



Supercritical CO₂ extraction device



1, Brief introduction

Supercritical extraction technology is the latest discipline emerging in modern industrial separation, and it is one of the internationally emerging advanced separation process.

The so-called supercritical fluid means that the thermodynamic state is above the critical point (P_c , T_c). The critical point is the state point where the gas-liquid interface has just disappeared. Supercritical fluid has a very unique physicochemical properties, its density is close to liquid, viscosity is close to gas, and the diffusion coefficient is large, the viscosity is small.

The characteristics such as large dielectric constant make it a better separation effect and a good solvent.

Supercritical extraction is under high pressure, at a suitable temperature, the solvent is contacted with the extract in the extraction kettle, the solute diffuses into the solvent, and then in the separator change the operating conditions to precipitate dissolved substances to achieve the purpose of separation.



Since the supercritical device has chosen CO₂ medium as the supercritical extraction agent, it has the following characteristics:

1. Wide operating range, easy to adjust.
2. With good selectivity, the required components can be extracted in a targeted manner by controlling the pressure and temperature.
3. The operating temperature is low, and the extraction is carried out near room temperature, which is particularly suitable for heat-sensitive components.

During the extraction process, the possibility of oxidation in the presence of oxygen and light reaction is excluded, and the extract can maintain its natural flavor.

4. One step from extraction to separation is completed, and the CO₂ after extraction does not remain on the extract.
5. CO₂ is non-toxic, odorless, non-flammable, inexpensive and easily available, and can be recycled.
6. Fast extraction speed.

2, Main technical parameters of the device

1. Design pressure: 50MPa;
2. Design temperature: normal temperature ~ 85 °C;
3. Extraction kettle: 0.5L, 50MPa;
4. Separation kettle: 0.3L, 30MPa, 2 sets;
5. CO₂ flow: 0 ~ 50L / h adjustable;
6. Power supply: 380V 50Hz;
7. Instantaneous maximum power: 16KW.

3, The main structure of the device

HB121-50-0.5 supercritical CO₂ extraction device consists of the following parts:

- (1) Extraction system: including extraction kettle, heat exchanger, heating bath, temperature control, safety protection, pressure measurement, etc.
- (2) Separation system: including separation kettle, heat exchanger, heating bath, temperature control, safety protection, pressure measurement, etc.
- (3) Pressurized system: including liquid CO₂ pump, cold water circulation, safety protection, electrical contact protection, pressure measurement, etc.
- (4) Voltage stabilization system: including filters, back pressure valves, etc.
- (5) Liquid CO₂ generation system: including refrigeration unit, liquid CO₂ storage tank, purifier (gas-liquid separation Device), pressure testing, etc.
- (6) Entraining agent system: including entraining agent pump, entraining agent addition tank, entraining agent flow meter
- (7) CO₂ circulation system: mixer, liquid CO₂ flow meter, etc.

4, Operation instruction

Preparations before starting up

1. First check whether the power supply, three-phase four-wire are intact. (AC380V / 50Hz).
2. Whether the cooling water source of the refrigerating device is unblocked (relative to the water-cooled unit), the cold box is 30% ethylene glycol+ 70% aqueous solution. The stirring pump in the cold box is used for stirring in the cold box and CO₂ pump head cooling.
3. The pressure of the CO₂ gas cylinder is guaranteed at a pressure of 4 to 6 MPa, and the food grade purity is 99.9%.
4. Check whether the pipe joint and the connection parts are firm.
5. Add purified water and deionized ionized water (distilled water) to each hot box.

Remember: Check the water level every time before starting the machine.

6. According to the needs of the experiment, the extracted raw materials are loaded into the barrel, and the raw materials should not be too full (according to the product process), max 2 to 3 cm away from the filter.
7. Put the barrel into the extraction kettle, install the barrel O-ring and put in the vent ring, cover the pressure ring and the plug.
8. If the liquid material is extracted or the entrainer needs to be added, put the liquid material into the carrying agent tank, and the carrying agent can be used pump into the extraction kettle.

