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Recent Advances in the Extraction of Spice Essential Oil and Oleoresin: A Review

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ABSTRACT

Essential oils and oleoresins are the secondary metabolites of spice origin and have wide application in the food industry because of their flavour and aroma. They are recovered by various extraction techniques. Both conventional and emerging techniques are being utilized for this purpose. Conventional methods of spice extraction include, distillation and solvent extraction. Continued search for better extraction techniques finally ended up in the development of various advanced technologies. Methods such as supercritical fluid extraction, microwave assisted extraction, enzyme assisted extraction and ultrasound assisted extractions fall under emerging extraction techniques. These advanced technologies helped to solve the limitations in conventional methods. Selection of extraction technique depends on the requirement and economic feasibility. This review deals with the emerging techniques involved in the extraction of essential oils and oleoresins.

Key words: Emerging techniques, Essential oil, Extraction, Oleoresin.

Spices are known for their bioactive compounds and have drawn attention because of their nutritional and nutraceutical properties. They are available from different plant parts such as root, stem, leaves, fruits, flowers *etc*. (Teng *et al*., 2019). and in various forms including whole, roasted, pureed, paste, crushed, powdered and extractives (Andrews *et al*., 1995). Compared to other forms of spices, spice extractives have better stability in terms of flavour and storage. The main two categories of extractives include, essential oils and oleoresins. They have been explored for their ability to replace the original ground spices and herbs with a standardized taste and aroma (Serrano *et al*., 2020) . In order to recover these extractives, cell matrix of the respective spice has to be disrupted. Generally, dried spices are used for the extraction purposes compared to fresh spices as they have better stability and are more concentrated (Kurup *et al*., 2020; Manousi *et al*., 2019). Dried material is crushed so that surface area is increased and solvent can very well diffuse into the cell matrices (Saxena *et al*., 2018). Efficiency of extraction depends upon the extraction time, solvent used, temperature of extraction and solvent-solid ratio (Zhang *et al*., 2018).

Asbahani *et al*. (2015) explained essential oils as aromatic oily liquids traditionally extracted by hydro or steam distillation of spices. They are complex mixtures of low molecular weight compounds and are responsible for the aroma produced (Raut and Karuppayil, 2014). According to Bakkali *et al*. (2008), there are approximately 3000 essential oils and among this around 300 are of commercial importance. Essential oil does not represent a spice completely as they contain only volatile components. Distillation is considered as the conventional method of extraction of essential oils and has limitations like thermal degradation, longer extraction time *etc.*

While essential oils are the volatile components in a spice, oleoresins are the non-volatile part. They are highly concentrated forms with wholesome flavour and aroma of the respective spice. Compared to fresh and ground spice, oleoresins are hygienic (Shaikh *et al*., 2006). Extraction of oleoresin involves the use of various solvents (Aziz *et al*., 2018; Lucas *et al*., 2020; Pawar *et al*., 2018) and among those, acetone is the most commonly used one (Doosthosseini *et al*., 2019). Solvent remaining in the extract after the extraction is removed by evaporation or distillation (Salzer and Furia, 2009). Solvent extraction is the conventionally followed method for the extraction of oleoresin. It also has limitations like, longer extraction time, need of costly solvents *etc.* (Azmir *et al*., 2013; Luque-García and Luque De Castro, 2004; Sovilj *et al*., 2011; Stoica *et al*., 2016).

