



Article

Preliminary Removal of Mercury from Depleted Coal Sorbents by Thermal Vacuum Method with Associated Extraction of Precious Metal Composite

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Abstract: This paper presents the results of laboratory studies for the distillation of mercury from depleted coal sorbents produced in gold recovery factories using CIP technology. The mercury content in these materials is more than 1%. The developed technology was tested in a large-scale laboratory on a pilot vacuum sublimation electric furnace with the rheological movement of dispersed material. The use of this equipment makes it possible to demercurize various materials with fairly high moisture (up to 20%). It eliminates the use of an additional technological operation—drying the material in a vacuum drying oven. It has been shown that a high degree of mercury distillation is achieved (more than 99.8%) at 350–400 °C in the reaction space and residual pressure in the system of less than 1.33 kPa, with residual mercury content in the material of less than 0.001% (10 mg/kg), which complies with the European environmental standards. Mercury-free coal sorbents are sent for combustion for the additional extraction of precious metal composites. The proposed vacuum technology is characterized by its environmental safety because the process is performed in sealed equipment, eliminating toxic emissions of mercury vapor into the atmosphere. The proposed vacuum technology equipment is characterized by reliability and ease of use.

Keywords: coal sorbent; mercury; vacuum; demercurization; rheology; technology; dispersed material; electric furnace; heating; precious metal composite



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1. Introduction

The approved gold reserves in the Republic of Kazakhstan as well as the volume of their production have led to the fact that Kazakhstan ranks third in these indicators. Only the Russian Federation and Uzbekistan are ahead in terms of mining and production amounts of the precious metal composite [1]. The existing gold deposits can be classified as so-called small (with reserves up to 25 tons) and medium (from 25 to 100 tons) deposits in the territory of the Republic. In total, there are currently about 293 gold deposits in Kazakhstan (including 38% complex, 60% gold deposits, and 2% placer deposits) [2]. It should be noted that gold deposits have been discovered in almost all regions of the Republic. However, the leading position is occupied by the fields of Eastern, Northern, and Central Kazakhstan [3–7].

Traditionally, most of the gold in Kazakhstan is mined from complex deposits. They contain complex ores in their chemical and phase composition. To date, many methods have been developed for the extraction of gold from relatively poor, finely disseminated ores, tailings, and other beneficiation products [8–16]. However, the basic process for the

extraction of gold from such raw materials is the cyanidation process [16–22]. This allows the selective release of gold and other noble metals into solution when it is treated with weak cyanide solutions (KCN, NaCN, and $\text{Ca}(\text{CN})_2$). The advantage of the cyanidation method is its significant economic efficiency due to the simplicity of the process intended to isolate anion complexes of noble metals. Furthermore, there is almost no corrosion of the equipment when the process is performed under natural conditions, and it eliminates the use of expensive acid-resistant equipment [23,24].

A gold-containing cyanide solution for the extraction of precious metal composites can be sent for sorption on anion exchangers or activated carbons [25–29] or for cementation using the example of zinc dust [30]. At the same time, the sorption process on activated carbons, so-called “carbon in pulp” (CIP) and “carbon in leaching” (CIL), has found wide applications in industry due to its efficiency and economic feasibility [31]. The process combines leaching with the sorption of metals on activated carbon with the subsequent elution of the metal. Economic indicators consist of the more complete recovery of gold from solution than the traditional Merrill Crowe process, with significantly lower capital and operating costs [32]. However, coal loses its sorption ability as a result of several sorption–desorption cycles. These depleted coal sorbents, containing a total of up to 0.1% noble metals, were sent for combustion, and the ash from combustion was sent for additional extraction of noble metals.

It should be noted that the sorption of other metals occurs in addition to that of noble metals, including metallic mercury and its compounds [33]. Due to the tightening of environmental requirements for non-ferrous metallurgy enterprises [34], as well as the fact that the Republic of Kazakhstan has joined the Rio Declaration on Environment and Development [35], the combustion of depleted coal sorbents without their preliminary purification is prohibited due to the contamination of the atmosphere by toxic elements, primarily metallic mercury and its compounds, which poses a significant danger to the environment and humans [36–45]. In this regard, depleted coal sorbents were sent to dumps; these are very rich materials that contain significant amounts of precious metal composites. First, it is necessary to remove mercury from them in the most environmentally safe way to solve the problem—how to use mercury-containing depleted coal sorbents in the processing.

