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Review of the Chemical Separation

Mokhtar Mohamedalamin Mokhtar [1, 2], Jianfeng Li [1], Zhiping Du[1], Fangqin Cheng[1*]

[1]Institute of Resources and Environmental Engineering, Shanxi University, China
[2]General Plant Protection and Biological Control Directorate -Ministry of Agriculture, Animal Resources and Irrigation, Khartoum State, Khartoum 11111, Sudan
* Corresponding Author

Abstract**—** *The chemical separation is used to reduce the quantity of potentially toxic or hazardous materials discharged to the environment. In addition, separations that lead to recycle, recovery, or reuse of materials also prevent discharge. It is can assists in resolving some of the environmental challenges, so it became more important for researchers, in particular, environmental specialists. Hence, this article aimed to collection information about the chemical separation to help the researchers to resolve the environmental problems. Overall, it can concluded that the extraction is the first step to separate the desired substance from the raw materials and there are various methods for extraction such as sublimation, distillation method and solvent extraction. Correspondently, there are many types of separation processes, the most widely used method is the chromatography method.*

Keywords**—** ***chemical separation, gas chromatography, separation methods, solvent extraction, environment.***

I. INTRODUCTION

Separation processes, or processes that use physical, chemical, or electrical forces to isolate or concentrate selected constituents of a mixture, are essential to the chemical, petroleum refining, and materials processing industries (NRC, 1998). Chemical separations are of central importance in many areas of environmental science, whether it is the clean-up of polluted water or soil, the treatment of discharge streams from chemical processes, or modification of a specific process to decrease its environmental impact (Noble and Terry, 2004). Hence, this report will supply the reader with principle information about separation processes; functions, and methods of separation, more detail about extraction and chromatography methods, in particular, gas chromatography; classification, fundamentals and advantages and disadvantages of gas chromatography.

II. THE FUNCTIONS OF SEPARATION PROCESSES

Separation processes are used for three primary functions: purification, concentration, and fractionation. Purification is the removal of undesired components in a feed mixture from the desired species. For example, acid gases, such as sulfur dioxide and nitrogen oxides, must be removed from power plant combustion gas effluents before being discharged into the atmosphere. Concentration is performed to obtain a higher proportion of desired components that are initially dilute in a feed stream. An example is the concentration of metals present in an electroplating process by removal of water. This separation allows one to recycle the metals back to the electroplating process rather than discharge them to the environment. Lastly, in fractionation, a feed stream of two or more components is segregated into product streams of different components, typically relatively pure streams of each component. The separation of radioactive waste with short half-lives from that having much longer half-lives facilitates proper handling and storage (Noble and Terry, 2004).

III. SEPARATION METHODS

There are many different separation techniques which may be broadly classified into processes of mechanical separation and separation by diffusion or others, include various types of separation processes (Taulbee and Maroto-Valer, 2000; Moskvin, 2016 and Ibrahim, 2018), for example: extraction, chromatography, crystallization, filtration, Decantation and Sublimation. We will discuss extraction and chromatography, because they are widely used.

3.1. EXTRACTION:

Extraction is the first step to separate the desired substance from the raw materials. There are many methods for extraction such as solvent extraction, distillation method, pressing and sublimation. However, the most widely used method is solvent extraction (Zhang *et al*., 2018). These methods were summarized in (Table 1.). Soxhlet extraction and distillation method will be discussed as sample for extraction methods.

Table 1: A brief summary of various extraction methods for natural products (Zhang, et al., 2018).

Method	**Solvent**	**Temperature**	**Pressure**	**Time**	**Volume of organic solvent consumed**	**Polarity of natural products extracted**
Maceration	Water, aqueous and non-aqueous solvents	Room temperature	Atmospheric	Long	Large	Dependent on extracting solvent
Percolation	Water, aqueous and non-aqueous solvents	Room temperature, occasionally under heat	Atmospheric	Long	Large	Dependent on extracting solvent
Decoction	Water	Under heat	Atmospheric	Moderate	None	Polar compounds
Reflux extraction	Aqueous and non-aqueous solvents	Under heat	Atmospheric	Moderate	Moderate	Dependent on extracting solvent
Soxhlet	extraction Organic solvents	Under heat	Atmospheric	Long	Moderate	Dependent on extracting solvent
Pressurized liquid extraction	Water, aqueous and non-aqueous solvents	Under heat	High	Short	Small	Dependent on extracting solvent
Supercritical fluid extraction	Supercritical fluid (usually S-CO2), sometimes with modifier	Near room temperature	High	Short	None or small	Dependent on extracting solvent
Ultrasound assisted extraction	Water, aqueous and non-aqueous solvents	Room temperature, or under heat	Atmospheric	Short	Moderate	Dependent on extracting solvent
Microwave assisted extraction	Water, aqueous and non-aqueous solvents	Room temperature	Atmospheric	Short	None or moderate	Dependent on extracting Solvent
Pulsed electric field	Water, aqueous and non-aqueous	Room temperature, or	Atmospheric	Short	Moderate	Dependent on extracting Solvent

