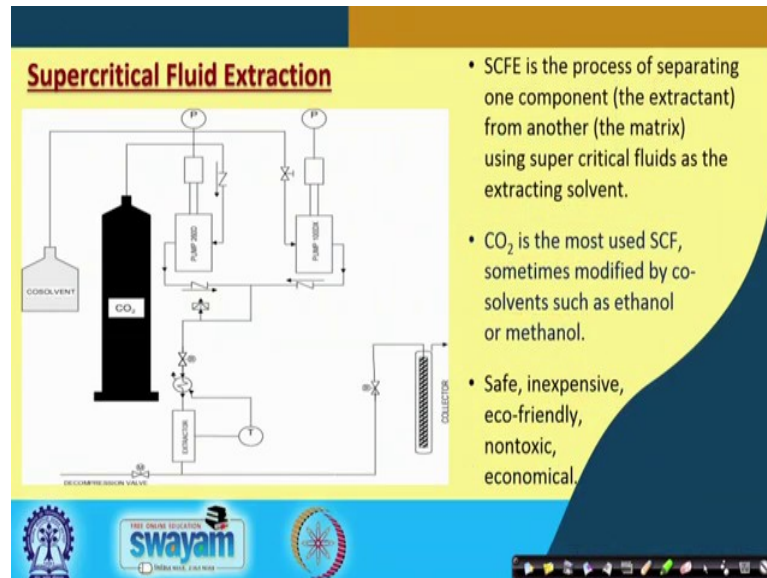


**Novel Technologies for Food Processing and Shelf Life Extension**  
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**Department of Agricultural and Food Engineering**  
**Indian Institute of Technology, Kharagpur**

**Lecture - 20**  
**Supercritical Fluid Extraction (Part 2)**

In this lecture, the application aspects of this process in food industry is discussed.



Supercritical fluid extraction (SCFE) is a process of separating one component from the other using supercritical fluid as the extracting solvent. The component which is separated is known as extractant and the material from which it is extracted is known as a matrix.

Carbon dioxide gas is the most commonly used supercritical fluid, sometimes it is modified by co-solvents like ethanol, methanol, etc. This process is safe, expensive, eco-friendly, non-toxic and economical. In this figure, carbon dioxide generating source, co-solvent store, extraction vessel, pump, etc. are shown. In the extractor, the material is loaded and carbon dioxide with some co solvent are pumped to mix with the material and extract different components. The desirable bio materials get extracted in the solvent, then the solvent is decompressed and finally, these components are separated from the mixture of the fluid and the extract.

### Applications of SCFE in Food Industry

SCFE has wide application in natural products and food industries such as

- ✓ Decaffeination of coffee and tea
- ✓ Spice extraction (oil and oleoresin)
- ✓ Deodorization of oils and fats
- ✓ Flavors, fragrances, aromas, and perfumes
- ✓ Decholesterolization of egg yolk and dairy creme
- ✓ Antioxidants from plant materials
- ✓ Food colors from botanicals
- ✓ Natural pesticides
- ✓ Denicotinization of tobacco












Applications of the supercritical fluid extraction in the food industry: SCFE has a wide range of application in the natural products as well as food industries. It can be used for decaffeination of coffee and tea, for extraction of oils and oleoresins from spices, for deodorization of oils and fats, etc. So, in the oil milling or oil refining industry, it has a good application. It can also be used for preparation or extraction of flavours, fragrances, aromas and perfumes, de-cholesterolization of egg yolk and dairy cream, etc. It is used for extraction of antioxidant from plant materials, food colours from botanicals, or nicotine from tobacco and all.

### SCFE technology can be used for extraction, purification, and separation of:

✓ Edible oils and fats	✓ Bioactive compounds, e.g. pyrethrum, caffeine, theobromine, cholesterol, capsaicin, etc.
✓ Hops extract	✓ Mono- & diglycerides
✓ Natural dyes: Annatto, Hibiscus	✓ Aroma compounds
✓ Vitamins (Tocopherols, Vit. E, Tocotrienols)	✓ Thiosulfinates
✓ Carotenoids	✓ Citrus oils
✓ Sterols	✓ Antioxidants such as vitamin E, ascorbic acid, polyphenoles, diacin, genicin (steroids).
✓ Essential fatty acids (EPA, DHA, DPA)	




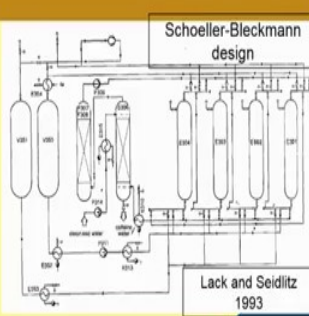




SCFE is applied for extraction, purification and separation of edible oils and fats, hops extracts used in the beer making industry. Hops is a flowering part from which the flavouring component is extracted that gives the characteristic flavour to the beer.