In order to solve the problems associated with conventional extraction methods, several non-conventional extraction techniques have been introduced. Some of those include, supercritical fluid extraction, ultrasound assisted extraction, enzyme assisted extraction, microwave assisted extraction *etc.* (Farooq *et al*., 2020; Strati *et al*., 2015; Wang and Weller, 2006; Zhang *et al*., 2018). These methods are safe and efficient separation of compounds can also be

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ensured. They require less energy cost over the solvents and are less damaging to environment (Gandhi *et al*., 2017). Essential oils and oleoresins are widely used in food application at industrial level. They can be used where whole spice or any other forms of spices cannot be directly added. They are being used in dairy products (like yoghurt, curd, cheese *etc*.), confectionary, beverages, bakery products *etc*. (Arimboor *et al*., 2015; El-Sayed and El-Sayed, 2020; Melgar-Lalanne *et al*., 2017). Other than food, essential oils and oleoresins have application in the development of cosmetics, household cleaning and hygiene products, air fresheners, medicinal uses *etc.*

This review focuses on the non-conventional extraction techniques used for the extraction of essential oils and oleoresins from various spices.

Historical perspectives of spice extraction

The record of origin of extraction from aromatic plants including herbs and spices has been lost to time. Man discovered the benefits of plants and the methods for separation of extracts very long ago. It was during Middle Ages, the understanding of extraction of aromatic plants spread to European region (Jacobs, 2020). Baser and Buchbauer (2019) explained that these extracts had been used in cosmetics by ancient Egyptians during 4500 BC. Mixtures of herbal preparations from various aromatic plants were used to prepare medicines and perfumes (Elshafie and Camele, 2017) also. According to Naeem *et al*. (2018), application of these oils changed with time and later on, it has been used for culinary purposes other than medicinal uses. It was during 4500 BC and 2000 BC the use of aromatic oils was first recorded in traditional Indian and Chinese medicine. Ali *et al*. (2015) reviewed that the use of spices such as cumin, peppermint *etc.* had been documented by Greek history between 500 BC and 400 BC. With the arrival of Arabs, a technique for essential oil distillation has been established. By the mid of 18th century, around 100 oils had been invented in Europe. But chemistry of these was not known. Towards the beginning of 19th century, chemists started studying the chemistry of these essential oils, which lead to the increased production all over the world (Essential Oil, 2019).

Continued search for better extraction techniques ended up in various conventional methods like, hydro distillation, steam distillation, solvent extraction, maceration, percolation *etc.* But these methods had several disadvantages, because of which improved techniques of extraction had to be invented. So, there was an enormous interest in searching for a method that give importance to sustainable environment (Fig 1).

Supercritical fluid extraction

The concept of supercritical fluid extraction (SFE) was introduced as a substitution to the conventional extraction techniques such as steam distillation and solvent extraction (Maharaj *et al*., 2018) because of its ability to extract the compounds of our interest with mild extraction conditions and minimum loss (Yousefi *et al*., 2019). (Williams, 2000) explained that the SFE are of two types; static and dynamic. In dynamic mode, supercritical fluid passes through the column that has sample in it. But in static mode, supercritical fluids are absorbed by the sample and as a result, no fluid pass through the column during extraction. (Shah *et al*., 2020; Yan *et al*., 2017) studied that extraction of a particular compound is dependent on the extraction time, pressure, percentage of co-solvent *etc.*

Gandhi *et al*. (2017) explains SFE as the process that utilizes supercritical fluid to extract the compound of our interest (Fig 2). Supercritical fluids are those having properties similar to both gas and liquid, but cannot be distinguished as either of the two. So, this helps in the solubilization of solutes in the raw material. Density of these fluids are similar to that of liquid and viscosity to gas. This technique utilizes certain solvents at pressure and temperature above their critical point. Critical point can be explained as the point on a phase diagram at which both the liquid and gas are indistinguishable. Solvents used in this are non-hazardous, so the process can be termed as 'green'. The most commonly used solvent is carbon dioxide as it is easily available, cheap and is GRAS for its use in food industry. Also, Sanchez Camargo *et al*. (2020) reviewed that the critical conditions of carbon dioxide (30.9°C and 7.38 MPa) are easy to attain. Another advantage of using

