Applications of New Generation Solvents for Extraction of Herbal Products prior to Atomic and Molecular Analysis

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Abstract: In this review, an up to date and current knowledge of some of the green solvents, which includes supercritical fluids extraction (SFE), switchable polarity solvents (SPS), and natural deep eutectic solvents (NADES) are discussed with more emphasis on the extraction of active components of herbal products. Different scientific articles and books have been researched and reviewed to explain the applications of new generation solvents for extraction of herbal products prior to atomic and molecular analysis from the past until now. Currently, the most of techniques used in processing herbal products involve the use of extraction methods. Therefore, trends in extraction methods focuses mainly on finding reasonable solutions that minimizes the use of toxic solvents and allows the usage of renewable and green solvents from natural products, which ensure high quality and safe extracts. In future, SFE is definitely going to be on the industrial scale due to its numerous applications in the large scale especially for herbal, food, cosmetics and pharmaceutical products etc.

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1. INTRODUCTION

The separation between molecular analysis and atomic analysis, which are regarded as two distinct and extremely divergent areas, has existed in analytical chemistry for many years. As a matter of fact, the tools and extraction techniques created for atomic analysis—which inevitably need vaporization, atomization, and occasionally even ionization—are extremely different from those used for molecular analysis, and the spectra seen in both situations show considerable differences (1). However, given the different types of works that connect these two disciplines nowadays, such a division needs to be reevaluated. High-resolution continuum source atomic absorption spectrometers (HR-CS-AAS), which use either flame (F) or graphite furnace atomizers (GF), have considerably revolutionized the science of atomic absorption (2). As the concept of using continuum sources instead of line sources for AAS can be virtually traced back to the invention of the technology, several devices have been developed based on such concepts (3). Molecular analysis is regarded as a laboratory procedure that entails the examination of various sample materials, including food, herbals, cells, tissues, and environmental samples, for the identification of various target analytes at a molecular level using various analytical techniques, including high performance liquid chromatography (HPLC) and gas chromatography (GC), using different detectors (4).

Generally, major industrial and conventional chemical processes involve the use of flammable, toxic, volatile, hazardous, and environmentally destructive solvents in chemical reactions and separation processes (1). These have a negative impact on the economic and environmental performance of such processes (2). Conventional or classical extraction methods are also time-consuming, laborious, and involve large amounts of energy-consuming solvents such as alcohols, chloroalkanes, and other hydrocarbons (3). Therefore, since most bioactive components are water-insoluble, this can ultimately aggravate the degradation of some target analytes (4). It is worthy of note that, despite the use of high amounts of solvents and high energy consumption, the yield is also lower in comparison to the alternatives (5). Therefore, a few decades ago, safer, effective extraction methods in accordance with a sound compromise of their environmental, economic, and social requirements began to be considered as replacements (6).

Green chemistry as a concept was first introduced in 1991 by PT Anastas when he launched a unique program in order to implement sustainable development in the area of chemical technology (7).

Green chemistry has 12 principles which serve as guidelines for designing chemical products and methods (8,9) that reduce or occasionally remove the application and generation of harmful and hazardous chemical materials altogether by using solvents and chemicals that are entirely non-toxic to human health as well the immediate environment in order to design and create effective and non-hazardous methods facilitating the use of renewable solvents known as' new generation solvents' or 'solvents of the future' (10). Gu and Jerome proposed the first requirements for a solvent and a process to be considered green based on their availability, biodegradability, flammability, grade, performance, price, recyclability, renewability, stability, storage, toxicity, and synthesis (11). There is no doubt that these are reasonable and sound conditions (12). Nevertheless, it can also cause a dilemma regarding classifying a solvent as a green solvent, because in most cases, a solvent cannot fulfill all these parameters. For example, a solvent can fulfill eleven or ten or nine out of the total twelve conditions of a green solvent (13). Therefore, can this solvent still be regarded as green? It is therefore crucial to be precise regarding the meaning of these green solvents. The term itself can therefore be misleading due to the its novelty and relative nature (14). Though Warner and Anastas defined "green chemistry" as a process of reducing the toxicity and hazards of processes or methods, they did not define it as a method that does not cause any damage or harm (15). Therefore, a solvent can be considered green if it is 'greener' when compared with the current classical solvent to be replaced, which can be supported with clear and strong evidence (1). In almost all cases, the choice will be based on a compromise between different conditions, as seen in Figure 1 below (17,18).