Application of SCFE is found in extraction of natural dyes like annatto, hibiscus; vitamins like tocopherols, vitamin E, tocotrienols; carotenoids, sterols, essential fatty acids like EPA, DHA, DPA; bioactive compounds like caffeine, theobromine, cholesterol, capsaicin; monoglycerides, diglycerides, aroma compounds, thiosulfinate; even essential ions from citrus fruits etc.; antioxidants such as ascorbic acid, polyphenols or even diacin, genicin, etc.

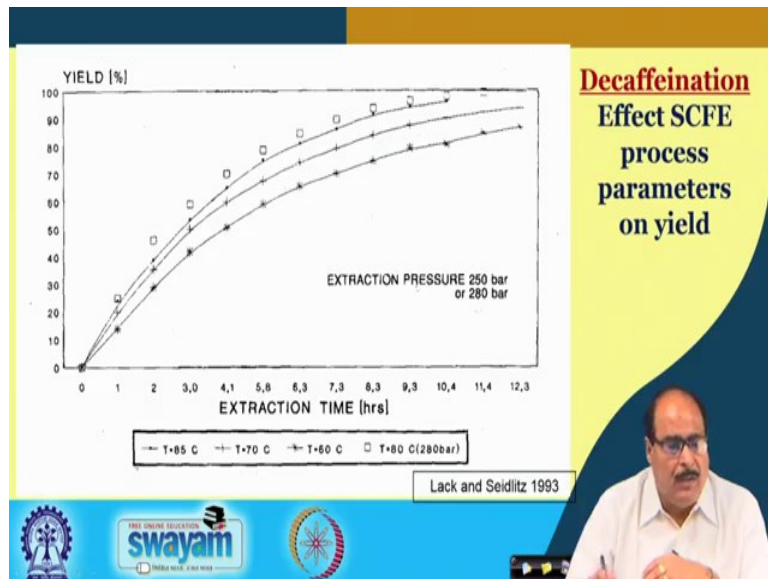
**Process flowchart for decaffeination of coffee & tea using SCFE technology plant**

- Coffee bean is wetted with water
- Wetting tend to dissolve & desorb caffeine from the solid material.
- The operating conditions are
  - ✓ Pressure 300 bar
  - ✓ Temperature 40 °C
- Green beans are loaded into the vessel & SCF CO<sub>2</sub> is introduced in the vessel.
- Extract is transferred through an expansion valve, to the separator which operates at low pressure and separates it into two phases – aqueous caffeine extract and CO<sub>2</sub>.
- The CO<sub>2</sub> is recycled.
- Caffeine can be recovered e.g. by adsorption on an activated carbon column.

