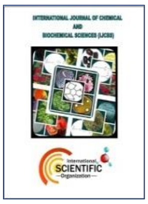
International
SCIENTIFIC
—Organization—

International Journal of Chemical and Biochemical Sciences
(ISSN 2226-9614)

Journal Home page: www.iscientific.org/Journal.html



Recent advances in nanoparticles formation techniques for essential oils

***Areeba Gul**[*1]**, Muhammad Riaz**[2]*

[1]Department of Chemistry, University of Agriculture, Faisalabad-38040-Pakistan.

[2]Department of Basic and Applied Chemistry, Faculty of Science and Technology University of Central Punjab, Lahore 54000, Pakistan

Abstract

Essential oils (Eos), volatile and thermolabile metabolites derived from various plant parts, play a crucial role in the biogenic synthesis of metal nanoparticles. Phytochemicals within essential oil are extracted through diverse classical and innovative methodologies. This sustainable synthesis method facilitates the reduction of metal ions to nanoparticles without the use of harmful chemicals, operating at low pressure and temperature with increased speed and simplicity. This approach mitigates the toxicity risks associated with conventional physical and chemical methods. In parallel, encapsulation procedures have been developed, encompassing biomolecule incorporation at nanoscale such as nanoemulsion and nanofibers. These strategies combined with biogenic synthesized nanoparticles, find applications in diverse industries, including pharmaceutical, cosmetics, textiles and food packaging. Encapsulation serves to control the release of active ingredients, ensuring targeted delivery and protection from environmental parameters. Validation of the success of essential oil-based nanoparticles is confirmed through analytical techniques such as FTIR, UV-Vis, SEM, EDX and TEM. These methodologies provide insight into the structural and morphological characteristics, further establishing the efficacy of the biogenic synthesis approach.

Keywords: essential oil, metal nanoparticles, phytochemicals, nanoencapsulation, nanoemulsion, nanoprecipitation.

Full length review article **Corresponding Author*, e-mail: *areebagul1122@gmail.com*

1. Introduction

In scientific research, nanotechnology as a revolutionary field has become a significant thrust for scientist in investigation, exploration and analysis of nanostructure based material and their tremendous applications in daily life [1]. In past, considerable efforts were done to produce the nano-meter sized particles with controlled morphology and effective characteristics [2]. Due to enhanced surface characteristics, nano-meter sized particles with large surface to volume ratio, nanotechnology found optimum use in number of applications such as agricultural [3], medicinal, military [4-5], food packaging [6], environmental remediation [7] and vice versa. Different applications of nanoparticles are displayed in Fig1.

Different technologies, including physico-chemical routes, have been utilized for nanoparticle synthesis. However, these methods often involve vacuum conditions, high temperatures, and the use of combustible and environmentally hazardous chemicals [8]. The chemicals including organic solvents (tollens reagent, ethylene glycol, N,N-dimethylformamide) having high vapor pressure are at higher risk to contaminate the surrounding biosphere and cause serious health risks [9]. The main objective is to synthesize nanoparticles using environmentally friendly and energy-saving technologies, resulting in the production of fast, stable, low cost and non-toxic nanoparticles [10]. Sustainable chemistry is solution to the problem that invigorate the advancement of methods that minimize the formation of harmful product. Green chemistry uses environmentally friendly precursors and energy efficient methods like supercritical fluid extraction and solvent free microwave extraction. The products formed are not harmful to the environment and the nanostructure formed can be used to remove the pollutants [11].

Essential oils are extracted from various plant parts having phytochemicals such as terpenes etc., as major constituent. The formation of nanoparticles is facilitated by a variety of stabilizing reactive sites found in essential oil [12]. Examples include, nanosized Palladium produced from banana peel [13], silica nanoparticle fabrication from Vietnamese rice husk [14], gold nanoparticles synthesis from *Punica granatum* [15] and the use of *Annona squamosa* peel for Ag nano-particle synthesis. These nanoparticles are thus characterized by various spectroscopic approaches such as UV-Vis, TEM, SEM, XRD, GC-MS and NMR [16].

Essential oils are unbalanced organic compounds with high vapor pressure. Nanotechnology involves in transportation of essential oils. Encapsulation increase the essential oil action duration and stability. It removes the difficulties associated with essential oils transportation. Nanoparticles are loaded with essential oils and protect this volatile organic liquid from UV rays' degradation, heat, volatility and ensure the stability [17].

2. Essential oil

Essential oils referred as ethereal oils, are hydrophobic, odorant, volatile liquids derived from plant raw material, either by dry distillation, appropriate mechanical process without heating or by water driven steam. This organic liquid represents complex chemical composition and withdrawn from aqueous film by physical approach without any symbolic change in its composition. Essential oils are commercially known as desequiterpenated, rectified or deterpenated according to Pharmacopoeia [18].

The fundamental processes in plants life, photosynthesis and respiration, called primary metabolism. The primary metabolism processes result in the production of organic compounds called primary metabolites such as nucleic acids, proteins, carbohydrates and lipids. In addition, plants carried out additional processes, by using these primary metabolites, called secondary metabolism. These are called secondary because these are not as essential as primary for plants life. These processes encircle a variety of secondary metabolite such as organic oils, flavonoids, steroids, polyamines, saponins, tannins and coumarins [19].

Oily aromatic liquids (essential oil) are retrieved from odorous botanical stuff. The essential oils (EO's) synthesized as secondary metabolites in various plant organs such as leaves (Eucalyptus, Salvia, Thym), flowers (jasmine, rose, violet and lavender), herbs, buds (clove), wood (sandal), seeds (*Elettaria cardamomum*), rhizome and roots (*Zingiber officinale*) [19]. These hydrophobic oils have a lower density than water and are lipophilic in nature, immiscible in aqueous medium and miscible in organic liquids. The main component of these lipophilic semi-liquid phytochemicals are terpenes and other self-assembled products [20]. Among wide diversity of plants only some plant species about 10% contain essential oil, and these plants are called aromatic plants. The aromatic plant families from which essential oils can be found traditionally are Lauraceae, Piperaceae, Rutaceae, Lamiaceae, Myrtaceae, Asteraceae, Poaceae, Cupressaceae, Zingiberaceae [19-21].

2.1 *Benefits of essential oil*

Essential oils as a mixture of different constituents in different concentrations have varying properties and modes of action. The role of essential oil in plant is speculative but plays role in plant-animal interaction (pest repellents and pollinator attractors) and plant to plant interaction (hindering the germination and development of more plants). Essential oil provides defensive character against insects, herbivores, fungi, pathogens and microorganisms. According to experts, essential oils control and regulate the plant environment by providing chemical signal to plants. [22]. Traditionally essential oils possess different benefits including antioxidant, anti-inflammatory, antidiabetic, antimicrobial, cardioprotective, antiviral, neuroprotectant, anticancer, and anti-hyperpigmentation as described in fig 2 [23].

2.1.1: Antioxidant

Reactive oxygen species as free radicals associated with various diseases like cancer, heart disease, inflammation, deterioration in immune system, brain dysfunction and age-related disorders. Thus, to prevent disease, antioxidant use is of great concern [24]. Anti-oxidants are the species that are used to prevent oxidation. Plants derivative such as essential oils have great antioxidative potential. Invitro assays were prepared by using essential oils (essential oil with different concentration) obtained from medicinal herbs to check their antioxidant aptitude as free radical scavengers in lessening the oxidative pressure by hindering reactive nitrogen or oxygen species. From results it was found that *Laurus nobilis* L., *Citrus hystrix* DC. essential oils have significant antioxidant potential [25]. The essential oil composition containing double bond, as secondary metabolites with phenolics contribute to antioxidant property. The active constituents of essential oil such as carvacrol, a monoterpenic phenol, carry out a basic function in elimination of free radicals as well as deterioration of peroxides [26].

2.1.2: Anti-Inflammatory

Inflammatory disease such as rheumatism, allergies, arthritis can be clinically treated by using several essential oils. Essential oil components such as citral, carvone, geraniol perform function against inflammation. Continuous application of geranium essential oil suppress the inflammatory symptoms of edema and neutrophil accumulation [27].

2.1.3: Anticancer

Essential oil active components also perform function against cancer cells. It was observed that in human malignant cells, a sesquiterpene namely α-bisabolol is a potent apoptosis inducer. α-bisabolol is non-toxic to normal cells. This apoptosis is accompanied by intrinsic pathway [28]. Besides, in several cancer cell lines the cytotoxicity is improved by using the combination of chemotherapy drugs and essential oil components. D-limonene as an essential oil component in combination with docetaxel, a chemotherapy drug, improve the anticancer action against DU-145 cells, human prostate carcinoma, without causing any toxicity to normal prostate epithelial cells [29].

2.1.4: Antidiabetic

Essential oils significantly display hypoglycemic effect, lessen lipid peroxidation activity and efficiently spruce up the anti-oxidant defense system. A natural dietary monoterpene, menthol, play an important role as antidiabetic. The menthol reduces the glycosylated hemoglobin, blood sugar level and increase the plasma insulin, total hemoglobin and levels of liver glycogen in diabetic rats. Moreover, the other biomarker related to damaged liver restored to normal [30]

2.1.5: Antimicrobial

The antimicrobial agents against pathogens have become a thirst among researchers around the globe. Essential oil antimicrobial potential has been widely investigated. Essential oils perform antibacterial activity by interrupting proton pumps and ATP exhaustion [31]. Tea tree oil increase the liberation of potassium ions (K^+) and interrupt internal respiration (cellular respiration) which result in hindering growth of *E. coli*. Essential oils also perform antifungal activities such as various *Candida* species (*C. glabrata, orthopsilosis, C. albicans, C. krusei*) [32].

