IJPRD, 2013; Vol 5(05): July-2013 (056 – 063) International Standard Serial Number 0974 – 9446

International Journal of Pharmaceutical Research and Development (IJPRD)
Platform for Pharmaceutical Researches & Ideas
www.ijprd.com

PREPARATION AND ENCAPSULATION OF SPICE OLEORESINS

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ABSTRACT

The title of our project is entitled as "Preparation and Encapsulation of Spice oils". It is a consultancy project given to us by INDO NACOP PVT Ltd., a spice manufacturing company. It is situated at Vinukonda, AP, India.

The spice oil and oleoresins industry in India has undergone phenomenal growth in recent years. From 3 tonnes of spice oils and oleoresins valued at Rs. 1.41 lakh during 1971-72 to 2625 tones valued at Rs. 293 core during 1998-99, the annual rate of growth works out to 19.27% in quantity and 28.46% in value. India ranks 26th in imports and 14th in exports in the world trade of essentials oils. Due to biotic contaminants and pesticide residues in whole spices, oleoresins are gaining significance. Encapsulated spice extractives extended on a salt or dextrose carrier, encapsulated spice oils and oleoresins, homogeneous free flowing oleoresins etc. are currently available.

Our project is mainly encapsulating chilly and ginger spices. Their oleoresins are prepared by extracting using an organic solvent. These oils are encapsulated in three different techniques. In micro-encapsulation, micro fine particles of essential oil or oleoresin are coated with an envelope of edible carrier. The coating ensures stability to the core product. On application, the coating dissolves off, releasing the flavor to the food. Most spice oils and oleoresins can be offered as microencapsulated/spray dried powder.

We hope that the spice oil production and encapsulation technique reviewed in this project would serve as a forerunner for determining best possible way of using spices. The factors' influencing their optimization provides a clear picture towards developing a suitable technique not only for chili and ginger but also for other food and cosmetic industry as well.

Keywords:- *Spice oil, Ginger oil, Solvent extraction, Methanol, Encapsulation.*

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INTRODUCTION

Spice oils and Oleoresins:

Spice oleoresins are the concentrated liquid from the spices that reproduce the character of the respective spice fully. The spice oils are the oils distilled off from the spices at the initial stage before subject to solvent extraction. Spice oleoresins are largely used for flavoring of food particularly by large scale food processing and flavoring industries like meat canning, sauces, soft drinks, pharmaceutical preparations, perfumery and soap, tobacco, confectionery and bakery. The demand of spice oils and oleoresins in the developed countries is increasing day by day as more and more spicy snacks are being introduced by fast food chains with standardized tastes. The spice oils and oleoresins are especially suitable for such snacks in that they can be used very conveniently (without any handling of the raw spice like ginger, chili, onion, etc.) and producing a standardized effect on taste. This is the reason practically all plants in India, numbering to more than twenty five are cent percent exporting their products to these nations. The demand is increasing and more and more plants are being commissioned for 100% export. The margins are high with the spice oil prices ranging between US $ 30 to 100 per Kg. made from equivalent raw material components of about US $ 1 to 5.

As already indicated, the spices have two main flavor attributes. The one that catches the immediate attention of a costumer is the spice aroma. This is contributed by essential oil or spice oil, which is detected by the olfactory organ of nose. Spice oils can be separated by steam distillation. The other flavor that attributes is the hot, pungent taste felt in mouth while masticating. Pungency is caused by chemicals that are non-volatile. Spices also have color, although some of them are considered as attractive as in case of paprika and turmeric. The coloring components are also non-volatile. If all the flavor attributes of aroma, taste and color are required, only solvent extracted oleoresin will be a complete extractive. Even the volatile spice oils will be found in the extract.

Before the improved two-stage method of preparation of oleoresins was introduced in India in the 1970s, oleoresins used to be produced in a single stage of a solvent extraction. However, there are drawbacks in that the quality of oil is not as good because of interference of solvent. During the removal of solvent, some fine aroma can be lost. In the two-stage process, spice oil is separated by steam distillation as the first step. The deoiled spice, after drying and lump breaking, is extracted with an appropriate solvent for non-volatile fraction. The solvent chosen can be the best suited for non-volatile components only, as the essential oil has already recovered. The resin fraction, so obtained after removal of solvent, is blended with an adequate quantity of oil collected in the first stage to obtain oleoresin.