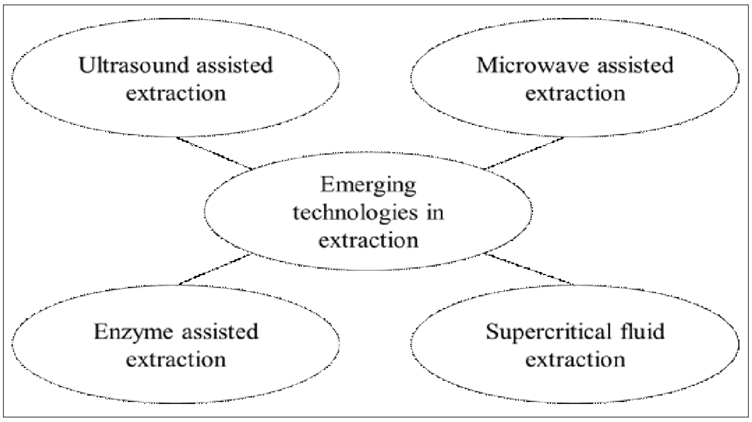

Fig 1: Emerging techniques in the extraction of plant materials.

supercritical CO_2 as solvent is solvent free product. i.e., the release of pressure by the system after the completion of extraction will help the CO_2 to convert in to gas and that will leave the sample.

But the major drawback of supercritical carbon dioxide is its low polarity, which can be solved by the use of co-solvents to change the polarity. Other than carbon dioxide, propane, ethanol *etc.* can also be used as supercritical fluids for the extraction (Table 1).

Supercritical fluid extraction of essential oil

Saleem *et al.* (2015) reported that compared to conventional methods such as steam and hydro distillation, cinnamon oil yield was high when extracted by SFE. Priyanka and Khanam (2018) studied that extraction of turmeric oil using SFE was found to be economical ate industrial level. Studies were carried out in order to investigate the anti-inflammatory effect of marjoram (*Origanum majorana* L.) and sweet basil (*Ocimum basilicum* L.) essential oil obtained by the supercritical fluid extraction (Sanchez Camargo *et al.*, 2020). *In vitro* studies showed that both essential oil had appreciable anti-inflammatory activity (Arranz *et al.*, 2015). Zeković *et al.* (2017) reported that, highest yield of essential oil from coriander was obtained at 200 bar pressure and 55°C. Yield of extraction was high when ethanol was used as co-solvent in the SFE of *Piper nigrum* by (Nagavekar and Singhal, 2018).

Supercritical fluid extraction of oleoresin

Devani *et al.* (2020) studied the supercritical fluid extraction of rotten onion oleoresin using carbon dioxide as the solvent.

Table 1: Solvents used in supercritical fluid extraction along with their critical temperature and critical pressure.

Solvent	Critical temperature (K)	Critical pressure (MPa)
Carbon dioxide	304.10	7.38
Water	647.30	22.12
Ethanol	513.90	6.14
Propane	369.80	4.25
Methanol	512.60	8.09
Acetone	508.10	4.70

Source: (Sapkale *et al.*, 2010).

Optimized parameters were 80°C temperature and 400 bar pressures. Study also helped in developing a method that will reduce the agricultural waste. Another study conducted by Dutta and Bhattacharjee (2015) in black pepper was by varying the temperature and pressure of the extraction process. Different combinations of temperature (40, 50 and 60°C) and pressure (200, 300 and 500 bar) were experimented and concluded that yield was high at 60°C and 300 bar pressure. Fitriady *et al.* (2017) also carried out experiments to optimize the temperature and pressure to get maximum yield by the SFE of ginger by using CO_2. According to their study, it was at 40°C temperature and 4500 psi pressure yield was high. A method by coupling SFE by fractionation was developed by Shukla *et al.*, (2019) to get maximum gingerol in the oleoresin and essential oil extracted. It resulted in 5.95% oleoresin yield which is 96.15% pure and 2.71% volatile oil which is 95.94% pure. Nagavekar and Singhal, (2019) experimented on *Curcuma longa* and *Curcuma amada* to study the optimization of extraction conditions of oleoresin by SFE. In the case of *Curcuma longa,* maximum extraction was attained at 65°C, 150 mins and 350 bar. But for *Curcuma amada*, it was at 40°C, 30 mins and 300 bar pressures. The study also reported that, addition of ethanol as co-solvent improved the extraction yield in both the varieties. SFE has been well explored for the extraction of bioactives from spices using various solvents (De Melo *et al.*, 2014; Herrero *et al.*, 2013) (Table 2).

