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A Review on the Green Technology and Innovative Techniques for the Extraction of Essential Oil and Active Constituents in Herbs and Spices

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ABSTRACT
Increasing demand for natural products from edible plants necessitated the need to replace conventional extraction techniques which are associated with the use of toxic solvents, high energy, and longer extraction time and solvent consumption. To address these problems, green extraction techniques are novel extraction methods that are presently receiving research attention. This study reports the review of green and innovative technology for extraction of essential oil and active ingredients from herbs and spices. The review describe the principle of operation of various extraction methods, merits and demerit of the different extraction approaches with a view of establishing the most suitable technique for extracting thermolabile compounds. The study revealed that the recovery of essential oil and active constituents depends on extraction processes largely used in chemical, biochemical and pharmaceutical industries. Among all methods reviewed, supercritical fluid extraction process (solid – fluid phase extraction) is new process that have the potential for efficient extraction of essential oil and active ingredients from herbs and spices in terms of non-usage of toxic solvent and energy saving.

Keywords: Extraction, Essential oil, Green Technology, Innovative Techniques.

1 INTRODUCTION

Essential oils are extracted from various parts plants (bark, seeds, fruits wood, roots, leaves, flowers, buds and branches). These essential oils are used in foods and beverages flavouring, as fragrances in perfumery, deodorants and cosmetics, in soap and detergents and as a component in pharmaceutical preparations, antiseptic and aromatherapy products (Xu *et al*., 2011).

Ginger (*Zingiber officinale)* is a common condiment for various foods and beverages. Traditionally it is used human ailments treatment, aid digestion. It used also used as an anthoxidant and anti-carcinogenic properties (Ghosh et al., 2011). Ginger rhizome bears ginger oil. The extraction of essential oil from ginger rhizome is mostly carried out using conventional methods.

The present increasing concern about the health and safety hazards strongly associated with the application and use of organic solvent in conventional extraction of soluble constituent of a solid matrix. These organic solvent often introduces toxic contaminants into the final products which must be removed. These concerns have stimulated research interest into finding a safer technique for the extraction of the desirable components of the solute as well reducing the thermal degradation and solvent contamination to the barest minimum (Asep *et al*., 2013).

Green technology is a technology based on a clean, eco-friendly and processing technique which serves as an alternative to organic solvents based extraction of oils and active constituents for the production of pharmaceutical drugs, cosmetics, perfumes and food industries. The principle of green technology is to reduce the use of harsh organic solvent whilst at the same time encourage the use of novel extraction techniques that are known to be friendly (Shah and Garg, 2014). This technology is an innovative technology which has to do with the design development and implementation of chemical products and processes to reduce or eliminate the use and generation of substances hazardous to human health and the environment (Shah and Garg, 2014).

This focus of this review to give explicit account of the principles, prospect and challenges of different green extraction technique for the extraction of essential oil from ginger rhizome.

2 METHODS OF EXTRACTION

The extraction of plant constituent from their matrix are usually carried out using numerous methods such as traditional, classical or conventional extraction methods and advanced, modern, non-conventional or green extraction methods.

2.1 TRADITIONAL EXTRACTION METHODS

The traditional methods of extraction of bioactive compounds are maceration, infusion, percolation, decoction, soxhlet (solid-liquid) extraction and hydro distillation. These methods are based on the

characteristics of solvent power (correct choices of solvent being used and the application heat and/or agitation to increase solubility of the desired compounds and to also improve mass transfer (Azmir *et al.*, 2013).

a. MACERATION, INFUSION, PERCOLATION, AND DECOCTION

Maceration is a well-known and inexpensive technique for obtaining essential oils and volatiles on a small scale. This process consists of the following stages:

- The first stage is to grind the plant materials into coarse or powdered sizes to increase the surface area. (Azmir *et al.*, 2013).
- Secondly, the pulverized plant is soaked in a suitable solvent in a stoppered vessel. The mixture is left for several days (3 days) at room temperature to open the plants cell wall to release the soluble components with occasional agitation. Occasional shaking facilitates extraction by increasing diffusion and removing concentrated solution from the sample surface so that a fresh solvent is contacted for more extraction yield (Azmir *et al.*, 2013; Chewaka, 2016).
- Thirdly, the miscella is strained off and the left over plant sample is squeezed out to obtain more of the liquid. The liquid is then mixed and separated by filtration (Azmir *et al.*, 2013).

