A World of supercritical fluid instrumentation and application.

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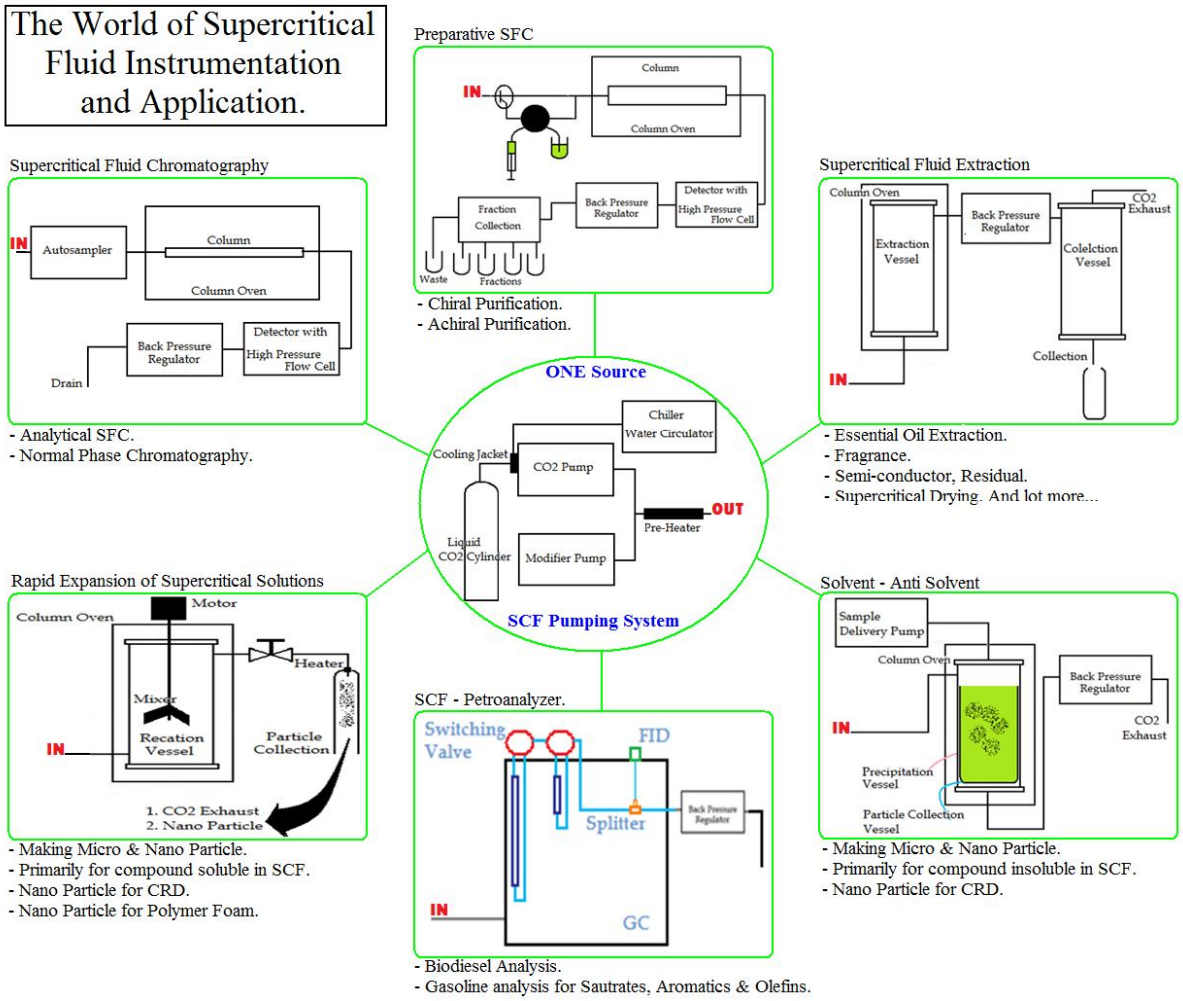
'A World of supercritical fluid instrumentation and application' explain about the various applications on SCF. This poster can be used as a single source reference to understand the design, instrumentation and field of application of various SCF applications. Supercritical Fluid Carbon dioxide applications are gaining interest in the field of research mainly because of its environment friendly properties and easily achievable parameter aprox of 31.3°C and 73 Bar. The high diffusivity and low viscosity gives wider region to work. The CO2 flow through the various phases of liquid to supercritical and to gas during each process making it unique in itself.

The supercritical fluid CO2 has the widest application like chromatography, extraction, nano particle, purification, dry-cleaning, human in-plants, semiconductors, green catalyst, reactions, ionic liquid, bio-diesel etc.