extraction	solvents	under heat				
Enzyme assisted extraction	Water, aqueous and non-aqueous solvents	Room temperature, or heated after enzyme treatment	Atmospheric	Moderate	Moderate	Dependent on extracting Solvent
Hydro distillation and steam distillation	Water	Under heat	Atmospheric	Long	None	Essential oil (usually non-polar)

3.1.1. SOXHLET EXTRACTION:

The extraction of organic compounds, including pesticides, polycyclic aromatic hydrocarbons and phenols from matrices (soils, sewage sludges, vegetables, plants), has historically been carried out by using Soxhlet extraction. The mode of operation of the extraction system is that organic solvent under the influence of heat (and pressure) will desorb, solvate and diffuse the organic compounds from the sample matrix allowing them to transfer into the bulk (organic) solvent (Dean, 2009).

The Soxhlet apparatus consists of a solvent reservoir, extractor body, an electric heat source and a water-cooled reflux condenser.

In the practice, the solid sample is loaded on the Soxhlet thimble and placed in the inner tube of the extractor body. The extractor body is then fitted to a round bottomed flask containing the chosen organic solvent and to a reflux condenser, then turn on heat source. The samples take few hours to extract, the process is repeated many times with new samples to get the required quantity (Dean, 2009; Elamin and Satti, 2012). Soxhlet extraction uses a range of organic solvents to remove organic compounds from samples, but solvent properties are very important to the extraction process (Prado *et al*., 2015 and Dean, 2009).

3.1.2. HYDRODISTILLATION:

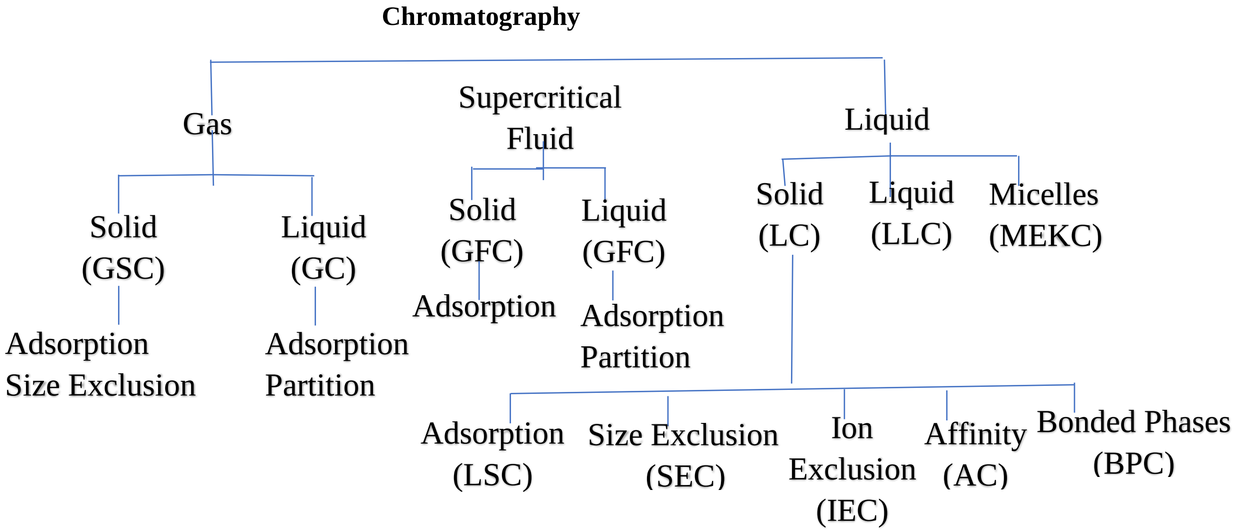
Hydrodistillation is a commonly used method of extracting essential oils from plant samples. This method is divided into the subcategories of steam distillation, water distillation, and a combination of water and steam distillation (Dilworth *et al*., 2017). The distillation apparatus consists of a vessel for plant material and water together (water distillation) or two vessels, one for plant material and other for water (water and steam distillation) or a vessel for plant material with steam inlet (steam distillation), a condenser to cool and condense the vapour produced and a method of collection, or ‘receiver’. The sample for extraction is placed in the distillation vessel. This is then heated to boiling point and the steam (water vapour) carries out the volatile oils (Clarke, 2008).