The process used for maceration also applies to infusion and decoction where both can be soaked in cold or boiled water. The period of maceration is shorter and the sample is boiled in specified volume of water for a defined time. Decoction is suitable for extracting thermolabile constituents resulting in a more oil-soluble compound when compared to maceration and infusion (Azwanida, 2015).

For percolation, the dried powdered material is soaked initially in a solvent in a unique equipment called percolator and macerated for 2 hours. Additional solvent is then poured on top of the plant material and allowed to percolate slowly out through the bottom of the percolator at a moderate rate. The mixture is evaporated to obtain a concentrated extract during the extraction process. Additional filtration is not required because there is a filter at the outlet of the percolator (Azwanida, 2015; Chewaka, 2016).

Advantages

- They are easiest and simple methods of extraction.

Disadvantages

- They can be quite time consuming from few hours to several days.
- Organic wastes are generated as the solvent has to be separated, hence it requires waste management.

b. SOLVENT EXTRACTION

This is also known as solid - liquid extraction or leaching or lixiviation or soxhlet extraction. It was developed by a German chemist physiologist Franz Ritter von Sohxlet in 1879. The apparatus was named after him as soxhlet extractor (Zygler *et al.*, 2012; Azmir *et al.*, 2013). The efficiency of extraction depends on a number of interdependent variables which includes the solubility of solute in a solvent, the matrix and mass transfer effects. Other factors include the nature of the matrix, choice of solvent, solid-liquid ratio, temperature, pressure and extraction time. The nature and type of sample goes a long way in determining the number of steps in the sample preparation process. According to Zygler *et al.*, (2011); Azmir *et al.*, (2013) soxhlet apparatus is primarily made up of a distillation flask, thimble (sample holder), extractor and condenser. The system is also supported by structures such as electro-thermal heating mantle and a chiller (air or water) unit

The first step is to homogenize, grind and weigh the sample. The ground sample is packed in a filter paper and transferred into a cellulose sample holder. The sample holder is placed into the extraction chamber. Flask, placed on a heating mantle and the extractor containing the thimble is placed on the round bottom flask and a condenser is mounted in the extractor. The in and out section of the condenser is connected to a tap and drain or a chiller. When the solvent is heated or vaporizes into the extractor through the side arm tube and is liquefied by condenser drop back into the thimble. When the liquid contents aspirates or is emptied into the bottom flask with the extractor solutes. Fresh solvent vapours are cycled again onto the samples. This process is repeated over and over again until complete extraction is attained. After extraction, the solvent is recovered using a rotary evaporator (Zygler *et al.*, 2012; Azwanida, 2015; Chewaka, 2016).

Advantages

- Equilibrium transfer is freely displaced due to the repetitive contact between solvent and the sample.
- Relatively high extraction temperature is maintained from the heat of the distillation flask during the entire extraction.
- Simplicity of process thereby requiring minimal training.
- Filtration of the extract is not required after leaching.
- Equipment capital cost is relatively low.
- Wide varieties of compound can be effectively extracted from solids materials (Zygler *et al.*, 2012; Shams *et al.*, 2015; Azwanida, 2015).

Disadvantages

- Long extraction time.
- Requires high volume of solvents and therefore a small amount of solvent residue is left behind.
- Possibility of thermal degradation of target compounds during the long extraction process.
- The extract has to be concentrated after extraction.
- Lacks selectivity and high purity is required.
- Emission of toxic fumes which causes allergies.
- Not environmental friendly thereby contributing to pollution problem in comparison to advance extraction method (Zygler *et al.*, 2012; Azwanida, 2015).

C. HYDRO-DISTILLATION (HD) METHOD

Hydro-distillation is one of the simplest and oldest traditional methods for extraction of essential oils from plant where organic solvents are not used. Hydro distillation can be subdivided into water distillation, water and steam distillation and direct steam distillation (Azmir *et al.*, 2013; Uitterhaegen, 2014).

i. WATER DISTILLATION

Plant material and water are added to the still and boiled for several hours. The vapour arising from the still contains both the vapour and volatiles which is passed through a condenser and thus both water and essential oil are collected in a separating funnel. The fluid forms an insoluble mixture, gravity separation occurs and essential oil accumulates at the top layer which can be collected (Uitterhaegen, 2014).