In addition, the implementation of measures to prevent and eliminate mercury emissions into the atmosphere associated with the combustion of depleted coal sorbents benefited with precious metal composites from gold mining factories will reduce the number of casualties on-site and reduce material damage from negative consequences in the future.

Scientific research in the field of the vacuum pyroselection of various types of mineral and technogenic raw materials has been carried out at the Institute of Metallurgy and Beneficiation JSC (Republic of Kazakhstan) in the laboratory of vacuum processes for a number of years. The research results have shown that the most rational and environmentally friendly method of demercurization of various types of minerals and technogenic raw materials is thermal vacuum. When this process is performed in sealed equipment, it eliminates the release of toxic elements into the environment, which in turn determines the environmental safety of the method and helps reduce the amount of waste process gas, which significantly reduces the cost of their sanitary cleaning. In addition, vacuum pyroselection enables improved conditions for vapor condensation, a more rational selection of components, and safe working conditions for operating personnel [46].

A number of works by the authors [47,48] have been devoted to solving the problem of how to study the behavior of mercury and its compounds when they are heated in an electric furnace at a reduced pressure as well as technological solutions for the preliminary demercurization of depleted coal sorbents. Technological tests of the developed technology were performed on the existing VVU-1M vibration-vacuum installation working under a continuous operating principle [49]. The fundamental possibility to use thermal vacuum technology in practice and a continuously operating VVU-1M vibro-vacuum installation, which allows demercurization of up to 500 kg of depleted coal sorbents per day with a

material moisture content of no more than 9%, was shown as a result of the technological tests performed. At the same time, the removal of mercury from the material was more than 99%, and its residual content in the sorbent less than 0.001%. This fully complies with the European sanitary requirements [50]. However, the complexity of the design of the VVU-1M vibration-vacuum installation [51] and, consequently, the high requirements for the qualifications of operating personnel prompted the authors of the work to conduct research to develop and create equipment that is simpler in design.

2. Experimental Part

The materials under study were depleted coal sorbents (activated carbons) after desorption produced in the gold recovery plant of Altyntau Kokshetau JSC, which is the largest in Kazakhstan in the field of gold mining and processing. According to the sample certificate from Altyntau Kokshetau JSC, the following contents of elements were determined in the depleted activated carbons (wt. %): 0.961—Hg; 0.0835—As; 0.00093—Cu; 0.2491—Fe; 0.00041—Zn; 0.00068—Pb; 0.00099—Bi; 0.4644—SiO₂; 0.0744—Al₂O₃; 0.6618—CaO; 0.011—MgO; and 0.2797—S. The Au content was 0.06 g/t. The sample was dried under natural conditions.

The content of mercury and other basic elements in the depleted coal sorbents and products obtained after heat treatment of the material under study at reduced pressure was determined by a chemical method on an Optima 8300 DV “Perkin Elmer” atomic emission spectrometer (Waltham, MA, USA) and by an X-ray fluorescence method on the wave-dispersive combined spectrometer Axios “PANalytical” (PANalytical, Almelo, The Netherlands). The phase composition of the starting materials and their processing products was determined on a D8 Advance “BRUKER” X-ray diffractometer (Bruker, Karlsruhe, Germany) under Cu-K α radiation and on a JEOL JXA-8230 scanning electron microscope (JEOL Ltd., Tokyo, Japan). Standardized parameter measurement systems were used in the experimental studies.

The moisture content of the samples was determined experimentally by heating them in a vacuum drying oven at a pressure of 7.8 kPa and a temperature of 70 °C until a constant mass of the sample was established. The initial mass of the coal placed in the drying cabinet was 0.25 kg. The moisture of the carbon sorbent sample before drying was 32.12%, and that of the sample after drying was 0.7%. Photographs of the initial (wet) carbon sorbent and after drying (drying) are shown in Figure 1.



Figure 1. Photos of depleted carbon sorbent: (a)—initial coal sorbent (wet—before drying); (b)—dried sorbent (after drying).

The bulk density of dried coal (after calcination) was determined using a measuring cylinder, and it was 0.593 g/cm³ without shaking and 0.644 g/cm³ with shaking; the angle of repose of the dry coal was 33°.

