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Gas Chromatography Application in Supercritical Fluid Extraction Process

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1. Introduction

There are two types of application for gas chromatography (GC) in the supercritical fluid extraction process. Gas chromatography is a type of supercritical extraction apparatuses which can separate a component from a multi-component mixture during supercritical extraction. Therefore, this application can be the alternative to conventional gas chromatography, which needs high temperatures for the evaporation of the feed mixture and for liquid chromatography, where liquid solvents may be replaced. This process results in a different transport velocity along the stationary phase for different molecules. Molecules having weak interaction forces with the stationary phase are transported quickly while others with strong interactions are transported slowly. Beside the interactions with the stationary phase, the solvent power of the mobile phase determines the distribution of the components. Furthermore, supercritical gases have a high solvent power and exert this solvent power at low temperatures.

Another application of GC in supercritical fluid extraction is consideration and analysis of extraction product. The obtained products from various types of supercritical apparatuses (such as phase equilibrium and rate test apparatus) should be analyzed. However, different types of analyzer can be used but, the conventional GC with a suitable column has widely been recommended. Although several columns for detecting a lot of components have been designed and fabricated by some companies but due to lacking of suitable columns for some components or unclear peaks obtained from some columns, an extra process (such as esterification of the fractionated fish oil) before GC analyze is sometimes required. In this application, the samples obtained from the supercritical extraction apparatus are not under pressure or their pressures have broken down by a damper (in online GC).

In this chapter both types of GC application in supercritical fluid extraction with examples will be illustrated.

2. Gas chromatography apparatus

In supercritical fluid chromatography (SFC) the mobile phase is a supercritical gas or a near critical liquid. Compared to gas chromatography (GC), where a gas is under ambient

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pressure (for example in the second type of apparatus applied in supercritical process), and liquid chromatography (LC), where a liquid is used as mobile phase, the solvent power of the liquid mobile phase in SFC can be varied by density, e.g., by pressure changes at constant temperature. Solubility increases in general with pressure under supercritical conditions of the mobile phase, temperature sensitive compounds can be processed. The chromatographic separation can be carried out at constant pressure (isobaric operation) or with increasing pressure (pressure programmed). In addition, temperature can be varied. SFC has one more adjustable variable for optimization of elution than GC or LC (Brunner, 1994). A supercritical fluid has properties similar to a gas and also similar to a liquid. While density and solvent power may be compared to those of liquids, transport coefficients are more those of a gas. SFC, because of its mobile phase, can cover an intermediate region between GC and LC, as illustrated in Figure 1 with respect to density and diffusion coefficient. For preparative and production scale operations, SFC has the advantage of easy separation of mobile phase from separated compounds. A disadvantage is that strongly polar and ionic molecules are not dissolved by supercritical gases, which can be advantageously used in SCF (Brunner, 1994).

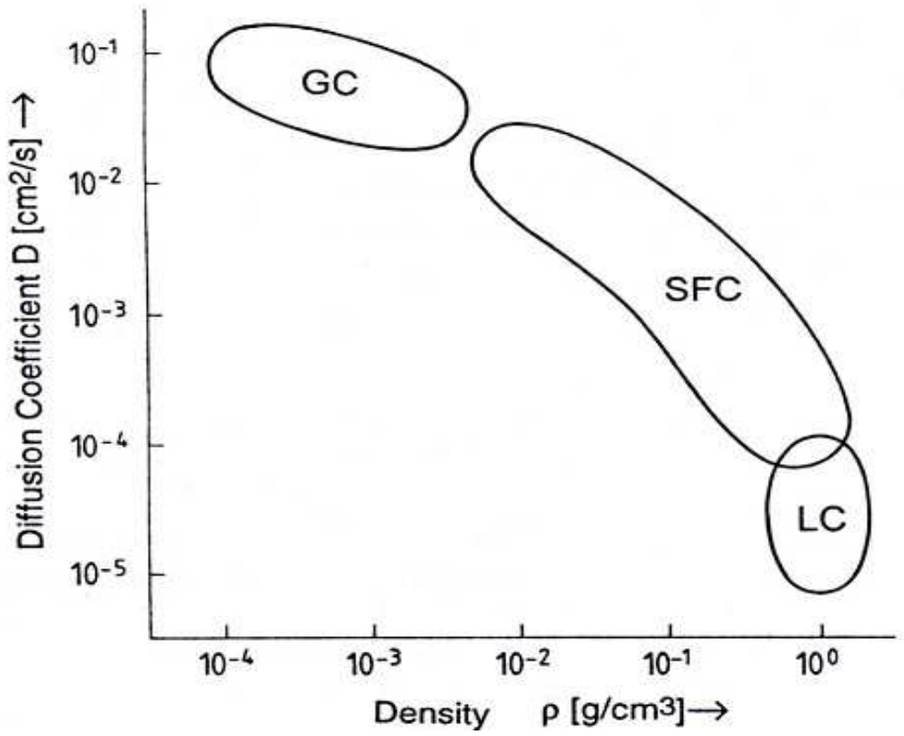

Fig. 1. Areas for the different mobile phases in chromatographic separations with respect to component properties (Schoenmakers and Uunk, 1987).