Advantages

- Method is simple and solvent is available and inexpensive.

Disadvantages

- The thermolabile compounds and oil risk being degraded Low quality) due to the fact that they are subjected to high temperature and water for a long time which causes hydrolysis and polymerization of sensitive compounds.

ii. WATER-STEAM DISTILLATION METHOD

This method is to resolve the problem of thermal degradation of some compounds associated with distillation. The plant material is placed on a perforated grid at some distance above the water level so that only water vapor or steam comes into contact with the material (Uitterhaegen, 2014).

Advantages

- Exhibits low capital cost, simple design and higher quality oil.

Disadvantage

- Lower capacity of plant materials and lower yields since lumping of materials form an obstruction to the material flow steam.

ii. STEAM DISTILLATION METHOD

In a separation process for temperature sensitive materials like oils, resins and hydrocarbons which are insoluble in water and may decompose at their boiling point. It enables a compound or mixture of compounds to be distilled at a temperature substantially below the boiling point of the individual constituents, since the steam is not produced within the distillate apparatus but in an external boiler (Uitterhaegen, 2014).

Advantages

- Relatively cheap process to operate.
- Controllable flow and quantity of steam.
- Faster and more energy efficient distillation possible.

Disadvantage

- Lower yields as a result of incomplete extraction.
- High operating temperature and consequent breakdown of thermally labile compounds.
- Requires post extraction process to remove water.
- Automated and commercially automated extractor system.

2.2 GREEN TECHHNOLOGY OF EXTRACTION

The principal goals of "Green Technology" referred to as sample preparation tools are to reduce or eliminate chemically toxic solvents or acids related impact on human health, search for alternative production methods that is environmentally friendly and energy efficient, extract multiple compounds at the same time and, increasing automation (Herrero *et al.*, 2012; Shah and Garg, 2014).

The conventional extraction methods have some major challenges which are longer extraction time, requirement of costly and high purity solvents, low extraction selectivity, thermal decomposition of thermolabile compounds, and distillation and use of the large volume of solvent present safety concern and environmental risk (Shams *et al.*, 2013). In order to overcome the problems of traditional extraction methods, green and clean methods of extraction known as advanced/modern extraction methods were introduced that will provide natural products free of toxicity (Shah and Garg, 2014).

The modern methods include

- Pressurized Liquid Extraction (PLE)
- Subcritical Water Extraction (SWE)
- Supercritical Fluid Extraction (SFE)

- Microwave-Assisted Extraction (MAE)
- Ultrasound-Assisted Extraction (UAE)
- High Pressure Soxhlet Extraction (HPSE)

a. PRESSURIZED LIQUID EXTRACTION (PLE)

PLE is also known by several names such as accelerated solvent extraction (ASE), pressurized fluid extraction (PFE) or pressurized solvent extraction (PSE) (Zygler *et al.*, 2012; Azmir *et al.*, 2013; Shams *et al.*, 2015; Chewaka, 2016).

The PLE relies on the basic principle of using liquid solvents at high temperatures and pressures to carry out extractions and these parameters are kept below their critical conditions so that its liquid state are maintained when the cell approaches the supercritical region. The application of these conditions results in a faster extraction processes where increased extraction yields are obtained with minimal amount of organic solvents hence improved mass transfer kinetics. When the temperature is increased, the solubility of the analytes in the solvent also increases while the solvent viscosity and surface tension decreases and hence the solvent will have a better penetration into the plant matrix (Herrero *et al.*, 2012; Azmir *et al.*, 2013; Chewaka, 2016). Figure 1 is a typical pressurized fluid extraction set-up.