The spice oils removed in the first stage are unaffected by the solvent. Generally, the yield of the oil is so high that only about half the quantity need be used for blending with the resin extract. In fact, during steam distillation of the first stage, oil can be collected as two fractions. The first fraction will be richer in the harsher smelling monoterpenes; the second fraction will be richer in sesquiterpenes and oxygenated compounds. The second fraction can be used as salable oil. The first fraction with its strong top note will be ideal for blending with nonvolatile resin obtained in the second stage by solvent extraction, Due to the improved two stage process, production of quality spice oils became a part of the oleoresin industry, thus making the process more commercially viable.

Encapsulation of spice oleoresins: When milled spices are stored, their essential oils evaporate over time. Micro-encapsulation preserves these valuable flavours. In this process, the milled particles are microencapsulated and then sterilized at a mild temperature to reduce the germ count. The process is suitable for a variety of spices and takes just a few seconds. Microencapsulated spices currently are making an outstanding contribution to targeted flavour development in end products, particularly when it comes to special manufacturing processes in the food industry.

Chemical encapsulation techniques typically yield particle dispersions that can be used as is or post-processed by other methods, such as spinning disk, spray drying or fluid bed to produce free-flowing powders.

METHOD OF PREPARATION

Raw materials taken:

There are many varieties of spices distributed throughout the world. Out of many spices we have considered 2 spices. They are Chili and ginger. From these spices, the spice oil is extracted and it is encapsulated. The other materials required are an organic solvent, Acacia gum and Gelatin gel.

Manufacturing process of Oleoresin:

The conventional method of soxhelet extraction is used to prepare spice oil. The idea was to use an organic solvent to extract the oil essence and prepare the spice oil. Before explaining about the experimental procedure, some data about soxhelet extraction is seen.

A Soxhelet extractor is a piece of laboratory apparatus invented in 1879 by Franz von Soxhelet. It was originally designed for the extraction of a lipid from a solid material. However, a Soxhelet extractor is not limited to the extraction of lipids. Typically, a Soxhelet extraction is only required where the desired compound has a limited solubility in a solvent, and the impurity is insoluble in that solvent. If the desired compound has a significant solubility in a solvent then a simple filtration can be used to separate the compound from the insoluble substance.

Normally a solid material containing some of the desired compound is placed inside a thimble made from thick filter paper, which is loaded into the main chamber of the Soxhelet extractor. The Soxhelet extractor is placed onto a flask containing the extraction solvent. The Soxhelet is then equipped with a condenser. The solvent is heated to reflux. The solvent vapor travels up a distillation arm and floods into the chamber housing the thimble of solid. The condenser ensures that any solvent vapor cools, and drips back down into the chamber housing the solid material. The chamber containing the solid material slowly fills with warm solvent. Some of the desired compound will then dissolve in the warm solvent. When the Soxhelet chamber is almost full, the chamber is automatically emptied by a siphon side arm, with the solvent running back down to the distillation flask. This cycle may be allowed to repeat many times, over hours or days.

During each cycle, a portion of the non-volatile compound dissolves in the solvent. After many cycles the desired compound is concentrated in the distillation flask. The advantage of this system is that instead of many portions of warm solvent being passed through the sample, just one batch of solvent is recycled. After extraction the solvent is removed, typically by means of a rotary evaporator, yielding the extracted compound. The non-soluble portion of the extracted solid remains in the thimble, and is usually discarded.

CHILI SPICE OLEORESIN PREPARATION:

The organic solvent considered for the extraction of chili spice oleoresin is Acetone.

First Run (trail run): Done mainly to observe weather extraction happens, if yes the physical consequences.Some chill's were picked and chopped and extraction was done. Time of contact was 1 hour.

Result: very little amount of oil was extracted.

Second Run: Chilis were finely grinded and the powder is prepared.

Weight of feed=15 gms,Weight of oil=0.6 gms,Weight of solid left=13 gms

% Extraction=30%

Third Run:

Weight of sample=50gms,Weight of left powder=39.2gms,Weight of oil=3.5gms

% Extraction=32.2%

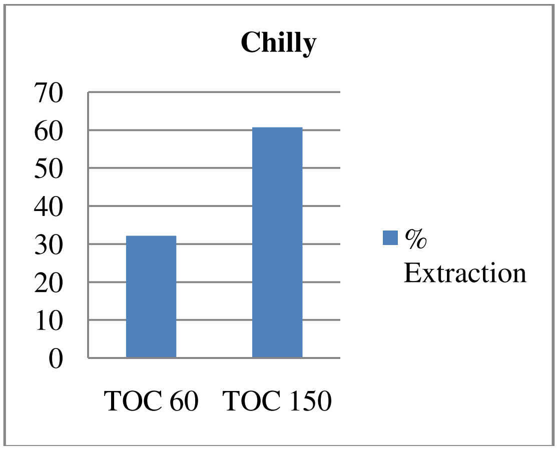
Time of contact=60mins

Fourth Run:

Weight of sample=50gm,Weight of left powder=36gms,Weight of oil=7.5gms

%Extraction=60.71%,

Time of contact=150mins



GINGER SPICE OLEORESIN PREPARATION:

The organic solvent used in preparation of Ginger spice oil is Acetone.