2.1.6: Antiviral

It is investigated that essential oil shows a striking antiviral activity. The essential oil monoterpenes show repressing activity against Herps Simplex Virus (HSV-1) above 80%. It was carried out by deactivating free virus particles and activating on HSV in dosage dependent manner, which result in deactivation of HSV-1 virus. The individual monoterpene cause toxicity in contrast to the combination of monoterpene with tea tree essential oil and this combination also increases the selectivity index [33].

2.1.7: Neuroprotectant

Synthetic drugs were used for treatment of nervous disorder causing side effects. Therefore, it up thirst the researcher to synthesize the drugs with no side effects and plants essential oils were greatly acknowledged for this purpose. Essential oil as antioxidants treats oxidative specie and free radical induced disorders, and thus control neurological and age-related disease. Lavender essential oil antioxidant activity is linked with neuroprotective and cognitive enhancement effects in Alzheimer's disease patients [34-35].

2.1.8: Cardioprotective

Essential oil and its components act as therapeutic agents against cardiovascular disease [36]. The essential constituents diminish the oxidative stress, regulate vascular contractility, obviate hypertension and prevent hypo or hyper responsiveness to vasodilatory and vasoconstrictor agents in aortic rings The control of oxidative stress result in decrease of blood pressure which can be achieved by using plant essential oils antioxidant potential [37].

2.1.9: Anti hyperpigmentation

A condition known as hyperpigmentation occurs when the certain areas of skin become darker than others. It is mainly caused by an increase in melanin production. Tyrosinase is an enzyme that catalyze the melanin production in skin commanding to epidermal hyperpigmentation which results in several dermatological related problems like melasma, freckles and aging signs [38]. Plants secondary metabolites have anti-tyrosinase potential and are great natural source of skin whitening agents and de-pigmentation. Eucalyptus flower essential oil has great potency to lessen the melanin production by suppressing the tyrosinase activity. Essential oils inhibit the signaling pathway that regulate the tyrosinase activity. Essential oils from some other plant species such as lime mint also possess the property of anti-hyperpigmentation [39].

2.2 Factors affecting the quality of essentials oils

Multiple factors (extrinsic and intrinsic) effect the yield and chemical content of essential oils. Different researches conducted to scrutinize the factors effecting yield and chemical contents of EOs. Some of the factors responsible for such variation are discussed here.

2.2.1 Geographical variations

Composition and yield of essential oil varies with geographical conditions. Altitude changes result in variation of temperature, humidity, water availability, wind speed and rate of radiation. The change in these condition result in variation of eco-physiological reaction in plants. In this regard the research was conducted in Turkey by growing *Kundmannia anatolica* fruits at different altitudes (1560, 1002, 820 and 400m) and it was observed that geographical factors influence the volatile constituents of essential oil extracted from fruits [40].

The results expose that, variation in altitude effects the biosynthesis of essential oil constituents. The terpenoids and oxygenated monoterpene synthesis was more significant at low altitudes and the synthesis of sesquiterpenes were significant at higher altitude. In addition, some constituents of sesquiterpene such as heptanol, α-cadinene and α-santalal were absent from fruits essential oil when fruits grown on lower altitude but present in fruits obtained from higher altitudes. In contrast the oxygenated monoterpenes found when growth take place at low altitude. Thus, the altitude changes as a geographical variation influence the chemical content and of essential oil [41].

2.2.2 Plant growth cycle

Researchers found that harvesting plant at different stage of growth result in variation of yield and composition of essential oil. Researches revealed that essential oil yield is high when plant fruits were harvested at pasty stages (moisture 54%) as compared to the plant fruit harvested at ripening (5 % moisture) in case of *Trachyspermum ammi* sprague. The yield is 7.1% w/w in case of pasty stage and 3.2% w/w when harvested at fruit ripening stage. 12 major constituent identified when essential oil was obtained from ripe fruit while 10 major compounds identified from fruit harvested at pasty stage [42].

2.2.3 Drying time and technology

Drying time, drying technology also effect quantity and the quality of essential oil obtained from different plant species. According to research findings essential oil obtained from fresh and dried flowers of *Lavandula angustifolia* had variation both in its yield and composition. 65 chemical compounds were extracted from fresh flower, and 73 compounds from dried flower after hydrodistillation. Dried lavender flower essential oil comprised of small quantity of monoterpene esters and accelerated amount of alcohol as compare to essential oil of fresh lavender flowers. Drying may enhance the activation of enzymes which result in production of volatile compounds [43].

3. Chemical composition of essential oil

These lipophilic, volatile, organic liquids contain a mixture of about more than 300 different chemical compounds [44]. Due to low molecular weight and high vapor pressure essential oils are partially found in vapor state at room temperature and atmospheric pressure [26]. Two to three chemical components present as major concentration in essential oils while remaining chemical constituents are present in trace amounts [45].

Essential oil major constituent is categorized into two main family's terpene and terpenoids on basis of hydrocarbon skeleton. The first one terpene, formed by combination of 2-4 isoprene units and are called as monoterpene, sesquiterpene and diterpene respectively and is wide occurring group of naturally occurring hydrocarbons. The general structural formula for terpene is $(C_5H_8)_n$ where n is number of isoprene units [19]. The monoterpene ($2 \times C_5$) has 10 carbon atoms and represent a major composition about 80% in essential oil while sesquiterpene has 15 carbon atoms having molecular formula $C_{15}H_{24}$. They represent cyclic as well as acyclic hydrocarbon structures. The second one terpenoids also known as isoprenoids. These isoprenoids are terpenes oxygenated derivatives such as phenol, ether, ketone, aldehyde, acids and esters [46]. Another oxygenated molecules class and its derivative also found in essential oils known as phenylpropanoids [47]. This is a special case as in Cinnamon bark, vetiver, Sassafras and clove. Essential oils main composition include terpene, terpenoids and phenylpropanoids and thus it results in representing a wide variety of chemical structures and compounds as shown in fig 3 [48]. Distinct primary metabolic precursors are used for synthesis of terpenoids and phenylpropanoids by using different pathways such as shikimate pathway for phenylpropanoid biosynthesis and mevalonate pathway for terpenoid synthesis [26].

4. Essential oil secretion

Essential oils are biologically produced, assembled and reserved in certain plant histology structure designated as secretory glandules. There is need to differentiate between essential oil and plant essence. Plant essence are natural secretions produced by specific secretory cells in plants. Volatile organic liquid (essential oil) is squeezed by hydro or steam distillation and therefore they can be referred as distilled plant essence. There are two main types of these histological structure, exogenous secretion associated with the secretory glandules located on plant surface and endogenous secretion associated with glandules located in internal organs inside the plants. They may also be confined in cytoplasm of some cells in more than one organ of plants [19]. Secretion is a general phenomenon occur in living cells and it may contain salts, latex, sugar, fat, waxes, resins as well as essential oils. We can discriminate different types of glandules as:

4.1. External secretory tissues

The excreting tissues located outer side of the plant are called external secretory tissues. The secretory structures involved in external secretion are epidermal papillae and glandular trichomes. The epidermal papillae are conical epidermal cells located in flower petals of different botanical families' plants which secrete essences. Example includes *Rosa damascene* of Rosaceae family and *Convallaria majalis* of asparagaceae. The glandular trichomes or secretory bristles are evolved from epidermal cells. Various types of these bristles include sessile and stalked trichomes [49]. There are three distinctive types of stalked trichomes: digitiform, capitate and peltate trichomes These glandules are sites for biosynthesis and collection of essential oil and a unique feature of family lamiaceae. The essential oil is collected in between common cuticle and secretory cells [50].

4.2. Internal secretory tissues

The secretory tissues present inside the plant are internal secretion tissue. These includes the secretory canals, schizogenous pockets and cells with intracellular secretions. Small secretory canals are formed by aggregation of secreting cells and extended throughout the length of plant such as stem. Example includes, *Petroselinum sp. Daucus sp. Pimpinella sp.* of apiaceae family. The schizogenous pockets are spherical intracellular spaces which are filled by essential oil droplets synthesized by cells such as epicarp of fruit. Example include: *Citrus sp*. of rutaceae and myrtaceae family. Intracellular isolated secretion cells have specific function of collecting and secreting essential oil within their vacuole. These cells die when the essential oil concentration reaches high level. The plant organs involved in this category are leaves, stem and rhizome. Example include *Cinnamomum, Laurus nobilis, Acorus calamus* of Lauraceae family [19].

5. Essential oil extraction methods

In order to extract essential oil from plant raw material many extraction techniques are used. These techniques can be divided into two major categories: inventive/advanced techniques and traditional/classical techniques as depicted in fig 4. New innovative technologies dominated over classical techniques due to the benefit of reduction in energy and time, improved extraction yield, and enhanced essential oil quality [51].

5.1. Conventional and classical methods

In classical methods the essential oils were squeezed from plant material by using water distillation technique based on heating. Several conventional methods used for extraction are discussed here [51].

5.1.1. Hydrodistillation

The simplest and oldest extraction method was hydrodistillation. Avicenna was the first scientist who caried out extraction by using alembic. Firstly, rose essential oil was extracted. Water and plant material both are placed in alembic apparatus and boiled. Extraction device components include source of heating, reaction vessel, condenser and decanter. Azeotropic principle-based extraction takes place. The plant material along with water introduced in vessel (alembic) and heated at atmospheric pressure. The essential oil and water are separated by co-distillation process [52].

