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Thermal Demercurization of Coal Sorbents

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Abstract: The extraction of mercury in the vapor–gas phase from coal sorbents, used to capture mercury from industrial waste gases, was studied herein to develop a unified technology. The behavior of mercury compounds (Hg_2Cl_2 and $HgCl_2$) under conditions of thermal demercurization in a fore vacuum and at atmospheric pressure was examined using partial pressure diagrams. It was established that the stable phases during the technological process are vaporous mercury and Cl_2. As a result of technological research and extensive testing with developed equipment at 400–800 °C and pressure in the range of 0.13–91.99 kPa, it was established that mercury in a vacuum under these conditions almost completely enters the vapor–gas phase (99.4–99.97%). A similar degree of mercury extraction from a coal sorbent was achieved at 600–800 °C at atmospheric pressure. A study was conducted, and it was established that the sorbent after thermal demercurization—in terms of its sorption capacity for gold—was practically comparable to fresh, unused Norit sorbent.

Keywords: mercury; calomel; mercuric chloride; coal sorbent; demercurization; vacuum; technology

Academic Editor: Petros E. Tsakiridis

Received: 29 April 2025
Revised: 4 June 2025
Accepted: 11 June 2025
Published: 13 June 2025

Citation: Kenzhaliyev, B.; Volodin, V.; Trebukhov, S.; Nitsenko, A.; Linnik, X.; Magomedov, D.; Kilibayev, Y. Thermal Demercurization of Coal Sorbents. *Metals* **2025**, *15*, 662. https://doi.org/10.3390/met15060662

1. Introduction

Due to tightening environmental requirements, much attention has recently been paid to the problem of removing mercury from the materials containing it. Mercury is present in fossil coals [1,2] and in various mercury-containing industrial and consumer wastes [3–6], including spent coal sorbents from gold mining factories, sorbents used for collecting mercury from waste gases from thermal power plants and industrial enterprises, and other materials.

In recent years, a number of studies have been conducted to increase the retention capacity of various carbon sorbents [7,8] by means of small additions of copper dichloride [9,10] and gold nanoparticles [11–14]. Studies have also been conducted to develop an effective sorbent intended to remove mercury with the use of biochars modified with Br [15].

One of the methods for removing mercury from contaminated gold concentrates, coal sorbents obtained in the process of gold extraction, and soils is thermal demercurization, developed with the participation of the authors [16]. It enables the removal of mercury up to a content of (3–9) $\times 10^{-4}$ wt.% [17].

However, to develop a unified technology for the demercurization of coal sorbents—in particular, those obtained by capturing mercury from industrial waste gases—the behavior of mercury and its compounds when they are heated in an electric furnace at low and

atmospheric pressures, the related technologies, and the choice of technical design for the process being developed need to be studied.

2. Behavior of Mercury Compounds During Thermal Desorption

The work in [3] showed that mercury was present in waste sorbents obtained at purification plants intended for process gases and ventilation gases. It was present there in the form of mercuric chloride ($HgCl_2$) in an amount of up to 3.5 wt.% and as calomel (Hg_2Cl_2) in an amount of up to 1.8%. The increased content of mercuric chloride in relation to calomel is due to the greater degree of [2] $HgCl_2$ capture from gases.

The behavior of calomel when heated in a vacuum was studied in the laboratory of vacuum processes of the Institute of Metallurgy and Ore Beneficiation. The kinetic characteristics of the process were established, and it was stated that calomel in a vacuum (less than 2.66 kPa) decomposes with the sublimation of mercury at temperatures greater than 573 K (300 °C); at atmospheric pressure, it does so at temperatures greater than 700 K (427 °C). However, the phase state of mercury present in the gas phase and the sequence of decomposition stages were not established.

It is known that the sequence of transformations in nonequilibrium dynamic systems is similar to that in equilibrium ones. Thus, the method of partial pressure diagrams applicable to equilibrium systems was used for our study.