Supercritical fluid extraction, which is a separation technique to extract bio-active components is a green extraction technology that meets all the regulations related to health and environment. Also, it has gained attention in many industries like, pharma, cosmetics, food *etc.* So, this can be considered as a promising technology for the coming years.

Microwave assisted extraction of essential oil

Microwaves are non-ionizing electromagnetic waves with a frequency ranging from 300 MHz to 300 GHz and will help in improving the extraction process (Martínez *et al.*, 2019; Shams *et al.*, 2015). According to Chaturvedi (2018), microwave assisted extraction can be used to extract the nutraceuticals such essential oils, oleoresins, dietary

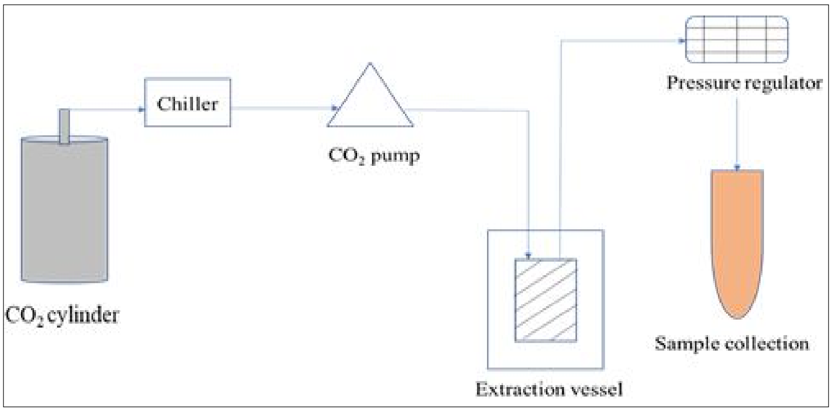

Fig 2: Diagrammatic representation of SFE that utilize CO_2 as supercritical fluid [Adapted: (Wei *et al.*, 2005)].

supplements *etc.* (Fig 3). Pre-treatment methods that helps in improving the extraction efficiency include, milling of the material, enzymatic pre-treatment *etc.* milling is to increase the surface area and enzymatic treatment is to break the cell wall (Vinatoru *et al.*, 2017).

There are mainly two categories of instruments used in the microwave-assisted extraction. First one is the multimode microwave ovens that make use of closed extraction. Here, extraction occurs by controlled pressure and temperature. Second one is the focused microwave ovens in which only the part of the vessel that includes sample will be focused for irradiation with microwave (Chaturvedi, 2018; Manousi *et al.*, 2019). Chan *et al.* (Chan *et al.*, 2011) listed the factors that depends upon the yield of extraction include, solvent to plant material ratio, nature of solvent, temperature, time taken for extraction *etc.* (Arya and Kumar, 2021; Dahmoune *et al.*, 2015; Kaderides *et al.*, 2019) reported that microwave assisted extraction requires lesser time. According to Mustapa *et al.* (2015), use of less solvent and consumption of less energy are the major advantages of microwave assisted extraction and also it helps in reducing the thermal degradation of the extracted compounds. Bayramoglu *et al.* (2008) introduced a novel microwave extraction technology without using the solvent for the extraction. They studied the extraction of essential oil from oregano (*Origanum vulgare* L.) by hydro distillation and microwave extraction and concluded that there was no much difference in the composition of oil obtained from these two methods.