Most gases which can be used in SFC are non-polar. Therefore, polar substances of a feed mixture can only be eluted by adding a polar modifier. Polar gases like ammonia or sulfur dioxide are reactive compounds under pressure the equipment must be able to withstand corrosive conditions. On the other hand, carbon dioxide is easy to handle and safe. Polar modifiers, which are easier to handle than ammonia or sulfur dioxide may instead be applied. To make effective use of the possibilities of SFC, allowable pressures should be high.

Composition of the mobile phase can substantially influence separation in SFC (Brunner, 1994). Retention times of substances may be very much different due to polarity or other physico-chemical properties of the components of the mobile phase. Pickel (1986) investigated large differences in the separation of aromatic hydrocarbons with CO_2, N_2O, C_3H_8 and C_3H_6.

Gases applied in SFC are mostly non-polar. The polarity of carbon dioxide at low densities is comparable to that of n-hexane and at higher densities to that of methylene chloride. Nitrous oxide and the alkanes butane or pentane behave similar. Polar substances are eluted only after long retention times and in broad peaks even not at all. In these cases, a polar modifier, added to the gaseous mobile phase, introduces the necessary polarity to the mobile phase. The modifier then determines the elution sequence, which can be changed by the amount and the type of modifiers (Brunner, 1994).

With increasing content of a modifier in the mobile phase, retention times become shorter. For polycyclic aromatic compounds, Leyendecker *et al.* (1986) investigated the influence of 1.4-dioxane as modifier on n-pentane as mobile phase.

Temperature and pressure can be employed in supercritical chromatography as parameters for influencing separation characteristics. Temperature directly determines vapor-pressure of the feed components and density of the mobile phase and, indirectly, adsorption equilibrium. With higher temperatures, vapor-pressures of the feed components increase exponentially. Density decreases proportionally to temperature if conditions are far from critical, but in the region of the critical point of the mobile phase, which is the main area of application of SFC, density varies dramatically with temperature. The solvent power of the mobile phase, which increases with density, is therefore changed substantially in this region. The influence on chromatographic separation depends on the relative importance of these two effects, if other conditions remain unchanged (Brunner, 1994).

Pressure mainly influences density of the mobile phase. With increasing pressure, the influence of temperature is diminishing, since density varies less with temperature at higher pressures (Brunner, 1994). SFC allows the variation of temperature and pressure for optimizing separation conditions as well as during the separation process itself. Such an operational mode is called pressure and temperature programming. Temperature programming is well known from gas chromatography, but is less common in SFC, since pressure programming can be very effective. Pressure and temperature programming may be combined to density programming (Brunner, 1994).

In preparative chromatography, conditions are kept constant during separation, since feed mixtures of several injections may be on their way at the same time in the column. The elution of substances of different molecular weight in isobaric SFC separations is better than in isothermal GC, since vapor pressure is not so important in SFC. Compared to LC, the tendency of peak broadening is lower in SFC, since diffusion coefficients are far higher (Brunner, 1994).

Flow rate of the mobile phase is a further important parameter which affects the number of theoretical stages in chromatographic separations. Due to the low viscosity of near critical mobile phases, flow rate in SFC can be high, and number of theoretical stages remains nearly constant over a wide range of flow rate. A more detailed discussion of

chromatographic fundamentals and especially analytical applications of SFC can be found in the abundant literature on analytical SFC (Gere et al., 1982; Lee and Markides, 1990; Smith, 1988; Wenclawiak, 1992; White, 1988).

The apparatus (as shown in Figure 2) consists of the separation column as central part in a temperature controlled environment (1), the reservoir for the mobile phase (2), a unit for establishing, maintaining and controlling pressure (3), an optimal unit for adding a modifier (4), the injection part for introducing the feed mixture (5), a measuring device (detector) for determining concentration of the eluted substances (6), a sample collection unit (7), a unit for processing the mobile phase (8) and another one for processing data and controlling the total apparatus (9) (Brunner, 1994).

The flow of the supercritical gas under pressure is maintained by long-stroke piston-pumps, reciprocating piston-pumps or membrane-pumps which deliver the mobile phase in liquefied form. The fluid is then heated to supercritical conditions before entering the column. Pressure and flow rate must be kept as constant as possible in order to maintain constant conditions for separation and to achieve a stable base line in the chromatogram. Oscillating pumps therefore can have three heads which deliver at different times or a pulsation dampener in order to minimize pulsation (Brunner, 1994).

