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Plunger pumps for supercritical extraction service

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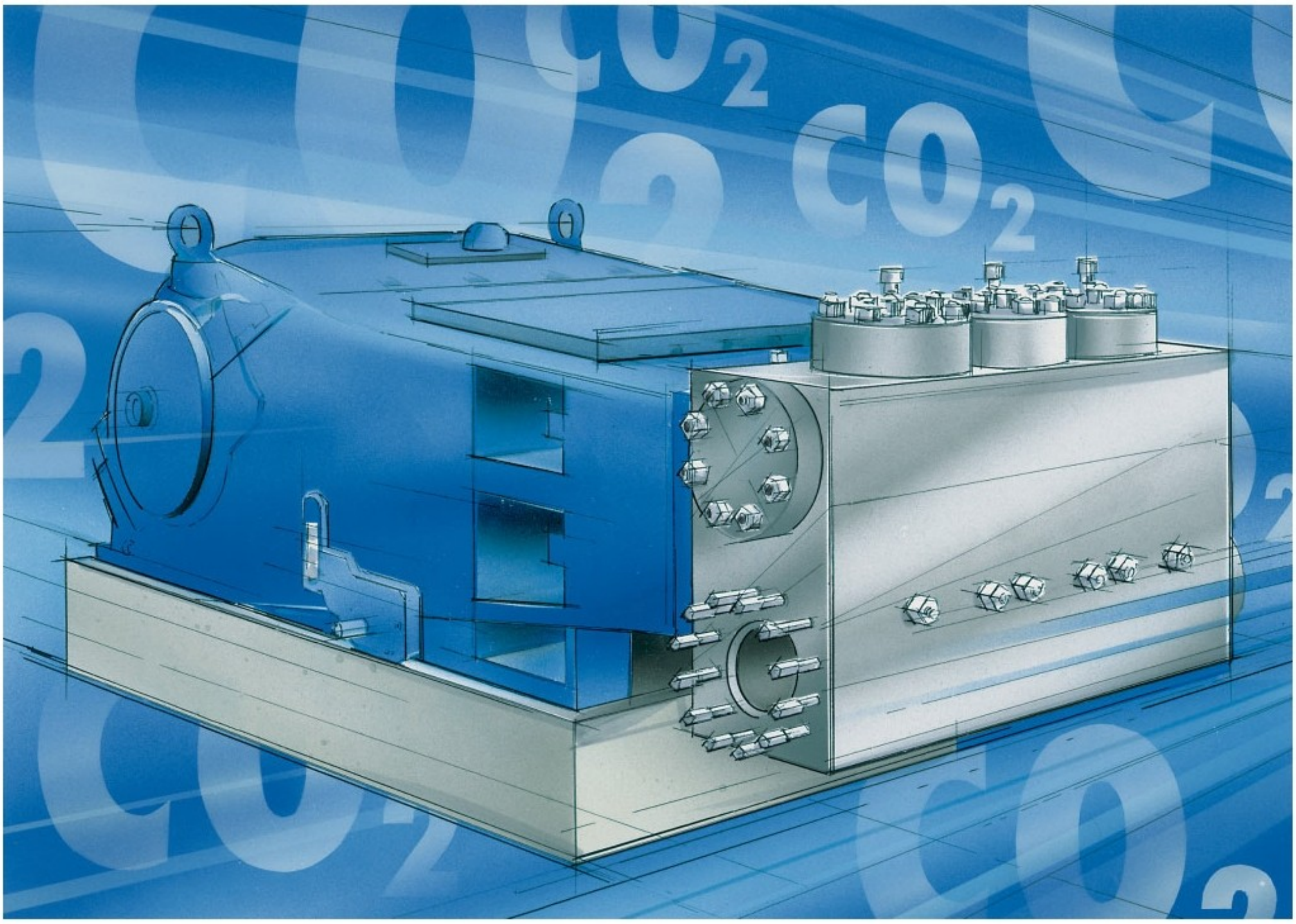

HIGH PRESSURE STATE OF THE ART TECHNOLOGY. WORLDWIDE.

Plunger pumps for supercritical extraction service

It is held that, through the utilization of carbon dioxide supercritical technology, purer and therefore more valuable end products may be produced. Optimal process results relative to product quality depend upon the controllability of the process parameters in the high pressure loop. As the control of the carbon dioxide process parameters requires sophisticated techniques and knowledge, so does reciprocating pumping equipment design to be used in these processes. The combination of these conditions give rise to the desired operating characteristics described herein.

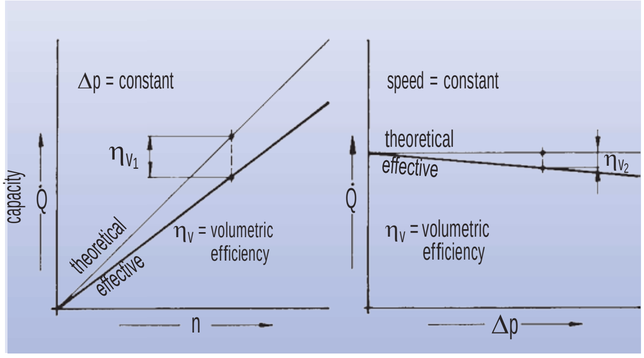

Figure 1 Dependency of volumetric flow on pump speed and discharge pressure of reciprocating plunger pumps.