The essence of the method is as follows: Calculate the Gibbs energy of the reaction (ΔG) as $\Delta G = \Delta H - T \times \Delta S$, where ΔH is the difference between the sum of the enthalpies of reaction products and the sum of the enthalpies of initial components; ΔS is the difference between the sum of the entropies of reaction products and the sum of the entropies of initial components; and T is the temperature in K. At the same time, $\Delta G = -RT \ln K_P$, where R is the gas constant and K_P is the equilibrium constant of the reaction. The equilibrium constant, in turn, is equal to the ratio of the product of the partial pressures or activities of the reaction products, to the degree corresponding to the coefficient in the equation, and to the product of the partial pressures or activities of the starting substances to the corresponding degree. Then, the order of reactions and thermodynamically stable phases at pressures corresponding to the technology being developed is determined by constructing a diagram in $\ln T^{-1} - \ln \overline{p}_{HgCl_2} - \ln \overline{p}_{HgCl_2}$ coordinates.

Thermodynamic constants from reference publications were used in the calculations [18,19]. These calculations were performed for the temperature range of 673 to 1083 K (400–800 °C), due to the fact that all components in this temperature range are present in a vaporous or sublimated state. The partial pressure of each substance or compound above its boiling point (sublimation) ($\overline{p}_t$) was found according to Charles's law: $\overline{p}_t = \overline{p}_o(1 + \beta \times \Delta t)$, where $\overline{p}_o$ is the pressure of a gaseous substance at its boiling point (sublimation); β is the thermal pressure coefficient; and Δt is the temperature difference between the boiling point and the determined temperature. Since the thermal pressure coefficients for the compounds under consideration are unknown, and those given in [19] for 12 substances and compounds differ only in their fourth decimal place, the average value, equal to 0.00367, was accepted.

The enthalpy and entropy of evaporation for mercuric chloride $HgCl_2$ were calculated based on the vapor pressure given in [20] and found to be $\Delta H^V_{HgCl_2}$ = 64.55 kJ/mol and $\Delta S^V_{HgCl_2}$ = 108.98 J/(mol × K).

Similar values for calomel were not found in the available sources of information. Therefore, the vapor pressure of $HgCl_2$ was determined using the boiling point method (isobaric version). The initial product was obtained by reacting mercury sulfate ($HgSO_4$) with hydrochloric acid, both of reagent grade, according to the method outlined in [21].

An X-ray diffraction analysis showed a monophase of Hg_2Cl_2 in the washed, filtered, and dried precipitate.

The results of determining the vapor pressure are given in Table 1.

Table 1. Sublimation temperatures (K) corresponding to certain vapor pressures.

Pressure	Temperatures, K			
1.33 kPa (10 mm Hg)	514	521	519	522
40 kPa (300 mm Hg)	615	620	614	628

The vapor pressure of calomel corresponds to the dependence $\ln \overline{p}_{Hg_2Cl_2}[\text{Pa}] = 28.077 - 10,831 \times T^{-1}$. The sublimation temperature calculated using this equation corresponds to 654 K (381 °C), which practically coincides with the data in a reference publication [19] (383.7 °C) and indicates the correctness of the measurements performed.

Hence, the enthalpy and entropy of sublimation correspond to the following values: $\Delta H^V_{Hg_2Cl_2}$ = 90.05 kJ/mol, $\Delta S^V_{Hg_2Cl_2}$ = 137.61 J/(mol × K).

The possible reactions between the components of the vapor phase, their equilibrium constants, and the calculated values of the logarithms of the constants are given in Table 2.

Table 2. Equilibrium constants for the decomposition and interaction reactions of mercury chlorides in the vapor phase.

No.	Reaction	Equilibrium Constant	Values of $\ln K_p$ at Temperature, K	
			673	1073
1	$Hg_2Cl_2 = Hg + HgCl_2$	$\overline{p}_{Hg} \times \overline{p}_{HgCl_2} / \overline{p}_{Hg_2Cl_2}$	14.625	17.015
2	$Hg_2Cl_2 = 2Hg + Cl_2$	$\overline{p}^2_{Hg} \times \overline{p}_{Cl_2} / \overline{p}_{Hg_2Cl_2}$	46.496	48.675
3	$HgCl_2 = Hg + Cl_2$	$\overline{p}_{Hg} \times \overline{p}_{Cl_2} / \overline{p}_{HgCl_2}$	35.311	32.729
4	$2Hg_2Cl_2 = 3Hg + HgCl_2 + Cl_2$	$\overline{p}^3_{Hg} \times \overline{p}_{HgCl_2} \times \overline{p}_{Cl_2} / \overline{p}^2_{Hg_2Cl_2}$	57.678	62.248
5	$Hg_2Cl_2 + Cl_2 = 2HgCl_2$	$\overline{p}^2_{HgCl_2} / \overline{p}_{Hg_2Cl_2} \times \overline{p}_{Cl_2}$	−6.780	32.174
6	$Hg_2Cl_2 + HgCl_2 = 3Hg + 2Cl_2$	$\overrightarrow{p}^3_{Hg} \times \overline{p}^2_{Cl_2} / \overline{p}_{Hg_2C_l} \times \overline{p}_{HgCl_2}$	80.876	83.777