3.2. CHROMATOGRAPHY:

Chromatography is one of the most important analytical techniques. It allows the separation and subsequently the qualitative and quantitative analysis of complex mixtures, as long as the samples are volatile or soluble in a suitable solvent (Meyer, 2013). Chromatography has been defined by Irving *et al.* (1978) as “a method used primarily for separation of the components of a sample in which the components are distributed between two phases, one of which is stationary while the other moves. The stationary phase may be a solid, or a liquid supported on a solid, or a gel. The stationary phase may be packed in a column, spread as a layer, or distributed as a film, etc.; in these definitions Chromatographic bed is used as a general term to denote any of the different forms in which the stationary phase may be used. The mobile phase may be gaseous or liquid”.

3.2.1. CLASSIFICATION OF CHROMATOGRAPHY:

There are many forms of chromatography based on the different mobile phases, stationary phases, and supports that can be used in this method, which has led to a wide range of applications for this technique, they are summarized in the figure, then are explained as follow (Irving *et al*. 1978 and Moskvin, 2016).



The figure. Family tree of Chromatographic methods.

Source: Poole, C. F. (2000). Chromatography. In: Wilson, I. D.; K. Cooke, M. and Poole, C. F. (Ed.), Encyclopedia of Separation Science. Academic Press.

3.2.1.1. CLASSIFICATION ACCORDING TO PHASES USED:

In this classification the first word specifies the mobile phase and the second the stationary phase. A liquid stationary phase is supported on a solid.

3.2.1.1.1. Gas Chromatography (GC), we will reach it in detail later.

3.2.1.1.1.1. Gas-liquid chromatography (GLC)

3.2.1.1.1.2 *Gas-solid chromatography* (GSC)

3.2.1.1.2. Liquid Chromatography (LC):

3.2.1.1.2.1. Liquid-liquid chromatography (LLC)

3.2.1.1.2.2. Liquid-solid chromatography (LSC)

3.2.1.1.2.3. Liquid-gel chromatography (LGC)

Liquid-gel chromatography includes gel-permeation and ion-exchange chromatography.

3.2.1.2. CLASSIFICATION ACCORDING TO MECHANISMS:

Main types of liquid chromatography based on their separation mechanisms include:

3.2.1.2.1. Adsorption chromatography

3.2.1.2.2. Partition chromatography

3.2.1.2.3. Ion-exchange chromatography (IEC)

3.2.1.2.4. Size-exclusion chromatography (SEC)

3.2.1.2.5. Affinity chromatography.

In addition to LC and GC, and the use of columns or open tubular supports, there are a variety of other chromatographic methods that can be used for chemical separation and analysis. Some important examples are supercritical fluid chromatography (SFC) and planar chromatography.

3.2.1.3. CLASSIFICATION ACCORDING TO TECHNIQUES USED:

3.2.1.3.1 Column chromatography (cc)

3.2.1.3.2 Open-tube chromatography

3.2.1.3.3 Paper chromatography (pc)

3.2.1.3.4. Thin-layer chromatography

Chromatography carried out in a layer of adsorbent spread on a support, e.g. a glass plate.

3.5 filament chromatography

3.2.1.3.2.2. GAS CHROMATOGRAPHY:

Gas chromatography comprises all chromatographic methods in which the moving phase is a gas.

The word chromatography itself implies that a stationary phase is present in addition to the moving phase, Includes:

- **A.** **Gas-liquid chromatography** comprises all gas-chromatographic methods in which the stationary phase is a liquid distributed on a solid support. Separation is achieved by partition of the components of a sample between the phases.
- **B.** **Gas-solid chromatography** comprises all gas chromatographic methods in which the stationary phase is an active solid (e.g. charcoal, molecular sieves). Separation is achieved by adsorption of the components of a sample.