Start-up operation sequence

1. Send air switch first, then start power (green) button.

2. Turn on the refrigeration switch and adjust the temperature controller of the cold box to about 5 °C (automatically stop and open, ready set up). The chiller has its own circulation to cool the CO₂ storage tank water and the CO₂ pump head.
3. At the same time as starting cooling, close valve 1, valve 19 and all other vent valves, open valve 2, and make CO₂ in the gas cylinder enters the purifier, cold coil and storage tank, and the storage tank is equipped with a pressure gauge to display the real-time storage tank Pressure value.
4. To start heating, first turn on the heating switches of extraction kettle, separation I and separation II, and turn on their respective temperature controllers. Adjust to the desired set temperature.
5. Open valves 19, 20, 22 (valve 22 is normally open and used for maintenance), and close the outlet valve of the CO₂ pump (open valve 25 when the CO₂ main pump is turned on, wait until valve 25 emits liquid CO₂, then close valve 25), open the panel Valve 3 (other valves on the panel are closed), CO₂ is heated by the mixer and the coil in the hot box, meanwhile the extraction kettle slowly opens the extraction kettle vent valve 4 and slowly releases the air remaining at the upper end of the extraction kettle. Close the vent valve 4 after emptying.
6. When the temperature of the chiller drops to 5 ~ 8 °C, and the temperature of the extraction kettle, separation I and separation II reach the set temperature, perform the following operations.

Pressure increase: first set the electrode point pressure gauge to the protection pressure overpressure and automatically stop the pump (the factory protection pressure setting

Set to 35MPa), start the green button of the main pump, and then manually press the green touch switch RUN in the frequency conversion digital operator,

If the flow rate is too small, press the touch switch ▲ manually, the pump speed will increase until the flow rate reaches the required release;

If the flow rate is too large, you can manually press the touch switch ▼, the pump speed decreases until the flow rate drops to the required release (digital

For a detailed description of the keys of the operator, please refer to the "Inverter User Manual"). When the pressure is close to the set pressure time (about 5MPa in advance), start to open the throttle valve at the back of the extraction kettle.

Adjust based on different flow directions:

① Extraction kettle → separation I → separation II → circuit

Open the valves 1, 5, 8, 11, 15, 18, CO₂ enters the separation I, separation II, loop cycle from the extraction kettle ring. Adjust the opening size of the back pressure valve 6, control the pressure of the extraction kettle, adjust the valve 11 to control the separation pressure, adjust valve 15 to control separation II pressure.

② Extraction kettle → separation I → separation II → rectification column → loop

Open the valves 1, 5, 8, 11, 12, 13, 15, 16, CO₂ from the extraction tank into separation I, separation II, 9/13 HB121-50-0.5 supercritical CO₂ extraction device instruction manual distillation column, loop circulation.

Adjust the opening size of the back pressure valve 6, control the pressure of the extraction kettle, and adjust the valve 11 Control the pressure of separation I and adjust the valve 15 Control the pressure of separation II and adjust the valve 16 Control the pressure of the distillation column force.

③ Extraction kettle → rectification column → separation I → separation II → loop

Open the valves 1, 5, 7, 9, 11, 15, 18, CO₂ enters the distillation column from the extraction kettle, separation I, separation II. Loop circulation. Adjust the opening size of the back pressure valve 6, control the pressure of the extraction kettle, adjust the valve 11 control separate I pressure, adjust valve 15 to control Separate II pressure, adjust valve 16 to control rectification column pressure.

Note: After the above three process settings are completed, CO₂ enters the automatic cycle extraction, which can be slowed down during the extraction process. Slowly open the discharge valve 28, 29, 30, 31, 32, 33 to take out the material under pressure, or take it after the extraction is completed material. 7. When stopping the pump halfway, just press the STOP button on the digital operator.

8. If you need to use the entrainer during the extraction process, first put the entrainer into the entrainer tank and open the valve 21. Turn on the power of the entrainer pump, and manually adjust the flow rate of the entrainer according to the process requirements.

9. After the extraction is completed, turn off the chiller, high-pressure pump and heating cycle switch, and then turn off the main power switch. Close valve 3, first fully open regulating valves 11, 15, 16 and then fully open regulating valve 6, put the pressure in the extraction kettle, separation kettle and rectification column into the storage tank, wait for the pressure in the extraction kettle and the back to balance.

After that, close valves 1, 2 and slowly open the discharge valves 27, 28, 29, 30, 31, 32, 33 to take the material, Open the vent valve 4, 10, 14, 17 to release the system pressure until there is no more material. Air speed moderate, not too fast, otherwise the material will be washed out of the barrel or frozen.

After there is no pressure, open the extraction kettle lid, remove the barrel, and clean the residual material in the barrel. Whole extraction The process ends.

10. After the pump outlet pressure is set, the pump will automatically stop when the working pressure of the pump is overpressure.

5, Trouble analysis and elimination

1. This device is a high-pressure mobile device. Those who are not familiar with the flow of this system should not operate it.

