IMPROVEMENTS OF THE SAFETY PERFORMANCE OF HIGH PRESSURE EXTRACTION VESSELS

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Preferentially smaller extraction vessels are equipped with cartridges to minimize down time between the extraction cycles. Such cartridges are simply made of a proper tube with reinforced and supported sieve plates at the bottom and the top to prevent that the material is swept along. A sealing directs the solvent through the basket. In the very case that both sieve plates are blocked by any means (fine dust, hardened extract,..) the cartridge can be destroyed because of the forces derived from the differential pressure unless no correctives are applied (shut down, bypass,..). Even more serious damages can occur in case the blocking crops up at the depressurization of the extraction vessel: Pressurized gas to an unknown level may remain in the cartridge although the pressure in the vessel is indicated to be Zero. The spontaneous release of the case after the opening of the lid of the vessel may cause damages to the machinery and – as a worst case - injuries to the operators. The paper presents remedies.

1. State-of-the-art procedures

Since several decades extraction using CO_2 as agent is applied in large scale to coffee (decaffeination) and hops (recovery of the extract used in the brewing business for preserving and bittering of the beer). Moreover there is a multitude of other applications.

The separation of components from e. g. hops by means of a solvent under high pressure happens step by step: The container is filled with the material to be processed, and then the lid (mainly a quick closure for the sake of saving time) is closed. After the required pressure has been achieved, the solvent is passed several times through the solid material until the desired result has been realised. The success of extraction can be determined by the progress of extraction (by weighing the extract for example) or by weighing and/or analysing afterwards the remainders.

Within the cycle, for obtaining the extract, the solvent is separated from the dissolved material. While this separation is made with liquid solvents by means of evaporation at increased temperatures, the separation from compressed gases is performed by decreasing the density by reducing the pressure and/or by increasing the temperature. The decrease of density by means of pressure reduction takes place directly in the valve. The following separator vessel serves for collecting and calming the phases which separate and for removing the individual phases. The cleaned solvent is discharged at the top, transported to the pump and from there returned to the extractor vessel. It might be necessary to repeat this procedure

several times depending on the extract quantity, the solubility of the extract in the solvent and the kind of transition from the solid matter to the solvent.

While in case of liquid solvents, a change of temperature is the only possibility of adapting the dissolving characteristics during dissolving and separating, these characteristics can be fixed in the case of compressed gases by varying the density. By means of temperature the diffusion out of the solid substance mixture, i.e. the kinetics of the extraction, can be established.

Referring to the above, an extraction unit will consist of two main groups, i.e. the extraction step and the separation step. The other devices are auxiliary equipment for the addition and elimination of heat and for transport and condensation. During the extraction step the soluble material is extracted with high pressure from the solid substance matrix and transported away by the solvent; during the separation step the dissolved material is removed from the solvent by decreasing the solubilising power of the solvent. Consequently a cleaning (regeneration) of the dissolved material is achieved.

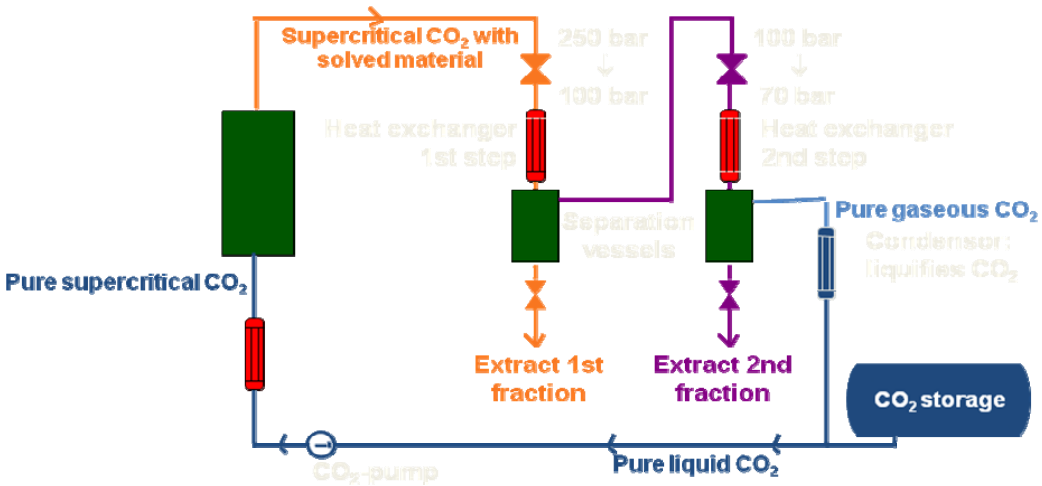

Figure 1: Flow sheet of a CO_2-extraction plant

High pressure vessels for extraction purposes are usually equipped with quick closures to minimize down time between extraction processing. Commonly used are cartridges (baskets) in the high pressure vessel that contain the material to be processed at least in smaller units (< 1 m^3). Such cartridges exhibit manifold advantages:

- The cartridges can be filled and unloaded outside of the pressure vessel thus saving time (economic advantage).
- In case of e. g. sensitive dust (hazard of explosion) the filling and unloading can be transferred to a place with corresponding requisites (safety issue).
- Similar arguments are valid in case the materials to be handled are sensitive to pollutions in the atmosphere (hygienic issue).

Prior to the opening of the quick closure the internal pressure must have been released completely. Customarily additional checks need to be performed to verify the absence of pressure: Pressure gauges have to prove the balance of internal pressure and atmospheric pressure, pressure controlled switches open interlocks at a set level, finally a probe valve is opened and controlled by an operator.

The measures described usually demonstrate also the absence of pressure in the internal cartridge. At least if there are free openings between cartridge and volume of the vessel.