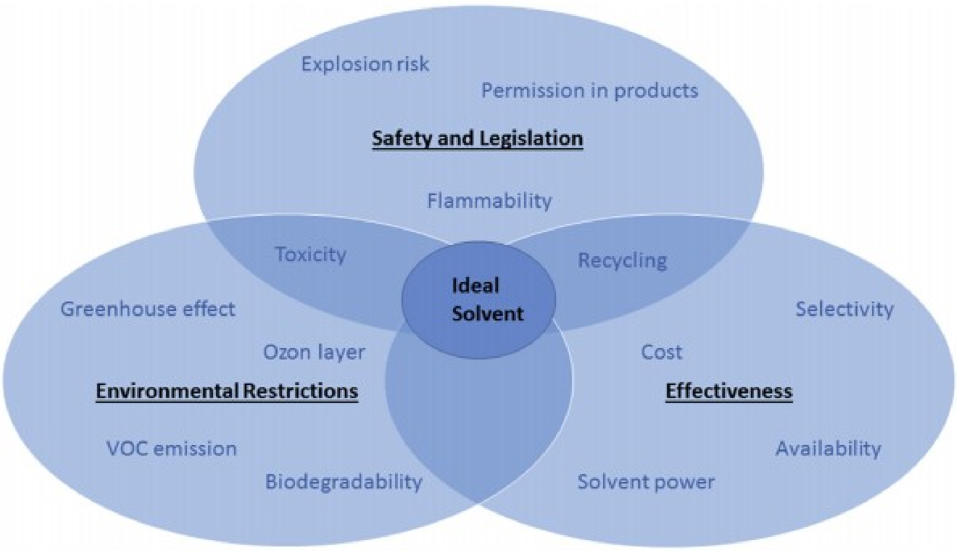

Figure 1: An ideal solvent is a combination and involves a compromise of different and multitude requirements (Adapted from Chemat *et al*. (11)).

These are termed "new generation solvents" owing to their tunable properties, which may prove to be a class of solvents that offer energy and material efficiency greater than existing solvents that need further exploitation to improve their application as a green chemical process (19).

Finally, given their limited context, there is a need to use their applicability in micro-extraction methods, particularly for switchable polarity solvents (SPS) (20).

In conclusion, the aim of this review is to show some applications and wide extraction methods using new generation solvents for herbal products prior to atomic and molecular analysis (21). In this regard, atomic analysis entails the identification and characterization of various sample materials, particularly environmental, cosmetic, food, and herbal samples, for the identification, isolation, and confirmation of analytes at the elemental state via atomization using various analytical techniques such as FAAS and inductively coupled plasma mass spectrometry (ICP-MS). While molecular analysis is considered a laboratory procedure that involves the study of different sample materials such as food, herbals, cells, tissue, and environmental samples for the identification of various target analytes at a molecular level using different analytical techniques such as HPLC and GC using different detectors.

2. SOLVENT-FREE SYSTEM

Sometimes the best solvent is no solvent at all. A solvent-free system completely satisfies the 5th condition of green chemistry. Numerous studies have shown that there is an abundance of industrial and chemical processes in which the use of solvents is completely avoided (22). However, this is not always possible. Especially if the absence of the solvent leads to dangerous overheating or results in a higher demand for energy (23). Therefore, these disadvantages would definitely surmount the advantages of using a non-solvent system (solvent-free process). Some decades ago, scientists tried synthesizing polymers using solvent-free conventional reactions (24). It is indeed notable that some industrial polymerizations have succeeded in using solvent free methods in synthesis as well as in separation, such as solid state polymerization and melt polymerization (25). For example, PET is produced commercially through melt polymerization and then followed by the solid state polymerization method (26). It is possible to produce polymers, for example poly (phenylene vinylene), by ensuring that there is close contact among the molecules of the starting materials for about five minutes in the ball milling process (27). This ball milling process is not only used in polymer science, but also has diverse applications in organic synthesis (28). Apart from avoiding the use of organic solvents, it also has numerous advantages, such as a reduction in reaction time and high energy efficiency (29). As far as the solvent-free process is concerned, mentioning the solvent-free microwave extraction method is significant, which is a well-designed technique introduced by Lucchesi and his group in the year 2004 (30). This sophisticated method can be applied to extracting essential oils from fresh plant material in a microwave-assisted dry distillation process (31). It is a quick, robust, and sensitive method that gives a strong alternative to the time-consuming and long-lasting conventional hydro-distillation method (32).