5.1.2. Entrainment by water steam

It's one of the most used official procedures for extracting essential oil from raw plant material. The working principle is same as hydrodistillation except that there is no direct contact between plant material and water. The chemical conversions are less due to shorten duration [53]. The other modifications include vapor hydrodistillation and steam distillation. In vapor-hydrodistillation the extraction takes place in alembic but the difference is that a perforated plate or grid is employed to keep up the suspended plant over the base of still holding water in order to prevent their direct contact. The water vapors are formed at bottom position and injected up to plant raw material which carries the volatile chemical compounds. The advantages of this method over the simple hydrodistillation are that, less time is required, loss of polar molecules is minimized and artifacts are reduced. In steam distillation or vapor-distillation the extraction is obtained in alembic similar as vapor hydrodistillation but the vapors are formed outer side of the alembic chamber. The steam used in this can be superheated or saturated above atm pressure. The steam that was introduced at the bottom and move upward, passes through plant raw material and extraction obtained. It has same advantage as that of vapor hydrodistillation to reduce artifacts over the simple hydrodistillation [54].

5.1.3. Organic solvent extraction

The plant raw material is dissolved in organic medium and mixture is concentrated by drawing out the solvent at moderate pressure. This technique involves the cold extraction and thus minimize the effects like chemical artifacts and alterations. In hydrodistillation the procedure involved the submerging of plant matter in water and it may result in solubilities of some plant constituents into water and thus lower the pH. In case of organic solvent extraction, the organic solvent may pollute the fragrance and food to which organic solvent added. Thus, this technique is harmful for medicine and food applications. The combination of organic solvent extraction technology with steam distillation may avoid this handicap [55].

5.1.4. Cold pressing

Cold pressing is a conventional technique for extracting oil. In this the oil is extracted mechanically by pressing the plant fruit which result in breaking of fruit sacs and thus the oil is released which is collected. The simplest example is the citrus fruit oil extraction. The resultant liquid is oily water which is then centrifuged to obtain oil. The obtained material is plant essence that is used in medicine and food industries [54].

5.2. *Innovative methods*

The major detriment of conventional extracting procedure is that the product obtain is highly thermolabile (a substance which undergo decomposition like hydrolyze, oxidation and isomerization or change under the action of heat like) due to high temperatures. The oil extracted is highly damaged especially when extraction time is elongated. The new techniques developed which result in reduction of time, solvent used, energy consumption along with keeping the chemical constituents of essential oil at its original state [56].

5.2.1. *Supercritical fluid extraction*

This extraction carried out at well-defined critical conditions, critical temperature (Tc) and critical pressure (Pc). When the extraction carried out under these conditions the liquids show fascinating properties like high diffusivity, low viscosity and density close to liquids. The mostly used solvent for supercritical fluid extraction is CO_2 because of its i. easily reachable critical points (Tc:31.2 C, Pc:72.9atm), ii. Nontoxic, iii. Chemically inert, iv. amiable to thermolabile molecules, v. can be easily removed from extract, vi. Non-flammable, vii. Less expensive, viii. High purity availability [57].

The supercritical carbon dioxide is obtained by compressing and heating the CO_2 at elevated conditions. Supercritical CO_2 is passed from plant raw material, it carries the botanical particles (volatile matter of plant) and passes through a no. of separators where the CO_2 is decompressed to separate extract from fluid. The released gas could be used again and again [58]. The disadvantage includes the high-cost equipment, difficulty in installation and maintenance operations. This technique produces essential oil with superior quality and better properties. Studies investigated that the supercritical oil carries better biological activity ac compare to hydrodistillation extracted essential oil. Example include the supercritical carrot essential oil's superior performance against Bacillus cereus [59].

5.2.2. Subcritical extraction liquids (CO_2 and H_2)

Subcritical water is used by certain researchers to extract essential oil. In order to maintain the liquid state of water, the subcritical condition is reached when the temperature is below the critical point and the pressure is higher than the critical point. It is a powerful, rapid extraction procedure involved lower temperature and the fluid obtained has very interesting properties. The instrumentation for subcritical water extraction (SWE) consists of an oven housing the extraction cell and extraction process, a valve to regulate pressure, and a water reservoir connected to a high-pressure pump for introducing the solvent into the system. At the end of the extraction system, a vial is used to collect the extracted materials. The mastery of this technology over traditional is that: cost effective, reduction in time, rapid extraction, environment friendly, softer, less solvent expenditure, prevent loss of volatile material and thermolabile components. The subcritical water extraction is less costly as compare to subcritical CO_2 extraction because subcritical CO_2 requires specific equipment's. The essential oil obtained is of high quality [19].

5.2.3. Ultrasound assisted extraction of EOs

This technique used laboratory scale size equipment and was developed in 1950. The botanical raw material along with any other solvent in subjected to ultrasound which result in acceleration and thus release of essential oil. This technique mostly employed for seeds oil extraction. Ultrasound wave used in this technology ranges in between 20 kH-1 MH. The ultrasound waves cause intense vibration to plant membranes which result in release of essential oil drops. The sonication produced easily destroy the walls of glands to which essential oil is stored in plant raw material. The technique is usually simple and has advantage over conventional method like reduction in time, inexpensive equipment, improved efficiency and rates [60].

5.2.4. Solvent free microwave extraction

Dry distillation and microwave heating energy are combined in this process. This process is performed at atmospheric pressure without the use of water or any organic solvent [61]. When in situ plant material water content is heated it result in swelling of glands and thus bursting. The essential oils are released which then evaporated by azeotropic distillation This technique completes the extraction process in only 30 minutes while the conventional hydrodistillation completes the process in 2 hours [62].

5.2.5. Microwave hydro diffusion and gravity

This method involves the placement of plant material in microwave oven and the direct contact of microwaves with biological water (the water present in plant material produce steam) takes place. This steam makes it easier for plant material to release its essential oils. A spiral condenser is attached at the bottom position of oven. The mixture moves downward to the condenser under the action of gravity where the oily essential oil condenses. The condensate oil is stored in a flask that resembles a separatory funnel, where it is kept until it is needed [63].

6. Nanoparticles

Human ideas and inspiration periodically give rise to novel science and technology. The term nanomaterial is defined as “a substance, whether natural or artificial, that accommodates loose, organized, or aggregated particles with outside diameters ranging from 1 to 100 nm” [64]. The composition, size, shape, and origin of the nanomaterials are used to categorize them. Nanomaterials earned eminence in technology development due to tunable biological, chemical and physical characteristics in contrast to their bulk content [65].

6.1. Nanoparticles – a historical perspective

Nanoparticles have stunningly long history. Nanoparticle preparation is not an entire result of recent research and nor narrow down to the man-made synthetic materials. Nanoparticles also occur in nature from a long time ago include inorganic compound (aluminosilicates, metals, iron oxyhydroxide and others), organic (viruses, proteins, polysaccharides and others) and nanoparticles produced accidently like volcano eruptions, microbial process, wildlife and weathering [66]. It is not necessary that nanoparticles only prepared in laboratory but they exist in nature for a very long time ago and dated back to prehistoric periods. Metal nanoparticles show more lustrous effects as color pigments regarding glass technique. Gold is used for imparting remarkable red color to glass. Ruby glass is one of the examples prepared by Roman and placed in British Museum as Lycurgus Cup [67]. Abraxane^{TM} (a human serum containing albumin nanoparticles) in 2005 was manufactured and discharged to pharmaceutical market [68]. In 2012 dye sensitized solar cells containing TiO_2 nanoparticles were available for commercial use. In 2014 up to 1814 items based on nanotechnology were accessible in 20 countries [69].

6.2. Methods for Synthesis of nanoparticles

It is possible to manufacture a wide range of nanoparticles using a multitude of synthetic techniques. In this section we discuss both physical and chemical ways for nanoparticle synthesis.

6.2.1. Physical methods

Different techniques for the synthesis of nanomaterials are selected depending upon the need. The particular method for synthesis of nanomaterial is selected depending upon the availability of facilities. Every method has its own advantage and limitations. For small scale production of nanomaterials physical methods are used [67].

6.2.1.1. Ball milling

A novel approach for fabrication of nanoparticles at room temperature. The apparatus used for ball milling consist of container which contains hard balls. The hard balls are made up of carbide or steel. Nanocrystalline Cr, W, Ag-Fe and Co can be manufactured by this method. The ratio of material to hard balls is 1:2. Air or inert gas is filled in the container containing hard balls and swiftly revolved around the center axis as shown in fig 5. The substance crushed between the container's walls and the hard balls. Size of nanoparticles can be controlled by duration and speed of milling apparatus. Different forms of mills including vibratory, planetary, vibrating and rod can be used [67].

6.2.1.2. Laser ablation

This method apparatus includes a vacuum chamber in which the target sample is placed. The laser beam of high pulse is concentrated on the sample, as a result plasma generated. The generated plasma is transfigured into colloidal solution of nanoparticles. The second type of laser in the form of harmonic group is consistently used to articulate nanoparticles. The factors which effect the final nanoparticle creation are type of laser, type of solvent, some pulses and pulsing time [70-71].

6.2.1.3. Sputtering

Through the use of an inert gas ion beam, solid target material is vaporized using this technique. By this process parallel beams of nanomaterial formed and nanostructured films are sedimented on silicon substrate. Sputtering procedure took place in vacuum chamber. The cathode is made of the targeted material and anode comprised of material on which deposition take place. A high voltage is put across the target (cathode) and inert gas is injected into the vacuum chamber. The free electrons move and collide with sputtering inert argon gas atoms and resultantly the gas ionized and plasma generated. The process continues and the positively charged gas ions captivate to the target cathode and strike continuously on this target. As a result, the surface energy of the target material increases then the binding energy and thus atoms from target released. The released ejected atoms collide with gas molecules in chamber and scatter which result in formation of diffuse cloud on specific substrate [72].

6.2.1.4. Flame pyrolysis

The working principle for synthesis of nanoparticles in flame pyrolysis involves the direct spraying of precursor (liquid, solid, gas) into the flame. This method is suitable for those precursors whose vapor pressure is very low in the state of vapor. The gas, liquid and solid are exposed to flame and permitted to form nanoparticles [73].