The partial pressure diagram $\ln T^{-1} - \ln \overline{p}_{HgCl_2} - \ln \overline{p}_{HgCl_2}$ is presented in Figure 1, with the partial pressure values of mercury and chlorine included in the numerical value of the equilibrium constant at the corresponding temperature. The diagram highlights the pressure region in the form of a vertical rectangular prism, implemented in the developed technology from 0.133 to 101.325 Pa ($1.32 \times 10^{-3} \div 1$ atm).

It follows from the analysis of the diagram that the decomposition reactions are practically independent of temperature—the reaction planes are practically parallel to the ordinate axis. At low pressures of mercuric chloride (less than 4.3×10^{-13} Pa (4.25×10^{-18} atm)), the decomposition of calomel proceeds according to the reaction $2Hg_2Cl_2 = 3Hg + HgCl_2 + Cl_2$; above this pressure, direct decomposition into mercury and chlorine occurs according to the reaction $Hg_2Cl_2 = 2Hg + Cl_2$. When the pressure increases in the range from 4.3×10^{-13} to 5.0×10^{-30} Pa, mercuric chloride also decomposes into mercury and chlorine. Thus, during the thermal demercurization of coal sorbents both in vacuum and at atmospheric pressure, the thermodynamically stable phases are vaporous mercury and gaseous chlorine. The latter requires an appropriately designed condensation and pumping system.

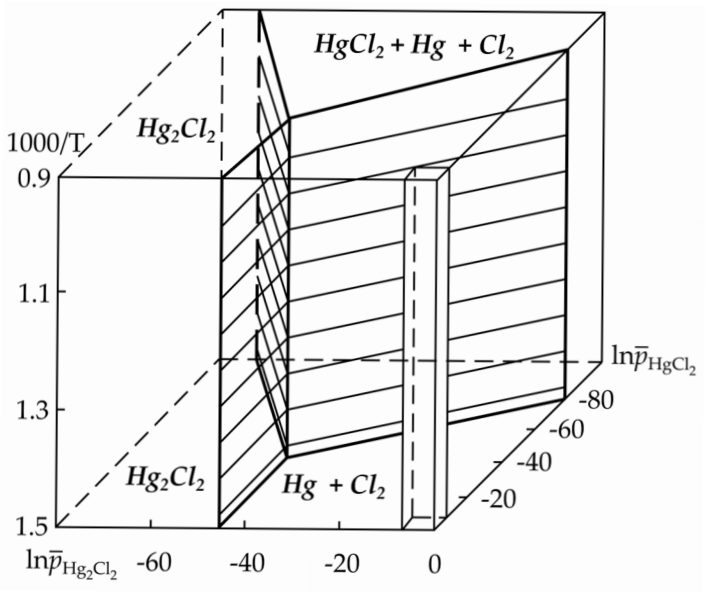

Figure 1. Partial pressure diagram T–Hg_2Cl_2–$HgCl_2$.

3. Technological Studies on Demercurization of Coal Sorbents

For technological studies on demercurization, a coal sorbent used at Altyntau Kokshetau JSC (Kokshetau, Kazakhstan) was chosen. The sorbent contained the following elements, in wt.%: As, 0.0835; Cu, 0.00093; Fe, 0.2491; Zn, 0.00041; Pb, 0.00068; Bi, 0.00099; SiO_2, 0.4644; Al_2O_3, 0.0744; CaO, 0.6618; MgO, 0.011; S, 0.2797. Its Au content was 0.06 g/t. The appearance of the original wet sorbent is shown in Figure 2.

Figure 2. Appearance of wet spent coal sorbent.

The sample was dried in air under natural conditions. The fractional composition of the dry spent sorbent is given in Table 3.

Table 3. Fractional composition of spent coal sorbent.