The brief information of instrumentation of the technologies explained in the poster are:

ONE Source : The pumping system for CO2. The chiller to maintain liquid condition of CO2 at pump, further pumped into Pre-Heater to convert to SCF, Modifier pump can be optionally

used. Common for all the SCF applications. A reciprocation pump mounted with non-return check valve is used. The Pump head is cooled with either Peltier or Water circulating cooling jacket to maintain the CO2 in liquid condition. Now you may think, why liquid condition! Its so that the pump can easily pump the Liquid CO2 in to the System. After the Pump, CO2 is passed through the heater, usually at 35°C to convert it to supercritical fluid. The is common for any supercritical fluid (CO2) instrumentation. The Pump take the CO2 directly from the liquid CO2 cylinder at pressure of 60 Bar.

I wish to go a step further and mention the information that is small but very useful about the heart of the process, the CO2 Cylinder. The Food grade Liquid CO2 with 31Kg of CO2 in a Cylinder with a dip tube. Effectively 70% of CO2 can be used from each cylinder. Cost is approx $1/Kg.

Supercritical Fluid Chromatography : SFC instrumentation is similar to HPLC. The outlet of the 'ONE Source' is connected to the Autosampler to inject the sample in the range of 5 - 100µL. The outlet of the Autosampler is connected to the SS Column (usually Silica, Cayano and Chiral Column.) with dimension 250mm x 4.6mm. Outlet of the column is connected to detector with high pressure flowcell with a BPR to regulate the pressure so as to maintain the supercritical condition.

Supercritical Fluid CO2 is Normal phase chromatography. SFC was developed 20 Years before HPLC and was earlier named as High Pressure Gas Chromatography. The first application of SFC was with FID, SCF has flexibility to use wide range of detectors from UV to MS, and ELSD, Chiral, FID, FTIR also.

Preparative SFC : Used for purification for normal phase and chiral applications. Flow rate range should be practically between 40ml/min to 120ml/min with column dimension id 20mm to 50mm. The Instrumentation of Prep SFC is similar to SFC except for a fraction collector. Low viscosity/high diffusivity increase the life of column. The gradient mobile phase has around 70 – 90% of SCF-CO2, so the fraction has only 10 – 30% of solvent, this reduce the drying time of the fraction. The purity and yield is better than the Prep LC in the range of 95% and above each, almost zero carry over. The process is approximately 6 – 10 times faster than Prep LC and equally cheaper. The only limitation as of now is, it has more normal phase application, still research is in process to develop column to use SCF in reverse phase.

Supercritical Fluid Extraction : SFE is most popular among the other SCF technique. This has the very simple instrumentation with "ONE Source" connected to the extraction vessel, a BPR to regulate pressure and a collection vessel. The compound is filled in the extraction vessel and SCF is passed through, extracting the desired component. Various components can be extracted from the same compound by modifying the pressure and temperature. Usual operating parameters are 50°C and 200 to 600 Bar at any Flowrate between Lab scale to pilot scale. Analytical scale extraction vessel can be as small as 10ml.

Widely used for extraction of essential oils, fragrance, fats, herbal & medicinal plant etc. also used for semiconductors, SCF drying. SCF-CO2 extracts do not have any residual impurity as compared to solvent extraction, reducing the time and process of purification.

Solvent Anti-Solvent : The sample dissolved in the solvent is introduced in the precipitation vessel of 1000ml at a low flow rate in range of 0.2ml/min making particle of the desired

sample. The particle can be in range of micro to nanometer. Precipitation vessel is filled with continuously flowing SC-CO2. The Nano particle are collected in the Precipitation vessel. The size and the yield can be improved by changing the pressure in range of 150 to 200 Bar, temperature between 40 to 50°C, flow of SC-CO2 in range 30 to 50ml/min and the flow of the solvent between 0.2 to 0.5ml/min on a lab scale instrument.

SCF Petro-analyzer : Analytical SFC is connected to a GC-FID. The mobile phase is SCF-CO2. Silica & Silver column are used. The detector is a FID. Used to analyze olefins, hydrocarbons & saturates. This process was developed for few years now, still very under utilized. The instrumentation is similar to SFC. Used for ASTM methods.

Rapid Expansion of Supercritical Solutions : RESS is the simplest of all SCF instrumentation. The sample that is soluble in SC-CO2 is filled in the reaction vessel (usually 20% of the vessel size). The sample is dissolved with SCF and released through nozzle at specific temperature. This process gives nano particles. The "ONE Source" outlet is connected to the RESS vessel, it is further connected to a nozzle. Usual operating parameters are 50 to 80°C with 200 to 250Bar, release at half of one circle turn of nozzle. Nano particle achieved in the size as low as 100nm.

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