2. Potential malfunction

Fine powders together with sticking, gluing constituents may block the mesh of the filter elements at both sides of the cartridge. Also waxy, greasy constituents that become solid at lower temperatures, e. g. at the release of the pressure, may cause similarly a blocking.

Under these circumstances the real pressure in the cartridge may no longer be equal to the pressure in the pressure vessel. The standard opening procedure will not detect any abnormalities and will start the operation. The lid can be opened and removed. Two cases can be expected as extremes:

- The cartridge is stable enough to manage the internal pressure. The pressure escapes e. g. when the lid of the cartridge is opened (preferably under controlled conditions).
- At the weakest point the cartridge will break. In the worst case the bottom sieve is rupturing. The cartridge will be ejected like a bullet.

3. Answer to the problem

How to avoid a built up of internal pressure when sieve plates are blocked?

Bursting discs or safety relieve valves at the cartridge would be first choice. Unfortunately such devices may be applicable in the controlled situation of a laboratory but are not practicable. Devices like mentioned can be blocked. Moreover the burst pressure is depending on temperature, shocks and pulsation of the pressure. The rating must be adjusted to the design pressure of the cartridge that means rather low. Then pulsations at the operation will already cause bursting.

Most effective way will be to measure both the pressure in the volume of the vessel and the pressure in the cartridge. At ordinary operation conditions there will be obviously no differential pressure. In case differential pressure is displayed special requisites must be started.

4. Final and optimal solution

The lid of the cartridge is fixed to the lid of pressure vessel; consequently the cartridge and the pressure vessel are closed simultaneously. Now it is possible to install a pipe from the lid of the cartridge through the lid of the pressure vessel to the outside. This pipe can be used to measure directly the pressure in the cartridge. This pipe cannot be blocked or even be destroyed at the closing procedure (what could not be prevented when e. g. a flexible hose would have been used between the ‘old fashioned’ cartridge and the outside of the pressure vessel).

This probe pipe can additionally be used to vent the cartridge in case of blocking.

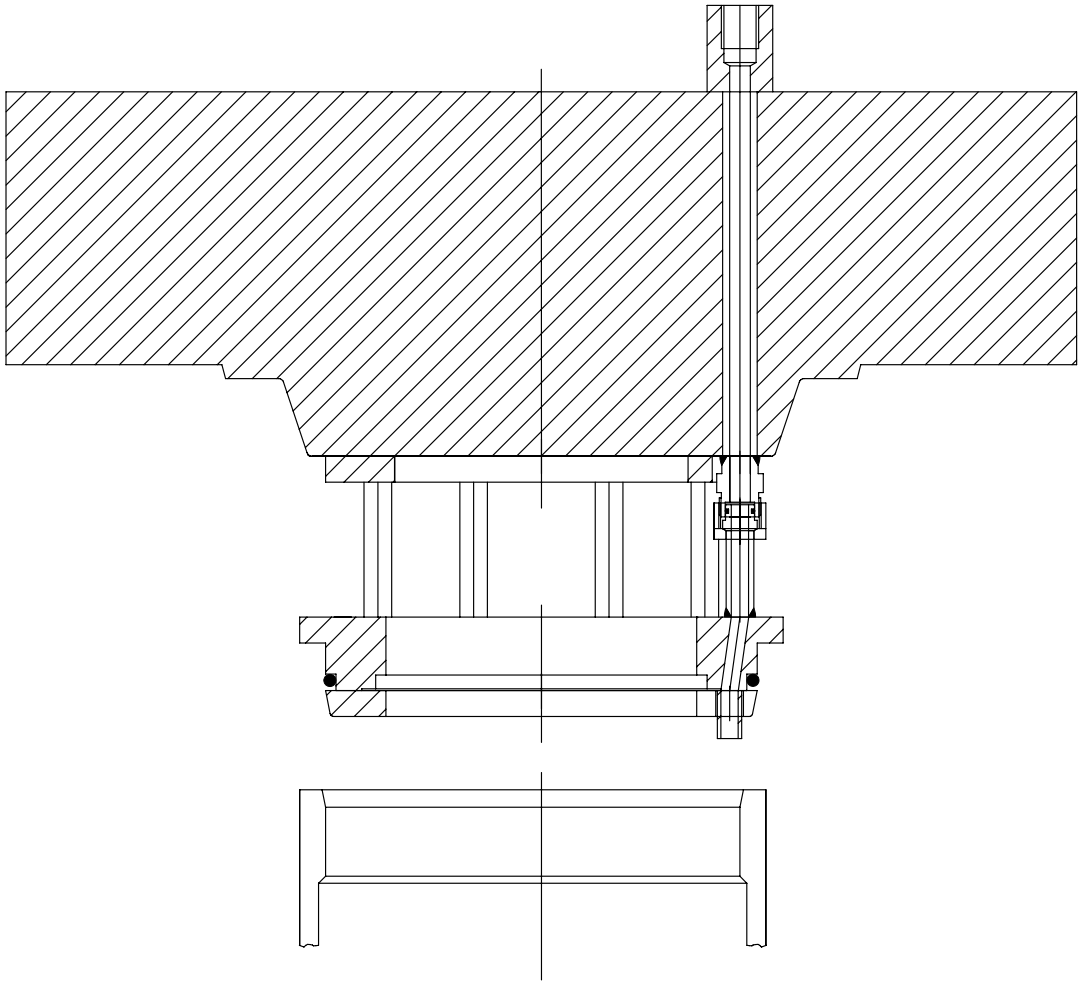

Figure 2: Lid of the cartridge fixed to the lid of the pressure vessel (hatched) and connecting pipe. Pressure vessel not exhibited.