Fraction size, mm	+2.5	−2.5 + 2.0	−2.0 + 1.0	−1.0
Quantity, wt.%	13.6 ÷ 19.5	34.7 ÷ 39.6	50.7 ÷ 40.6	1.0 ÷ 0.3

The bulk density of the dried sorbent was determined using a measuring cylinder and was 0.593 g/cm^3 without shaking or 0.644 g/cm^3 with shaking. The angle of repose of the dry sorbent was 33°.

An RA-915M mercury analyzer (Lumex-Marketing LLC, Saint Petersburg, Russia) was used to determine low mercury contents. The original optical–electronic circuit of the analyzer provides an ultra-low detection limit for mercury in direct measurement mode (without preconcentration) with high selectivity for analysis and a wide dynamic range of measurements.

For these technological studies, the sorbent was doped with mercury in the form of Hg_2Cl_2 according to the method described in a previous study [17]. Activated carbon particles were saturated in a solution of corrosive sublimate at 80 °C and then dried at 10 °C. The mercury content in the solution prepared in this way was 1.31%. Only the distribution of mercury was monitored in the experiments.

The experiments were conducted on a unit structured as shown in the diagram in Figure 3. The unit was a long horizontal retort made of quartz glass. Its length was more than twice the length of the electric resistance furnace heating it. There was a vertical pipe at the end opposite to the open end of the retort. It was lowered down, connecting the space of the retort with a condenser located below and connected, in turn, to the pumping system. The vertical pipe was equipped with a caisson for water-cooling.

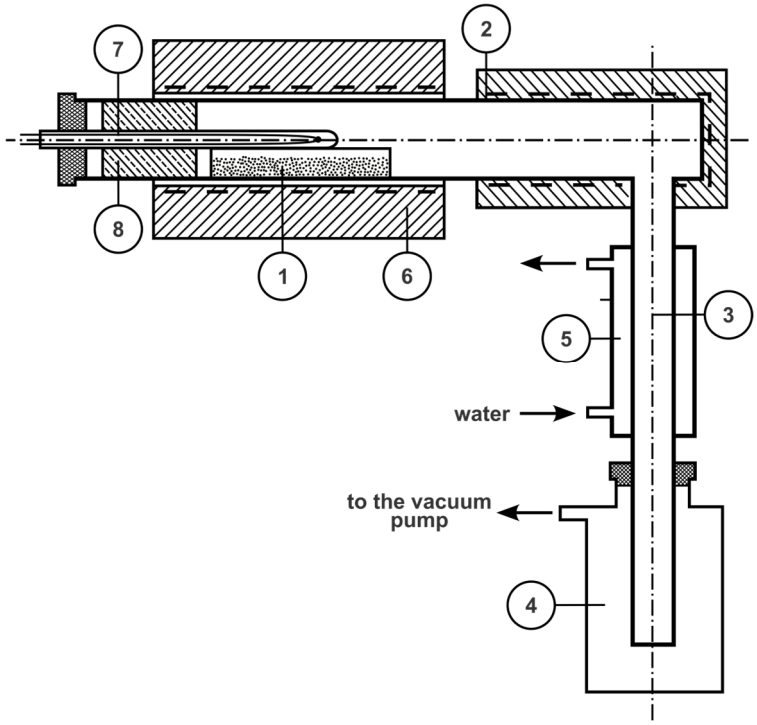

Figure 3. Schematic diagram of unit for demercurization of carbon sorbents: (1) container with sorbent; (2) retort; (3) pipe; (4) condensate collector; (5) caisson; (6) electric oven; (7) thermocouple; (8) heat-insulating screen.

The procedure was as follows. The electric furnace was moved to a part of the quartz retort closer to the vertical pipe and heated to the temperature specified for the experiment. A quartz container with a sample of carbon sorbent (~50 g) containing mercury chloride was placed in the retort. A thermocouple was placed in the open part of the unheated retort, close to the surface of the sorbent, and sealed in with a heat-insulating screen. After the gases were evacuated from the retort to the pressure specified by the technology, the electric furnace was moved onto the part of the retort where the low-pressure quartz

container with sorbent was located, such that the container was in the isothermal region of the electric furnace. The time at which the sorbent sample reached the set temperature was considered the beginning of the experiment. Mercury-containing vapors were discharged into a condenser to be transferred into the condensed phase. The non-condensable gas was evacuated from the condenser using an exhaust cleaner. After a certain exposure time, the electric furnace was shifted to its original state. The container with the sorbent was cooled under natural conditions; after the pressure in the retort was equalized with atmospheric pressure, the sorbent was removed from the retort and analyzed for its mercury content. The extraction of mercury into the vapor phase was calculated from its concentrations before and after the experiment. The condensate after each experiment was not analyzed for its mercury content due to the small amount of material.