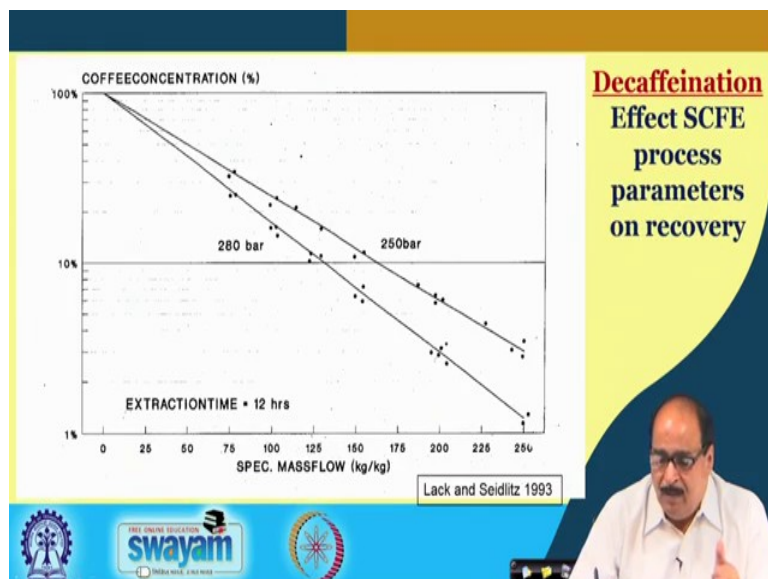


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Decaffeination of coffee or tea using SCFE technology: First, the coffee bean is wetted with water and this wetting of the bean tends to dissolve and desorb the caffeine from the solid matrix. Then, the wetted green beans are loaded into the extraction vessel after which supercritical carbon dioxide is introduced into the vessel. The operating conditions normally maintained are 300 bar pressure and 40 °C temperature. The extract obtained is transferred through an expansion valve to the separator which operates at a comparatively lower pressure. It separates the extract in 2 phases i.e. aqueous caffeine extract and carbon dioxide. The carbon dioxide is again recycled further to improve the efficiency and the caffeine is sent to the recovery unit where it can be recovered either by adsorption or by decompression. So, even adsorption on an activated carbon column can be used to recover the caffeine from the extract.



The extraction process variables i.e. extraction pressure, time, and temperature influence the yield of the caffeine. Y-axis (in graph) is the yield of the caffeine that increases with the increase in the time, temperature and pressure. For maximum yield or maximum recovery one should always standardize the process and optimize it.




In the graph, it is shown that the concentration of the material which is extracted i.e. caffeine is initially taken as the 100 % concentration which was fed into the extractor and the extraction time maintained was 12 hours. The concentration of the material that is remaining increases with both increase in the mass flow rate (kg per kg solid) as well as the pressure.

So, operating pressure, mass flow rate of the material in the continuous system, gas temperature, pressure etc. influence the yield of the product as well as the concentration remaining in the material.

### Extraction of natural food antioxidants

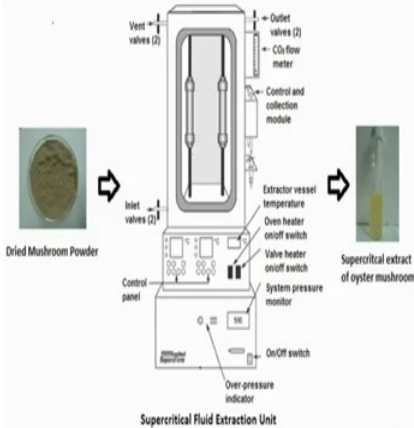
- SFE has been widely studied by several authors to obtain highly active rosemary antioxidant extracts.
- ✓ Topal et al. (2008) demonstrated that the antioxidant activity of supercritical extracts of different Turkish plants (rosemary among them) were higher than those obtained by steam distillation.
- ✓ Better results in terms of antioxidant activity were also achieved when compared to liquid solvent sonication (Tena et al., 1997).





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The extraction of natural food antioxidants have been widely studied by several authors to obtain many common or important bio materials like rosemary antioxidant extracts etc. Some researchers have standardized the process and demonstrated that the antioxidant activity of supercritical extracts of rosemary or other different Turkish plants were higher than those obtained by conventional processes like steam distillation etc. Even better results are reported in terms of antioxidant activity when particularly compared to liquid solvent sonication.

### Extraction of antioxidants from dried mushroom



- Ethanol modified supercritical CO<sub>2</sub> (SC CO<sub>2</sub>) was used for the extraction of L- ergothioneine, an antioxidant and total phenolic (TP) from dried mushroom *Pleurotus ostreatus*.
- About 1.35 mg/g dw of ergothioneine and 5.48 mg GAE/g dw of TPC were recovered.

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*Pleurotus ostreatus*

Extraction of antioxidants from dried mushroom: The mushroom is taken, dried and made into the powder. Then in the powder form, it is introduced into the extraction vessel and the rest thing follows as usual like in any other process. It is given the desired time to contact with super critical carbon dioxide at desired temperature and pressure. Finally, ergothioneine 1.35 mg/g dry weight and total phenols about 5.48 mg GAE/g dry weight have been obtained.


**Extraction of bioactive components from Microalgae**

- SCFE technology was applied on microalgae *Chlorella vulgaris* for the extraction of lutein, an important carotenoid, total phenolic (TP), and antioxidants (AO).


✓ About 6.6 mg/100 g dw lutein, 22.8 mg GAE/100 g dw TP, and 5.3 mg/100 g dw AO could be obtained using ethanol modified SC CO<sub>2</sub>.






