material); B – cooling water; W – water; G – conversion gases

At the output of the furnace there is a heat exchanger (6), which is used for cooling gaseous products. Cooling is carried out by feeding water to the heat exchanger (flows B and W). After the heat exchanger there is a container for collecting condensate (14), with an opening for taking the samples (8) [17].

In the course of the study we examined stone coal (Lisichansk, Ukraine) of the following composition, % by weight: C – 86; H – 4.5; N – 1.5; O – 3.1; S – 3.2; impurities – 1.7.

4. 2. Procedure for conducting the experiment

Before the experiment, we placed a catalytic system and the examined mass of stone coal in the reactor. Volume of the reactor was 90 cm³. This coal mass was shredded to a fraction the size of particles 1.2–0.3125 mm. During experiments, we studied coal with a weight of 19 g and 1 g. Density of the dust fraction of coal was 633 kg/m³ [17].

The catalytic system was composed of a dispersive material (glass balls with a diameter of 0.8–1.2 mm) and a catalyst – Cr₂O₃. The system takes up 30 % of the volume of the reaction zone (at 19 g of coal) and 50 % (at 1 g). Preparation of the catalytic system was performed in line with a standard procedure for the technology of aerosol nanocatalysis [18].

The experiment was carried out at 550 to 750 °C. To remove air from the reactor, the reactor before the experiment was blown with nitrogen [19].

Rotation of the reactor creates an aerosol of nanoparticles of the catalyst and enables its mechanochemical activation [20].

Reactor rotation velocity was accepted at 3 Hz, which proved to be the best according to preliminary results of the research. Increasing rotation velocity of the catalytic system is inapplicable because it is pressed against the walls of the cylindrical reactor by centrifugal force [21].

Upon reaching the examined temperature, water supply starts via a syringe dispenser. Water feed rate was 3.43 ml/min and 1.15 ml/min (0.2058 and 0.069 l/h, respectively).

Conversion gases were cooled in the refrigerator with water and were collected in the condensate collector. Conversion gases contained the unreacted water and the water that had formed during chemical process. In reality, however, it is rather problematic to completely remove water vapor from the conversion gases, which is why vapor was partially present in the analyzed samples [22].

A gas sample was selected for the further analysis.

4. 3. Procedure of the analytical control

Analytical control is carried out to determine chemical composition of the resulting reaction products. The data obtained are then estimated, analyzed and systematized. Based on the received information, one can correct a procedure for conducting the experiment.

Analytical control process is performed using the chromatograph “LHM-8” (made in the USSR). The employed technique of analysis belongs to the methods of gas chromatography. Among the determined percentage content of components of the analyzed mixture the most important indicator is the volumetric part of hydrogen and carbon monoxide in a gas sample.

Gas chromatography is a method for separation of volatile components in which a movable phase is the inert gas (gas-carrier) that flows through the motionless phase with a large surface.

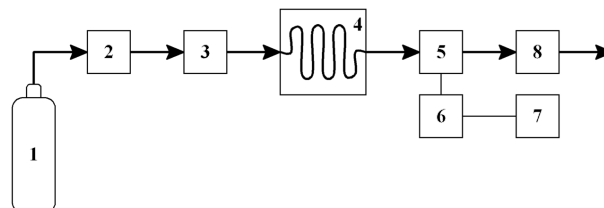


Fig. 2. Schematic of a gas chromatograph: 1 – the source of gas carrier (movable phase); 2 – gas-carrier flow controller; 3 – device for sample introduction; 4 – chromatographic column in the thermostat; 5 – detector; 6 – electronic amplifier; 7 – registering device (recorder, computer); 8 – flow meter

Hydrogen, helium, nitrogen, argon, carbon dioxide are used as a mobile phase (in this case, we applied nitrogen). A gas-carrier does not react with the stationary phase and the substances that are separated.

A gas sample is taken from a sample-collector into an air-tight container with a volume of 20 ml. Next, the sample to be analyzed is brought for chromatographic analysis. The gas sample, via the dispenser, is introduced to the flow of gas-carrier. Duration of analysis of one sample is 40 minutes. The chromatograph determines concentrations of the following substances: H₂, O₂, CO, CO₂, H₂S, CH₄, C₂H₂, C₂H₄, C₂H₆ [22].

Concentrations of organic substances (except for CO and CO₂) were determined with accuracy not less than 0.001 % by volume, the concentrations of H₂, O₂, CO, CO₂ – not less than 0.01 % by volume [22].

5. Experimental study of the process of obtaining synthesis-gas by stone coal steam conversion

The target product of the process is the synthesis-gas (CO+H₂). Design of the study technique is aimed at increas-

ing volumetric fractions of these products in the gas sample. According to data on chromatographic analysis, along with these substances, many other substances are produced in the course of chemical reactions. This indicates that the process occurs with a large number of side reactions [21].

Results of study of the process are given in Table 1, 2.