Laboratory technological studies were performed in the temperature range of 300–800 °C at pressures of 0.13–91.99 kPa for 30 min with samples of coal sorbent weighing ~100 g. A pressure of 91.99 kPa (690 mm Hg) corresponds to atmospheric conditions in Almaty. The results of the experiments are shown in Table 4.

Table 4. Results of technological demercurization of coal sorbent.

Temperature, °C	Content in Residue and Extraction of Mercury at Pressure, kPa:							
	0.13		1.33		13.33		91.99	
	Content, %	Extraction, %	Content, %	Extraction, %	Content, %	Extraction, %	Content, %	Extraction, %
300	8×10^{-4}	99.94	1×10^{-3}	99.92	8×10^{-3}	99.40	8.7×10^{-2}	93.43
400	5×10^{-4}	99.96	6×10^{-4}	99.95	6×10^{-3}	99.55	5.0×10^{-2}	96.22
500	5×10^{-4}	99.96	6×10^{-4}	99.95	2×10^{-3}	99.85	1.6×10^{-2}	98.79
600	3×10^{-4}	99.98	5×10^{-4}	99.96	3.4×10^{-3}	99.74	3×10^{-3}	99.77
800	4×10^{-4}	99.97	3×10^{-4}	99.98	1.9×10^{-3}	99.97	2×10^{-3}	99.85

It became clear during the analysis of the experimental results that mercury in a vacuum almost completely (99.4–99.97%) entered the vapor–gas phase. A similar degree of mercury extraction from the coal sorbent was achieved at 600–800 °C at atmospheric pressure. The maximum permissible concentration of mercury in solid waste according to European standards is 10 mg/kg (1×10^{-3}%) [22]. This value exceeds the residual mercury contents in the experiments in vacuum at 0.13 and 1.33 kPa and is slightly lower that those at pressures of 13.33 and 91.99 kPa; the latter can be leveled out by increasing the heat treatment time (Table 5).

Table 5. Mercury content and its extraction with an increase in the thermal demercurization time.

Temperature, °C	Time, min	Content in Residue and Extraction of Mercury at Pressure, kPa:			
		13.33		91.99	
		Content, %	Extraction, %	Content, %	Extraction, %
600	30	3×10^{-3}	99.77	3×10^{-3}	99.77
600	60	1×10^{-3}	99.92	1×10^{-3}	99.92
600	120	3×10^{-4}	99.98	1×10^{-4}	99.98
800	30	3×10^{-3}	99.77	2×10^{-3}	99.85
800	60	3×10^{-4}	99.98	1×10^{-3}	99.92
800	120	1×10^{-4}	99.99	1×10^{-4}	99.99

It can be seen that an increase in the thermal demercurization time of the sorbent to 60 min or more in all cases makes it possible to obtain a material that meets the permissible standards for mercury contents in solid waste.

It should be borne in mind that the process at 600–800 °C can also be used to restore the sorption properties of activated carbon [23].

The positive results of these laboratory technological experiments served as the basis for extensive tests on equipment developed by the authors to process bulk materials without the forced movement of the bulk material in the reaction zone [24].

Technically, the solution is in the form of a shaft formed of pairs of plates with inclined slots, the angle of which (to the horizon) is greater than the angle of repose of the bulk material. There are gaps between the pairs of plates so that the material falling at the angle of repose does not "flow" over the upper edge of the plates below.

The unit (Figure 4) as a whole comprises a sublimator (1), heated by an electric furnace (2), inside of which there is a shaft of inclined surfaces (plates). At the first stage, the sublimator body is used as a heater to simplify the design. The sublimator space is connected by a heated steam line (4) to a water-cooled condenser (5). A water-cooled pipe (6) is coaxially mounted inside the condenser, and a fabric filter (7) is placed inside this pipe to capture fine condensate.