3. SUPERCRITICAL FLUID EXTRACTIONS

These are substances which exist as a single phase above their critical points of temperature and pressure (33). This critical point can be defined as the point at which liquid and vapor can be distinguished without the need for boundaries (34). For example, the supercritical point of water was discovered to be 374 °C and 22.7 MPa C (Figure 2A), while the supercritical point of CO_2 was discovered to be 31 °C and 7.3 MPa C (Figure 2B) (35).

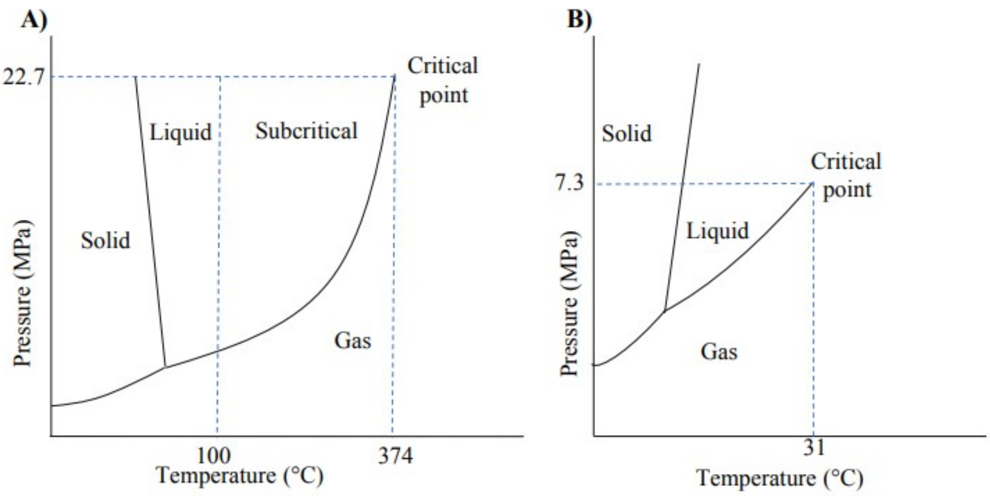

Figure 2: Phase diagram of water and CO_2 (Adapted from Lucchesi et al., 2004 (35)).

Due to their tunable properties of low viscosity (gas-like) and high density (liquid-like), these are considered to be important new generation solvents that improve solubility and mass transfer properties (36). The properties of certain supercritical fluids are mentioned in Table 1. In analytical chemistry, supercritical propane–butane, water, ammonia, and CO_2 have also been used. Because of its low critical temperature and pressure (31 °C and 7.3 MPa), inertness, purity, non-toxicity, and availability, CO_2 is the most widely used supercritical fluid. Furthermore, the strength of **supercritical carbon dioxide** ($ScCO_2$) solvation can be adjusted by changing the temperature and pressure (37).

Another benefit of CO_2 is that it is gaseous at room temperature and pressure, making active product recovery relatively quick and cheap, as well as the ability to produce solvent-free extracts. $ScCO_2$'s ability to operate at low temperatures while using a non-oxidizing medium is also advantageous for sample processing of medicinal, food, biological, and natural products, as it allows for the extraction of thermally labile or readily oxidized compounds with minimal degradation. $ScCO_2$ has solubilizing properties similar to n-hexane and n-heptane due to its low dielectric constant and dipole moment near to zero. Due to charge isolation and its electronic composition, it has a quadrupole moment, allowing it to behave as both a Lewis acid and a Lewis base. Despite having a quadrupole moment, $ScCO_2$ acts like a nonpolar solvent, limiting its use in removing hydrophilic analytes. A polar modifier or (co-solvent) is normally applied to tune the polarity and increase the solvating ability to address this constraint. Chemical solvents such as methanol, ethanol, and ethyl acetate are applied to $ScCO_2$ in small amounts (1–20 vol percent) to broaden its extraction spectrum to include more polar analytes.

Table 1: Physical properties (density, diffusion and viscosity) of gaseous, supercritical and liquid states. (35).

State	Density (g/cm^3)	Diffusion (cm^2/s)	Viscosity (g/cm•s)
Gas	10^{-3}	10^{-1}	10^{-4}
Supercritical	10^{-1} - 1	10^{-4} - 10^{-3}	10^{-4} - 10^{-3}
Liquid	1	<10^{-5}	10^{-2}

Supercritical fluid extraction of herbal and biologically active components is now a growing area of interest. It gives an ability to process plant and herbal products at a very low temperature, in absence of toxic and harmful solvents, in addition to limiting thermal degradation of the analytes (38). Any fluid can be used as a supercritical fluid if it fulfils the characteristics under critical conditions. However, cost, solvation power and toxicity determine the best and suitable solvent to be used in a particular and specific application. It has been reported that propane, dimethyl ether and ethane have been used in extraction of herbal products and biologically active compounds as supercritical solvents (39).