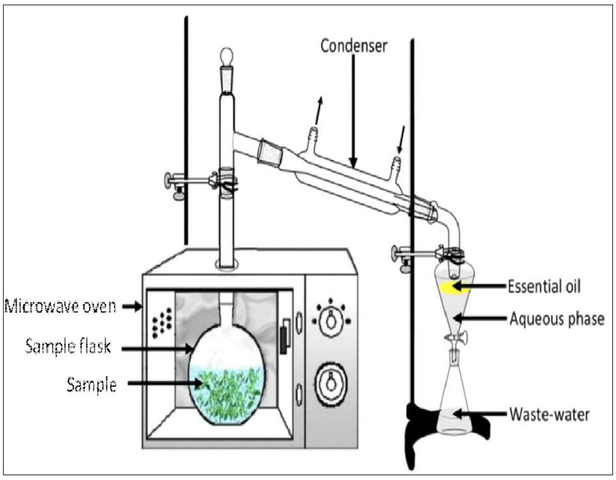

Fig 3: Schematic representation of microwave assisted extraction of essential oil [Source: (Kusuma and Mahfud, 2016)].

Extraction is carried out by combining microwave heating with dry distillation under atmospheric pressure. If the sample used is fresh, that can be directly taken for extraction. Otherwise, it has to be rehydrated and excess water should be drained off. An efficient microwave assisted extraction technique was developed by Bener *et al.* (2016) for the fast extraction of curcumin from turmeric by using methanol as solvent. Similar study was conducted by Mazzara *et al.* (2021) on ajowan seeds and reported that the thymol content was maximized by the use of microwave assisted extraction with reduced extraction time. In situ microwave assisted extraction of clove bud carried out by Gonzalez-Rivera *et al.* (2021) explained that it is a green and economical approach that can be applied to wide range substance like, spices, herbs, seeds *etc.*

Table 2: Various supercritical fluids used for the extraction of spices.

Spice	Essential oil/ oleoresin	Supercritical fluid used	References
Black pepper (*Piper nigrum*)	Oleoresin	CO_2	(Nagavekar and Singhal, 2018)
Cardamom (*Elettaria cardamomum*)	Essential oil	CO_2	(Hamdan *et al.*, 2008;
		CO_2+co-solvent (ethanol)	Topal *et al.*, 2008)
Clove (*Eugenia caryophyllata)*	Oleoresin	CO_2	(M. Das *et al.*, 2021; Ivanovic *et al.*, 2013)
Coriander (*Coriandrum sativum* L.)	Oleoresin	CO_2	(Zekovic *et al.*, 2017)
Cumin (*Cuminum cyminum*)	Essential oil	CO_2	(Saha *et al.*, 2016)
Fennel (*Foeniculum vulgare*)	Oleoresin	CO_2	(Hatami *et al.*, 2018)
		CO_2+co-solvent (ethanol)	
Ginger (*Zingiber officinale* R.)	Oleoresin	CO_2	(Leal *et al.*, 2003;
		CO_2+co-solvent (ethanol/ propane)	Sánchez-Camargo *et al.*, 2020)
Malagueta peppers (*Capsicum frutescens*)	Oleoresin	CO_2	(de Aguiar *et al.*, 2018)
Marjoram (*Origanum. majorana* L.)	Essential oil	CO_2	(Arranz *et al.*, 2015)
Oregano leaves (*Origanum vulgare*)	Oleoresin	CO_2	(Cavero *et al.*, 2006;
		CO_2+co-solvent (ethanol)	Ivanovic *et al.*, 2013)
Rosemary (*Rosmarinus officinalis*)	Oleoresin	CO_2 + co-solvent (ethanol)	(Cavero *et al.*, 2005; Sánchez-Camargo *et al.*, 2020)
Turmeric (*Curcuma longa*)	Oleoresin	CO_2	(Leal *et al.*, 2003; Priyanka and Khanam,
		CO_2+co-solvent (ethanol/propane)	2018) (Gopalan *et al.*, 2000); (Abdul Haiyee
		CO_2	*et al.*, 2016; Carvalho *et al.*, 2015; Lv *et al.*, 2018)

Enzyme assisted extraction

According to Cheng *et al*. (2015), enzyme assisted extraction is the process by which compounds of interest are extracted by the hydrolysis of cell wall. It is based on the catalytic property of enzyme to bind the cellular matrix (Fig 4). Solvent used for extraction can either be an organic solvent or water. According to Schweiggert *et al*.(2008), enzymatic hydrolysis of plant material is being widely used for the extraction.