Mercury in coal is mainly in metallic form; the content is still 1.06%, while the maximum permissible concentration of mercury for CIS countries in solid waste corresponds to a value of 2.1 mg/kg or 0.00021% [52,53]. It was semi-quantitatively determined that the gold content in the coal was 0.0036% (36 g/t), that of carbon was $\approx 95.36\%$, and that of sulfur was 0.53%.

Using X-ray fluorescence analysis, the main elements in the carbon sorbent sample were determined, and they are presented in Table 1. The determined total amount of impurities was 15.995 wt.%. The carbon content was 84.005 wt.%.

Table 1. Composition of impurities determined by the X-ray fluorescence analysis of a representative sample of coal sorbent.

Components	Na	Mg	Al	Si	P	S	Cl	K	Ca	O
Content, wt.%	2.183	0.248	0.397	0.923	0.074	1.064	0.562	0.115	2.354	5.475
Components	Fe	Ni	Cu	Zn	As	Hg	Ag	Au	Pb	Bi
Content, wt.%	0.387	0.031	0.03	0.005	0.066	1.103	0.205	0.023	0.699	0.051

Chemical analysis established that the mercury content in coal after drying was 0.036% mercury (360 mg/kg) and the mercury content in coal before drying was 1.06%. The sulfur content of the coal was 1.064%. X-ray phase analysis found that mercury was mainly in metallic form.

Additionally, express analyses were carried out using a mercury analyzer RA-915M (Lumex Ltd., St. Petersburg, Russia). The original optical–electronic circuit of the analyzer provides an ultra-low detection limit of mercury in direct measurement mode (without pre-concentration), a high selectivity of analysis, and a wide dynamic range of measurements.

To confirm the previously obtained data [47–49] and determine the optimal conditions for the removal of mercury from depleted coal sorbents, we designed a large-scale laboratory-size vacuum unit, as shown in the diagram in Figure 2. A quartz container (11) with a boat (3) where the depleted coal sorbent was poured was placed inside the quartz reactor (2). The quartz reactor was sealed with rubber seals (4), in front of which heat-insulating screens (5) were installed. The temperature in the reaction space was measured using a chromel–alumel thermocouple located in a cover (6) directly above the material under study. The quartz reactor was connected through rubber seals (8) to a water-cooled “refrigerator” (7) designed for the condensation of mercury vapor. Condensed metallic mercury was collected in a special tank (9). A Bunsen flask connected to a vacuum pump was used as a mercury collector. The electric furnace was equipped with movable slides on a platform (10), and during the experiment, it was moved onto a permanently installed quartz reactor. This design of a large-scale laboratory-size unit eliminated the contamination of the test material with metallic mercury after the experiment since the condensation of mercury vapor during the heat treatment of the sorbent is not excluded in the “cold” parts of the quartz reactor, and mercury may enter the carbon sorbent with a mechanical impact on the reactor during loading and unloading operations.

The dependence of the metallic mercury removal degree on the heat treatment temperature and its duration was determined by the following method. A sample of the test material (depleted coal sorbent) weighing 200–250 g was placed in an alundum boat placed inside a quartz tube (container). The quartz container was placed inside a quartz reactor. The quartz container served as a protective screen during the unloading operation, excluding the possible ingress (contamination) of mercury condensed in the cold parts of the retort into the boat with the coal sorbent under study. The quartz reactor was sealed and connected to a mercury vapor condensation system. Air was pumped out of the reactor using a 2NVR-5DM rotary vane pump (Vacuummash, Kazan, Russia) until the specified residual pressure was established in the system. The residual pressure in the system was measured with the use of an M110 aneroid barometer with an accuracy class of ± 0.5 . When the set temperature of the experiment was reached in the electric furnace, an electric furnace

mounted on a special movable skid was moved onto the quartz reactor. The start time of the experiment was fixed from the moment the required temperature was established above the sample of the material being studied.