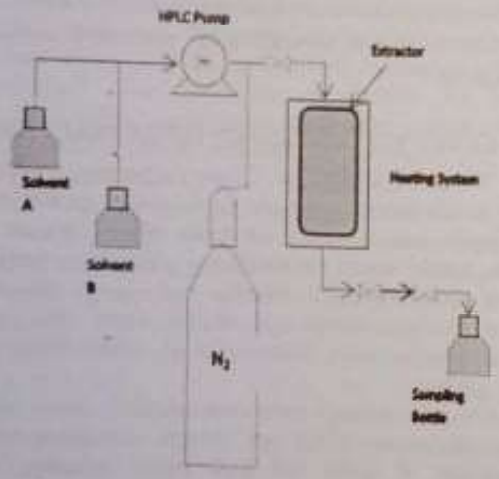

Figure 1: Basic pressurized liquid extraction set-up (Osorio and Meireless, 2013)

Advantages

- The process is relatively simple compared to SFE, offers a better precision, reproducibility, more economical and environment friendly as a result of the use of minimal amount of organic solvent and no solvent disposal (i.e. enable a solvent free extractable matter/residue hence its recognition as a green extraction method).
- Solvent penetration into matrix is increased when the temperature and pressure is elevated and metabolite solubility is efficiently achieved, hence there is reduced solvent consumption, decreased extraction time, and high extraction efficiencies (yield) as it accelerates extraction kinetics in terms of its mass transfer and diffusion rates (Zygler *et al.*, 2012; Herrero *et al.*, 2012; Shams *et al.*, 2015; Chewaka, 2016).
- Elevated temperature accelerates the extraction kinetics, mass transfer or diffusion rate because increases in the analytes solubility in the solvent decreases while high pressure maintains the solvent in its liquid state (below its boiling point) thus analyzing safe and rigid extraction at elevated temperatures and pressures (Herrero *et al.*, 2012; Shams *et al.*, 2015; Chewaka, 2016).
- The selectivity of extracted components is increased when processing operating parameters are tuned.
- Inert atmosphere reduces risk of oxidizing of compounds compared to conventional extraction process.
- Possibility of automation is achievable at low extraction cycle temperature since loss of low boiling compounds is avoidable).

Disadvantages

- The efficiency of the extractor is drastically reduced for extraction process taking place at a condition close to supercritical state of the extractant.
- Variable factors such as extraction time, pressure, temperature, solvent suitability and sample amount have to be optimized and determined for each sample.
- Thermal degradation is a cause for concern especially for thermolabile compounds when the temperature is elevated depending on the solvent used.
- Post extraction clean up step is necessary (Azmir *et al.*, 2012; Shams *et al.*, 2015; Chewaka, 2016).

b. SUBCRITICAL WATER EXTRACTION (SWE)

SWE is also known as pressurized hot solvent extraction (PHSE), enhanced solvent extraction (ESE) and high pressure solvent (soxhlet) extraction (HPSE). SWE uses the principle of PLE by employing hot water as pressurized liquid (under pressure) in addition to providing high extraction yields. To maintain the water in its liquid in the course of the extraction process, temperatures between the boiling point of water i.e. 100°C and lower than its critical temperature of 374°C are usually used while pressures between 3.5 to 20 MPa are

employed. Analytes diffusion is facilitated when the temperature is increased to 200°C than at 25°C which favours mass transfer kinetics as a result of the disruption of intermolecular forces (van der Waals forces, H_2 bond and dipole interactions), decreases the viscosity of water enabling better penetration of the solid sample and decreases the surface tension. Factors that influence SWE in terms of yield are extraction temperature and pressure, time, water flow rate and addition of a modifier. Dielectric constant is the most important to be taken into consideration (ε) as increasing the water temperature weakens the H_2 bond resulting in a lower dielectric constant. Water is a polar solvent at room temperature (25°C) with dielectric constant of about 80, however, this value is reduced considerably to between 25–27 (similar to methanol and ethanol) when water is heated to about 250°C under enough pressure to maintain its liquid state (Herrero *et al.*, 2012; Shams *et al.*, 2015). See figure 2 of a schematic diagram of SWE.

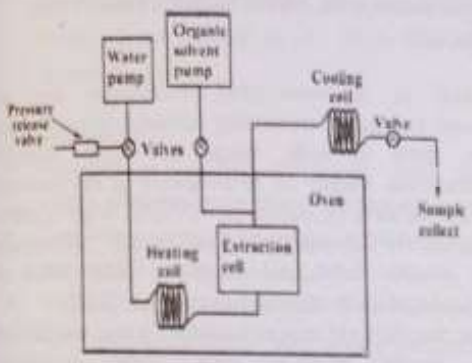

Figure 2: Schematic diagram of the basic subcritical water extraction equipment (Shams *et al.*, 2015).