6.2.1.5. Chemical etching

In nanostructure formation etching procedure involves the removal of material by physical or chemical means. The basic principle follows in etching is that the material which is to be etched, selectively exposed to etchant, etchant reacts with parts of the material to be expunge and dissolve it leaving subsequently pattern or structure. Two approaches involved in etching process: dry etching and wet etching. Inductively coupled plasma ICP is most commonly used dry etching approach. ICP are roomy equipment, require expensive setup and dangerous gases used during operational procedures. Due to these negative aspects, it is not affordable by laboratories and used at very low scale in laboratories. Wet etching involves metal assisted chemical etching and it requires only metal catalyst along with etchant in a beaker. Due to this simple setup its application increases day by day.

The design of nanostructure can also be tailored by regulating etching process [74].

6.2.1.6. Sonochemical method

Ultrasound waves are element of acoustic spectrum. The sonic spectrum ranges from 10 MHz to 20 kHz and the region of acoustic spectrum ranges from 1 MHz to 20 kHz is experienced in sonochemistry. Sonochemistry deals with the ultrasound acoustic waves which irradiate the aqueous solution and the sound energy in sufficient amount result in formation of nanostructures under the influence of ultrasound irradiation. The sufficient sound energy play role in breaking the bonds which facilitate the formation of nanostructures [75]. Pd-CuO nanohybrids are formed by using sonochemistry procedure. In this the copper salt sono-chemically fused in presence of Palladium and water. The metal salts revolutionized into their oxides with the assistance of ultrasound energy in the prevalence of water and Palladium [70].

6.2.2. Chemical methods

6.2.2.1. Sol gel

A chemical method employs to fabricate nanoparticles especially metal nanooxides. The sol gel process concern to the transition of sol (liquid) into gel (solid) phase. This can be attained by modifying the concentration or prepared solution pH. The techniques which are employed to attain such alteration are drying or deportation of solvent [76]. The sol gel method includes the steps of hydrolysis followed by condensation and then thermal decomposition (aging and drying) of metal precursors solution. A balanced solution of metal precursors or metal alkoxide is prepared known as sol. When this solution hydrolyzed and then condensed the gel is formed, as a result the viscosity increases [70]. The obtained gel is wet which to be dried by using different methodologies and it may take several days. The process of drying determines the properties and application of the gel formed. The process of drying is accompanied by shrinkage of gel which may result in formation of cracks, the most important issue here is to prevent the formation of cracks. The obtained gels are then powdered and calcined. The gels can be grinded by specific mills and result in creation of nanoparticles [76].

6.2.2.2. Hydrothermal

Hydrothermal approach is extensively used procedure for nanoparticle synthesis. This approach is based on chemical reaction in water as solvent and requires a broad temperature range (room temperature to very high temperature) [70]. The precursors and solvent placed in autoclave at elevated temperature and pressure. Chemical reaction takes place in the vessel at increased temperature and pressure. After definite time the autoclave containing product is cooled and the solution containing product is centrifuged. Then the upper layer is poured off and washed with double de-ionized, distilled water. The washing procedure is reciprocated to clear away the impurities of product, the product is dried and then studied under different characterization techniques [77].

6.2.2.3. Superfluid

Helium extremely cold droplets are superfluid and used for the synthesis of nanoparticles. Nanodroplets of helium are extremely small consisting of liquid helium. The temperature of the helium droplets is as low as 0.38k. When any atom or molecule come across the helium droplet they will be captured. In many cases the atoms or molecules will migrate inward to the helium droplets. The binding forces between the atom migrate to inward and the helium droplet is very weak, therefore, picking up of more than one molecule or atom result in the formation of clusters. When more atoms or molecules added to the inward portion of helium droplet in sequence nano onions (core shell nanoparticles) formed. The properties of these nanoparticles are extremely different from other techniques formed nanoparticles [78].

6.2.2.4. Microwave method

Microwave method involves the production of noble nanoparticles within a short time range. The plant extract or precursors extract is prepared according to the nanoparticles to be formed using this technology. The prepared extract is placed in the oven where the sample is irradiated with microwaves. The microwaves provide suitable heat and other optimum conditions required for nanoparticle growth. The sample solution is centrifuged and dried which result in formation of suitable size nanoparticles [79].

6.2.2.5. Electrodeposition:

It is a well acknowledge technique for synthesis of nanoparticles. Electrodeposition involves the impeachment of metal or other material on a surface by process of electrolysis. The electrolysis used for formation of nanoparticles comprise of cathode, anode, current source and electrolytic bath. Electrolyte solution is prepared by using the reactants which are to be deposited. External current either direct or pulsed is applied to the system and it result in the interference between electrodes and electrolytic solution. The nanostructures in the form of 2D and 3D deposited on the respected electrode successfully [80-81].

7. Recent advances in nanoparticles formation techniques of essential oil

Nanoparticles are usually made by physical and chemical means on a massive scale, which could potentially result in chemical release to the environment [82]. Toxic chemicals are used in nanoparticle synthesis. These compounds when released into environment cause dangerous metabolic processes [83]. The biological synthesis techniques have been developed to overcome the challenges associated to old typical methods and for safe usage in various applications to lower the risk associated with the discharge of chemical-based nanoparticles into the environment [84]. In contrast to conventional methods, Fig. 10 illustrates the need for essential oil in the fabrication and improvement of nanoparticle attributes [85]. The harmony between nanotechnology and biotechnology as bio nanotechnology materialize an efficient substitute for several sustainable approaches to environment. Bio nanotechnology skillfully articulate the worth of plant extracts, as obtained by aforesaid techniques, with the assistance of high-power nanostructures. The essential oil as plant extract obtained can be used to fabricate nanomaterials by using different protocols [86].