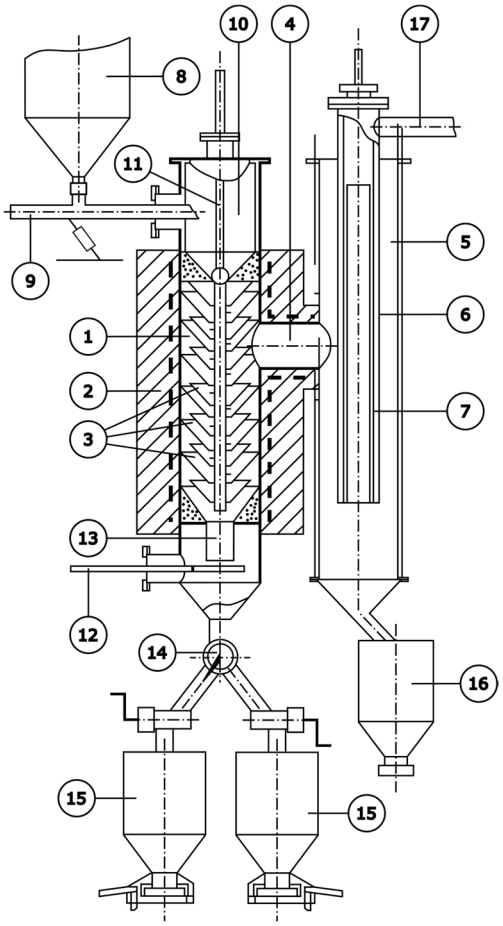

Figure 4. Scheme of vacuum sublimation electric furnace with rheological movement of dispersed materials: (1) sublimator; (2) electric furnace; (3) inclined surfaces; (4) steam line; (5) capacitor; (6) pipe; (7) fabric filter; (8) bunker for raw materials; (9) vibration loader; (10) intermediate capacity; (11) rod with conical valve; (12) vibration unloader; (13) mouth; (14) flow switch; (15) residue-receiving bins; (16) condensate-receiving hopper; (17) vacuum tube. Reprinted from Ref. [25].

The unit is equipped with a hopper (8) for feedstock to be fed by a vibrating feeder (9) into an intermediate container (10). The intermediate container is separated from the sublimator space by a movable hollow rod with a conical valve (11), where a thermocouple is located. A vibration unloader (12) is mounted below the shaft, assembled from plates. It forms a shutter that separates the unloading system consisting of a flow switch for the processed material (14) and receiving bins (15) for the residue from processing together

with the bulk material at the mouth (13). The condensate falling from the walls accumulates in a bunker (16). Gases are evacuated from the apparatus through a vacuum line (17).

The processed material is heated by means of radiation onto open areas of the surface formed by dispersed material due to natural pouring and facing an external heater. The surface area for steam release (open surface areas) is ~5×10^{-2} m^2. The sorbent processing time—the time of residence in the reaction space—is regulated through the action of a vibrating unloader, with its plane located with a gap relative to the mouth. The dispersed material at the mouth and on the unloader forms a seal with the sides, separating the sublimation volume from the bunker space, which prevents the penetration and condensation of the vapor phase in the latter. The vapor phase is sent for condensation into a cyclone-type condenser.

A carbon sorbent with a mercury content of 1.21% and a humidity of less than 1% was used in large-scale technological tests. The sorbent samples weighed 2.0–2.2 kg at 400, 600, and 800 °C in vacuum (1.33 kPa) and at atmospheric pressure (91.99 kPa). The conditions and results of the technological testing are given in Table 6.

Table 6. Conditions and results of technological tests for demercurization of coal sorbents with rheological movement of material.

No.	Temperature, °C	Pressure, kPa	Sorbent Output, %	Hg Content in Residue, %	Hg Recovery Rate, %
1	400	1.33	98.34	7.0×10^{-4}	99.94
2	400	91.99	96.77	5.0×10^{-2}	96.00
3	600	1.33	97.64	6.0×10^{-4}	99.95
4	600	91.99	96.31	3.0×10^{-3}	99.76
5	800	1.33	97.13	3.0×10^{-4}	99.98
6	800	91.99	95.43	3.0×10^{-3}	99.76

It follows from the above results that the extraction of mercury into the vapor–gas phase was not sufficiently complete (96.00%) at 400 °C and atmospheric pressure. Demercurization proceeded quite completely, with the transfer of 99.76% to the gas phase, when the temperature was increased to 600 °C or higher (800 °C).

When this process is industrially implemented, the pressure value in the technological process should be chosen based on economic feasibility, taking into account the energy consumption and the cost of vacuum pumping systems, as well as the limited amount of processed sorbent and the possibility of also restoring the sorbent's sorption capacity during heat treatment at high temperatures.