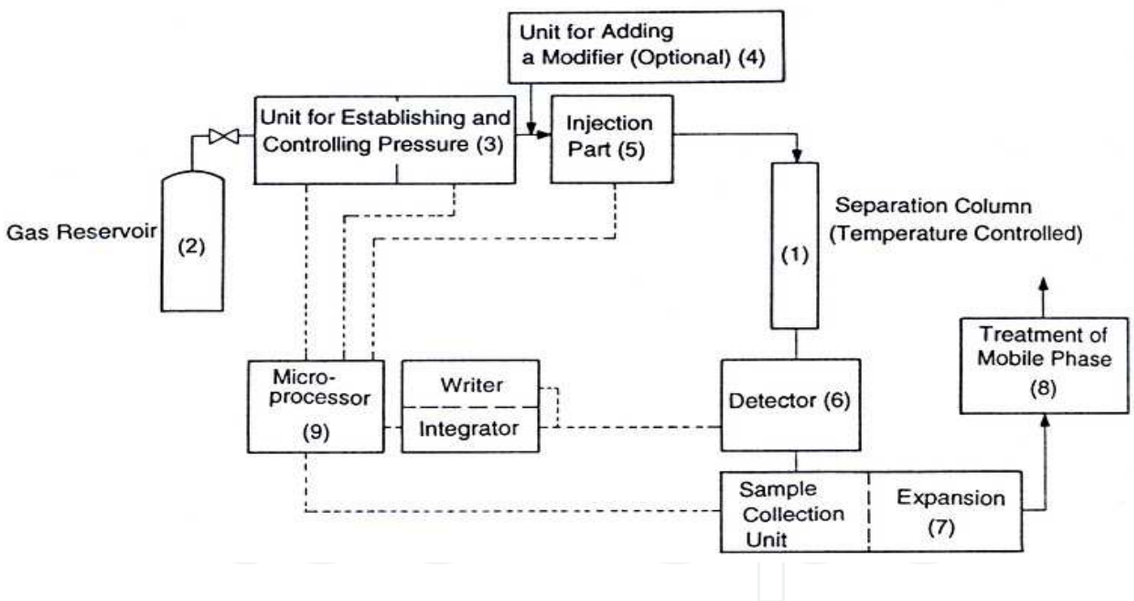

Fig. 2. Flow scheme of apparatus for SFC (Brunner, 1994).

2.1 Columns

Columns for chromatographic separation with supercritical are chosen, like other chromatographic columns, according to the needs of the separation. For analytical purposes the choice is between packed and capillary column. Capillary columns are used with a length between 10 m and 25 m. Pressure drop is low compared to liquid mobile phases. Therefore, capillary columns with inner diameters of 50 to 100 μm can be used and a high

number of theoretical stages verified. Separations with capillary columns can be nearly as effective as in gas chromatography. While in early applications steel capillaries had been used in SFC, since 1980, fused silica capillary columns have replaced the steel capillaries. Stationary phases mostly stem from polysiloxanes and polyglycols. Frequently used stationary phases have been listed in the literature (Brunner, 1994).

Under conditions of SFC, the compounds of the stationary phases may be slightly soluble in the mobile phase and are therefore fixed by linking them by chemical reactions. Numerous packed columns are available, many from HPLC applications. Normal phase chromatography (polar stationary phase, non-polar mobile phase) and reverse phase chromatography (non-polar stationary phase, polar mobile phase) are applied, but are not as important in SFC as in HPLC, since a polar mobile phase in SFC involves a polar modifier. Most separations in SFC are carried out with unmodified silica gel or chemically modified silica gel as stationary phase (Brunner, 1994). For packed columns particles are available with diameters in the range of 3 to 100 μm. For analytical purposes particles in the range of 3 to 5 μm have a high separation power in a packing and enable a high linear velocity of the mobile phase leading to short retention times. For preparative purposes particles in the range of 20 to 100 μm are used (Brunner, 1994).

Special filling techniques are necessary to ensure a homogeneous packing. Saito and Yamauchi (Saito *et al.*, 1988; Saito and Yamauchi, 1988; Saito et al., 1989) and Yamauchi and Saito (Yamauchi et al., 1988; Yamauchi and Saito, 1990) applied columns of 7 to 20 mm diameter, Perrut (1982, 1983, 1984) a column of 60 mm inner diameter and 600 mm length with particles of 10-25 μm, Alkio *et al.* (1988) a 900 mm long column with 40-36 μm diameter particles.

The length of the column is dependent on the allowable pressure drop. Pressure drop usually is in the range of 1 to 4 MPa for 250 mm. In this range for the pressure drop capillary factors are nearly independent of pressure drop as demonstrated by Schoenmakers *et al.* (1986). To avoid unacceptable pressure drop, Saito *et al.* applied a recycling technique (Saito *et al.*, 1988; Saito and Yamauchi, 1988; Saito *et al.*, 1989). A cycle pump transports the eluted substances several times to the beginning of the column. Thus, the separation power of the column can be enhanced, without increasing pressure drop. Peak broadening occurs due to the cyclic operations.

2.2 Detectors

Detection of a substance is necessary in analytical and preparative chromatography. In general, the same detectors are used as in gas and liquid chromatography. Selection of a detector depends on the quantity of substance available and the chemical nature the compound. A flame ionization detector (FID) detects substances down to nanogram quantities. Between two electrodes a voltage of 300 V and a hydrogen flame are maintained. If a substance with at least one carbon-hydrogen bonding is eluted from the column to the detector, it is burned and ions are formed, which leads to a current between the electrodes. The current is amplified and processed as a signal for the concentration of the substance. Nearly all substances can be detected. Response factors mainly differ according to number of carbon atoms, therefore calibration is easy (Brunner, 1994).

The ultraviolet spectroscopy detector (UV) is a nondestructive detector, which can be applied at column pressure. It is widely used, but is limited to substances with chromophoric groups. Saturated hydrocarbons, fatty acids and glycerides may be difficult to detect quantitatively. These substances may be detected with a refractive increment detector (RID), where the variation of refractive index of the mobile phase caused by dissolved substances is applied for detection. Other detectors are the fluorescence detector and the light-scattering detector (Brunner, 1994).