Advantages

- Low extraction times.
- Higher quality of extracts.
- Lower cost of extractant agent.
- Environmentally cleaner technique.
- Better and adjustable selectivity by tuning the extraction temperature.

Disadvantage

- Lower water solubility of certain compounds and instability of some of them and/or matrices towards increase in temperature.

c. SUPERCRITICAL FLUID EXTRACTION (SFE)

Production of natural extracts with high potency of active ingredients uses SFE process which is a modern and environmentally benign separation technology that uses supercritical fluid as the extracting solvent for the production of essential oil and active constituents for use in perfumes, cosmetics, food and pharmaceutical industries (Vieira, 2014; Chewaka, 2016).

Supercritical fluid extraction (SFE) is the process of separating our component (the extractant) from another (the matrix) using supercritical fluid as the extracting solvent (Bhusmure et al., 2015). Supercritical fluid (SF) is defined in terms of its critical temperature and critical pressure known as critical point where the fluid is subjected to conditions that exceed its critical temperature and critical pressure (Xu *et al.*, 2011; Azmir *et al.*, 2013; Vieira, 2014). Critical point is defined as that characteristic temperature (Tc) and (Pc) above which both the gas and liquid phases cannot be distinguished. All fluids are characterized by critical points which are the conditions that determine the critical region of a substance defined in terms of critical temperature and pressure (Xu *et al.*, 2011; Azwanida, 2015).

Any fluid in this region has the physical properties of both the gas and the liquid state where the specific properties are no longer tenable. This infers that SF cannot easily be liquefied by temperature and pressure modification. SF when in critical region possess a gas-like property of high diffusivity, low viscosity and zero surface tension, and liquid-like property of density and solvation power attributed the suitability of the extracted compounds within a short time with higher yields (Azmir *et al.*, 2013; Vieira, 2014). The flow diagram is shown in Figure 3.

Choice of SF as a solvent

- Solubility and diffusivity of the solute in the SF
- Viscosity of the fluid in the supercritical region
- Heat and mass transfer parameters of the solvent and
- All the conditions necessary to achieve the supercritical region of the fluid with regards to economic and safety aspects.

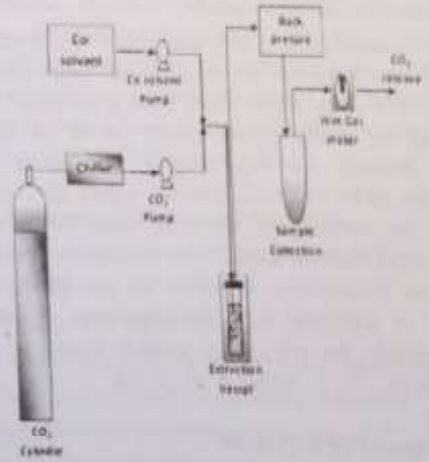

Figure 3: Flow diagram of supercritical carbon dioxide ($SC\text{-}CO_2$) extraction (Salleh *et al.*, 2013).

CO_2 is considered an ideal and most acceptable supercritical solvent for SFE and its choice is based on advantages such as its low critical temperature and pressure, inexpensive, non-toxic, odorless, tasteless, non-flammable gas, environmentally friendly and hence regarded as safe (GRAS). Thermal degradation is does not occur as the extraction takes place in the absence of oxygen and light. Because CO_2 is gaseous at room temperature makes analytes recovery very simple and extracted material is obtained by simply depressurizing or recycled to reduce Green House Gas emission to the atmosphere. The main drawback is that its polarity is low, therefore efficient extraction for polar solvents is difficult to obtain due to its poor ability to displace analytes from the matrix. This problem can be successfully overcome by employing a chemical modifier/entrainer/co-solvent. The addition of 1 to 10% or even 20% modifier (methanol or ethanol) enables CO_2 expands to extract polar compounds (Xu *et al*, 2011; Azmir *et al*., 2013; Moraes *et al*., 2013; Vieira, 2014; Shams *et al*., 2015; Azwanida, 2015; Bhusnure *et al*., 2015).