Chlorella vulgaris



Lutein



Structure of Lutein



Extraction of bioactive components from microalgae: SCFE was applied on microalgae *Chlorella vulgaris* for the extraction of lutein, an important carotenoid, total phenolic (TP) and antioxidant (AO). About 6.6 mg/100 g dw lutein, 22.8 mg GAE/100 g dw TP and 5.3 mg/100 g dw AO could be obtained using ethanol modified supercritical carbon dioxide.

**Low cholesterol whole milk powder and cream powder**

- SC CO<sub>2</sub> alone and ethanol (co-solvent) modified SC CO<sub>2</sub> were employed to extract cholesterol from whole milk powder (WMP) and cream powder.
- About 55.8% and 46% of cholesterol removal from WMP could be achieved by using SC CO<sub>2</sub> alone and ethanol modified SC CO<sub>2</sub>, respectively.
- Addition of ethanol led to enhanced extraction rate.
- About 39% cholesterol could be removed from cream powder using SC CO<sub>2</sub> alone.

Whole milk powder      Supercritical Fluid Extraction Unit      Cholesterol Extract

Low cholesterol whole milk powder and cream powder: SC CO<sub>2</sub> alone and ethanol modified CO<sub>2</sub> were employed to extract cholesterol from whole milk powder (WMP) and cream powder. About 55.8 % and 46 % of cholesterol removal from WMP could be achieved by using SC CO<sub>2</sub> alone and ethanol modified SC CO<sub>2</sub> respectively. Addition of ethanol led to enhanced extraction rate.

**CHOLESTEROL FREE CREAM & MILK POWDER**

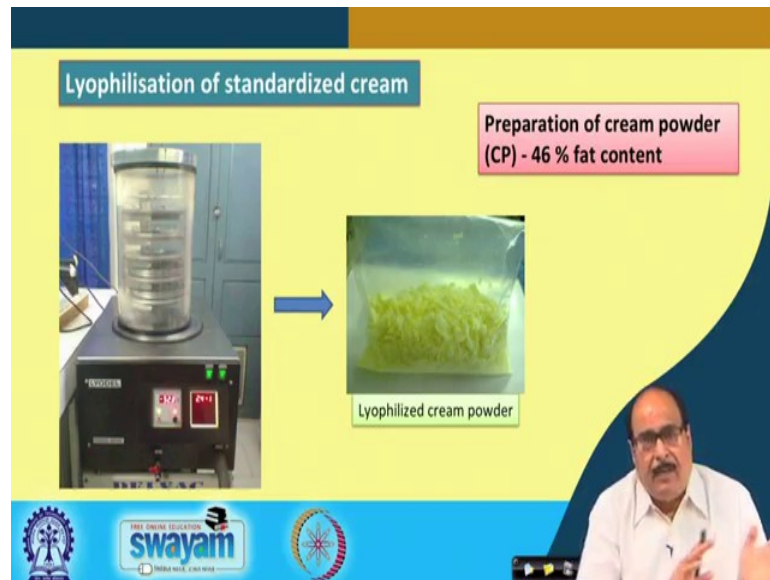
**Process flowchart for preparation of low cholesterol milk powder using SCFE technology**

- SAFE
- ENVIRONMENT FRIENDLY
- SELECTIVE
- NO DIFFERENCE IN PROPERTIES/TASTE

MILK POWDER      VESSEL      EXTRACTION CHAMBER      CO<sub>2</sub> CYLINDER & CHILLER      SEMI-LIQUID STATE OF CO<sub>2</sub>

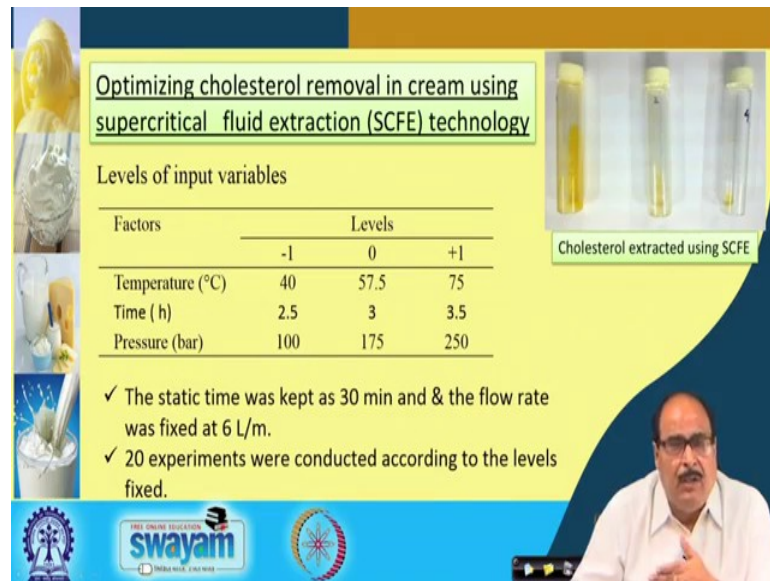
Process flow chart for the preparation of low cholesterol milk powder using SCFE technology: In the pictorial flow diagram, the milk powder is introduced into the extraction vessel and this vessel is fitted in to the SCFE equipment. The characteristics of low cholesterol dairy cream and milk powder obtained were also analyzed and found that