4. Determination of the Sorption Capacity of the Sorbent After Demercurization

A productive cyanide leaching solution from Altyntau Kokshetau JSC with a gold content of 2.564 mg/L was used to determine the sorption capacity of the carbon sorbent after thermal demercurization. The pH of the initial solution was 10.85 but was increased to 11.5 to reduce the degree of absorption of Cd, Pb, Zn, Ni, and Cr impurities. The gold sorption extraction experiment included a supply of 1.0 L of solution to each sample of sorbent. No preliminary treatment of the coals was performed in either variant of the comparative experiments, since a sample of fresh Norit coal, which had not previously been subjected to sorption/desorption processes and associated washings, was used to compare the efficiency of coal sorption after demercurization.

Two samples of coal were used in the experiment: one sample was a sorbent after thermal demercurization at 800 °C at a pressure of 1.33 kPa for 1 h; the second was a sample of fresh "Norit" activated carbon. Coal sorbent in an amount of 50 g was added to 1.0 l of gold-containing solution and mechanically stirred for 4 h at 45 °C. The solutions after sorption were analyzed for their residual gold concentrations, on the basis of which the efficiency of the process was calculated. The results of this gold sorption extraction are given in Table 7.

Table 7. Results of gold sorption extraction.

Carbon Sorbent	Au Content in Initial Solution, mg/L	Au Content in Solution After Sorption, mg/L	Au Recovery, %
After thermal demercurization	2.564	0.202	92.12
Original "Norit"	2.564	0.198	92.28

It was established based on the test results that the sorption properties of coal after thermal demercurization are not practically inferior to those of a sample of fresh activated carbon of the Norit brand: 92.28% Au was extracted during the sorption treatment of a gold-containing solution with Norit coal, whereas the extraction rate of the noble metal reached 92.12% on coal after thermal demercurization. Taking into account the fact that fresh coal not previously used in sorption or desorption processes was used as a comparative option, the difference in recovery of 0.16% is extremely insignificant. Thus, carbon sorbent after demercurization can be used to extract noble and non-ferrous metals.

5. Conclusions

It was established, through thermodynamic analyses using partial pressure diagrams, that calomel (Hg_2Cl_2) and mercuric chloride ($HgCl_2$) captured by sorbents from industrial waste gases decompose into vaporous mercury and gaseous chlorine at 400–800 °C, and at pressures from fore vacuum to atmospheric.

Laboratory and large-scale technological tests established the possibility of the demercurization of coal sorbents at 400–800 °C, both at atmospheric pressure and under rarefaction, to the maximum permissible concentration corresponding to European standards; that is, 10 mg/kg (1×10^{-3}%).

The suitability of the developed equipment for processing bulk materials without forced movement in a sublimator was confirmed by laboratory research data. As a result of technological research and extensive testing with the developed equipment at 400–800 °C and pressure in the range of 0.13–91.99 kPa, it was established that mercury in a vacuum under these conditions almost completely enters the vapor–gas phase (99.4–99.97%). A similar degree of mercury extraction from coal sorbent was achieved at 600–800 °C at atmospheric pressure. However, the residence time of the sorbent in the sublimator must be increased to 60 min or more to remove mercury at pressures of 13.33 and 91.99 kPa.

The determination of the sorption capacity of coal sorbent after thermal demercurization (800 °C and 0.13 Pa), in comparison with that of an unused sorbent of the Norit brand, showed a negligible difference between them.

In the industrial application of the thermal demercurization of coal sorbents, selecting the appropriate pressure value should be guided by economic considerations, including energy consumption and the cost of vacuum pumping systems. It is also important to account for the limited amount of sorbent being processed and the potential to integrate the regeneration of the sorption capacity during heat treatment at high temperatures.

Author Contributions: Conceptualization, V.V. and S.T.; methodology, V.V.; investigation, B.K., S.T. and D.M.; data curation, V.V., A.N., B.K. and X.L.; writing—original draft preparation, V.V., S.T., A.N., X.L. and Y.K.; writing—review and editing, S.T., A.N., X.L., Y.K. and D.M.; visualization, S.T., A.N. and X.L.; project administration, B.K. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant BR21882140).

Data Availability Statement: The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

Conflicts of Interest: The authors declare no conflicts of interest.

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