Table 1
Composition of conversion gases
(loaded: 19 g of coal, water feed rate: 3.43 ml/min)

Temperature, °C	Composition of conversion gases, % by volume							CO:H ₂	Selectivity	
	H ₂	CO	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	H ₂ S		(CO)	(CO ₂)
550	1.28	1.17	9.36	3.05	0.48	2.31	5.38	1:1.09	4.53	36.26
600	3.87	2.79	12.57	3.51	0.66	2.56	6.52	1:1.39	7.81	35.21
650	6.54	3.12	14.67	3.98	0.73	3.28	8.27	1:2.10	7.00	32.90
700	8.48	4.11	15.64	4.85	0.89	4.71	8.94	1:2.06	7.72	29.40
750	9.53	4.29	17.66	5.16	0.98	4.93	9.82	1:2.22	7.36	30.31

Table 2

Table 2
Composition of conversion gases
(loaded: 1 g of coal, water feed rate: 1.15 ml/min)

Temperature, °C	Composition of conversion gases, % by volume							CO:H ₂	Selectivity	
	H ₂	CO	CO ₂	CH ₄	C ₂ H ₄	C ₂ H ₆	H ₂ S		(CO)	(CO ₂)
550	1.25	1.08	9.13	2.96	0.43	1.96	5.35	1:1.15	4.40	37.21
600	3.23	2.76	11.74	3.47	0.65	2.45	6.47	1:1.17	8.15	34.68
650	5.88	3.08	13.56	3.91	0.71	2.87	8.26	1:1.91	7.35	32.40
700	7.91	3.42	15.01	4.64	0.84	4.15	8.86	1:2.31	6.87	30.13
750	9.17	3.92	17.84	5.04	0.96	4.90	9.80	1:2.34	6.81	31.04

Dependences of the composition of conversion gases on temperature are shown in Fig. 3–5 (at different loading of coal and different water feed rate).

Fig. 3 shows effect of temperature on the direct yield of synthesis gas at different ratios of C:H₂O.

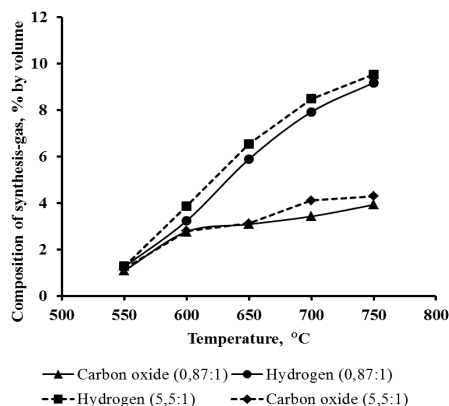


Fig. 3. Yield of synthesis-gas dependent on temperature at different ratios (C:H₂O=0.87:1) and (C:H₂O=5.5:1)

Fig. 4 shows effect of temperature on the yield of basic products and by-products.

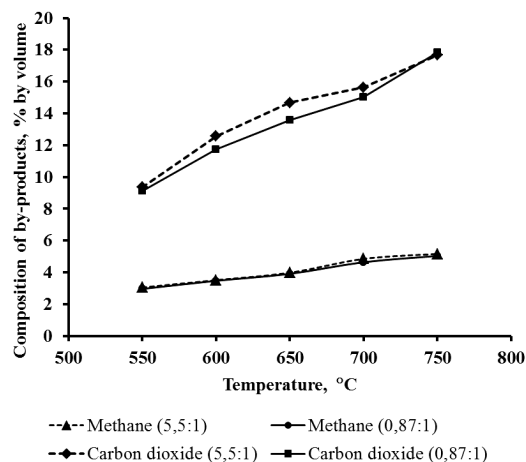


Fig. 4. Yield of by-products dependent on temperature at different ratios (C:H₂O=0.87:1) i (C:H₂O=5.5:1)

Fig. 5 shows dependence of change in the ratio of CO:H₂ on temperature at different loads of coal and varying water feed rate.

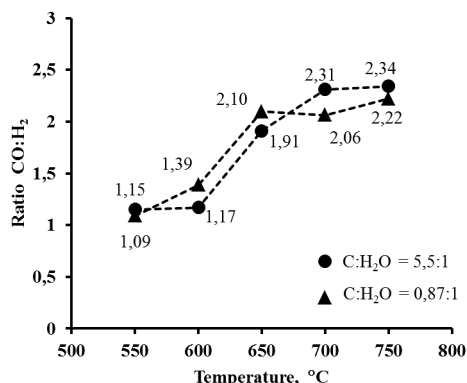


Fig. 5. Dependence of change in the ratio of CO:H₂ on temperature

We can note the increase in carbon monoxide on temperature, which is typical for the processes of conversion of any raw materials. Temperature is one of the factors that affect the composition of synthesis-gas, allowing it to vary depending on the needs in the industry.

6. Discussion and analysis of the obtained results

In the course of the study, we modeled two modes:

- 1) with a large excess of coal (C:H₂O=5.5:1) in the reactor, which leads to a zero-order reaction, and carbon (Table 1);
- 2) with a relatively small amount of coal (C:H₂O=1:0.87) to intensify mechanochemical activation (Table 2).