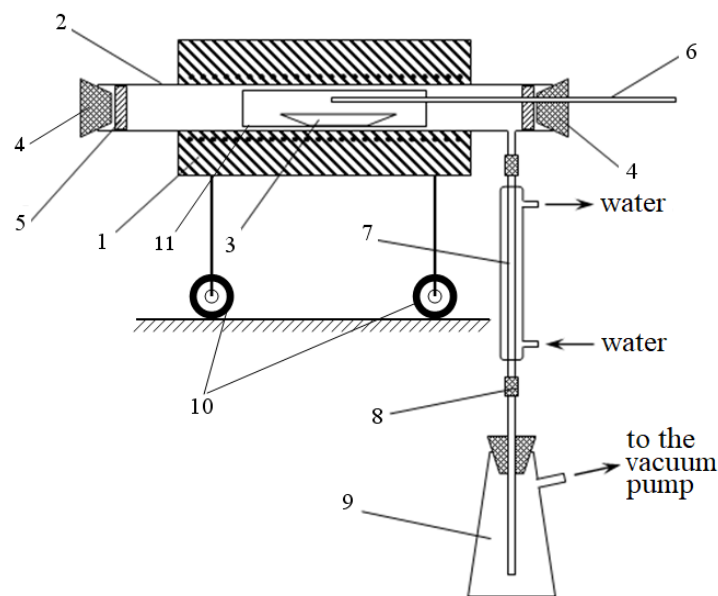


Figure 2. Scheme of a large-scale laboratory-size unit: (1) tubular electric resistance furnace, (2) quartz reactor, (3) boat with a weighed amount of material up to 250 g, (4) rubber tap (plug), (5) heat-insulating screen, (6) case with XA thermocouple, (7) water-cooled condenser (refrigerator), (8) rubber seal, (9) condensate collector, (10) movable slides on the platform, (11) quartz container.

After the end of the experiment, the electric furnace, on a skid, was moved away from the quartz reactor, and the latter was cooled under natural conditions to room temperature. The pressure in the system was balanced, the reactor was disassembled, and the depleted coal sorbent after heat treatment was analyzed by chemical and X-ray fluorescence analysis. The degree of mercury distillation was calculated on the basis of the results of analyses of the mercury content in the starting material and the residue from heat treatment.

Laboratory tests were performed with waste coal sorbents from the gold recovery plant of Altyntau Kokshetau LLP pre-dried in a vacuum drying oven at 60 °C and a pressure of 39.9 kPa to constant weight. The mercury content in the starting material was 1.06%.

Laboratory research was performed in the temperature range of 250–450 °C with system pressures of 0.13, 0.67, 1.33, 2.67, and 91.98 kPa for 20 min with 200–250 g samples. The results of laboratory tests are shown in Table 2.

Table 2. Results of laboratory tests on the distillation of mercury from depleted coal sorbents (sample weight—200–250 g).

$t, ^\circ\text{C}$	Time, τ , min	Pressure, kPa									
		0.13		0.67		1.33		2.67		91.98	
		Hg, %	Extraction, %	Hg, %	Extraction, %	Hg, %	Extraction, %	Hg, %	Extraction, %	Hg, %	Extraction, %
250	20	0.0065	99.387	0.0014	99.867	0.0021	99.801	0.0033	99.684	0.1678	84.173
300	20	0.0008	99.928	0.0008	99.918	0.0009	99.913	0.0011	99.901	0.0873	91.765
350	20	0.0006	99.944	0.0007	99.934	0.0008	99.923	0.0009	99.918	0.0595	94.383
400	20	0.0005	99.954	0.0006	99.944	0.0006	99.939	0.0007	99.934	0.0496	95.321
450	20	0.0002	99.979	0.0003	99.974	0.0004	99.964	0.0005	99.954	0.0349	96.709

The results of the laboratory studies given in the table show that it is possible to remove more than 99% of the mercury, resulting in heat treatment residues containing mercury within the European MPC standards (less than 10 mg/kg) [54] when depleted mercury-containing coal sorbents from gold mining factories are heat-treated using a vacuum, even at very low temperatures of material processing (300 °C and above).

Previously conducted pilot tests with the VVU-1M vibration-vacuum unit [49] for the demercurization of depleted coal sorbents from gold mining factories showed the high efficiency of the thermal vacuum technology for mercury removal. The extraction of mercury into condensate at a material processing temperature of 350–400 °C and a residual pressure in the system of 1.33–4.0 kPa from the vibro-fluidized layer was 99.95–99.98%. Because the design of the VVU-1M vibration-vacuum unit is quite complex from a technical point of view and there are technological limitations on the use of dispersed material with a moisture content of more than 7–9%, the authors of the work developed a design for a sublimation oven with the rheological movement of dispersed material [55–58] in which large-scale laboratory-size tests were performed to distill mercury from depleted coal sorbents. The unit diagram is shown in Figure 3.

