Development of methods for thermal desorption of iodine from carbon sorbent

E A Shapovalova, R A Hlopotov
Tyumen Industrial University, 38, Volodarskogo St., Tyumen, 625000, Russia
E-mail: shapovalovaelan@yandex.ru

Abstract. The paper studies and proposes four circuits of thermal iodine desorption from coal, which excludes the use of chemical reagents. The method allows for the sublimation of iodine from coal, avoiding the stage of pre-concentration and crystallization of crude iodine-concentrate. The proposed solution allows carrying out the process of thermal desorption of iodine without unloading it from the reactor.

1. Introduction
Today in the world, there is an actual problem of recycling wastewater produced from oil and gas fields, which is a source of valuable elements, such as iodine, bromine, boron, magnesium, lithium, etc. Therefore, development of innovative technologies for complex processing of hydromineral resources into the commercial chemical product is an important task of sustainable nature management.

The comparative analysis of the existing methods for the production of iodine from groundwater has shown that the stage of acidification of industrial water with mineral acid to suppress iodine hydrolysis and the oxidation stage of iodides with chlorine is general for these methods.

This is due to relatively high costs of reagents, which in turn affects the final cost of the product. Special attention must be paid to the system of industrial waste effluents, which when passing the stage of iodine extraction contains a substantial amount of toxic impurities, such as chlorine and sulfuric or hydrochloric acid. As a result, additional effluent wastewater purification is required after iodine extraction using reagents to neutralize them.

2. Materials and methods
As an optimal variant, based on the analysis of the state of the investigated problem, a combined non-reagent method [1] combining sorption and electrochemical processes was developed. The essence of the proposed method is as follows: the sorption unit where the iodine adsorption process takes place on activated carbon is simultaneously a flow-through electrolyzer, as the supplied DC provides acidification of the incoming water in the anode space of coal and the oxidation of iodide to molecular iodine with a constant potential. The suggested method received a patent of RF № 2550405 [2]. This approach ensures conducting all chemical processes in a single unit, eliminating the preliminary acidification and oxidation steps.

Then, after a complete saturation of the sorbent with iodine it is offered to start iodine desorption without unloading coal from the reactor, thus completing the process without additional desorption units.
As part of our work processes more research iodine desorption from coal have been carried out. Traditionally, chemical desorption is used in the production of iodine, namely eluting the sorbent with various solvents. During chemical desorption by washing iodine from the sorbent, iodine concentrate is obtained that goes to the subsequent selection of the crude iodine from it. This process is quite lengthy and requires the use of various reagents. It is proposed to use thermal desorption, which provides sublimation evaporation of iodine from the volume of coal. The method of thermal desorption is based on the fact that during heating the surface of the sorbent to certain temperatures bonds of physically and chemically adsorbed molecules with the surface are destroyed and the concentration of the molecules near the surface is reduced. Thermal desorption of volatile compounds is carried out by purging with superheated steam, inert gas, regenerating in furnaces [3-5]. On this subject, patent studies were carried out. The results of the analysis of the main Russian and foreign patents on the subject are presented in the following patents [6-10].

3. Results and discussion
Solving the problem, four schemes of thermal desorption of iodine from coal were developed, which will be considered below.

Figure 1 shows a circuit of thermal desorption of iodine from coal with superheated steam. The circuit consists of reactor 1 that is a cylindrical column inside of which is carbon saturated with iodine. Steam generator 3 and superheater 4 are sources of superheated steam for coal thermal desorption. Immediately after the column, steam desublimer 5 of the first level is installed which produces an iodine paste. Desublimer 6 of the second level to obtain crystalline iodine (commercial product).

Figure 1. Circuit of thermal iodine desorption from coal with superheated steam
The method of thermal iodine desorption using the device is as follows. Water taken from the tank by a pump built into the steam generator is heated and converted to steam with a temperature of 130-150°C, and then the steam is reheated in the superheater to a temperature of 380-420°C and is supplied to the thermally insulated reactor with carbon saturated with iodine.

Figure 2 is a circuit of thermal desorption of iodine from coal by means of hot compressed air.

The circuit consists of a reactor that is a cylindrical column inside of which is carbon saturated with iodine. Compressor and superheater are sources of hot compressed air for coal thermal desorption. Immediately after the column, gas desublimator of the first level is installed which produces a highly concentrated iodine paste. Desublimator of the second level is necessary to obtain crystalline iodine (commercial product) from the iodine paste.