Table 1, 2 show that the investigated mode of steam conversion of stone coal in a temperature range from 550 to 750 °C contributes to obtaining such by-products as carbon dioxide (about 9–18 % by volume) and hydrogen sulfide (approximately 5–10 % by volume). The yield of these products increases with an increase in temperature.

Obtaining hydrogen, which is one of the target products, with an increase in temperature in the range of 550–750 °C, grows from 1.25 to almost 10 % by volume. (Fig. 3).

It should be noted that the increase in temperature results in an increase in the rate of obtaining hydrogen by 7 times, that of hydrogen sulfide – by only 1.8 times (Table 1, 2). This indicates the sensitivity of the process of hydrogen evolution depending on temperature.

Another target product is carbon oxide, which forms along with hydrogen and by-products. In this case, the rate of its formation increases by 3.6 times with a rise in temperature.

We also noted obtaining such side hydrocarbons as methane, ethane, and ethylene in smaller quantities (Table 1, 2).

Next, we shall examine the process under the mode implying a shortage of carbon, that is, $C:H_2O=0.87:1$. In this case, we observed two temperature regimes under which we registered almost a stability at the ratio of target products $CO:H_2$, which is equal to 1.2 in a temperature range of 500–600 °C, and 2.3 in a temperature range of 700–750 °C (Fig. 5).

The sum of the yield of H_2 and CO, that is, a mixture of target products in a given process, increases from 1.5 up to 13.8 % by volume at a change in temperature from 550 to 750 °C, respectively.

When considering a process under the mode of excessive carbon, that is, $C:H_2O=5.5:1$, we registered that in the temperature range of 650–750 °C, practical stability is achieved at ratio $CO:H_2$, which is approximately in the range of 2.1–2.2.

These preliminary results can be predicted in the following manner: a temperature of about 700 °C at ratio $CO:H_2\approx 1:2$ is applied to obtain methanol or other alcohols; a temperature of about 600 °C at ratio $CO:H_2\approx 1:1$ is employed to obtain esters.

If we compare it with the analog of the process of steam conversion of methane, which proceeds at 800–900 °C and at 2–2.5 bar, then the examined process of coal steam conversion reaches stability at 600–700 °C and 1 bar.

The obtained preliminary results could answer the question whether it is possible to conduct the process of steam conversion under conditions of aerosol nanocatalysis. And our answer is positive. The process, however, is complicated and technological conditions have specific control parameters. That is why, while further determining the optimal mode of the process of steam coal conversion, it is necessary to investigate the influence of reactor rotation frequency, of the concentration and composition of the catalyst, and to extend the interval of ratios between starting substances.

The composition of conversion gases in Table 1, 2 is explained by conditions when conducting the process, and the composition of the starting raw materials. Specifically: the high yield of hydrogen sulfide is due to the appropriate level of sulfur content in the examined samples of stone coal. The fact that the increase in temperature leads to a change in the

hydrogen yield with a larger growth compared with hydrogen sulfide is explained by a certain chemism of the process. Its special feature is the increased part of chemical reactions with hydrogen evolution at an increase in temperature.

The difference between yields of products at different load of coal and at varying water feed rate is due to the influence of change in the molar ratio of the utilized raw materials on a change in the part of certain reactions in a total quantity of reactions that occur in a given process.

The sum of yields of H_2 and CO, reported in the present study (Table 1, 2, Fig. 3), is explained by a low, for this process, temperature of its course. The process of stone coal steam conversion is typically conducted at temperatures 1,000–1,100 °C. In a given work, the temperature is much lower, by 350–450 °C. This particular pattern is the advantage of carrying out this process using the technology of aerosol nanocatalysis.

7. Conclusions

1. We prepared a laboratory installation with a rotating reactor in order to study the process of coal steam conversion. Specifically, the improvements were made to the node of water vapor feed and to the node for selecting the resulting gas mixture. The study is conducted using a mixture of solid and steam phases, which introduces certain adjustments to the procedure of research. This scientific method implies that the reactor contains a solid phase, which consists of coal and a catalyst. Chemical conversion proceeds on the solid surface of the catalyst during interaction between solid coal, dispersed to the nano level, and the steam phase.

2. The results obtained show the possibility of conducting the process under conditions of the technology of aerosol nanocatalysis. In this case, we employed the new type of reactor – a rotating reactor. The results showed that at almost stoichiometric ratio we obtain two temperature intervals that could prove to be optimal.

3. When considering the impact of temperature on the yield of target products of the process of coal steam conversion, it is possible to assume the existence of several modes, which would contribute to receiving different resulting products. That is, at a temperature of 600–700 °C and at ratio $CO:H_2\approx 1:2$, it can be used for the production of alcohols, specifically methanol; at a temperature of 550–600 °C and at ratio $CO:H_2\approx 1:1$, it can be used for the production of complex esters. These are only preliminary results and conclusions, however; it is required to consider the impact of other parameters that control the process. This would make it possible to approach the optimal mode for obtaining a specific product.

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