The vacuum electric furnace consists of a vertically located reactor (1) and a capacitor (2), which are connected to each other by a steam line (3). A column of inclined surfaces (4) is installed in the reactor. There is a receiving hopper (5) hermetically connected to a vibrating feeder (6) and a loading unit (7) in the upper part of the reactor, above the column of inclined surfaces. A gate (8) and a rod with paddles (9) are installed in the receiving hopper. The rod (10) is removed from the reactor with the possibility of rotation and vertical movement. The column of inclined surfaces rests on a receiving cone (11) equipped with a pipe (12) facing downward. The lower section of the pipe (12) is made with a gap between it and the plane of the vibrating loader (13), equipped with sides (14). There is an unloading unit (15) for the processed material under the plane of the vibration unloader. The receiving cone rests on a heat-insulating partition (17). The reactor is equipped with an electric resistance heater (17) and thermal insulation (18). The condenser (2) is connected to the gas evacuation system via a vacuum line (19).

This design of a vacuum sublimation furnace with the rheological movement of dispersed material makes it possible to process material with a moisture content of more than 20% [59], in contrast to the VVU-1M vibration-vacuum unit.

A design in the form of vertical water-cooled pipes connected to each other in series is used as a mercury vapor condenser. In this case, the first pipe at the exit of the furnace space is air-cooled to prevent the formation of “mercury fog” due to the sudden cooling of the gas phase.

The methodology used to conduct large-scale laboratory-size tests was as follows. The original depleted coal sorbent was loaded into the loading unit and subjected to degassing for 5–8 min. Residual gases inside the reactor were evacuated in parallel through a vacuum line. The reactor was heated to the temperature specified by the technological parameters. The temperature was measured using an XA-type thermocouple inserted through a cover into the reaction space in the isothermal zone of the furnace. Then, the material was fed into the receiving hopper when directed vibrations were transmitted to the vibrating feeder. When the receiving hopper was filled, the shutter was raised using a rod, and the bulk material filled the quadrangular shaft formed by the inclined surfaces of the plates. The coal sorbent, filling the shaft and reaching the lower cut of the pipe, formed a shutter with the plane of the vibration unloader, separating the volume of the reactor from the volume of the unloading unit of the processed material. When the plane of the vibration unloader communicated with the sides of horizontal vibrations, the processed material in the form of a coal sorbent was poured over the sides of the plane of the vibration unloader and poured into the unloading unit of the processed material.

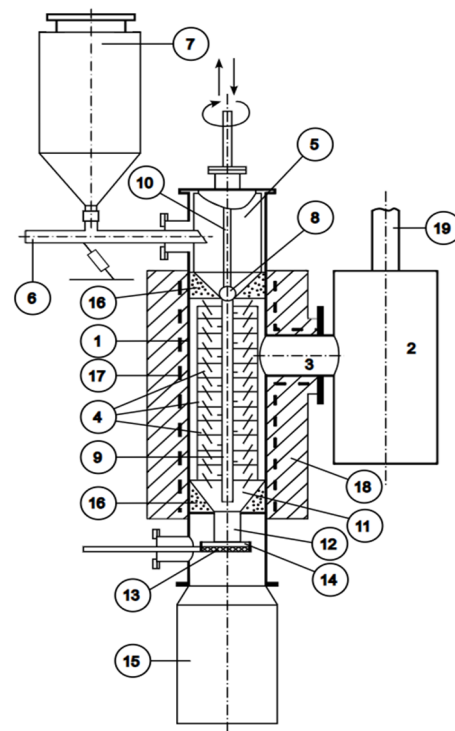


Figure 3. Diagram of a vacuum sublimator (oven) with the rheological movement of dispersed material: (1) furnace reactor, (2) condenser, (3) steam line, (4) column of inclined surfaces, (5) receiving hopper, (6) vibrating feeder, (7) loading unit hopper, (8) shutter, (9) rod with paddles, (10) rod, (11) receiving cone, (12) pipe, (13) vibration unloader, (14) sides of the vibration unloader, (15) residue-unloading bunker, (16) heat-insulating partition, (17) heater, (18) thermal insulation, (19) vacuum line.

