



Studies on Extraction, Microencapsulation and Potential Applications of Ginger Oleoresin

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The aim of this study is to develop stable formulations of ginger oleoresin using microencapsulation and emulsification technology. Different formulations of microencapsulated ginger oleoresin were formulated using spray drying technique. The advantage of encapsulation is that the formulations are water soluble unlike the oleoresin; which helps in reducing the use of solvents and promotes the use of natural product and widens its application range. The wall materials like polyvinylpyrrolidone (PVP) and maltodextrin (MD) showed the maximum encapsulation efficiency of 81.043%. The flow properties of microencapsulated powder were studied by analyzing the compressibility index and Hausner ratio for different combination of wall materials. The optimization of microemulsion was studied using pseudo-ternary phase diagram and the microemulsion with the maximum area was found in 3:1 (Surfactant: Co-Surfactant) ratio of Tween80- Propylene Glycol with caprylic-capric triglycerides as the oil phase and rest is the water phase. The ginger oleoresin diffusion study was carried out using Franz diffusion cell to study the active permeability, and stability of the formulations.

Keywords: Food, Formulations, Microemulsion, Rhizomes, Surfactant

Introduction

Ginger (*Zingiber officinale* Rosc.) belongs to the family Zingiberaceae. It originated in South-East Asia and used in many countries as a spice and flavoring agent in food, also the rhizome of ginger has been used in traditional herbal medicine. The health-promoting properties of ginger are attributed to its rich phytochemistry.¹ The rhizomes contain essential oil, some mixture of sesquiterpene hydrocarbons, ash, starch, water-soluble proteins, fat which includes free fatty acids, sterols, crude fiber and some trace elements like vitamins, reducing sugars, water soluble, and minerals. Ginger rhizomes contain two kinds of products: i) Volatile compounds containing the essential oils ii) oleoresin which includes bioactive such as gingerols, shogaols, and other products that are the pungent principles of ginger. These bioactive compounds are known for variety of remarkable pharmacological and physiological activities.^{2,3} The yield of the oleoresin is greatly dependant on solvent extraction conditions, and the state of rhizomes (fresh or dried). Shogaols are very similar to gingerols in structure with the only difference being a double bond which replaces the

hydroxyl group in gingerols. They affect the quality of pungent flavour of ginger causing volatile off-flavours. Ginger has starring potential for treating a number of ailments including degenerative disorders (arthritis and rheumatism), digestive health (indigestion, constipation and ulcer), cardiovascular disorders (atherosclerosis and hypertension), vomiting, diabetes mellitus, and cancer.^{3,4} It also has anti-inflammatory and anti-oxidative properties for controlling the process of aging. Furthermore, it has antimicrobial potential as well which can help in treating infectious diseases¹. Ginger oleoresin possesses the whole organoleptic properties of the spice, i.e aroma, flavour, pungency and finds similar applications as the ground spice in flavouring of processed foods.^{5,6} Ginger is widely used in processed foods in meat, and fish fatty sauces, in mixture with other spices and flavourings and in alcoholic beverages like cosmetics and skin protectants.² It contains around 40 antioxidants that prevent free radical damage and protect against aging.⁶ Ginger increases circulation to the scalp, which is essential for stimulating hair growth and the plethora of vitamin, minerals, and fatty acids also strengthen strands and combat hair loss. The nutraceutical compounds obtained from ginger has garnered great interest in the food processing and pharmaceutical industry.⁷⁻⁹