In a light-scattering detector the mobile phase is intensively mixed with an inert gas and heated while flowing downward a tube (Upnmoor and Brunner, 1989; Upnmoor and Brunner, 1992). The inert gas and the temperature increase reduce solvent capacity of the mobile phase. The eluted substances precipitate and are carried out droplets or particles into the detection chamber. Into this chamber a tungsten lamp delivers visible light, which is dispersed by droplets or particles. The dispersed light is detected by a photomultiplier under an angle of 60^{o}. The signal is proportional to the mass of light-scattering particles. Therefore, the light-scattering detector acts as mass detector and its signal is independent on chromatographic groups. It can be applied for detection of chromatographic and non-chromatographic substances in a mixture, as for example, in fatty acids and glycerides (Brunner, 1994).

2.3 Expansion of mobile phase and sample collecting system

In analytical SFC, the mobile phase is either expanded after or before detection. Downstream to a detector, which is operated under column pressure, expansion can be achieved by normal expansion valves. They can act as back pressure regulators may be controlled by a central unit. At interesting alternative to an expansion valve was designed by Saito and Yamauchi, who use time-controlled opening and closing of an unrestricted tube for expansion. This has the advantage that blocking of the tube by precipitating substances is avoided. Another expansion technique was adapted from GC: A glass capillary is formed into long, thin capillary, as so-called restrictor. Problems with blocking and difficulties with reproducible manufacturing of the restrictors are disadvantages of this solution. In analytical SFC expansion techniques are determined by detection needs. The amount of substances is small and can easily be handled. The quantity of the mobile phase is not small and it must be recycled. To avoid backmixing, the recycled mobile phase must be totally free from any dissolved substances. In most cases they will be in the range of 0.1 or even 0.01% (Brunner, 1994). Then, separation methods for the dissolved substances from the mobile phase become important. Figure 3 shows a chromatographic system proposed by Perrut (1982, 1983, 1984). After elution and detection, the mobile phase together with the dissolved substance is heated and expanded. By these means the solvent power of the mobile phase is reduced and the substance precipitates; it is collected in one of several collecting vessels, one for each substance. The substances are removed after sufficient quantities of each of the substances have accumulated after several injections. Before the expanded mobile phase can be recycled by a cycle pump, it is passed through an adsorbing bed, where remaining quantities of the dissolved substances and other un wanted substances (as, for example, water) are removed. As in any solvent cycle, make up gas must be added, and a small part of the solvent must be removed for disposal or for special cleaning (Brunner, 1994).

In preparative SFC so far mostly extracts from plants like lemon peel oil, tocopherols from wheat germ or ubichinones have been treated. Unsaturated fatty acids from fish oil, mostly processed as esters, is a subject investigated heavily in recent years (Davarnejad *et al.*, 2008).

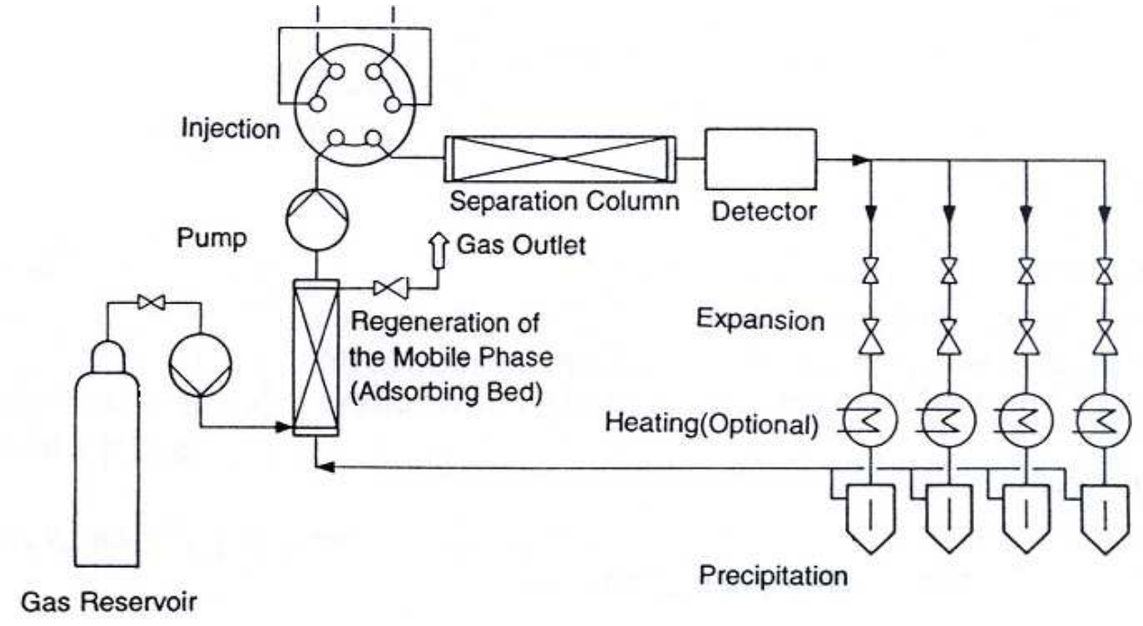

Fig. 3. Flow scheme of a preparative SFC with recycle of the mobile phase (Perrut, 1982, 1983, 1984).

More specialized applications deal with polymers or the fractionation of coal tar. Berger and Perrut (1988) have reviewed the applications of preparative SFC.

2.4 Injection techniques

Injection of the mixture to be separated is accomplished for analytical purposes by sample loops which may be filled at ambient pressure and are injected into the flow of the mobile phase by switching a multiposition valve in the appropriate position. Such valves can be manufactured as linear moving or rotating valve, as shown in Figure 4.