Run 1:

Weight of ginger taken=25gms,Weight of solid left=22gms,Weight of oil obtained=1.2gms

Time of contact=60mins

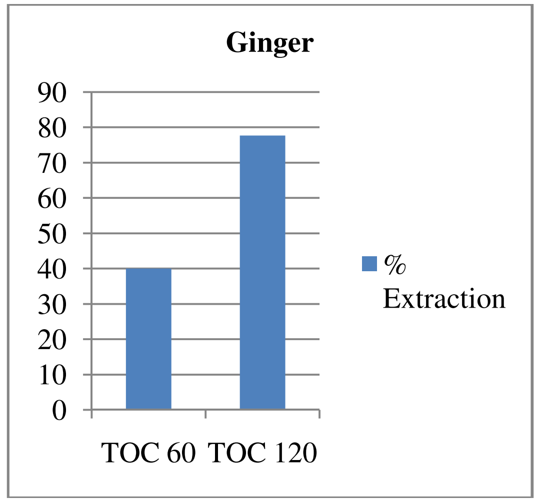
%Extraction=40%

Run 2:

Weight of ginger taken=50gms,Weight of solid left=41gms,Weight of oil formed=7gms

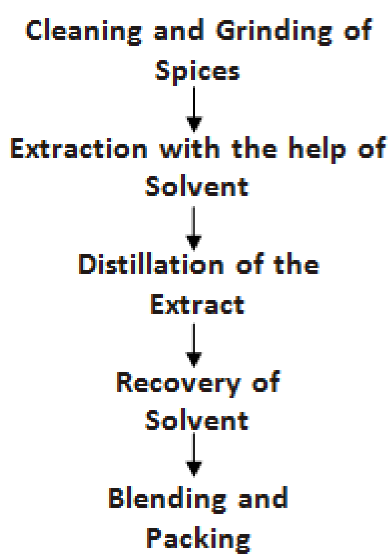
Time of contact=120mins

% of extraction-77.7%



Thus we are ready with oleoresins of chili and ginger spices. The next step in the project is to encapsulate the spice oleoresins.

FLOWSHEET:



ENCAPSULATION TECHNIQUES

Encapsulation of Spices:

The next stage in the project is to encapsulate the chili and ginger oleoresins prepared. The main encapsulation technique employed is microencapsulation. There are many types of microencapsulation techniques which are already discussed in detail.

The microencapsulation techniques in which the required oleoresins are encapsulated are:

1. Matrix polymerization using gelatin gel
2. Micro Encapsulation using spray drying
3. Centrifugal extrusion process

The procedure of encapsulation of the above three methods will be discussed sequentially.

Matrix polymerization using gelatin gel:

A stable liquid suspension of an spice oleoresin in a gel matrix with high shear mixing of colloidal gelling agent is made at a temperature of about 15^{0}C and dispersing the oleoresin in the gel is done for the formation of the gel matrix

The main objective of the above process is to develop a method that can encapsulate the oleoresin in a gel with good stability, which can be distributed into the food system uniformly. This process is flexible enough to encapsulate wide variety of food ingredients in the processed food systems. The colloidal gel obtained in the process can be used directly in the food systems or further processed and then incorporated in the food systems.

Briefly, a colloidal gel is made from water and gelatin powder. 10 grams of gelatin powder is mixed with 100 milliliters of water. The above mixture is agitated vigorously using an agitator at 200 RPM at 35^{0}C. A viscous gel of gelatin is obtained. The spice oleoresin which is initially prepared is added in the gel. About 15 grams of oleoresin is added and the resulting mixture is shaken for 120 minutes. The final product obtained is a spice enriched polymer. It is refrigerated for few hours to get a perfectly thick gel.