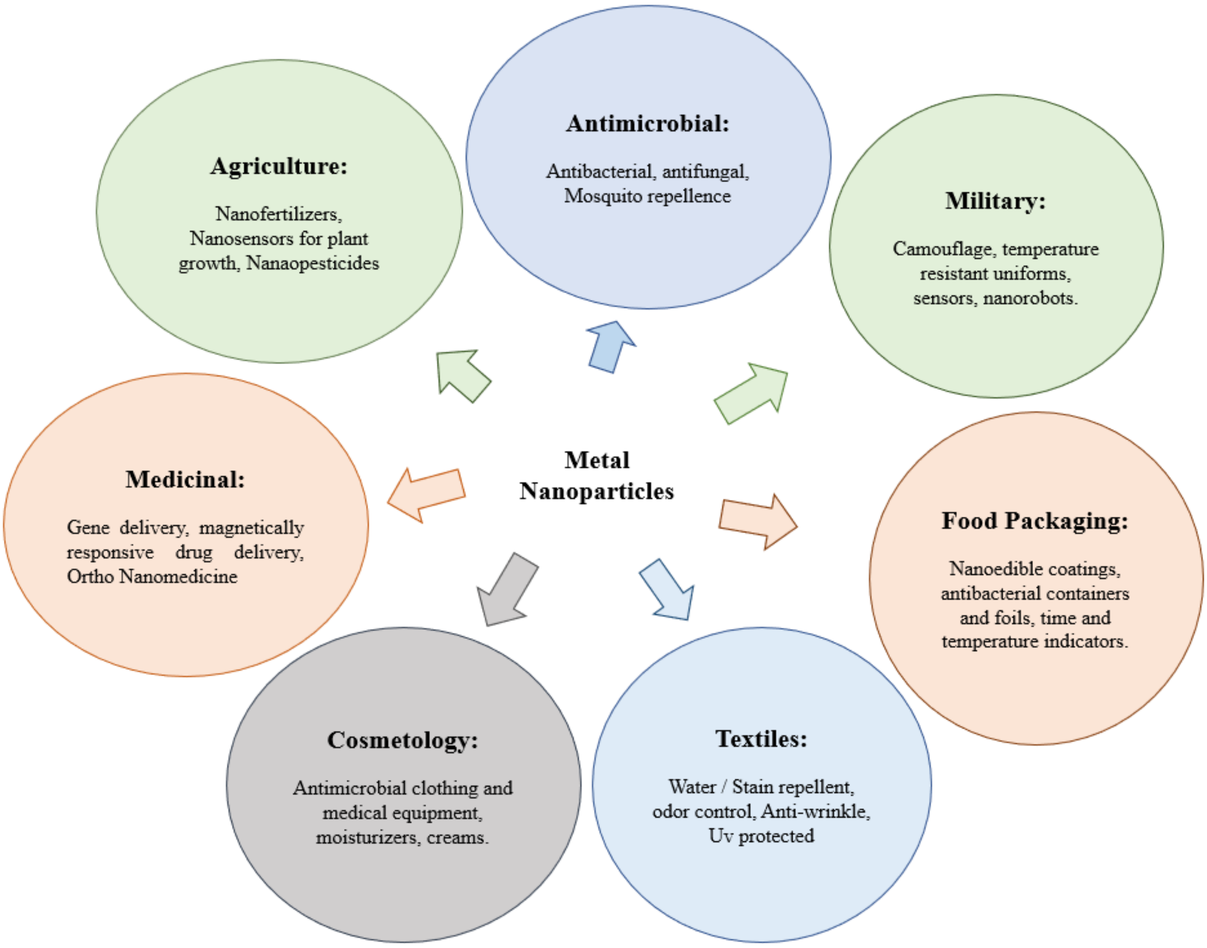


Figure 1: Applications of nanoparticles

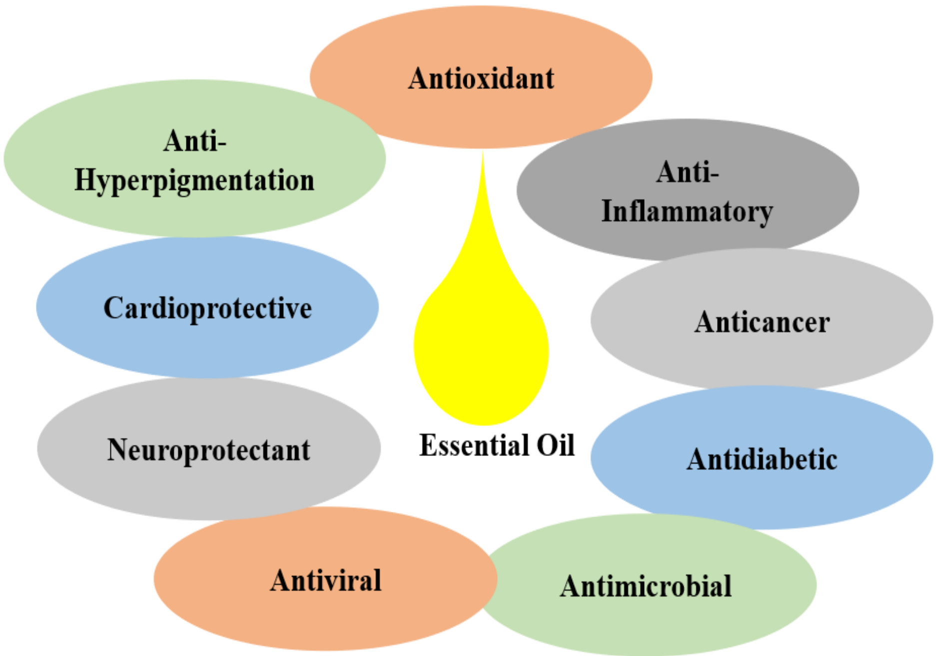


Figure 2: Benefits of essential oil

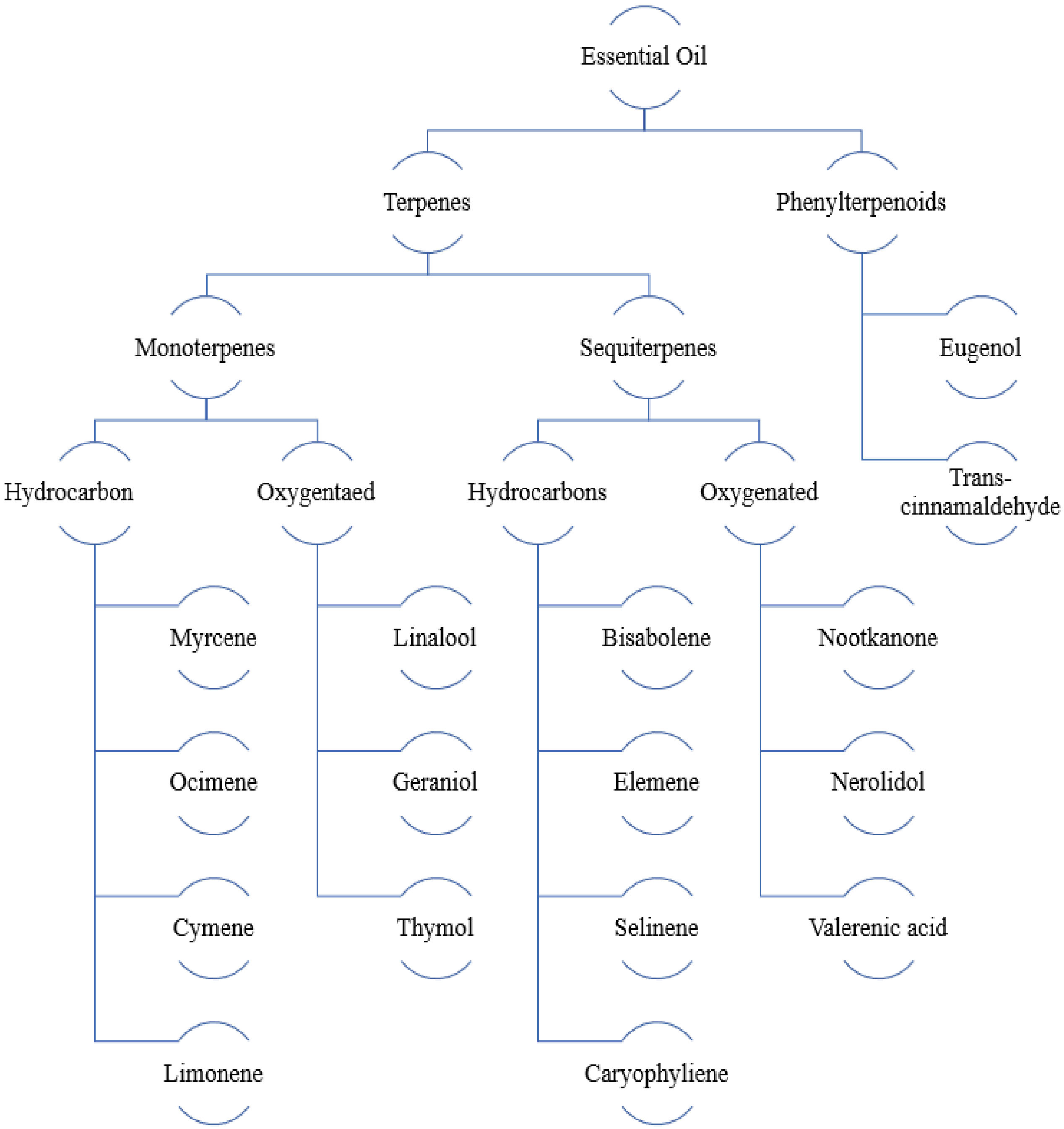


Figure 3: Some building blocks of essential oil

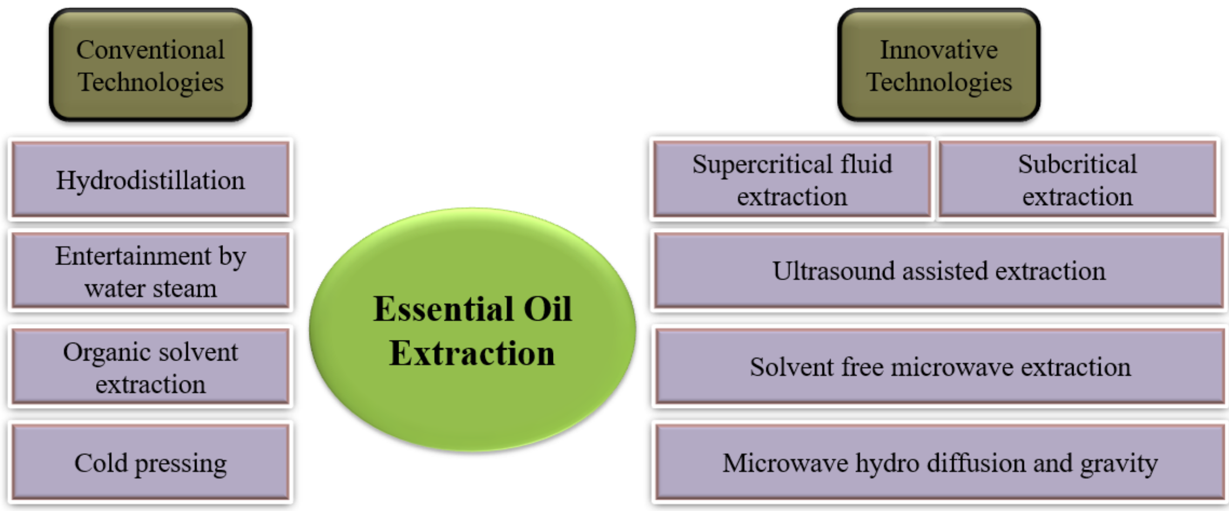


Figure 4: Essential oil extraction methods

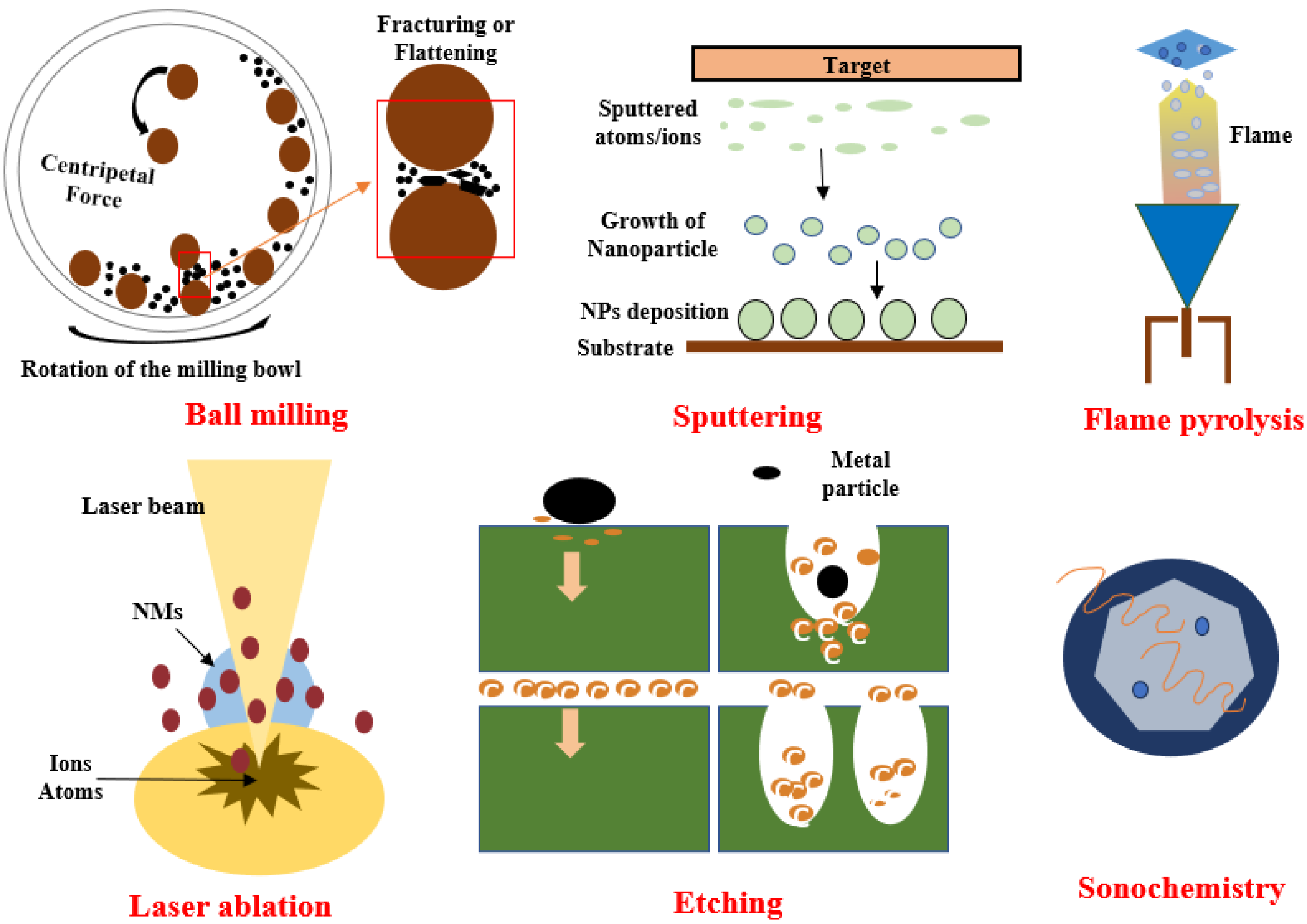


Figure 5: Physical methods for synthesis of nanoparticles

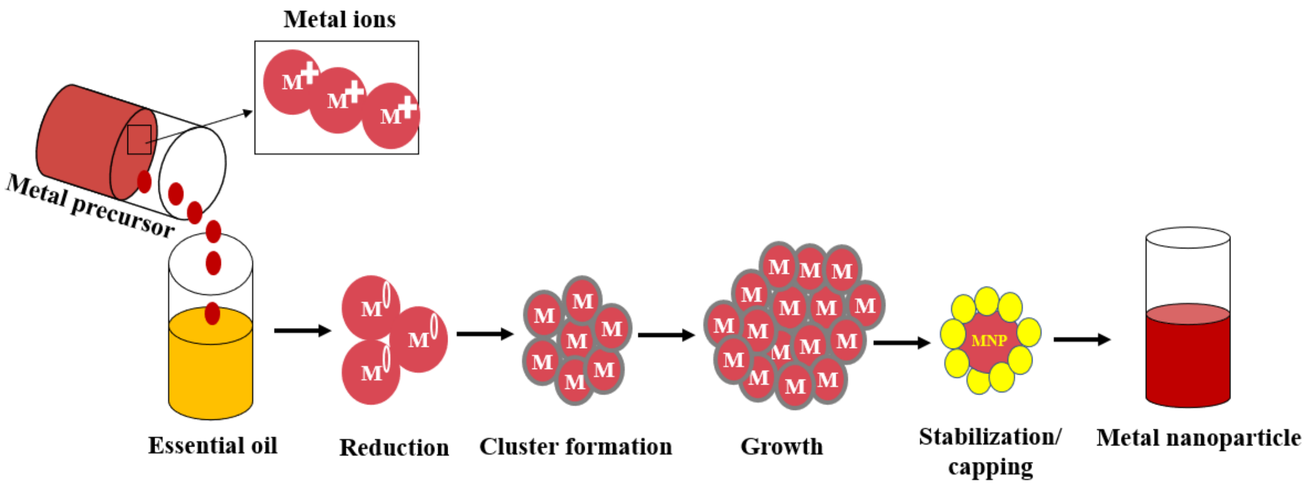


Figure 6: Biosynthesis of essential oil-based metal nanoparticles

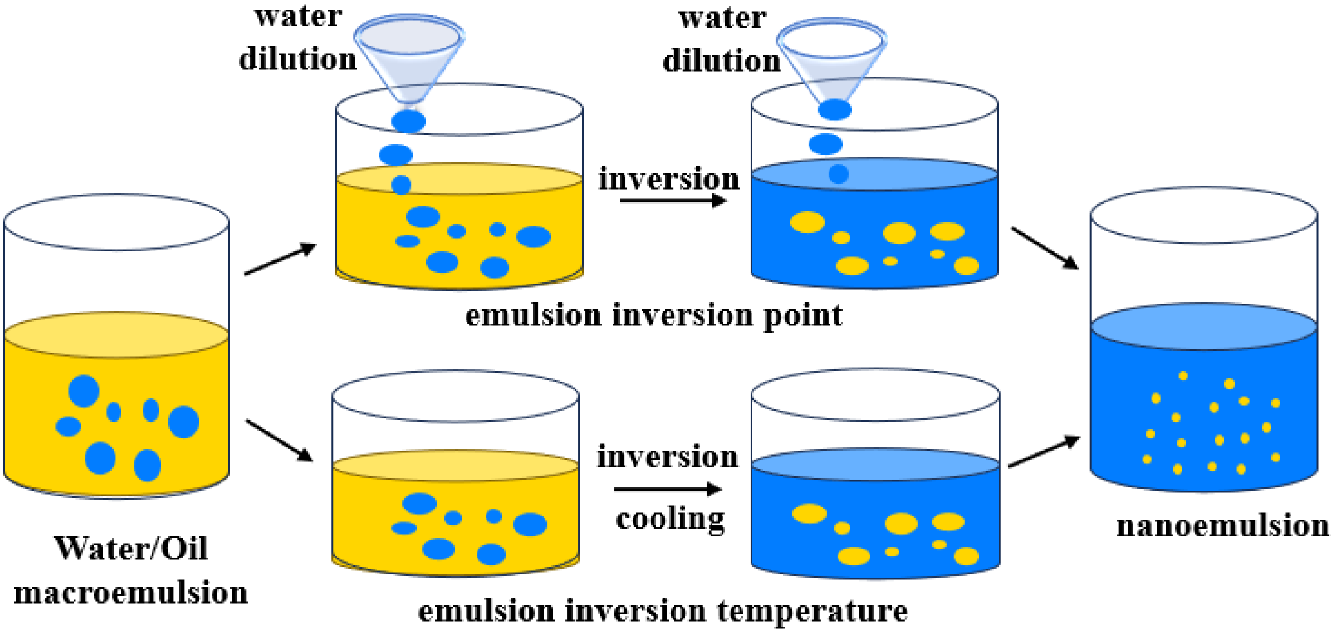


Figure 7: Nanoemulsion preparation by using low energy methods

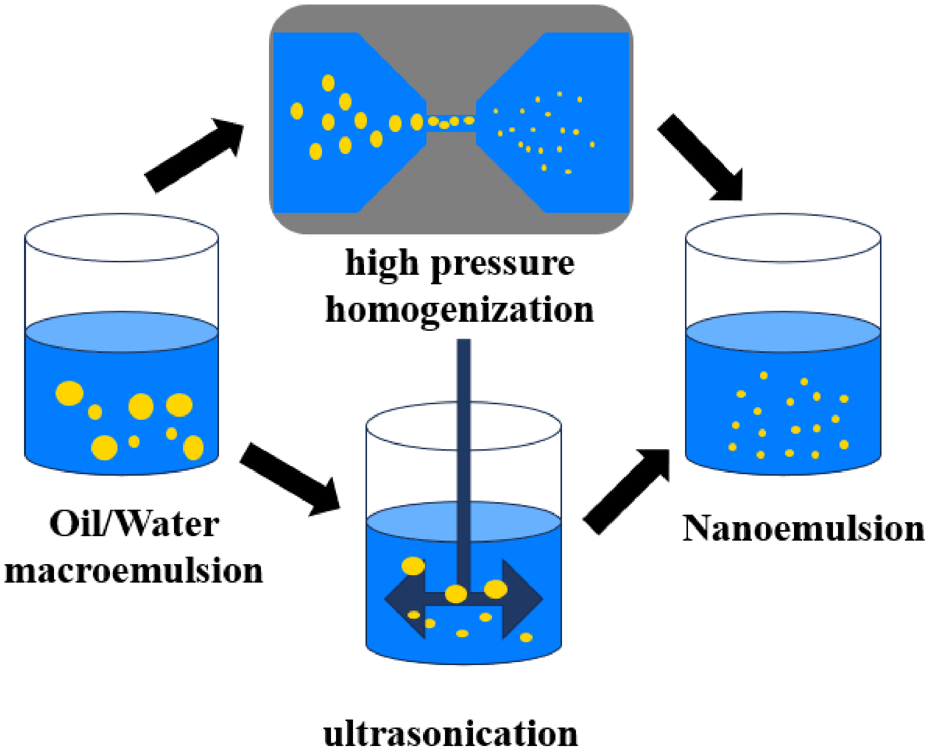


Figure 8: Nanoemulsion preparation by using high energy methods

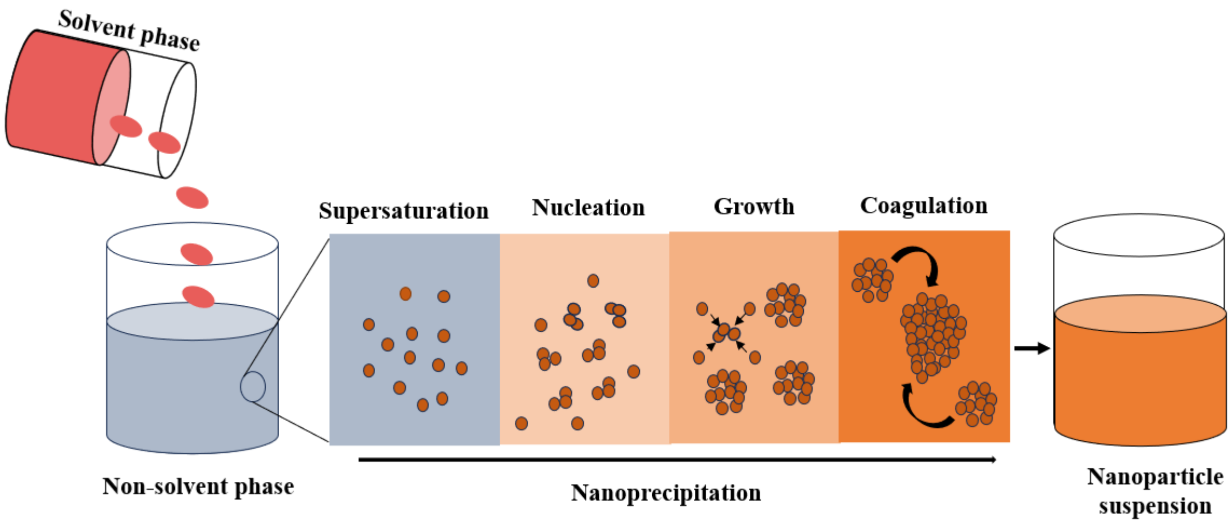


Figure 9: Nanoprecipitation mechanism

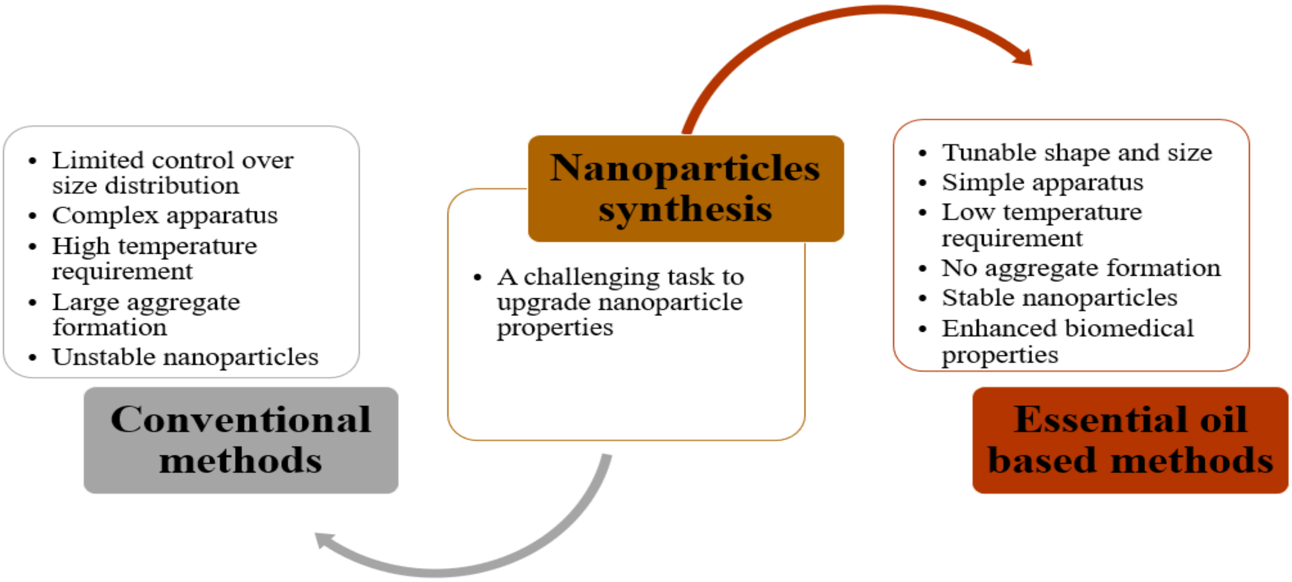


Figure 10: Need of essential oil for nanoparticles preparation

Table 1: Essential oil-based production of metal nanoparticles

S. No.	NPs	Plant Name	Applications
1	Au	*Mentha piperita*	Antifungal
2	Au	*Curcuma pseudomontana*	Antioxidant, cytotoxic
3	Au	*Nigella sativa*	Anticancer, Antibacterial
4	Ag	Rosemary	Antioxidant
5	Ag	Orange peel	Catalytic
6	Ag	Thymus syriacus	Antioxidant

7.1. Biosynthesis of essential oil-based metal nanoparticles

Nanoparticles are accomplishing prominence day by day due to higher surface to volume ratio, which has a consequence in rapid dissolution as compare to other particles. Industries devote great consideration to nanostructures based on transition metals due to their remarkable properties including low melting temperatures, high mechanical resistance, specific magnetic and enhanced optical properties [2]. The interest of nanoparticle formation increases and the main objective is to prepare stable nanoparticles. The most efficient method for synthesis of nanoparticle, free from toxicity and other deterioration is biosynthesis which involves the use of plant extracts or essential oil. This vital process is known as green synthesis or green process of nanoparticle manufacturing [70]. Two things, metal ion solution and biological reducing agents, are most important in green synthesis of nanoparticles [87].

7.1.1. Metal ion solution (metal precursor)