Continuous rheological movement through the high-temperature zone of the reactor was ensured due to the physical and mechanical properties of the material, even with high moisture (more than 20%), when the gate was open and the vibration feeder and vibration unloader were operating. The volatile components of the material (in particular, mercury and its compounds) sublimated into the intergranular space of the carbon sorbent and were brought to the surface of the material. Next, pairs of volatile components from the surface of the material were removed through the free space between the reactor and the quadrangular shaft formed by the paired plates. The productivity of the vacuum sublimation oven was determined experimentally, and it amounted to 230 kg per day during technological testing.

During the technological tests, the same depleted coal sorbent without drying with a mercury content of 1.06% was used as the test material as in the laboratory studies. The starting material was dried under natural conditions, and its moisture was 19.7%. Tests were performed with samples weighing 2.0 kg to 2.2 kg at different temperatures in the reaction space of the sublimation oven and different residual pressures in the system. The conditions and results of the large-scale tests are given in Table 3. The material balance of the mercury distribution among the products of vacuum thermal treatment of the depleted coal sorbents is given in Table 4.

Table 3. Conditions and results of large-scale tests with depleted coal sorbents in a sublimation oven with the rheological movement of material.

No.	T, °C	P, kPa	Material Yield, %	Hg Content in the Residue		Degree of Hg Distillation, %
				%	g	
1	350	0.4	78.5	0.0008	0.0126	99.926
2	350	1.33	78.55	0.0012	0.0189	99.889
3	350	3.32	78.56	0.0017	0.0287	99.843
4	400	0.4	78.43	0.0003	0.0049	99.973
5	400	1.33	75.5	0.0005	0.0078	99.954
6	400	3.32	75.54	0.0011	0.0191	99.898

Table 4. Material balance of Hg distribution among the products of vacuum thermal treatment of depleted coal sorbent in a sublimation electric furnace with the rheological movement of material.

Material	Quantity, kg	Dry Weight, kg	Yield, %	Hg Content		Hg Distribution, %
				%	g	
Sorbent of the first batch	2.0	1.606	100	1.06	17.024	100
Sorbent of the second batch	2.0	1.606	100	1.06	17.024	100
Sorbent of the third batch	2.15	1.726	100	1.06	18.296	100
Sorbent of the fourth batch	2.1	1.686	100	1.06	17.872	100
Sorbent of the fifth batch	2.0	1.606	100	1.06	17.024	100
Sorbent of the sixth batch	2.2	1.767	100	1.06	18.73	100
Total:	12.45	9.997	100	1.06	105.97	100
Residue from the first batch	1.57	1.57	78.5	0.0008	0.0126	0.074
Residue from the second batch	1.571	1.571	78.55	0.0012	0.0189	0.111
Residue from the third batch	1.689	1.689	78.56	0.0017	0.0287	0.157
Residue from the fourth batch	1.647	1.647	78.43	0.0003	0.0049	0.027
Residue from the fifth batch	1.57	1.57	75.5	0.0005	0.0078	0.046
Residue from the sixth batch	1.728	1.728	75.54	0.0011	0.0191	0.102
Condensate (mortar)	2.557	—	20.54	0.505	103.727	97.883
Total received	12.332	9.775	99.05	—	103.829	97.98
Residual error	−0.118	−0.222	−0.95	—	−2.141	−2.02

3. Conclusions

An environmentally safe thermal vacuum method and a vacuum unit with the rheological movement of dispersed material inside the reaction space for the demercurization of depleted coal sorbents with high moisture from the CIP process are proposed as a result of this study. The mercury removal was more than 99.8% with a residual mercury content in the material of less than 0.01 (10 mg/kg) at material processing temperatures of 350–400 °C and a residual pressure in the system of less than 1.33 kPa. This fully meets the European standards.

On an industrial scale, the use of the developed technology and equipment will significantly reduce the emission of toxic mercury into the atmosphere with the associated extraction of precious metal composites, thereby improving the environmental situation around gold mining enterprises using the CIP process in their technological cycle.

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