The major variable parameters are temperature, pressure, time of extraction, sample size, flow rate and modifier (type and amount) and solvent to feed ratio (Azmir *et al*., 2013).

BASIC SFE SYSTEM

It consists of the following

- CO_2 tank or vessel and pump to pressurize the solvent.
- Heat exchanger to sub-cool CO_2 before it reaches the pump to avoid cavitation.
- Flow Meter (dry/wet).
- Co-solvent vessel and pump.
- Mixer and gauge.
- Extraction cell/oven.
- Extract collection vessel.

The supercritical CO_2 and co-solvent were pumped extraction cell after the desired has been achieved at certain flow rates respectively for a given time. The CO_2 carrying the crude extract flows out of the extraction vessel through the top passing through a pressure reduction valve to precipitate the solute in a collection vessel. To ensure that all compounds precipitate, the supercritical fluid is heated in a separation vessel above saturation temperature to attain the gas phase and the material is collected from the separation vessel at the bottom while the gas phase solvent leaves at the top (Salleh *et al*., 2013; Azmir *et al*., 2013).

Advantages of SFE Process

- Higher extraction efficiency and reduced time of extraction.
- Increase solvent strength by tuning the temperature and pressure.
- Little organic solvent consumption makes it environmentally friendly, recycling and reflux of SF of SF which provides complete extraction and separation step is bypassed by depressurization of the SF through minimizing waste generation.
- Preservation of the thermolabile compounds is made possible because SFE is operated at low temperature.
- On-line integration with chromatographic process which is useful for detection of highly volatile compounds (Azmir *et al*., 2013; Shams *et al*., 2015).

Disadvantages

- Initial cost of the equipment is high.
- Several parameters are considered for optimization.
- Difficulties in extracting more polar compounds (Azwanida, 2015; Shams *et al*., 2015).

d. MICROWAVE ASSISTED EXTRACTION (MAE)

MAE is an innovative technology that uses microwave energy for extracting essential oils and natural extracts from a wide range of plant materials. Electromagnetic energy of microwaves in the frequency source of 300MHz (0.3GHz) to 300GHz when absorbed by materials is converted to heat energy. Microwaves possess electric field and magnetic field which are perpendicular to each other (Moraes *et al*., 2013).

The electric field causes heating via two mechanisms namely dipole rotation and ionic conduction simultaneously. Heat is generated when a solution offers a resistance to the migration of ions during ionic conduction. Dipole rotation is the realignment of the electric field of the molecules possessing a dipole moment in both the solvent and solid sample. The oscillation produces collisions within the surrounding molecules leading to liberation of thermal energy into the medium. During extraction, microwave heating disrupts weak hydrogen bonds enhances the migration of ions and solvent penetration into the matrix (Azmir *et al*., 2013; Azwanida, 2015; Shams *et al*., 2015; Chewaka, 2016).

Sample components absorb microwave energy according to their dielectric constants. Solvents employed for MAE are those that have high dielectric constant (Zygler *et al*., 2012; Azwanida, 2015).

The mechanism of MAE consists of three sequential stages:

Firstly, solutes are separated from the active sites of sample matrix under elevated temperature and pressure; Secondly, solvent diffuses across the sample matrix; and thirdly, the solutes are released from sample matrix to solvent (Azmir *et al*., 2013).

Factors affecting microwave extraction

- Solvent nature and volume.
- Extraction time.
- Microwave power and Irradiation time.
- Matrix characteristics.
- Temperature (Shams *et al.*, 2015).

Advantages

- It offers quicker heating and reduced extraction time for the extraction of bioactive materials, consumes less volume of solvents and less sample amount.
- Increased yield and selectivity than conventional extraction process.
- Essential oil composition free from residual solvents and contaminants.
- Minimal organic solvent use which makes it environment friendly and therefore recognized as green solvent (Azmir *et al.*, 2013; Chewaka, 2016; Azwanida, 2015).