If you leave the post, you should immediately shut down the main power supply for inspection if an abnormal situation occurs.

2. Refrigeration system

① Before starting the machine and during normal operation, check whether the oil level of the compressor is normal. In general, there is no shortage of oil, if too low to add special oil 25 # (The new model is 40 #).

② When the chiller is running normally, the high-pressure meter indicates 1.5-2Mpa in summer and 1-1.5Mpa in winter Protection 2.2MPa), low pressure table is 0.2-0.3Mpa. If the cooling effect is too low, R22 Fluoride can be added appropriately. (Can be added from the low pressure valve port)

③ Before the cooler is turned on, the high and low meters are under pressure, but after starting, the low pressure

meter is 0, and the cold machine is frequently started, stop, the possible reason is: the filter, expansion valve or solenoid valve is clogged.

The processing steps are as follows:

- A. Close the storage tank supply valve, start the chiller switch, recycle freon, and close when the low pressure gauge drops to minus cold machine.
- B. Open the filter and clean the filter screen at the lower port (filter core) of the expansion valve.
- C. After cleaning, after installing the filter and expansion valve, close the high-pressure valve and open the vent joint for cooling pump down until the low pressure gauge is less than 0 and there is no air in the high pressure outlet.
- D. Tighten the high-pressure vent connector (cap), and then open the high-pressure valve and the liquid supply valve.

Note: The above situation is abnormal, if it appears, it is best to ask a professional to solve it.

3. CO2 fluid system

- ① If the CO2 pump is running, check whether the pump head has cooling circulating water (supplied in the cold box).
- ② While starting to pressurize, wait for the temperature of the cooler to reach the requirements, and at the same time open the vent valve at the pump outlet 25 vent and close valve 25 as soon as valve 25 begins to emit CO2.
- ③ Due to CO2 or water in the material, ice blocking of the high-pressure coil in the cold box may occur. The fault phenomenon is the pressure of the storage tank display is low (below the outlet pressure of the CO2 cylinder) and cannot be circulated.

Solution:

- A. Frequently drain water from valve 23 at the bottom of the purifier and waterproof from valve 24 at the bottom of the storage tank.
- B. If there is ice blockage, let the temperature of the storage tank rise to room temperature naturally. Use normal temperature CO2 or nitrogen to import from the storage tank.

Close the valve at one end, open the valve 2, purge to the other end, close the valve 19 and open the valve 24 until the cold dry the moisture in the freezing coil.

4. Heating temperature control system

- ① When starting the machine, check whether the three-phase four-wire power supply is correct. Prohibit the lack of phase operation.
- ② Check the water level of each heated water tank every time (every shift) (very important). Insufficient should be filled in time charging (due to high temperature evaporation), otherwise it will burn the heating tube, and at the same time, check whether the pump motor is running to prevent water dirt stuck to the shaft and burned the motor.
- ③ If the measured temperature is much higher than the set temperature, or the water in the water bath is boiled, the possible cause is solid state relay is broken down and does not play a control role. At this time, only the corresponding solid state relay needs to be replaced.

5. The pump should be replaced with lubricating oil within a certain period of time.

6. Maintenance of heating water tank:

- ① If it is not used for a long time, please discharge the water to prevent the insulation jacket and corrosion cycle from freezing in winter water pump.

② Generally check the water level of the water tank before starting, if it is not enough, it should be replenished in time (due to temperature evaporation), otherwise it will burn heating tube is damaged.

At the same time, check whether the circulating water pump and the rotating shaft rotate flexibly to prevent the scale from sticking to the rotating shaft to burn out the electricity.

7. When the temperature changes greatly (room temperature exceeds 30 °C), the CO₂ storage tank gas should be vented if the equipment is not used, or reduce the press to below 3MPa. To prevent gas from expanding at high temperature and increasing pressure.

6, Maintenance

Usually pay attention to the cleaning of the equipment, clean the reactor and pipeline in time after each use, you can use alcohol to do circulated cleaning.

7.Matters needs attention

1. This device is a high-pressure mobile device. Those who are not familiar with the flow of this system shall not operate it.

If you leave the post, you should immediately shut down the main power supply for inspection if an abnormal situation occurs.

2. Pay attention to the pressure and temperature of the reactor during the experiment. If there is an emergency, turn off the main power supply and turn on the vent valve.

3. During the experiment, the reaction kettle is heated all the time. Note that during operation, water must not touch the heating area.

4. At no time shall conductive fluid flow to the place of electrical components.