3.2.1.3.2.2.1. FUNDAMENTALS OF GAS CHROMATOGRAPH:

A typical gas chromatograph consists of an injection port, a column, carrier gas flow control equipment, ovens and heaters for maintaining temperatures of the injection port and the column, an integrator chart recorder and a detector (Halord and Miller, 1998; AGC, 2002; Stauffer, 2008 and Qian *et al*., 2017).

3.2.1.3.2.2.1.1. INJECTION PORT:

A sample port is necessary for introducing the sample at the head of the column. Modern injection techniques often employ the use of heated sample ports through which the sample can be injected and vaporized in a near simultaneous fashion. A calibrated micro-syringe is used to deliver a sample volume in the range of a few microliters through a rubber septum and into the vaporization chamber.

3.2.1.3.2.2.1.2. CARRIER GAS:

The carrier gas plays an important role, and varies in the GC used. Carrier gas must be dry, free of oxygen and chemically inert mobile-phase employed in gas chromatography, Helium is most commonly used.

3.2.1.3.2.2.1.2. DETECTION SYSTEMS:

The detector is the device located at the end of the column which provides a quantitative measurement of the components of the mixture as they elute in combination with the carrier gas. These detection properties fall into two categories: bulk properties and specific properties. Bulk properties, which are also known as general properties, are properties that both the carrier gas and analytic possess but to different degrees. Specific properties, such as detectors that measure nitrogen-phosphorous content, have limited applications but compensate for this by their increased sensitivity. There are many detectors, but Mass Spectrometry Detectors detector are most powerful of all gas chromatography detectors (Table 2.).

Table 2: Typical gas chromatography detectors and their detection limits (Skoog, et al., 2007).

Type of Detector	**Applicable Samples**	**Detection Limit**
Mass Spectrometer (MS)	Tunable for any sample	0.25 to 100 pg
Flame Ionization (FID)	Hydrocarbons	1 pg/s
Thermal Conductivity (TCD)	Universal Detector	500 pg/ml
Electron-Capture (ECD)	Halogenated hydrocarbons	5 fg/s
Atomic Emission (AED)	Element-selective	1 pg
Chemiluminescence (CS)	Oxidizing reagent	Dark current of PMT
Photoionization (PID)	Vapor and gaseous Compounds	0.002 to 0.02 µg/L

3.2.1.3.2.2.1.3. COLUMN OVEN

The oven controls the temperature of the column. In GC, one takes advantage of both an interaction of the analyte with the stationary phase and the boiling point for separation of compounds. Oven temperature program rates can range from as little as 0.1 °C/min to the maximum temperature heating rate that the GC can provide. A rate of 2–10 °C/ min is most common.

3.2.1.3.2.2.1.4. OPEN TUBULAR COLUMNS AND PACKED COLUMNS

Open tubular columns, which are also known as capillary columns, come in two basic forms. The first is a wall-coated open tubular (WCOT) column and the second type is a support-coated open tubular (SCOT) column (Table 3.).

Table 3: Properties of gas chromatography columns (Thet and Woo, 2019).

	Type of Column			
Property	**Fused-silica wall-coated (FSWC)**	**wall-coated open tubular (WCOT)**	**support-coated open tubular (SCOT)**	**Packed**
Length	10 to 1000 m	10 to 1000 m	10 to 100 m	1 to 6 m
Inner Diameter	0.1 to 03 mm	0.25 to 0.75 mm	0.5 mm	2 to 4 mm

Efficiency (plates/m)	2000 to 4000	1000 to 4000	600 to 1200	500 to 1000
Sample Size	10 to 75 ng	10 to 1000 ng	10 to 1000 ng	10 to 10^6 ng
Pressure	Low	Low	Low	High
Speed	Fast	Fast	Fast	Slow
Flexibility	Yes	No	No	No
Inertness	Best	Good	fair	Poor

IV. CONCLUSION

Solvent extraction is widely used among the extraction methods. Chromatography is one of the most important analytical techniques. It is classified based on three types classification according to phases used includes gas chromatography (GC) and liquid chromatography (LC); according to mechanisms includes adsorption chromatography, partition chromatography, ion-exchange chromatography (IEC), size-exclusion chromatography (SEC), and affinity chromatography and according to techniques used includes column chromatography (CC), open-tube chromatography, paper chromatography (PC) and thin-layer chromatography.

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