there was no difference in the properties as well as in the taste of the low cholesterol containing cream or even after the removal of the cholesterol.



The image shows a lyophilizer machine on the left with a glass vial inside. An arrow points to a vial on the right containing a yellow, porous substance labeled "Lyophilized cream powder". A text box in the upper right corner reads "Preparation of cream powder (CP) - 46 % fat content". The slide also features the Swamyam logo and a speaker in the bottom right corner.

Similarly, the cream was lyophilized and converted into cream powder.



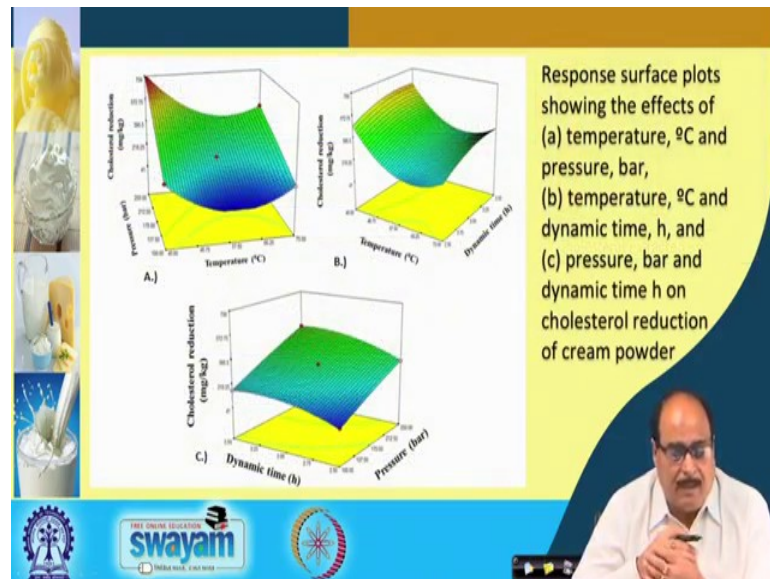
The slide is titled "Optimizing cholesterol removal in cream using supercritical fluid extraction (SCFE) technology". It includes a table of input variables and a list of experimental conditions. To the right, three vials show the extracted cholesterol. The Swamyam logo and a speaker are also present.

Factors	Levels		
	-1	0	+1
Temperature (°C)	40	57.5	75
Time (h)	2.5	3	3.5
Pressure (bar)	100	175	250

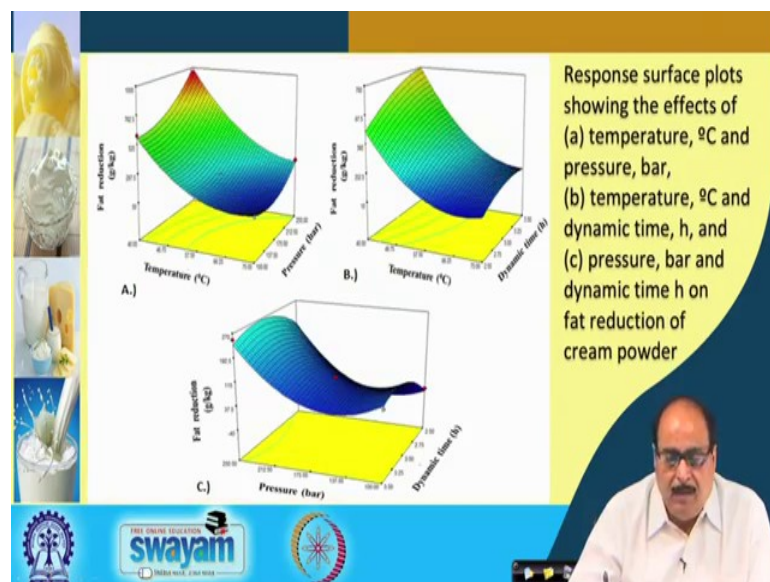
- ✓ The static time was kept as 30 min and the flow rate was fixed at 6 L/m.
- ✓ 20 experiments were conducted according to the levels fixed.