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Microencapsulation is a process in which enzymes and volatile oils, are coated by a material to form microcapsules.^{10,11} The coated material is called core and the coating material is called the shell. Microencapsulation helps in protecting the ginger oleoresin from the effects of ultra violet rays, moisture, and oxygen; also helps in increasing the storage life of all the volatile compounds present in the oleoresin. Microencapsulation decreases the transfer of active material from the core to the medium which helps in reducing the stringency of the oleoresin when applied on to the skin in the form of any cosmetic product.¹² The major industrial process for microencapsulation is spray drying and extrusion. Microencapsulation was carried out by spray drying, which helps in transformation of liquid form into a dry solid form. Spray drying was advantageous due to its high encapsulation efficiency of hydrophobic components, reducing the particle size, large surface area, and ease in operation as well as mass production. The problem of low solubility and membrane permeability of ginger oleoresin was the main reason to develop formulations based on spray dryer.^{12,13} The spray-drying assembly consisted of an aspirator that circulated air through the dryer, an ultrasonic spray nozzle which atomized liquid feed into fine droplets and a drying chamber where the atomized liquid encountered the hot air followed by two cyclone separators.¹⁶ The first cyclone separator collected coarser particles and the second trapped the fine and ultrafine particles. The drying media was passed through the scrubber, which prevented particles from entering the aspirator¹⁷ and avoided any possible blockages in the process. The powder obtained from spray drying, both in the chamber and the cyclone, was then subjected to further analysis. The optimized formulations have much more water solubility and membrane permeation rate which helps in incorporating the microcapsules in cosmetic uses. Microemulsion incorporate a wide range of drug molecules increasing their solubility and also act as a promising delivery system in most of the cosmetics application.^{14,15} Hence, a cosmetic oil-in-water (O/W) microemulsion was developed and a comparative study using oleoresin and the formulated spray dried powder were carried out.

Materials and Methods

Buffer, chemicals and reagents used for analysis were all analytical grade. Dried ginger was purchased from the local market. Dried rhizomes had a moisture

content of 8.32% that was used for the extraction of oleoresin. The solvent extraction of ginger oleoresin was carried out using an ultrasound bath sonicator model-10 L 2304, with the frequency of 50 Hz and voltage of 230 V AC. The ginger oleoresin was spray dried using a lab-scale Spray Dryer LU-227, Advanced spray drier Labultima, Mumbai, India. The operating parameters of spray drying were inlet temperature 135°C, outlet temperature 55°C, aspirator rpm 40/100, flow rate 8 kg/h, and air pressure of 2 kg/m². Instrument used for SEM analysis was JOEL JSM-6380LA, high performance scanning electron microscope with a resolution of 3.0 nm. The samples to be analysed were silver coated and placed on the disc for scanning. The images were taken in different magnifications ranging from 1500x to 5000x. Homogenizer Polytron PT6100D was used for preparation of emulsion of ginger oleoresin for microencapsulation.

Result and Discussion

Yield of Ginger Oleoresin

Dried ginger powder (100 g) was loaded in a 500 ml round bottom flask fitted with a condenser, 250 ml of solvent was added and extraction was carried out using methanol and n-hexane. The extraction was carried out under ultrasonication with a frequency of 50Hz and voltage of 230V AC. The bath temperature was maintained at 38°C as at higher temperatures the main pungent principle (gingerol) of the oleoresin may decompose and form shagaol. The temperature was maintained constant throughout the experiment and yield was calculated at 30 min, 60 min, and 120 min. The resinous extract was filtered and concentrated on rotary evaporator where the solvent is removed under vacuum. The yield and composition of the oleoresin is a function of time, as the time increases the yield of the oleoresin also increases as higher extraction time leads to more contact of the raw material with the solvent. The yield obtained is shown in Table 1 and it showed that methanol gives a better yield than n-hexane, hence the oleoresin extracted from methanol was carried forward for further development.

Time (min)	Oleoresin yield (%)	
	Methanol	n-hexane
30	1.6	1.2
60	2.3	1.9
120	3.5	2.7