For preparative separations the feed is pumped by metering pumps into the flow of the mobile phase. Intensive mixing can be achieved in line by static mixers. Other possibilities comprise a column, where the mixture is placed under ambient pressure and is then eluted by the mobile phase and transported to the separation column, or a combination with a gas extraction unit. The extract of the gas extraction process can be directly passed through the chromatographic column. The separated substances can be collected. The extract from an extraction unit is diluted with respect to the interesting compounds. It can be collected on a column and after some time transported to the chromatographic separation (Brunner, 1994). This operational mode is illustrated in Figure 5.

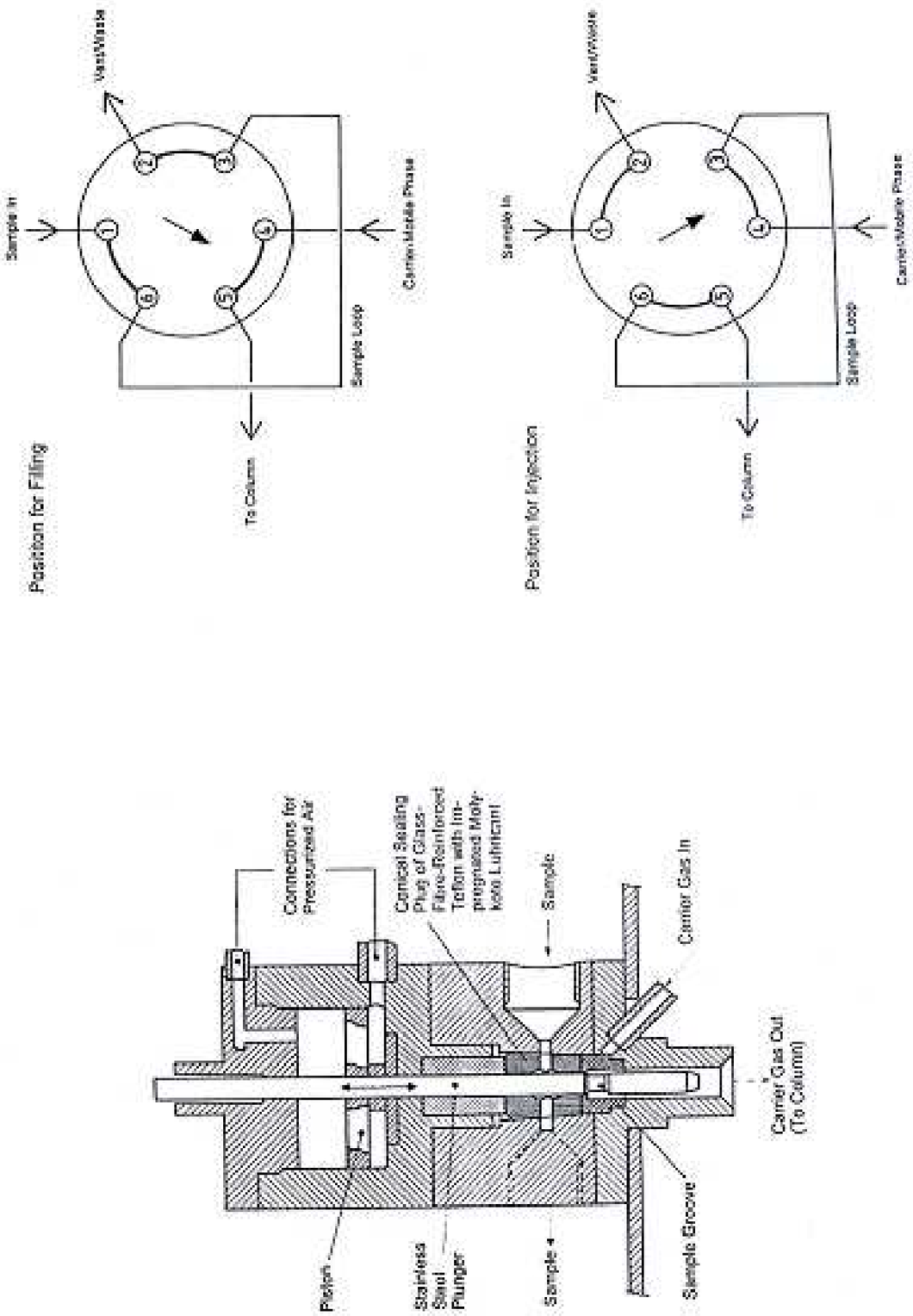

Fig. 4. Multiposition-valves for injection of samples into a SFC (Brunner, 1994).