The method of thermal iodine desorption using the device is as follows. The injected air is heated via a compressor flow-through heater to a temperature of 400-500°C and is supplied to the thermally insulated reactor with carbon saturated with iodine. Downstream of the reactor is a gas desublimator that captures the hot air with iodine vapor and crystallizes it in a highly concentrated iodine paste that is further processed into a finished product in the second level desublimator. It should be noted that with this method coal must withstand temperatures up to 500°C without its ignition. These include coal grade "KAD iodine".

![Figure 2. Circuit of thermal iodine desorption from coal with hot compressed air.](image)

Figure 3 is a circuit of thermal desorption of iodine from coal by means of heated inert gas. The circuit consists of a reactor that is a cylindrical column inside of which is carbon saturated with iodine. Cylinders with argon and superheater are a source of heated inert gas for coal thermal desorption.
Immediately after the column, gas desublimer of the first level is installed which produces a highly concentrated iodine paste. Desublimer of the second level is necessary to obtain crystalline iodine (commercial product) from the iodine paste.

The method of thermal iodine desorption using the device is as follows. Argon in the gaseous state under slight pressure of 1-2 atm is heated in a flow-through heater (steam superheater) to a temperature of 400-500°C and is supplied to the thermally insulated reactor with carbon saturated with iodine. Downstream of the reactor is a gas desublimer that captures the hot air with iodine vapor and crystallizes it in a highly concentrated iodine paste that is further processed into a finished product in the second level desublimer. It should be noted that with this method coal must withstand temperatures up to 500°C without its ignition. These include coal grade "KAD iodine".

Figure 3. Circuit of thermal iodine desorption from coal with heated inert gas.

Figure 4 is a circuit of thermal desorption of iodine from coal by means of heating coal in the reactor.

The circuit consists of a reactor, that is, a cylindrical column inside of which is carbon saturated with iodine. Simultaneously, the reactor is a heat carrier with a thermal jacket that is heated with superheated steam generated from the steam generator and superheater. With the vacuum pump negative air pressure is created in the reactor required to conduct thermal desorption processes. Immediately after the column, gas desublimer of the first level is installed which produces a highly concentrated iodine paste. Desublimer of the second level is necessary to obtain crystalline iodine (commercial product) from the iodine paste.

The method of thermal iodine desorption using the device is as follows. The reactor is heated to 184°C (iodine sublimation temperature) and above, with a vacuum pump creating a negative pressure in the reactor through the gas desublimer. There is a constant flow of air through the bottom valve of
the reactor. Vapors of evaporated iodine enter the gas desublimer and are converted into an iodine paste that enters in the second-level desublimer.

![Diagram of thermal iodine desorption from coal with heating reactor in vacuum](image)

**Figure 4.** Circuit of thermal iodine desorption from coal with heating reactor in vacuum

4. Conclusions
The studies obtained and presented solutions with different iodine desorption circuits. Carrying out pilot tests on selected circuits will allow making conclusion on the best option of thermal iodine desorption and recommending it for implementation at experimental industrial production.

References
[1] Ganyaev V P, Shapovalova E A 2013 Removing and safe disposal of iodine from natural geothermal and formation water of oil and gas fields (IV International Innovation Forum "NeftGazTek) Tyumen pp 61-63
[2] Shapovalova E A, Ganyaev V P, Latysheva T I, Andrianova L I 2015 The method of extraction of iodine from underground artesian water (Patent RU 2550405 C02F9/06 , C02F9/06 )
[3] Harlampovich G D,  Kudryashova R I, 1978 Wasteless processes in the chemical industry. (Moscow: Himiya) p 280
[4] Ksenzenko V I, Stasinenvich D S 1995 Chemistry and technology of bromine and iodine and their compounds. (Moscow: Chemistry) p 460
[5] Laperdin A N, Kozintsev A N, Plotnikov A A 2005 Use of West Siberian underground pressure water for the production of iodine. (Novosibirsk: Izdatelstvo SB RAS) p 127
[6] JX Nippon Mining & metals corp 2013 Method for eluting iodine adsorbed activated carbon (Japan patent: JP2013001634)
[7] Amaya Takayuki, Shibuya Mamoru, Kodama Hiroshi 2002 Method for eluting iodine from used iodine adsorbent (Japan patent: JP2002066318)
[8] Nagata Yusuke, Ohashi Shinichi 2009 Method and apparatus for recovering iodine from iodine-containing waste liquid (Japan patent: JP2009142764)
[9] Miyoshi Fumihiro, Osugi Hitoshi, Shibata Masaru, Yokoyama Kazuyuki 2005 Method for removing iodine from salt water (Japan patent: JP2005305265)
[10] Kiekpaev M A, Stroeva E V, Ponomareva P A 2009 Method of desorption of iodine from weakly basic anion exchangers (Patent RU 2397142)