Microencapsulation of Ginger Oleoresin

Microencapsulation is the process of shielding the active ingredients that is vulnerable to environmental changes by using a coating. Microencapsulation of ginger oleoresin was carried out using different ratios of polymers as wall materials like gum acacia, maltodextrin, whey protein Isolate, and polyvinylpyrrolidone (PVP).^{18,19} All the wall materials were taken as with different ratios and homogenized with 100 ml distilled water. The mixture was allowed to homogenize at 800 rpm for 10 min. Further the binder capsul, surfactant tween 80 and medium chain triglyceride (MCT) oil was added into the mixture and homogenized for the next 5 min. 15 g of ginger oleoresin was incorporated into the mixture and the emulsion was stirred for another 20 min until a homogeneous emulsion was obtained. Then emulsion was allowed to stay overnight and spray dried in optimized conditions. The powder obtained after spray drying was blended with anticaking agent like sipernat and further analysis was carried out. Different ratios of wall materials were taken to optimize the method for preparation of microencapsulated ginger spray dried powder. Several trials using different wall materials were performed as shown in Table 2.

Flow Properties of Encapsulated Powder

Flow properties of encapsulated powder depends on the moisture retention in spray dried powder. Loose and tapped bulk density of the encapsulated powder showed the distribution of the particle size and the adherence of the particles to each other. Hausner's ratio and compressibility index showed the ease of flowability of the powder mixture. Due to the oily nature of the ginger oleoresin, it might hinder the flowing consistency of the encapsulated powder, because at higher temperatures there might be a

chance of degradation of gingerol which also restricts the further processing of spray dried powder. Different flow property values were evaluated and shown in Table 3.

Particle size played a major role in any product formulation and the particle size of the encapsulated powder sample was evaluated for the content uniformity and administration of the active component in formulation.²⁰ The particle sizes of the microcapsules were in micro range with the MD: PVP (1:2) showing the lowest particle size which can be incorporated in to topical applications as well. The particle size of the encapsulated powder with different wall materials was determined and shown in the Table 4 and Fig. 1–3 for sample GA:WP (2:1), GA:PVP (1:2) and MD:PVP (1:2) respectively.

Encapsulation Efficiency

Microencapsulation of ginger oleoresin was carried out by spray drying, and it was important to find out the encapsulation efficiency to ensure the amount of active compounds being encapsulated. Encapsulation efficiency gave an insight into the shelf life of the product, and efficiency of the whole process. MD: PVP wall materials showed the better encapsulation efficiency of 81.04% when compared to GA: PVP as shown in Table 5; and hence this was carried forward for further application

Surface Morphology of Encapsulated Powder

The SEM analysis of encapsulated powder for surface morphology having maximum encapsulation

Table 4 — Particle size of encapsulated powder

Sample	Weighted residual (%)	Laser obscuration (%)	Dv (50) μm	Dv (90) μm
GA:WP (2:1)	0.732	2.28	3.47	44.7
GA:PVP (1:2)	0.66	2.86	2.84	38.9
MD:PVP (1:2)	0.63	2.09	3.07	23.4

Table 2 — Different wall materials for microencapsulation

Trial	GO	GA	MD	Tween 80	Sipernat 22S	Capsul	Water		
Trial 1	15	40	20	2	0.5	15.8	100		
Trial 2	GO	PVP	MD	HICAP 100	MCT Oil	Tween 80	MT	SSG	Water
	15	35	17.5	10	10	2.5	2	0.4	125

GO = Ginger oleoresin; GA = Gum Acacia; PVP = Polyvinylpyrrolidone; MD = Maltodextrin; MCT Oil = Medium Chain Triglycerides; SSG = Sodium Starch Glycolate; MT = Mixed tocopherol

Table 3 — Flow Properties of spray dried powder

Sample	Wt. (g)	Untapped volume (ml)	Tapped volume (ml)	Bulk density (g/ml)	Tapped density (g/ml)	Compressibility Index	Hausner ratio
MD:GA (1:2)	10.05	30	21	0.335	0.47	30	1.42
GA: WP (2:1)	10.03	29	20	0.345	0.501	31.0	1.45
GA:PVP (1:2)	10.03	27.5	20	0.364	0.501	27.2	1.37
MD:PVP (1:2)	10.03	28	21	0.358	0.49	25	1.33