Micro Encapsulation using spray drying:

Gum acacia is used as carried material for microencapsulation of ginger oil due to its high water solubility, low viscosity and ability to act as an oil-in-water emulsifier. Wall material (feed mixture) was prepared by dissolving the desired amount in deionized water at 50ºC stirring overnight to enhance hydration. In a beaker 80 gm of gum acacia was dissolved with 200 ml of distilled water, solution was kept on stirring to dissolve gum acacia. Further 20 gm of ginger oil was added and homogenized vigorously (10000rpm/5min) with an Ultra Homogenizer at ambient temperature (22°C) for 15 min. Maintain the pH of the solution around 4.5-5 and viscosity at 25^{0}C measured by Brookfield viscometer was 35Cp. The prepared feed mixture was slowly agitated till spray drying and the feed mixture was spray dried at the specified conditions.

In the present investigation efforts were made to standardize the process for microencapsulation of ginger oil by spray drying under different drying conditions. The prepared ginger oil and gum acacia feed mixture was encapsulated by spray drying by using LSD – 48 MINI SPRAY DRIER JISL. The microencapsulation of ginger oil was standardized by monitoring effect of different Inlet temperature combinations while other parameters such as feed flow rate, rational speed of atomizer and flow rate of air are kept constant at the values of 500ml/hr, 30,000 rpm and 110kg/hr, respectively.

Preliminary experimental work was done with different inlet temperatures for spray drying of feed mixture at the feed flow rate of 500ml/hr so as to select a range of temperature could be used in ginger oil encapsulation. In order to become better acquainted with spray drying equipment, numerous practice attempts were made and through trial and error, coupled with continued informal sensory evaluation by a panel of food scientists. As result of these preliminary trials, it was observed that at the lower inlet temperatures (between 110 to 140^{0}C), the powder so prepared was of inferior quality with larger particle sizes having lower flow ability while at the inlet temperatures above 180^{o}C were resulting in very minute particle size causing dusting of prepared powder with lower flavour profile. Hence on the basis of these results, it was decided to systematically study the spray drying of ginger oil encapsulation feed mixture in the range of 150 to 180^{0}C. Further systematic studies have been carried out by observing the different physico-chemical properties of encapsulated ginger oil powder so as to optimize spray drying inlet temperature. All the trials were done in triplicate and the averages of these triplicate measurements recorded. Additional determinations were carried out if the single values from the triplicates deviated by more than $\pm$0.6% from the triplicate mean. Results from microencapsulation of ginger oil by using a laboratory scale spray dryer are shows in Table below.

Inlet Temperature (^{O}C)	150	160	170	180
Outlet Temperature (^{O}C)	**99**	**108**	**112**	**126**
Moisture Content (%)	5.90	4.01	3.67	2.21
Bulk Density (g/ml)	0.79	0.74	0.71	0.69
Microencapsulation efficiency	92%	91%	87	82
Average particle size (µm)	55.9	41.2	37.8	32.1
Overall Acceptability	7.9	8.5	8.1	7.8

The physico-chemical properties of powder are important in judging its suitability for microencapsulation. Various physico-chemical properties of encapsulated ginger oil powder prepared with varying inlet temperature of from 150oC to 180oC are given in Table-3.

Moisture content of powder is one of the most crucial factors in justifying the suitability of powder for its end use. During present investigation, moisture content of spray dried powder found to be inversely related to inlet temperature of spray drying.

Moisture content of the spray dried encapsulated ginger oil powder at varying temperatures found to be in the range of 5.90 to 2.21 per cent. It was observed that if moisture content of powder will be higher (as in case of sample dried at 150oC), the powder have susceptibility for microbial spoilage while too much lower moisture (as in case of sample dried at 180oC), dustiness of powder is observed which is undesirable. The moisture content of sample dried at 160oC is observed to be 4.01 per cent which is sufficient enough for the long term of storage of powder without any microbial deterioration under specific conditions while at this moisture, no dusting of powder was observed. With respect to bulk density, it was observed that bulk density of spray dried encapsulated ginger oil powder decreases with increase in inlet temperature and vice versa. The encapsulation efficiency is defined as the proportion of ginger oil that was not available to the extracting solvent and, consequently, less exposed to the environment. It is related to the commercial powder quality so it has an effect on volatability and flow powder properties. This reflects the amount of oil recovered from powder particles during the solvent-extraction process, which is composed of both surface and internal oil entrapped within the wall matrix. In this study the encapsulation efficiency was highest at lower temperature and found to decrease with increase in temperature. Finally, Overall quality of prepared microencapsulated ginger oil powder was assessed on the basis of sensory evaluation. During sensorial evaluation, overall acceptability was decided on the basis of major quality characteristics including overall appearance and flavour profile of the powder. It was

found that maximum sensorial score was reported in case of powder at 150oC, slight clamminess was observed due to higher moisture content while at the temperature of 180oC flavour properties has shown minimum values. Maximum overall acceptability of powder was observed at the inlet temperature of 160oC, which may be due to higher flavour characteristics and better flow ability of powder with acceptable particle size.