Disadvantages

- Removal of solvents physically from the sample matrix after the extraction prior to further analysis.
- Possibility of inducing contaminants during subsequent purification step.
- Thermal degradation occurs if proper conditions are not used (frequency and longer extraction time) (Azwanida, 2015).

d. ULTRASOUND ASSISTED EXTRACTION (UAE)

The UAE technique uses mechanical vibration caused by sound waves with frequencies ranging from 20khz to 200khz. Compression and expansion cycles are created producing a phenomenon called cavitation, which means production, growth and collapse of bubbles. These bubbles convert kinetic energy of motion into heat. Mechanical disruption of the cells' wall occurs as a result of production, growth and collapse of bubbles to release the content and intense local heating of the liquid increases the diffusion of the extracts. The kinetic energy is introduced as a result of the collapse of cavitation bubbles near walls or at interface thus increasing the mass transfer across the solid-liquid interface. The mechanical effects of ultrasound facilitate penetration of solvent into cellular membrane walls thereby releasing the contents of the cells and improve mass transfer (Azwanida, 2015). The schematic diagram is shown in figure 4.

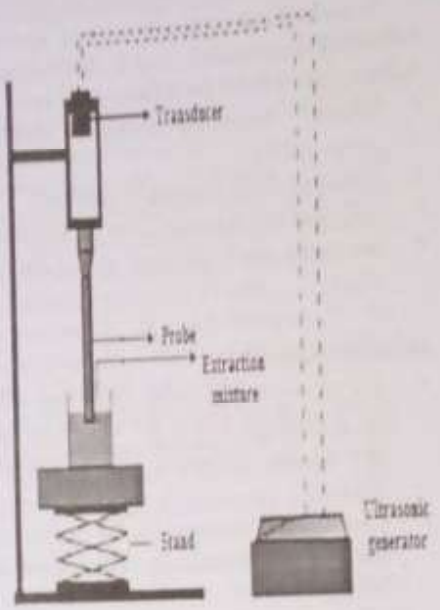

Figure 4: Schematic Representation of UAE Setup (Gupta et al., 2013).

Extraction Technique

UAE utilizes glassware that resembles sohxlet apparatus with some modifications as shown in the diagram above. The distillation flask is placed in a thermostatic bath and a sohxlet chamber is connected via Teflon connector. Ultrasound is applied by means of a sonifier equipped with a probe immersed in the thermostatic bath 1mm from the surface of the soxhlet chambers included as 45^{0}C from the bottom of the bath. The soxhlet chamber is filled with vapours which condense using a cooling system and drops on the sample. The sample in the extraction chamber is irradiated by ultrasound for a set period of time. The contact by the chamber is then unloaded once the extractant has reached the siphon height (Shams *et al.*, 2015).

Factors affecting Extraction Efficiency

The seven parameters identified that can affect extraction efficiency are:

- The nature of the tissue being extracted.
- Pretreatment of the tissue prior to extraction.
- The nature of components being extracted.
- The ultrasonic irradiation time.
- The intraparticle diffusion.
- The angle of inclination of the probe and
- The number of extraction cycles.

However, detailed optimization research shows that the number of cycles and the ultrasonic radiation

amplitude are the main valuables affecting the extraction process (Shams *et al*, 2015).

Advantages

Advantages of UAE include

- Shorter reaction/preparation time and solvent consumption.
- Usage of small amount of materials.
- Efficient and minimum cost on solvents and
- Increased yield (Kothari *et al*, 2013; Shams *et al*, 2015).

Disadvantages

- Use of ultrasound energy more than 20KHz may have an occasional effect on the active phytochemicals through the formation of free radicals (Azwanida, 2015).
- Undesirable changes on the active phytochemicals through the formations of free radicals (Azwanida, 2015).

e. HIGH PRESSURE SOXHLET EXTRACTION (HPSE)

This is also called fluid-phase extraction (FPE) with a solvent in a near critical state (sub- or supercritical fluid), an energy efficient, environmentally friendly method (Sovilj et al., 2011). It was first invented in 1980 and patented in United States in 1981 by G.W Jennings, R.H Wohleb and N.W Wohlers. Extraction is carried out under high pressure using heavy pyrex glass or stainless steel. Two major components in the extractor are a glass soxhlet extractor and the high pressure vessel, which comprises of a vertically elongated chamber housing the extractor to house the extraction process. The pressure inside the extractor assembly is the same as that outside it. Different solvents can be used with a high pressure extractor as in other modern methods so that several hundred compounds of different polarity can thus be extracted. Sample is loaded into an extraction thimble and the sealable pressure cylinder is filled with CO_2. The cylinder is heated in a water bath or electro thermal heating mantle for a period of time and the pressure rises. The system is connected to a coolant such as water or air-chilled from a chiller and passed through the cold finger to condense the CO_2. The CO_2 is vapourized again leaving the analyte in the flask and the whole extraction cycle is repeated. The whole procedure depends mainly on these factors which should be optimized before the extraction proper is started, these are temperature and pressure, the extraction time and the amount of sample used (Zygler *et al*., 2012).