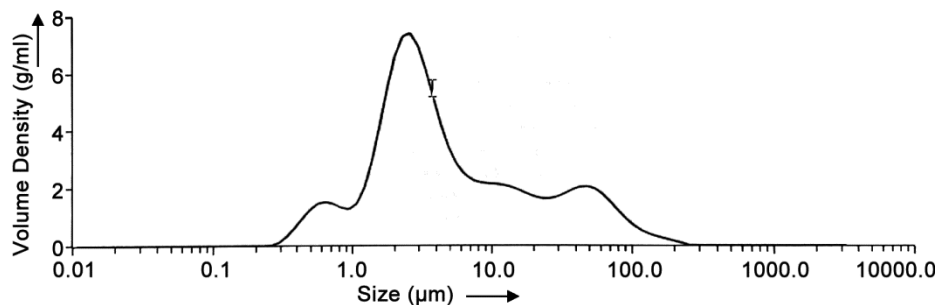


Fig. 1 — Particle size analysis of GA: WP (2:1)

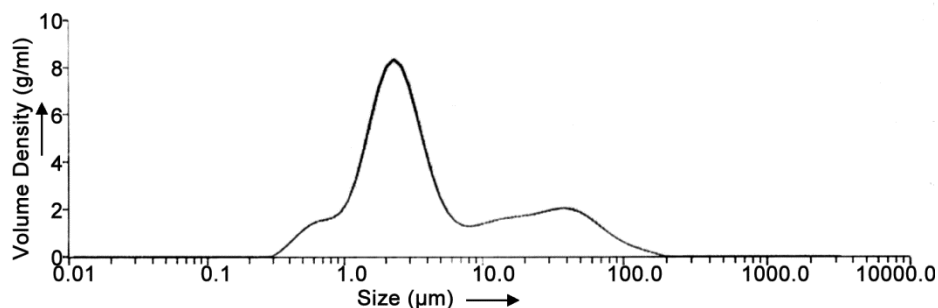


Fig. 2 — Particle size analysis of GA: PVP (1:2)

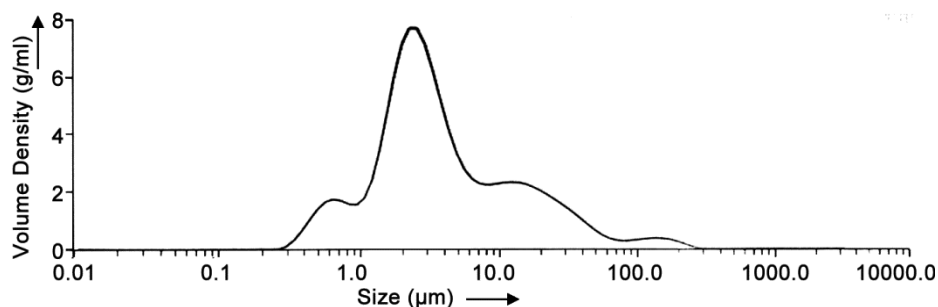


Fig. 3 — Particle size analysis of MD: PVP (1:2)

Table 5 — Encapsulation efficiency with different wall material

Sample	Encapsulation Efficiency (%)
GA: WP (2:1)	71.94
GA:PVP (1:2)	76.15
MD:PVP (1:2)	81.043

efficiency has been carried out with magnification ranges from 1500X to 5000X. As shown in Fig. 4, a clustered image of the powder; the particles were seen to cling on to each other because of the presence of moisture. 2700X and 5000X showed a maximized image of the powder with spherical and smooth surface. The SEM images showed

Antibacterial and antifungal activity

Antibacterial and antifungal activities of the encapsulated powder with ginger oleoresin were indicated by zone of inhibition with organisms like *Staphylococcus aureus*, *Escherichia coli*, and

Aspergillus niger. The Fig. 5 showed that Ginger oleoresin has zone of inhibition in all the organisms like *Staphylococcus aureus*, *Escherichia coli*, and *Aspergillus niger* when used in 100 µl quantity. The zone of inhibition of *Staphylococcus aureus*, *Escherichia coli*, and *Aspergillus niger* for ginger oleoresin was found to be 22 mm, 13 mm, and 13 mm respectively.