The solution of metal ion whose nanoparticles are to be synthesized is prepared by using suitable solvents like deionized water. This solution act as metal ion source. The metal ion is then reduced to metal nanoparticle by some biological reducing agent [70].

7.1.2. Biological reducing agents

The plant extract as essential oils are lipophilic in nature and contain many organic compounds. The organic compounds comprised of different functional groups with varying structures. The aldehydes, ketones, alcohol, lactone of terpenoids (major constituent of essential oil) possess active functional group. These functional group includes -CH_2, -CH_3, C=C, C-H, C-OH, C-O-C, C=O, C-O, C-N and many more present at different position of organic compounds in essential oil which involve in reducing and stabilizing metal ions and thus result in formation of metal nanoparticles as described in fig. 6 [2]. The particles in nano size range tend to agglomerate to lower surface energy. Terpenoids cling to the surface of nanoparticles and stop agglomeration, a significant issue with conventional techniques [85].

Metal nanoparticles prepared by using suitable essential oil obtained from different plants. Nano gold particles (NGP) are synthesized by using the essential oil extracted from the *Anacardium occidentale* plant fresh leaves. During experiment at 100℃ auric acid is reduced which actually involves the reduction of Au^{3+} to Au^0 and at that time the solution color altered to pink. FTIR analysis shows the actual mechanism involve in the reduction of gold (III) to gold nanoparticle (Au^0) and then its stabilization. It shows that the acetone is oxidized to carboxylic acid at high temperature which result in reduction of Au (III) to nano gold particles (NGP). The carboxylic acid formed release a proton and form a carboxylate ion. This carboxylate ion attached to the positive NGP surface and stabilize it. This negatively charged carboxylate ion stabilize the nanoparticle and prevent it from aggregation [2-88].