For easy understanding, we need to know that the supercritical fluid methods for most of the natural compounds are mainly categorized into two main divisions: (1) undesired or unwanted chemical substances that need to be removed from the plant materials (matrix), for example, removal of caffeine from tea and coffee, defatting of press cakes, as well as removal of various factors such as porosity, particle size, nature of the matrix, and moisture, as well as removal of some parameters from some processes, such as temperature, pressure, and solvent flow rate, which can have an effect on the supercritical fluid result, (2) the extraction of biologically active components from plant material (40). Table 2 shows a selection of common applications of supercritical fluid extraction from herbal and plant origins, along with extraction properties such as pressure, modifiers, and temperature.

Table 2: Supercritical-CO_2 extraction of some selected herbal bioactive compounds (selected from plant origin) (6).

Material	Extract	Modifier	T (°C)	P (bar)
Almond	Oil, tocopherols	Methanol	35–50	350–550
Aloe Vera leaves	α -tocopherol	Ethanol	40–70	300–600
Black pepper	Oleoresin, piperine	-	35–55	200–300
Ginger	Oleoresin	Ethanol, isopropanol	25–35	200–250
Thyme	Total extract, thymol,	-	40	80–400
Walnut	Oil	Ethanol	40–60	300–500
Grape skin	Resveratrol	Ethanol	40	150
Ginkgo leaves	Terpenes, flavonoids	Ethanol	60–110	242–312

4. SWITCHABLE POLARITY SOLVENTS (SPS)

Generally, switchable solvents (SS) are mixtures of compounds that have the ability to abruptly change their physical properties, such as polarity (hydrophilicity), conductivity, solubilizing capability orviscosity (41,42). The switching of polarity is induced through bubbling of CO_2 gas at atmospheric pressure into the reaction vessels (43). The CO_2 will further reacts and associates with a compound in the reaction system forming an ionic liquid (IL) having different properties from the initial molecular liquid (44). This process can simply be reversed through removal of CO_2 by bubbling inert gasses, N_2 gas, NaOH or sometimes through applying mild heat to the liquid (45).

It has been reported that various materials have the ability to switch some of their characteristics, for example polymers, solutes, and surfactants (46). With the aid of suitable reagents such as organic bases, water also has the ability to switch to higher ionic strength from lower ionic strength (47). SS are very important for processes that need various solvent characteristics in consecutive steps, such as product recovery or extraction (48). The use and application of switchable solvents reduces the number of solvents required during a chemical process (49,50).

Switchable polarity solvents (SPS) were first introduced by Jessop in the year 2005, which contain either only one component, for example, secondary amines, or are composed of multiple components (51). Usually, two components of switchable polarity solvents are composed of amidine or sometimes guanidine together with a primary amine or an alcohol (52). Other combinations, such as guanidine or diamines/acidic alcohol chemical mixtures, are possible (53).

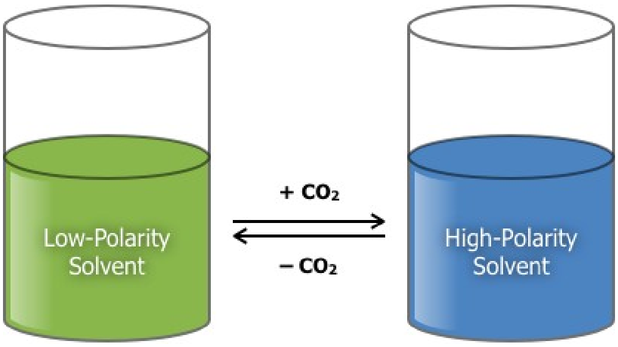

Figure 3: Switchable polarity solvent switching from low-polarity to high polarity by bubbling CO_2 (Adapted from Jessop et al., (56)).

Each SPS has its own properties (54,55). These characteristics have to be taken into consideration when selecting the best SPS for a certain chemical process (56). Systems containing amidine 1,8-diazabicyclo-[5.4.0]-undec-7-ene, for example, are extremely sensitive to moisture, whereas some secondary amines and other amidines SPS are less sensitive (57). One of the advantages of single component SPSs is that they do not need any operator to monitor the mole ratio of these two liquids. Furthermore, secondary amines are cost-effective when compared with amidines (58, 59).