Optimization of Microemulsion

The optimization of microemulsion was carried out by constructing a pseudo-ternary phase diagram which was used to predict the compositions of surfactants, oil and water in the development of microemulsion. The microemulsion was formulated using various oil phases like medium-chain triglycerides (MCT), Caprylic Capric Triglycerides (CCTG); Surfactant- Tween 80; co-surfactant-

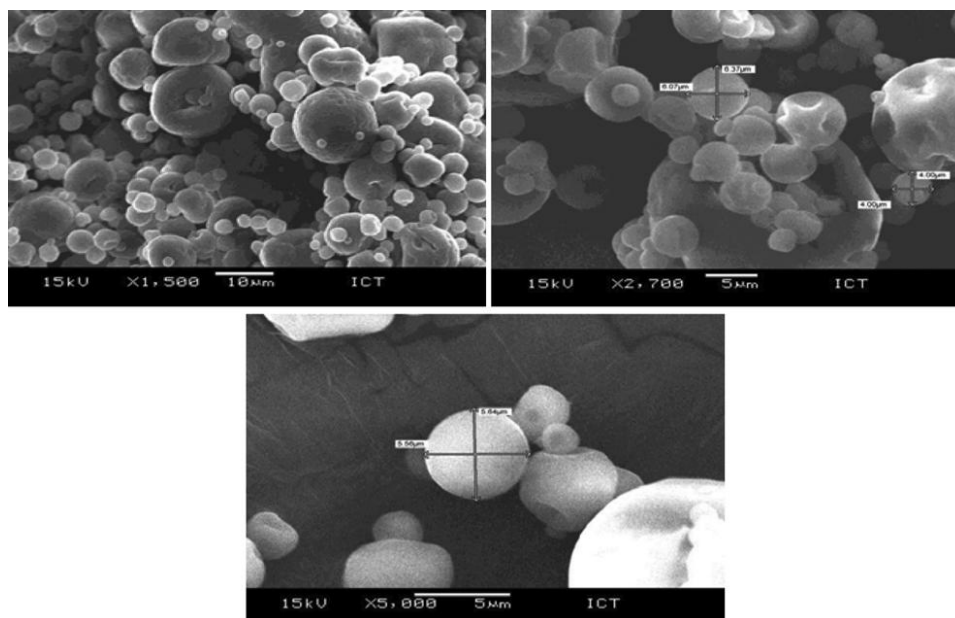


Fig. 4 — SEM images of micro-encapsulated ginger oleoresin

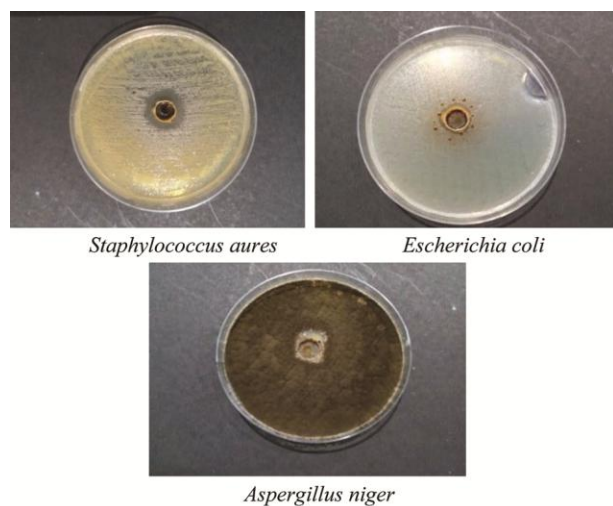


Fig. 5 — Ginger oleoresin showing zone of inhibition

Propylene glycol, and ethanol. Pseudo-ternary phase diagrams were constructed using TRIPLLOT software. Surfactant was blended with cosurfactant in fixed weight ratios like 2:1, 3:1 and aliquots of each surfactant and cosurfactant mixture (S-mix) were then mixed with oil at room temperature. For each phase diagram, the ratios of oil to S-mix were varied as 9:1, 8:2, 7:3, 6:4, 5:5, 4:6, 3:7, 2:8, and 1:9 (w/w). Water was added drop wise to each mixture under vigorous stirring by using magnetic stirrer²² and maintaining a temperature range of 40–45°C and each mixture was visually observed for transparency. The samples were marked as points in the phase diagram as shown in Fig. 6. The area covered by these points