Advantage of this technique include, improved yield of extracts, reduced time, mild processing conditions *etc*. (Wijesinghe and Jeon, 2012). Das *et al*. (2021) reported that, enzyme assisted extracted is purely dependent on the morphology and biochemical aspects of the plant material used for extraction and also the physio-chemical properties of the compounds to be extracted. In depth knowledge regarding these factors will aid in the right selection of enzyme. Enzymes are proteins with specialized catalytic functions and are produced by all organisms and are essential for all the metabolic activities. Prior to conventional methods of extraction, these enzymes are used for the pre-treatment of the plant material used for extraction by which the yield can be improved (Baby and Ranganathan, 2013).

Enzyme assisted extraction of essential oil

According to the study conducted by Sowbhagya *et al*. (2010) the yield of volatile oil from celery seeds was higher when it was pre-treated with enzymes and extracted oil did not have any physical or chemical changes. Similar study was carried out by Sowbhagya *et al*. (2011) on the extraction of cumin oil using enzyme assisted extraction. Study revealed that the pre-treatment of cumin seeds with enzymes led to improved yield of oil compared to the conventional methods. Evaluation of volatile oil indicated that compounds such as α-pinene, β-pinene and ρ-cymene was found to be slightly changed, but no change was observed in cuminaldehyde content. Enzyme pre-treatment of garlic also resulted in the improved yield of garlic oil and this was explained by Sowbhagya *et al*. (2009). Enzymes used include cellulase, pectinase, protease *etc*.

Enzyme assisted extraction of oleoresin

A novel technology for the production of paste-like and spray-dried ginger condiment was developed by the enzyme assisted liquefaction of ginger rhizome by Schweiggert *et al*. (2008). Varakumar *et al*. (2017) studied on the enzyme assisted three phase partitioning of oleoresin from ginger rhizome and reported that the yield of oleoresin was higher compared to conventional solvent extraction. Three phase partitioning is basically used for protein separation and recently has been applied for the oleoresin extraction also. The three phase consisted of water, ammonium sulphate and *t*-butanol. Kurmudle *et al*. (2011) studied on the enzyme assisted three phase partitioning for the extraction of oleoresin from turmeric. Ammonium sulphate and *t*-butanol was added to the slurry of oleoresin pre-treated with enzymes. Study revealed that the yield was improved and the time taken for extraction was lower compared to the conventional acetone extraction.

All these studies show that, the use of enzymes at optimum concentration will result in the enhancement of yield of essential oils and oleoresins. Because of which, enzyme assisted extraction can be considered as a better extraction technique compared to the conventional methods.

Ultrasound assisted extraction

Ultrasound assisted extraction is considered as a green technology and has been proved to be efficient as it uses fewer toxic chemicals. It provides wide range in the selection of solvent as toxic ones can be replaced by GRAS solvents (Rao *et al*., 2021). Solvent selection is another factor that is of higher importance when it comes to extraction and it depends upon the solubility and polarity of the compounds to be extracted.