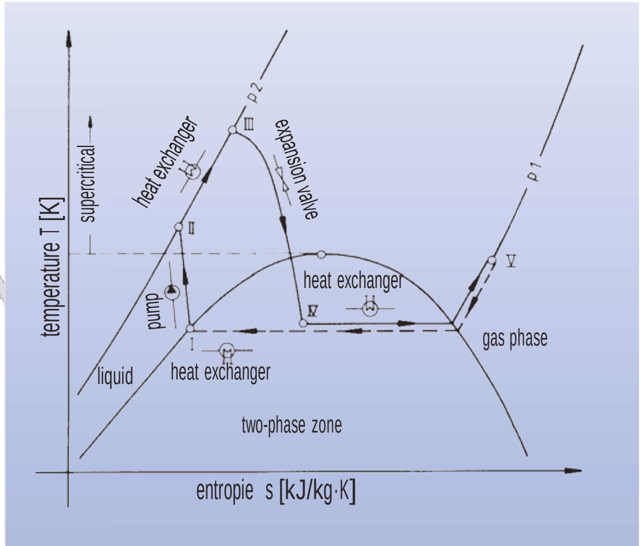

Figure 2 Temperature-Entropy Diagram (CO_2 Extraction Process).

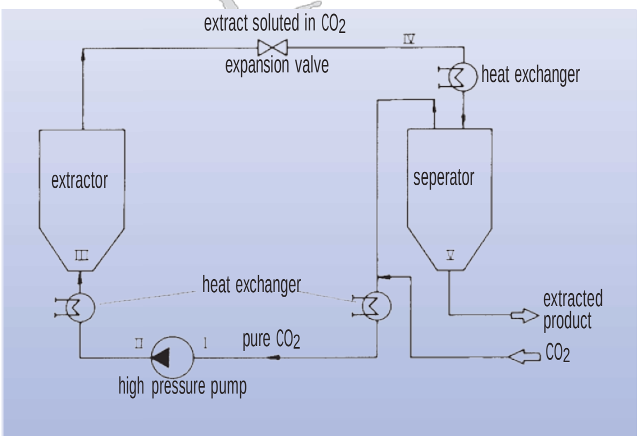

Figure 3 Process arrangement of the components of a supercritical extraction system.

Carbon dioxide supercritical extraction is a far reaching separation oriented type process that is growing in commercial application. One of the first commercial plants was designed in 1973 and went into service in 1979. Since that time many additional and larger plants have come into operation.

The efficient application of this process technique requires highly reliable and functional capability of the more important system components. Thus the pumping equipment required to pressurize the carbon dioxide liquid is a key process component.

Food and pharmaceutical industries require extraction of basic, rare, aromatic and/or taste substances for economic productivity and high product quality. Although the utilization of supercritical separation technology has been well known for many years, it has only recently been utilized for scale manufacturing applications. The prime utilization application is the extraction of substances from plant life raw materials.

The prime benefits of the supercritical extraction process relate to the non-destructive handling of the raw material and in addition the desired end product has an extremely high degree of purity. Also, the process can be made extremely selective as to extracted material(s). In fact, depending upon the selected combinations of pressure and temperature several different materials may be extracted from a common raw material. As can be imagined, the amount of the supercritical fluid throughput must be exactly maintained, controlled, and monitored.

Pressurization of the solvent stream is usually accomplished via a positive displacement plunger type pump, that must be designed to accommodate the extraction mediums physical properties. Plunger pumps are especially adaptable to this application in light of the required high discharge pressures and small throughput capacities. As an example, because of the compressibility of liquid CO_2, an especially critical necessity is the reduction of the pump's internal »dead space« to achieve optimum volumetric efficiency.

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Experience indicates that plunger pumps deliver an excellent overall efficiency and thus are highly economical machines to develop the required pressures and capacities. This result is in part attributable to the proportionality of pump speed vs. capacity and its relative indifference to end pressure. Performance is as illustrated in figure 1.

A high pressure supercritical recycle process

Figure 2 depicts the temperature/entropy curve for a carbon dioxide loop for a typical extraction process. A pump pressurizes the carbon dioxide from I to II (suction pressure »P1« to discharge pressure »P2«). At pressure »P2«, the carbon dioxide is heated in a heat exchanger from »II« to »III« i. e. the process temperature. A separator is employed to achieve the desired carbon dioxide combination of pressure and temperature and is let down from III to IV. An evaporator is utilized to proceed from »IV« to »V« at which point the heavier product is separated out. The »purified« carbon dioxide proceeds from »V« to »I« and is condensed, subcooled and at process point I sent on at »P1« to the pump suction.

Potential variations in the pressure, temperature and capacity process conditions permit the extraction of diversified substances from various raw materials. Therefore, the process is extremely flexible and lends itself to many potential applications. Assuming a common raw material, it is also possible to selectively extract several different components as previously indicated.