For optimization, the input variables were temperature (40-75 °C), time (2.5-3.5 h) and pressure (100-250 bar). About 20 experiments having combinations of these variables were conducted as per the experimental design; the static time was kept 30 min and the flow rate was fixed at 6 l/min.





These are the response surface plots showing the effect of temperature and pressure on the cholesterol removal, effect of temperature and dynamic time as well as effect of pressure and dynamic time on the reduction in cholesterol from the dairy cream or cream powder. If time increases, the increase in reduction is more, but with the temperature, it first increases and then decreases. At higher pressure, it is high and when the pressure is reduces, it may become low.

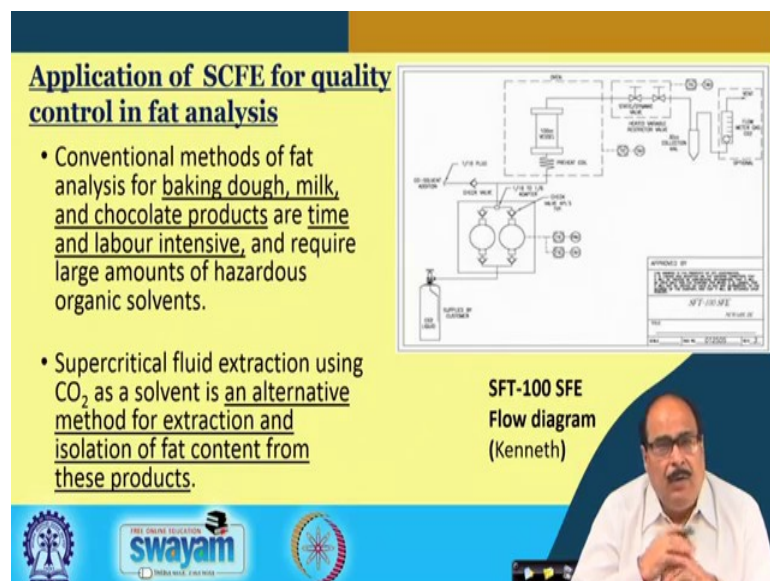


When the cholesterol is removed, there should not be substantial reduction of the fat. In the dairy cream these cholesterol which are basically phospholipids are generally found associated with the other lipid molecule. So, it is quite likely that the fat also get

extracted with the cholesterol. So, one has to optimize the process so that maximum cholesterol removal with minimum fat content can be obtained.



The process has been standardized and parameters are optimized in the laboratory experiments. With the help of the modifier, at least around 65-70 % reduction in the cream was achieved. This cholesterol free cream powder (see Fig.) was further used in the preparation of products like butter and ghee etc. It was observed that there was no significant variation in the characteristics of the products prepared from the low cholesterol.



SCFE is a unique and a very good advancement for the quality control particularly in the case of fat analysis because in the products like baking dough, milk, chocolate products,

etc., the conventional methods which are used for the removal of the fat and determination of the fat content in these products are normally labour intensive, require much time and large amount of hazardous organic solvents.

So, applying this supercritical fluid extraction technology using carbon dioxide as a solvent, one can get the complete extraction of lipids or fat molecules sometimes even selective fats like monoglyceride, diglyceride etc. can be obtained. So, this makes SCFE technology an alternative method for all extraction, isolation and purification of such materials. So, it becomes a very useful tool for the industry and this should be propagated.

This technology appears to be promising particularly where the products of interest are high value products, heat sensitive product and health ingredients in the material need to be remained intact. So, they can be easily extracted using supercritical carbon dioxide without any adverse effect on the health component or on the environment.

There is no by product, no residue and this is a completely green technology; however, for every product and for every process depending upon the characteristics of the raw material as well as depending upon the ingredient which is being extracted, one need to properly optimize the process and standardize it. And accordingly suitable plant, machinery and equipment need to be designed to facilitate the extraction process.