By using green synthesis process the nano silver particles (NSP) are prepared. The essential oil obtained from *Myristica fragrans* is used which reduced the Ag (I) silver ion to Ag (0) silver. In this preparation, $AgNO_3$ solution along with essential oil are mixed at 100°C. During the reaction the pH is varied (7,8,9,10) and solution turns from yellow to golden yellow in color. Finally, the golden yellow color change into red which indicate the formation of nano silver particles (NSP). The FTIR and GC-Mass analysis shows the mechanism involved in NSP formation. In the presence of acetone at 100℃ weak tertiary alcohol act as good reducing agent and form nanoparticle. Many other plants essential oils also used for preparation of silver nanoparticles [89]. Numerous gold and silver nanoparticles with spherical shapes and distinct properties are synthesized from essential oil of different plants as described in table 1 [2].

Essential oil enhances the biopharmaceutical action of nanoparticles. Nanoparticle shapes play an important role in treating pathogens. The nanoparticles with tunable size, specific shape and extraordinary properties can be synthesized by using essential oil. Researcher reported that the activity of triangular shape nanoparticles is considerably greater in comparison to that of the spherical, nano-rods and nanoparticles against gram negative bacteria [90]. Essential oil prepared nanoparticles exhibit enhanced antioxidant efficiency in comparison to synthetic antioxidants [91].

7.2. Nano encapsulation of essential oil

Encapsulation is the process of wrapping a biologically active substance in a carrier matrix to regulate the rate at which the bioactive substance releases its active ingredients. Essential oil contains 95% volatile and 5% non-volatile components [92]. Nanoencapsulation is a promising channel to address the instability of essential oil with respect to UV radiation, pH, and temperature. Particle size of carrier matrix plays an important role in determining the functional characteristics of the capsules [93]. The particle of carrier in the range of nanometer result in nanoencapsulation. The encapsulation increased the surface area which enhance the dispersion rate. The nano capsules are efficiently dispatched to the targeted cells due to their small size which assist them to penetrate through capillaries into tissues such as liver [94]. Nanoencapsulation found a number of applications in the field of medicine and food. Various procedures involved in nanoencapsulation of essential oil are discussed in following section.