Ultrasound waves can be successfully used for the extraction of oleoresin from various spices (Melgar-Lalanne *et al*., 2017; Morsy, 2016; Muhammad *et al*., 2021). According to the study conducted by Li *et al*. (2005), ultrasound can cause cell disruption and particle size reduction resulting in a larger contact area between the solid and liquid phases. In comparison to conventional procedures, this resulted in improved solvent access within the substance to be extracted (Fig 5). Arya and Kumar (2021) reported that use of ultrasound assisted extraction resulted in improved extraction yield as it uses lower temperature and short time. Ultrasonic effects are induced

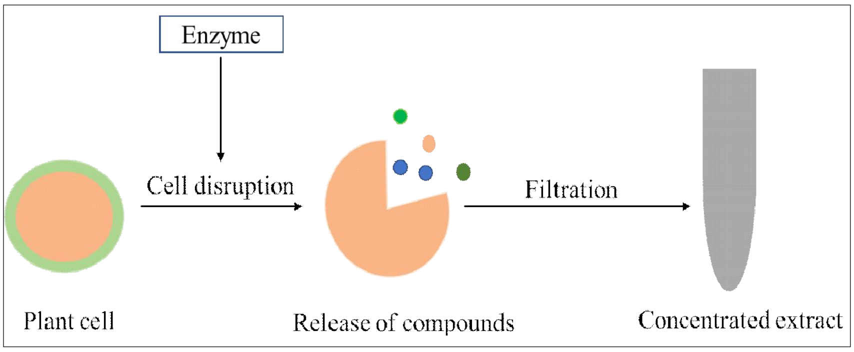

Fig 4: Schematic representation of enzyme assisted extraction [Adapted: (Sridhar *et al*., 2021)].

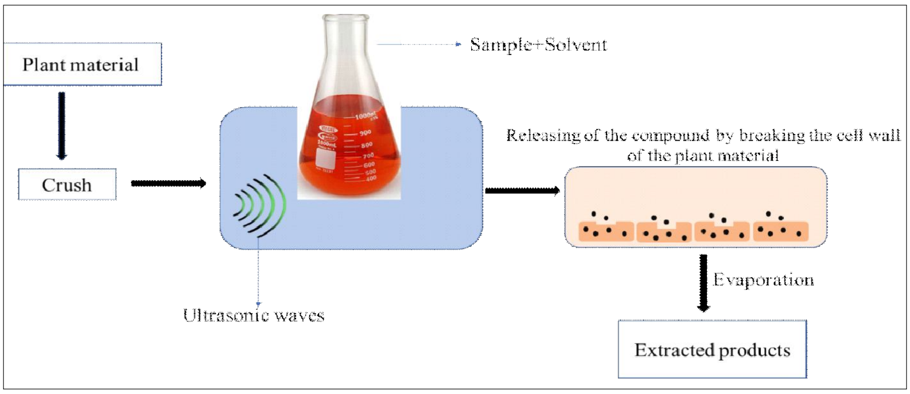

Fig 5: Schematic representation of ultrasound assisted extraction of plant material [Adapted: (Chen and Yang, 2020)].

within a material with the help of intense pressure and temperature gradient generated by high intensity shock waves. This will lead to the solvation of chemical constituents in the material used for extraction.

Ultrasound assisted extraction of oleoresin

Morsy, (2016) studied the difference between conventional maceration with ultrasound assisted extraction of oleoresin from nutmeg. GC/MS analysis indicated a great difference between the two oleoresins obtained and oleoresin obtained by maceration had less amount of myristicin compared to the other. Another study by Supardan *et al*. (2012) reported that ultrasound assisted extraction can be used to obtain oleoresin from ginger and GC analysis of the same indicated that there was no difference in the composition of oleoresin. Martínez *et al*. (2019) compared Soxhlet extraction with ultrasound assisted extraction and explained that the latter required shorter time and yield was quite higher. Thus it helps in fast-tracking the targeted extraction.

CONCLUSION

The demand for natural products is increasing day by day which will finally lead to the search of convenient extraction methods. To extract these compounds from various spices, it is of importance to optimize the process involved in the extraction. Considering the requirement of compounds and characteristics of the materials used, hybrid technologies are also being investigated as discussed in this review. This will help to overcome the limitations of conventional methods and will ensure a fast and a more selective method of extraction. Also, these methods should give importance to a more environmentally friendly approach.

Conflict of interest: none.

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