Table 6 — Ginger oleoresin diffusion study

Time	Oleoresin emulsion	Powder emulsion
1	0.032	0.055
2.5	0.057	0.082
3	0.09	0.096
5.5	0.125	0.158
7	0.135	0.193
8.5	0.1426	0.205
10	0.153	0.236
13	0.169	0.264
14.5	0.17	0.29
18	0.176	0.33
20.5	0.18	0.3542
25	0.185	0.36
30.5	0.185	0.36

was considered as the microemulsion region of existence.²³ The preparation of selected microemulsion was simply performed by adding the weighed components together and loading the encapsulated ginger oleoresin with 100 mg active and stirring to form a clear microemulsion.

Ginger Oleoresin Diffusion Study using Franz Diffusion Cell

Ginger oleoresin release study of microemulsion using Franz diffusion cell is performed.²¹ The diffusion cell of standard size of receptor volume up to 20 ml was used. Media used for release study was phosphate buffer with 35% methanol was selected having pH of 5.5. 2 g of the microemulsion was added into the sampling port and 2 ml of aliquot was taken on timely basis. The absorbance values are shown in Table 6. It was observed that maximum drug release

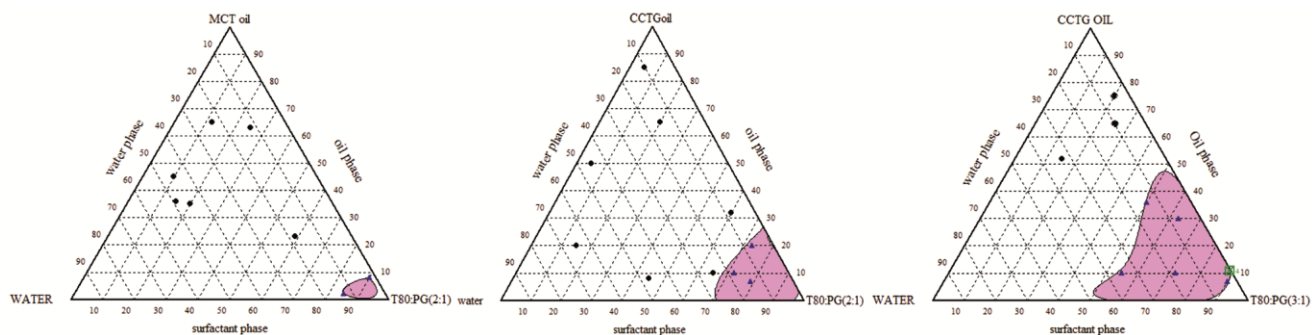


Fig. 6 — Pseudo ternary diagrams of microemulsion

obtained from the microemulsion loaded with spray dried powder by the end of 30 min which was found to be 28.31% followed by microemulsion loaded with oleoresin which was about 12.37%.

Conclusions

The present study focused on the uses of ginger as a topical applicant for foods and cosmetics formulations. Microencapsulating the ginger oleoresin helps in preserving the pungent constituents like gingerol from getting degraded from any external factors. Also, microencapsulated formulations are water soluble which makes it easier to be used in cosmetics. The results from in-vitro comparative drug diffusion study done using Franz Diffusion cell was supportive of the fact that the microemulsion incorporated with ginger formulations show increased drug diffusion activity than compared to emulsion loaded with ginger oleoresin. This can act as excellent natural cosmetics and would garner maximum consumer choice as natural/herbal cosmetics in the present scenario.

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