7.2.1. Nano emulsions

Emulsions are described as dispersion of two liquids that are not miscible into each other, and one of dispersed phase is present as droplet form in continuous phase. Oil and water are most broadly experienced elements in emulsion along with stabilizer. The dispersed droplet size determines the type of emulsion. The nanoemulsion droplet size ranges from 20 to 500nm [93]. Nanoemulsion found in different forms like water (W) in oil (O) or oil (O) in water (W) depending upon hydrophilic and hydrophobic proportion. Water/oil nanoemulsion contain lipophobic droplets dispersed in lipophilic phase (organic medium). This emulsion act as hydrophilic components transporter. Oil/water nanoemulsion incorporate lipophilic components dispersed in continuous hydrophilic aqueous phase [95]. The nanoemulsion has gained great potential in different fields due to increased interfacial zones. The process of nano emulsion involves first the formation of water/oil macroemulsion in stirrer system by mixing water, oil and surfactant. This macroemulsion is then transformed into nanoemulsion. On the basis of energy consumption, the nano emulsions can be prepared by different methods including low energy method and high energy methods. Oil in water nanoemulsion used due to essential oil hydrophobic character in preparation of essential oil base nanoemulsion [96].

7.2.1.1. Low energy methods

Low energy methods are spontaneous and require less amount of energy. Mostly involved low energy methods are Phase Inversion Temperature (PIT) and Emulsion Inversion Point (EIP). In phase inversion temperature, the water/oil macroemulsion is prepared at temperature greater than room temperature. The emulsion which is prepared is then cooled down to room temperature. During the cooling process an inversion point reached where the mixture transformation from water/oil to oil/water occur as viewed in fig 7. During this process of transformation, the oil/water interfacial tension is very low and small size droplets formed by the use of very small amount of energy [97].

Emulsion inversion point (EIP), the first step involves the preparation of water/oil macroemulsion at room temperature. The prepared emulsion is then slowly diluted with water. During the process of dilution an inversion point reached which transform the water/oil emulsion to oil/water emulsion. The interfacial tension of oil/water at inversion point is low and thus result in formation of small droplets with small energy consumption. In phase inversion temperature (PIT) the phase inversion is induced during cooling while in emulsion inversion point (EIP) the phase inversion is induced during water dilution process [98].

7.2.1.2. High energy methods

In high energy methods a high-pressure pump homogenizer is used. In this method the prepared water/oil macroemulsions are introduced into narrow gap where large droplets are braked into small sized droplets as presented in fig 8. This process is repeated multiple time to obtained a homogenized droplet particle size. The other high energy method involves the use of ultrasonicator. The high energy shock waves rupture the droplets into small size. The process is continued until constant particle size obtained [98-99].

Nanoemulsion with minute droplet size, outstanding durability, transparent look and flexible rheology is an attractive option for utilization in drug delivery, the food, cosmetics and pharmaceutical applications [100]. Studies declared that small size nanoemulsion formulation distribute lipophilic drugs more effectively than drug suspensions [101]. Nanoemulsion antibacterial property improves the food shelf and preserve it from water inherent diseases [102].

7.2.2. *Chitosan based nanostructures*

Chitosan is a polymer material used for nano-encapsulation of essential oil. The demand of chitosan as nanoencapsulation material is increased due to its undeniable properties like bioavailability, naturally abundant, biocompatibility, non-toxicity and essential oil antibacterial properties. These chitosan base nanostructures can be prepared by using different methods including: ionic gelation, nanoprecipitation and vice versa [103].

7.2.2.1. Ionic gelation

This is an effective chitosan-based nanostructures preparation method which involves the opposite charged macromolecules interaction. It's an environmentally friendly method that doesn't require high temperatures. This method involves ionic bonding between the positively charged chitosan and the negatively charged cross-linking chemicals. Sodium tripolyphosphate is mostly used cross linking chemical. It is biocompatible to environment and easily biodegradable [104]. The process involves the dissolution of chitosan, surfactant, cross linker and essential oil with continuous stirring in order to promote the precipitation. *Cynometra caulifora* essential oil were loaded into chitosan polymer using ionic gelation method. the prepared nanoparticles showed enhanced cytotoxic effects against breast cancer as compare to free *Cynometra caulifora* essential oil [105]. The gelation process is affected by many factors including the way of mixing, pH, ratio of ingredients. These factors result in different size of the nanostructures and their stability which should be managed during procedure [106].

7.2.2.2. Nanoprecipitation

Nanoprecipitation also designated as interfacial deposition or solvent displacement. Solvent and non-solvent, two immiscible phases are involved in nanoprecipitation. This is based on solubility properties. The polymer has ability to dissolve in organic phase (solvent) but not in aqueous phase (non-solvent). The non solvent is mainly water and solvent is organic medium. The chitosan (polymer) and essential oil (active ingredient) dissolve in solvent phase (organic) and show no dissolution in non-solvent phase (water). In nanoprecipitation method the polymer and essential oil are solubilized in an organic solvent. The prepared organic solution is poured into water under moderate magnetic stirring. This result in interfacial deposition of a polymer after displacement of organic solvent. The surfactants added to enhance the nanostructure stability [107].

Four stages are involved in nanoprecipitation including supersaturation, nucleation, growth and coagulation as demonstrated in fig 9. Following that, the organic solvent is allowed to evaporate using rotavapor or at room temperature, forming a suspension of nanoparticles in water. Additionally, the water was eliminated entirely or in part, resulting in films or powders [108]. The chitosan and essential oils are in contact with two solvent system but remain undissolved in non-solvent phase which result in formation of supersaturation condition, an essential point for nanostructure formation. The four stages and shearing speed effect the kinetic stability and sizes of capsules [109]. The nanoparticles prepared by nanoprecipitation method shows controlled invitro release of essential oil without any deterioration [110].

8. Efficiency of essential oil-based nanoparticles

The nanoparticles synthesized from essential oil has a number of benefits over classical methods, which include the use of plants essential oil active components as reducing and stabilizing agent. The nanoparticles formed by biogenic method are free from toxic chemicals and safe to use in numerous applications [10]. Although many imperfections associated to essential oils like speedy deterioration, huge volatility, greater lipophilicity, and narrow permeability to membrane can be diminished by nanotechnology. The essential oil formed polymeric nano capsules and nanospheres are used in commercial products like creams, gels where they improve the gels, creams efficiency and also act as preservative for pharmaceuticals and cosmetics [111]. Antimicrobial essential oil encapsulated in polymeric nanoparticles increases the solubility in aqueous media and lesser their toxicity towards cells and enhances antimicrobial efficacy. The targeting efficiency of drug and drug biological activity increased by assimilating the essential oil with nanosized lipid particles (nanosized solid liquid particles, lipid nanocarriers and liposomes) in drug delivery system. Essential oil when encapsulated in nanostructures, it prevents the essential oil from thermal degradation, evaporation and photo oxidation [112].

9. Essential oil-based nanoparticles characterization techniques

For proper characterization of nanoparticles, a single technique is not enough. Different techniques are used to characterize the nanoparticle. The actual aim of characterization is to ensure the size of the nanoparticles. Transmission Electron Microscopy (TEM) can characterize the morphology and structure of nanoemulsion. TEM images shows that particles are separated by fairly uniform interparticle distance. This reveals the presence of plants extract as stabilizing agent for nanoparticles [113].

Fourier Transform Infrared Spectroscopy (FTIR) identify the material composition either it is inorganic, organic or polymer. The materials are identified due to change in the absorption band patterns. The presence of potential reducing agents in essential oil biomolecules that convert metal ions into metal nanoparticles is evaluated via FTIR analysis. The appearance of bands at specific position indicates the presence of particular essential oil and biosynthesized metal nanoparticle. The characteristic C-H group peak at 2923 cm^{-1} indicates the existence of monoterpene structure [114]. Analysis interprets the functional groups and chemical bonds linked to biomolecules and reveals their reducing and stabilizing potential. The encapsulation of essential oil in nanoparticles result in increase of FTIR stretching frequency when compared with simple essential oil peaks. The FTIR signal produced represent a unique fingerprint for molecule which make FTIR a valuable tool for chemical identification [115].

Ultraviolet – visible spectrometer used for preliminary characterization of metal nanoparticles. Surface plasmon resonance phenomenon is involved which result in formation of specific color of metal nanoparticles. An increase in the reducing agent's concentration accelerated the NPs' production and the transition of light to dark to darkest color [116]. The particle size effect the band by changing the position of plasmon absorbance in spectra. With an increase in particle size, the absorption band shifts toward longer wavelength [117]. Scanning Electron Microscopy (SEM) involves the measurement of surface area and particle size. An electron beam is used instead of light beam. The metals show electrical conduction with electrons and no need to prepare the sample for metal analysis. But in case of non-metals the samples are prepared with sputter coater. The removal of water from sample is most important factor in SEM analysis [118].

10. Conclusions

The fabrication of metallic nanoparticles using essential oils stands out as an inexpensive, readily scalable and environmentally favorable method. This approach not only fulfills the essential requirement of creating metal nanoparticles without hazardous substances but also harnesses the power of phytochemicals present in essential oils, particularly terpenoids. It is crucial to recognize that the active functional groups within these phytochemicals play a pivotal role in the reduction and stabilization of metallic nanoparticles, highlighting their significance over the entire chemical structure. The versatility of essential oils extends beyond nanoparticle synthesis, finding applications in diverse filed such as cosmetics, agriculture, pharmaceutical and the food industry. This natural product's sensitivity to environmental factors has led to the development of an alternative method-encapsulation of essential oil at nanoscale. This innovative approach enhances the stability, protection, targeted release, and efficacy of essential oils, making them more adaptable to various applications. As we move forward, there is pressing need for broad investigation to provide fresh perspectives on biosynthesis through the use of essential oils. Special attention should be given to producing metal nanoparticles with precise control over their sizes and forms. Additionally, exploring the utilization of essential oils and their active compounds in creating colloidal particles, particularly for dermatology and localized skin therapy, as well as the emerging field of cosmetic-textiles, is of great concern in the present scientific landscape. This concluding reflection underscores the promising trajectory of research involving essential oils, offering sustainable and versatile solution that extend beyond the realm of nanoparticle synthesis.

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