One of the major challenges of industrial processes is solvent removal from hydrophobic materials without the use of distillation processes (60). However, the idea is possible provided that there is a solvent which can reversibly switch from hydrophobic state to a more hydrophilic one (61). Therefore, the discovery of SPS plays a major role in solving this negative inconvenience in industrial processes, because the removal of solvents through distillation is the most common industrial activity that suffers from the main drawbacks that lead to environmental hazards and damage (62). Firstly, because distillation usually employs the usage of a volatile substance, it results in smog formation through great vapor emission. Secondly, it needs a high input of energy (63). Therefore, there is a need to find a new and efficient non-distillative approach for the separation of solvents from their products, in order to avoid the usage of volatile solvents (64,65).

Soylak *et al.* reported that N,N,N-tributylpentanamidine was first used as an SPS that can be applied for extracting low polarity products such as vegetable oils, and then the solvent is removed from the product using carbonated water (66,67). Carbonated water has the ability to extract the solvents from the products due to the fact that the CO_2 can convert the solvent into the polar form. Subsequently, the solvent is then separated from the carbonated water by removing the CO_2, because the removal triggers the conversion of the solvent into its non-polar form. And finally, the removal of the solvent from the herbal product does not require distillation (68,69).

Memon *et al.,* (2017) proposed a green and novel switchable solvent, which was hyphenated with liquid phase micro extraction (SS-LPME) for extraction and preconcentration of the nutritionally and biologically important element Co(II) from tobacco and food samples using flame atomic absorption spectrometry. To improve conversion from the deprotonated form to the protonated form, N, Ndimethyl-n-octylamine bicarbonate was synthesized and used as a switchable solvent in the presence of CO_2 to improve conversion from the deprotonated to the protonated form, and then examined for analyte extraction. A quantitative recovery was achieved (70,71). This study involves the formation of a complex at a pH of 4.0 between Co(II) and 1-nitroso-2-naphthol, which serves as a ligand, and then extraction through conversion of the solvent to a nonpolar N,N-dimethyl-n-octylamine phase. The accuracy and validity of this method were checked using standard reference material (IC-INCT-OBTL-5) and additional recovery verification. The LOD and LOQ were also found to be 3.2 µg L^{-1} and 10.6 µg L^{-1} respectively. Finally, this method was efficiently used for the atomic analysis of tobacco, herbal products, and food samples (72).

5. NATURAL DEEP EUTECTIC SOLVENTS (NADES)

In 2003, Abbot and his co-workers introduced deep eutectic solvents (DES) for the first time (73). They reported excellent properties for some eutectic mixtures of a range of quaternary ammonium salts and urea (74). DES are now widely regarded as a novel class of sustainable solvents that serve as green alternatives to ionic liquids (ILs) (75). Because of their similar properties such as starting materials, non-flammability, non-volatility, and high viscosity, DES are considered the 4th generation of ionic liquids (78). However, DES are not entirely made up of ionic species (76,77). DES are created by combining two or more components, such as organic salts (phosphonium salt or quaternary ammonium) and hydrogen bond donors (HBD) or metal salts that can associate with one another via hydrogen bonding (78). The charge delocalization that occurs results in a decrease in the melting point of the final product when compared with the individual melting points of the starting materials (79). Nevertheless, DES has numerous advantages and applications over ILs, such as lower economic and environmental impact (80,81). This is more pronounced for DES that are produced naturally from primary metabolites of living cells, known as natural deep eutectic solvents (NADES), which are mostly found in abundance in our diet, such as amines, sugars, carboxylic acids, polyalcohol and choline (82). These NADES satisfy all the principles of green solvents, which produces many advantages, such as readily available starting materials, reduced toxicity, cost-effectiveness, and simplicity of preparation, among the others. Moreover, they possess excellent physicochemical properties such as adjustable viscosity, wide range of polarity, liquids even at lower temperatures, and high solubilizing capacity for a wide range of compounds that can be fine-tuned for a certain application because of their different structural possibilities (83).

Due to the above properties of NADES, there is a rapid increase in their application for the extraction of biological ingredients of herbal materials such as phenolic acid, saponins, flavonoids, alkaloids, terpenoids and anthocyanin, which clearly indicated the possibility of using NADES in extracting different hydrophobic and hydrophilic naturally occurring chemical compounds (84,85).