High pressure soxhlet extractor modification was done by Lentz on the Jennings-type autoclave to enable visual control of soxhlet apparatus inside it through the provisions of a small glass window on its upper cover as shown in the diagram below (Karaj and Mele, 2015).

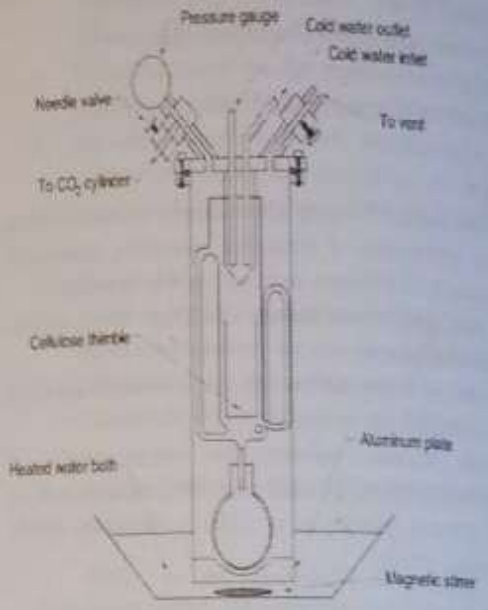

Figure 5: High pressure soxhlet Extractor (Zygler *et al*, 2012).

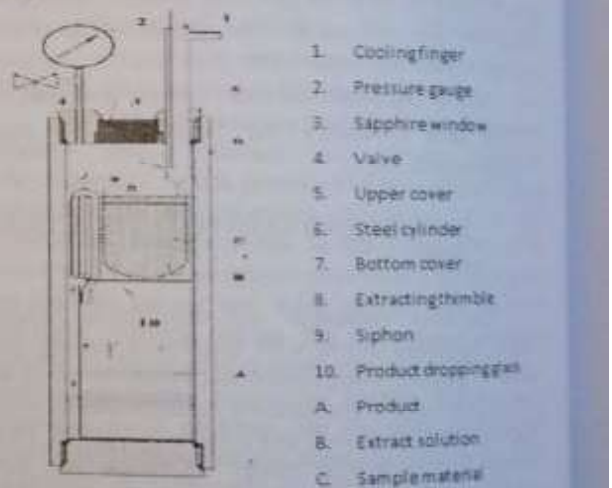

Figure 6: High pressure soxhlet extractor using liquid CO_2 (Karaj and Mele, 2015).

Research works has been carried out by Karaj and Mele in 2015 on the extraction of lycopene and β-carotene from tomato skin by near critical liquid carbon dioxide under the pressure of 64 bar and 299K. The extraction yields were determined after 0.5, 1, 3 up to 24 hours and presence of modifiers improves the extractability of lycopene.

This is an improvement on the sophisticated supercritical fluid extraction equipment characterized by cost. The above equipment is simple to use and almost all

the functions performed by SFE equipment can be carried out on the autoclave and specialized soxhlet apparatus.

4 CONCLUSION

A review on green technology and innovative extraction techniques has been presented for obtaining essential oil and active constituents in herbs and spices which covers a range from conventional to modern (non-conventional) extraction methods. It has been shown in this review that conventional methods have certain disadvantages concerning elevated temperature which causes degradation of some heat sensitive thermolabile (compounds) and huge energy consumption. Besides this, the organic solvent must be evaporated which makes the process usually time consuming and expensive (cost consuming) and solvent traces in pairs the quality thereby making it environmentally toxic for human use. In conclusion, the growing demand for extraction of bioactive compounds free of toxicity led to the search for nonconventional extraction methods using supercritical fluids such as carbon dioxide often referred to as green, clean, sustainable technology and environmentally friendly process.

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