In general, a supercritical extraction system consists of four major components:

- A pump to pressurize the extraction medium.
- A pressure vessel (»extractor«) where the extraction process takes place.
- A pressure vessel (»separator«) where the extract and medium are separated.
- Heat exchangers to control the extraction and separation temperatures.

Figure 3 shows a typical process arrangement for these elements. The Roman numerals correspond to the entropy diagram in figure 2.

Liquid carbon dioxide pump requirements

There are specific requirements, in regard to the design of a high pressure pump for utilization on liquid carbon dioxide. Initially, the pump must be capable of pressurizing the medium to pressures over the supercritical threshold. Liquid carbon dioxide is an extremely compressible fluid as may be seen from the thermodynamic table. There is also the problem that the temperature gain during the pressurizing process is not precisely controllable. As such, especially in large pump applications, there is the question of not being able to accurately predict pump capacity. This problem must be solved through experience and empirical measurement. To further understand these pump issues, a research project was undertaken to explore what happens in the pressurizing chamber of a plunger pump such that this data could be utilized to further advance design technology.

Abb. 4 URACA plunger pump KD 827 for supercritical extraction service.

The pump was fitted with instrumentation to measure the following: pressure in the space between the suction and discharge valves, inlet and outlet temperatures, cooling medium temperatures and ambient temperature. See figure 5.

Figure 6 is the developed indicator diagram through the synchronization of the functions in the liquid cavity and plunger stroke. In this diagram, »A« to »B« is the suction stroke. »B« to »C« is the pressurization to the required discharge pressure and »C« to »D« is the discharge valve opening permitting the liquid carbon dioxide to enter the discharge line. The relationship of »C« to »D« and »B« to »D« demonstrates a volumetric efficiency »VD«, considerably less than that obtained when utilizing water. This is explained by compressibility. »D« to »A« shows the expansion of the liquid carbon dioxide remaining in the pump's »dead space«. The cycle is repeated starting at »A«.

Repetitive testing under varying process parameters provided extremely meaningful empirical data to permit the design of pump equipment to exactly supply the required process conditions of service.

In addition to concerns relative to strength of materials, cavitation free operation is an absolute necessity to maintain trouble free operation. Also, to maintain a high degree of efficient »on line« operation, the seals on the suction and discharge valves are an important consideration.

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Test results indicate that high pump efficiency may only be ob- tained by the careful design of the high pressure liquid end to abso- lutely minimize dead space. Fur- ther design matters of interest relate to the stuffing box sealing system in light of liquid carbon dioxide lubrication and cooling characteristics.
A means to monitor the function- ing of the seal system is a prime design consideration.

In summary, pumping liquids used in supercritical applications require special suitable materials of con- struction and design technology based upon practical application and creative research and develop- ment.

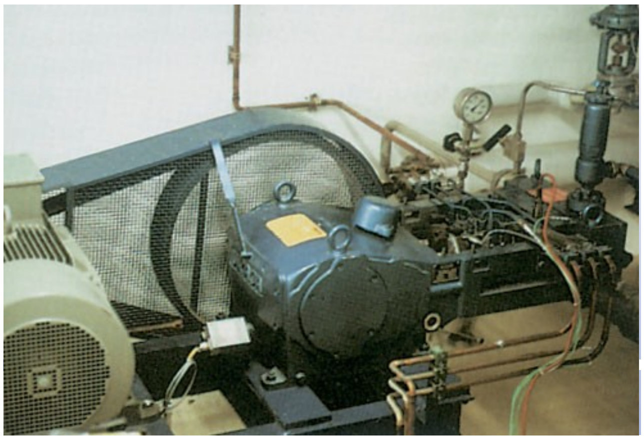

Figure 5 Liquid carbon dioxide pump with measuring instrumentation.

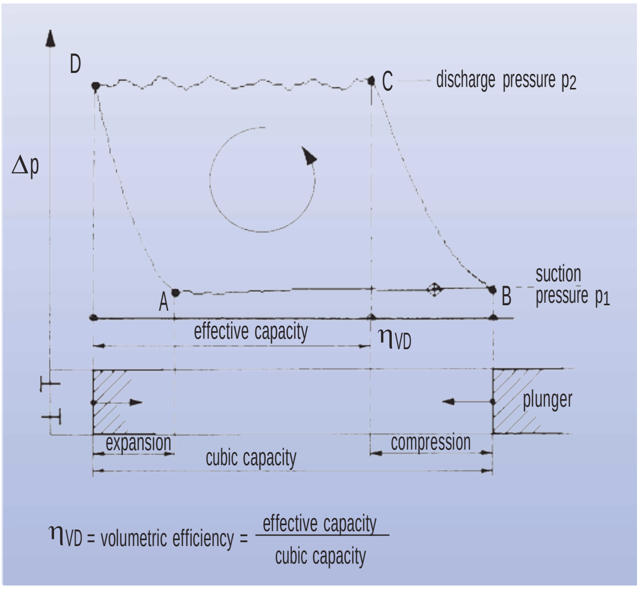

Figure 6 Indicator diagram for a reciprocating plunger pump in liquid carbon dioxide service.

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