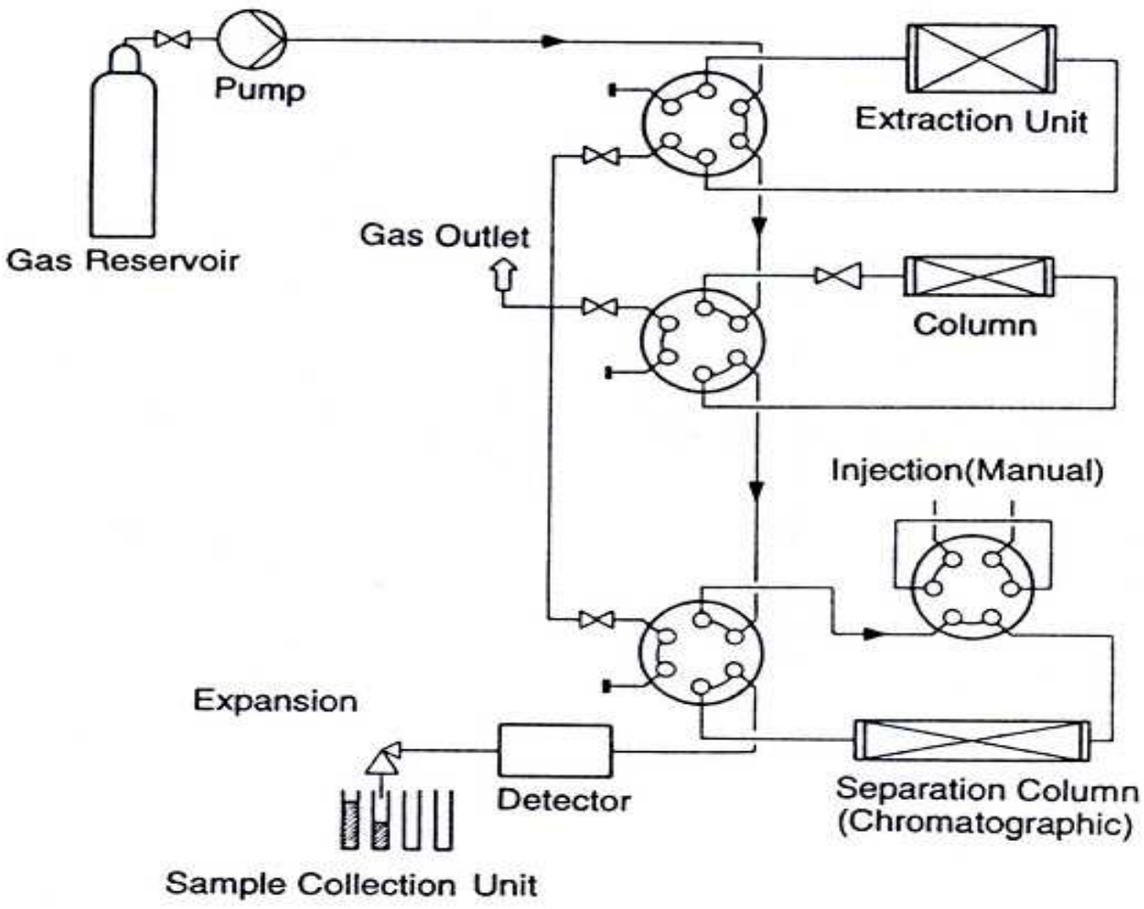

Fig. 5. Coupling of SFE with SFC. Concentration of compounds in a collecting column (Yamauchi and Saito, 1990).

According to this type of apparatus, some examples in details have been shown by Brunner (1994).

3. General gas chromatography apparatus

The obtained products from various types of supercritical apparatuses (such as phase equilibrium and rate test apparatus) should be analyzed. However, different types of analyzer can be used, but the conventional GC with a suitable column has widely been recommended. Although several columns for detecting a lot of components have been designed and fabricated by some companies, but due to lacking of suitable columns for some components or unclear peaks obtained from some columns, an extra process (such as esterification of the fractionated fish oil) before GC analyze is required. In this application, the samples obtained from the supercritical extraction apparatus are not under pressure or their pressures have broken down by a damper (in the online GC).

Since this type of apparatus has been explained in detail in the other chapters, therefore an example from its application is illustrated in this section.

3.1 Triacylglycerols analysis

3.1.1 Introduction

Most of the fatty acids of palm oil are present as triacylglycerols (TAGs). The different placements of fatty acids and fatty acid types on the glycerol molecule produce a number of different TAGs. About 7 to 10 percent of saturated TAGs are predominantly tripalmitin (Karleskind and Wolff, 1996) and the fully unsaturated triglycerides constitute 6 to 12 percent (Karleskind and Wolff, 1996; Kifli, 1981). The TAGs in palm oil are partially defined most as of the physical characteristics of the palm oil such as the melting point and crystallization behavior (Sambanthamurthi *et al.*, 2000). Detailed information about Malaysian tenera palm oil TAGs have been given in various references (Sambanthamurthi *et al.*, 2000; Kifli, 1981; Sow, 1979). Fatouh *et al.* (2007) studied the supercritical extraction of TAGs from buffalo butter oil using carbon dioxide solvent. They concluded that increasing the pressure and temperature of the extraction led to increasing the solvating power of the supercritical carbon dioxide. In these studies, the TAGs were extracted during the early stage of the fractionation thereby creating low-melting fractions. Conversely, TAGs were concentrated in the fractions (i.e. high-melting fractions) obtained towards the end of the process.

According to the literature, mole fraction solubility data of pure triacylglycerols in CO_2 were reported at temperatures of 40, 60 and 80 ^oC in the range of 10^{-10} to 10^{-2} (Soares *et al.*, 2007). These data depended on the type of triacylglycerols and operating pressure. That means high pressure had a good effect on solubility of triacylglycerols in CO_2. Furthermore, tricaprylin had the higher solubility in CO_2 (around 10^{-2} at high pressures) than the rest (Jensen and Mollerup, 1997; Bamberger *et al.*, 1988; Weber *et al.*, 1999).