In order to select a suitable NADES for extracting active compounds from herbal materials, it is very important to try different combinations having different physicochemical parameters. For instance, Dai et al., reported that seven NADES were used in the extraction of aromatic pigments having wide range of polarity from *Carthamus tinctorius* L. showing that NADES with low polarity possess the lowest efficiency for polar active compounds but higher extraction ability for non-polar active compounds and vice versa (86). This corresponds to the rule of “like dissolves like”. Various researchers draw the same conclusion when extracting phenolic compounds from grape skin. In the study, they used choline-chloride based NADES that contains organic acids, polyalcohol or sugars as the hydrogen bond donors (HBD). However, polyalcohol and sugar based NADES are less polar than organic acid based NADES, having a polarity almost equal to that of methanol (87,88).

Furthermore, to improve the extraction efficiency of NADES, there is a need to optimize the NADES content of water. Increasing the water content decreases its viscosity, thereby increasing the transfer of mass from the herbal matrices to a solution and hence increasing the efficiency of the extraction (89). The main problem with using NADES for extraction is its viscosity, which is generally high at room temperature. For instance, some NADES cannot be utilized directly for extraction without diluting them with water due to their high viscosity. However, increasing the water content decreases the interaction between the solvent and the target analyte. Also, an excess of water in NADES can lead to halide-HBD supramolecular complex breakage and thereby form a single aqueous solution of each of the initial components. In general, a NADES with high water content are more suitable for the extraction of more polar compounds, while those with low water content are more suitable for extracting non-polar compounds. However, water content has a significant effect on the yield for both non-polar and polar-active components. Furthermore, the stability of the target analytes while using NADESs should be taken into consideration as an important factor when selecting a specific solvent. In a study to determine the stability of some phenolic components of safflower extracts, the results showed that using NADES improved compound stability over using conventional solvents (39). The stability in NADES is due to the strong hydrogen bonding that exists between the molecules of the solvent and solute, whereby among the studied NADES, sugar-based NADES showed the highest stability (90). This interaction is responsible for decreasing the mobility of the solute molecules, and this reduces the contact time with oxygen and air, hence minimizes oxidative degradation, which is the major factor that causes degradation of the active compounds (91). There is a need for further research on the reasons behind bioactive ingredients' stability in NADESs for a full understanding of the mechanism (92,93).

Furthermore, before final selection of NADES, its environmental effects should be checked and examined, because the assumption that NADES are non-toxic is based on toxicity results for individual components used in preparing NADES, which are naturally occurring biomaterials that are pharmaceutically accepted (94). The assumption does not take into account the probability of the existence of combined and synergistic effects of the individual components that make up the NADES (95).

According to the current research trend, there is little or no application of the NADES extraction method on an industrial scale for green extraction of herbal active components on a large scale. Therefore, to achieve such industrial implementation, environmental and economic factors should be considered (96). Though the price of NADES is comparable to that of conventional and classical solvents, their production is regarded as a sustainable process (97). Nevertheless, other factors should be considered, such as the recovery of the target bioactive component as well as the NADES recyclability, before implementing this technique in large scales. Nonetheless, researchers (98-107) reported that NADES is a suitable alternative technique that involves renewability and sustainability for the extraction of value added compounds as well as other precious analytes prior to both atomic and molecular analysis.

CONCLUSION

Currently, most of the techniques used in processing herbal products involve the use of extraction methods. Therefore, trends in extraction methods focus mainly on finding reasonable solutions that minimize the use of toxic solvents and allow the use of renewable and green solvents from natural products that ensure high quality and safe extracts. In the future, SFE is definitely going to be on an industrial scale due to its numerous applications on a large scale, especially for herbal, food, cosmetics, pharmaceutical products etc. Due to its various applications, as mentioned in this research, CO_2 is a vital chemical compound. SFE shows diverse advantages over conventional solvents. Also, switchable polarity solvents (SPS) have shown excellent applications on an industrial scale through switching one of their characteristics from the non-polar form to the polar form simply by bubbling CO_2, which enables complete miscibility and ensures the extraction of the bioactive components. However, there is a need for more development and transference of this technique into the micro extraction method as there is less data and research in that field. Moreover, NADES show unique physicochemical parameters and completely satisfy all the principles of a green solvent that can be used for extracting bioactive compounds from herbal materials, due to their low environmental and economic impacts. However, there is a need for transferring this technique to the industrial scale owing to the meager amount of research conducted in the published technical literature that is used on a large scale.

Finally, the need for the identification, analysis, and standardization of herbal products is of paramount importance, owing to their significant application in society and to verify fraudulence, fraud, counterfeit, and adulteration.

CONFLICT OF INTEREST

None reported.

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