Centrifugal extrusion process:

Centrifugal extrusion process of encapsulation is suitable for specialty applications requiring protection of food ingredients. This technique offers a process for formulating with a variety of shell materials including gelatin, alginates, carrageenan, starches, fats, and waxes. Shell systems may be a blend of two or more polymers to obtain the desired characteristics. Capsule size capability of the process is in the range of 150-2000 µm, with the "comfort range" being in the 500-1000 µm range.

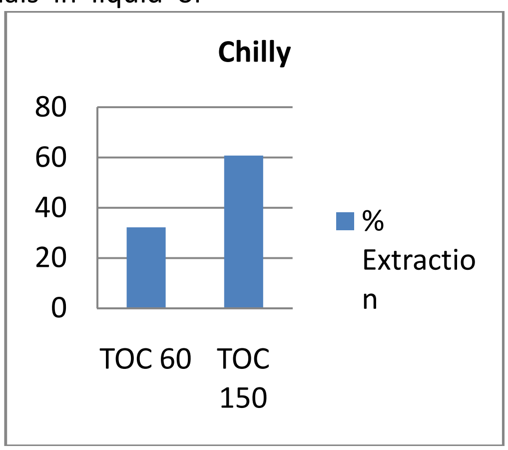
The Centrifugal extrusion process is a liquid co-extrusion process where a rotating extrusion head that contains concentric nozzles are used. Through a concentric feed tube, both coating and core material are pumped separately to the many nozzles mounted on the other surface of the device. Core material flows through the centre of the tube, while coating material flows through the other tube. The entire device is attached to a rotating shaft such that the head rotates around its vertical axis. As the head rotates, the core material and coating material are co-extruded through the concentric orifice of the nozzles as the core is enclosed in coating material. As the extrusion head rotates, centrifugal force impels the rod outward, causing it to break into tiny spherical particles. By the action of surface tension, the coating material encircles the core material, forming a continuous coating. While the droplets are in flight, the molten coating wall is hardened through solvent evaporation from the wall solution. Since the drops are formed by the breakup of a liquid jet, this process is only suitable for materials in liquid or slurry state. The capsules collected are collected on a moving bed, which cushions their impact and absorbs unwanted coating moisture.

Centrifugal Extrusion Applications are:

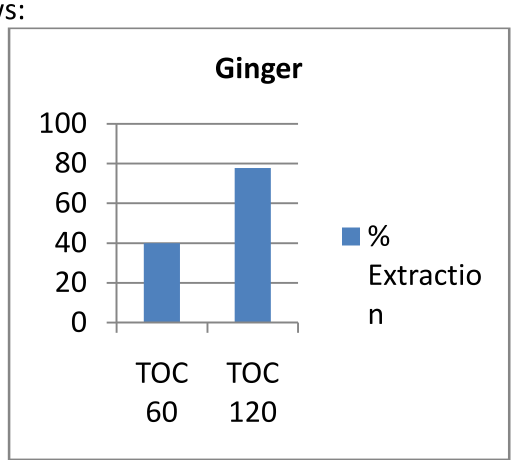
- Narrow size distributions
- Core-shell morphologies
- Gas, liquid or solid cores
- Variable shell thickness
- Variable payload composition

RESULTS AND DISCUSSIONS

This is the extraction rate graph. We can clearly observe that the % of extraction of spice oleoresin depends upon surface area available and time of contact.



The graph for the ginger is as follows:



The change in efficiency of microencapsulation with respect to temperature difference is given below:

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1. Chili spice oleoresin %extraction=60.71%
2. Ginger spice oleoresin% of extraction-77.7%
3. The microencapsulation techniques in which the required oleoresins are encapsulated are:
1. Matrix polymerization using gelatin gel
2. Micro Encapsulation using spray drying
3. Centrifugal extrusion process

CONCLUSION

After working for almost six months and trying out encapsulating spices in three different techniques, we find the process of encapsulation very important and handful in preserving and transporting spices. There are many other techniques of encapsulation as well. Few more research in these fields can give us an optimum encapsulation technique which is economical, useful and practically feasible in industrial scale.

- We hope that the spice oil production and encapsulation techniques reviewed in this project would serve as a forerunner for determining best possible way of using spices.

The factors influencing their optimization provide a clear picture towards developing a suitable technique not only for chili and ginger but also for other food and cosmetic industry as well.

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