In this research, phase equilibrium of TAGs from crude palm oil in sub and slightly supercritical CO_2 is studied. For this purpose, the samples obtained from the phase equilibrium supercritical fluid extraction apparatus are carefully analyzed by a HPLC in terms of TAGs.

3.1.2 Experiment

3.1.2.1 Materials and methods

Crude palm oil and CO_2 (99.99%) respectively as feed and solvent were purchased from United Oil Palm Industries, Nibong Tebal, Malaysia and Mox Sdn. Bhd. 1,3-dipalmitoyl-2-oleoyl-glycerol (99%) (POP), 1,2-dioleoyl-3-palmitoyl-rac-glycerol (99%) (POO) and 1,2-dioleoyl-3-stearoyl-rac-glycerol (99%) (SOO) as standards were purchased from Sigma-Aldrich. Acetone (99.8%) and acetonitrile (99.99%) as solvent and mobile phase were obtained from J.T.Baker and Fisher Scientific, respectively.

The phase equilibrium supercritical fluid extraction apparatus and calculations procedure have been shown and explained in detail in the references (Davarnejad, 2010; Davarnejad *et al.*, 2010; Davarnejad *et al.*, 2009).

The operating conditions were set at 10.8, 7.0, 2.7 and 1.7 MPa for temperature of 80 ^oC, 11.1, 7.6, 6.1 and 1.1 MPa for temperature of 100 ^oC and 7.4, 5.4, 3.3 and 0.6 MPa for temperature of 120 ^oC.

For TAGs analysis, the following stages are carried out step by step using a HPLC (brand: Shimadzu, Japan; model: 10 Series) which is equipped with a capillary column (Aglient Lichrosphere RP-18250×4 mn) and oven temperature is also set at 50 ^{o}C.

1. The standard solutions of POO, POP and SOO with concentrations of 10, 25 and 50 ppm are prepared by diluting these chemicals with acetone separately. These solutions are prepared by diluting these chemicals which are initially prepared at a concentration of 200 ppm.
2. A mobile phase containing 75% acetone and 25% acetonitrile (v/v) is prepared.
3. The chromatography interface, vacuum degasser, pump (LC-8A pump with maximum flow rate of 150 cm^3/min) and refractive index (RI) detector are switched on respectively.
4. The computer, printer and GC are switched on.

The HPLC diagram obtained from each sample is shown as following and compared with the diagrams obtained from the standard materials. Then, by applying the standard equation:

Samples concentration (ppm)=sample area/standard area ×standard concentration

Samples concentration in terms of each substance is calculated.

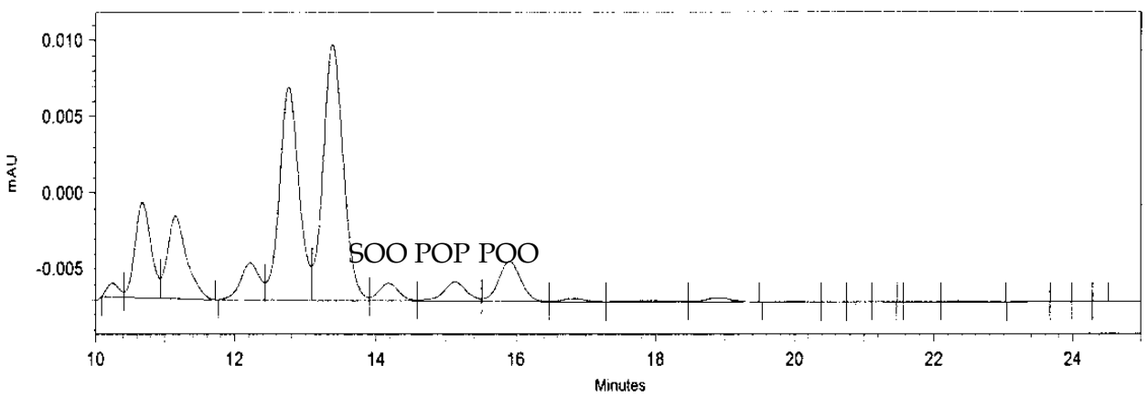

Fig. 6. HPLC chromatogram of crude palm oil TAGs for vapor phase at pressure of 5.4 MPa and temperature of 120 ^{o}C.

3.1.3 Results and discussion

Two-phase equilibrium data based on different TAGs are illustrated in Tables 1-3 as following:

Pressure (MPa)	Liquid phase, CO_2 mole fraction	Vapor phase, CO_2 mole fraction
T=80 °C		
10.8	0.9	1
7.0	0.9	1
2.7	1	1
1.7	1	1
T=100 °C		
11.1	1	1
7.6	1	1
6.1	1	1
1.1	1	1
T=120 °C		
7.4	0.9	1
5.4	0.9	1
3.3	1	1
0.6	1	1

Table 1. Two-phase equilibrium calculated data based on CO_2 at 80,100 and 120 °C in order to POP analysis

Pressure (MPa)	Liquid phase, CO_2 mole fraction	Vapor phase, CO_2 mole fraction
T=80 °C		
10.8	0.9	1
7.0	0.9	1
2.7	1	1
1.7	1	1
T=100 °C		
11.1	1	1
7.6	1	1
6.1	1	1
1.1	1	1
T=120 °C		
7.4	0.9	1
5.4	0.9	1
3.3	1	1
0.6	1	1

Table 2. Two-phase equilibrium calculated data based on CO_2 at 80,100 and 120 °C in order to POO analysis

Pressure (MPa)	Liquid phase, CO_2 mole fraction	Vapor phase, CO_2 mole fraction
T=80 °C		
10.8	0.9	1
7.0	0.9	1
2.7	1	1
1.7	1	1
T=100 °C		
11.1	0.9	1
7.6	1	1
6.1	1	1
1.1	1	1
T=120 °C		
7.4	0.9	1
5.4	0.9	1
3.3	1	1
0.6	1	1

Table 3. Two-phase equilibrium calculated data based on CO_2 at 80,100 and 120 °C in order to SOO analysis

The mole fractions of CO_2 in the equilibrium supercritical extraction of the POP substance for the liquid and vapor phases are shown in Table 1. The POP substance mole fractions in the vapor phase increased with increasing the pressure at 80 °C. This trend was also observed in the liquid phase. A regular trend was not observed in the vapor and liquid phases at 100 °C and 120 °C. It seems that high temperatures (such as 100 °C and 120 °C) to cause this irregularity however, the maximum solubility was carried out at 120 °C which is reasonable because high temperature increases TAGs solubilities in CO_2 (Soares *et al.*, 2007). The maximum solubility of POP substance in CO_2 was observed at 80 °C and 10.8 MPa, at 100 °C and 7.6 MPa as well as at 120 °C and 5.4 MPa, respectively. The optimum conditions around these operating regions for the maximum solubility of the POP substance were at 120 °C and 5.4 MPa.

The mole fractions of CO_2 in the equilibrium supercritical extraction of the POO substance for the liquid and vapor phases are shown in Table 2. The POO substance mole fractions in the vapor phase increased with increasing the pressure at 80 °C. This trend was also observed in the liquid phase. A regular trend was not observed in the vapor and liquid phases at 100 °C and 120 °C. Since POO is from TAGs group, this subject is reasonable as it was legitimized for POP. The maximum solubility of the POO substance was observed at 7.6 MPa and 100 °C as well as at 5.4 MPa and 120 °C, respectively. The optimum conditions around these operating regions for the maximum solubility of the POO substance was observed at 120 °C and 5.4 MPa.

The mole fractions of CO_2 in the equilibrium supercritical extraction of the SOO substance for the liquid and vapor phases are shown in Table 3. The SOO substance mole fractions in

the vapor phase increased with increasing the pressure at 80 °C; this trend was also observed in the liquid phase. A regular trend was not observed in the vapor and liquid phases at 100 °C and 120 °C. Since SOO also is from TAGs group, this subject is reasonable as it was legitimized for POP and POO. The maximum solubility of the SOO substance was observed at 7.6 MPa and 100 °C as well as at 5.4 MPa and 120 °C. The optimum conditions around these operating regions for the maximum solubility of the SOO substance was observed at 120 °C and 5.4 MPa.

The experimental results showed that although highest extraction using CO_2 was observed at 120 °C and 5.4 MPa for all of the TAGs substances but, maximum solubilities at the mentioned operating conditions were obtained for POP (y=1), POO (y=1) and SOO (y=1) respectively. The obtained result was confirmed by the overall conclusion which stated that the fats and oils composed of shorter chain fatty acids are more soluble than those with longer chain fatty acids (Fatouh *et al.*, 2007). Soares *et al.* (2007) also illustrated that high temperature had a desired effect on TAGs solubility in CO_2 as this output was obtained from the current research as well where this statement supported the results above.

Tan *et al.* (2008) also confirmed that high temperature had a positive effect on TAGs extraction using CO_2. They observed that monounsaturated fatty acids (such as POP) had a maximum yield of extraction in supercritical CO_2 as this output was obtained from current research. That means POP had a higher solubility in CO_2 in comparison with POO and SOO. Although they proposed high pressures for supercritical extraction of TAGs but they conducted their experiments at operating pressures more than 10 MPa and they studied yield of extraction.

3.1.4 Conclusions

In the mutual solubility study of TAGs from crude palm oil which is related to the experiment, it was shown that by using the supercritical fluid extraction process, the highest mole fraction percentage of TAGs solubility was obtained at approximately 2.2% (at 5.4 MPa and 120 °C). According to our calculations, the data slightly varied in seventh or eighth decimal points for the reported data due to the chromatography influence. In order to the calculations procedure, the significant part of these calculated data (solubility data) is prepared from the pressure increments. Furthermore, high temperature increased TAGs solubility in CO_2.

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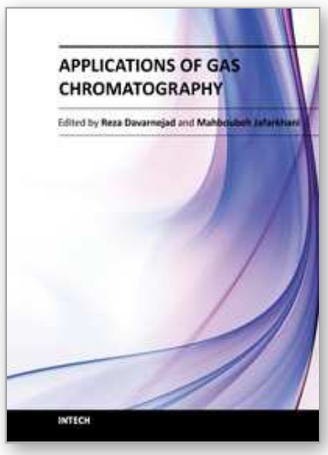

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This valuable book aims to provide a connection between various chromatography techniques and different processes. Authors applied these techniques in supercritical technology, medical, environmental, physique and chemical processes. Most of them prepared mathematical support (such as correlation) for their original results obtained from the chromatography techniques. Since chromatography techniques (such as GC, HPLC & etc) are separating and analyzing methods, this chapters will help other researchers and young scientists to choose a suitable chromatography technique. Furthermore, this book illustrates the newest challenges in this area.

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