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Effect of carrier blend proportion and flavor load on physical characteristics of nutmeg (*Myristica frangrans* Houtt.) oleoresin microencapsulated by spray drying

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Abstract

The effect of proportion of maltodextrin and gum arabic as carrier material and the percentage of nutmeg oleoresin as flavor load on microencapsulation by spray drying was studied. The proportions of 0, 20, 40 and 60 percent gum arabic (w/w) in the carrier blend and 10, 20 and 30 percent nutmeg oleoresin (w/w) as flavor load were studied. Emulsions were microencapsulated in a vertical co-current spray drier equipped with a rotary wheel atomizer operated at 18000 rpm. The inlet air temperature was set at 190 ± 2^{o}C. The microcapsules formed were then analyzed for their physical characteristics. Flavor load was found to have no influence whereas blend proportion was found to influence the moisture content. True density increased until 40% gum arabic and decreased thereafter. Solubility increased with increase in flavor load and blend ratio. Though the highest encapsulation efficiency of 74.8% was obtained at a flavor loading of 10% at a carrier proportion of 40% gum arabic, further decrease was marginal with increase in flavor load up to 20%. Considering the product characteristics, a 40% gum arabic in the carrier blend and a flavor loading of 20% nutmeg oleoresin were found to be optimum. The micrographs of optimum combination showed uniform spherical powder without surface dents and a central void with microdrops distributed in the solid matrix.

Introduction

Nutmeg (*Myristica frangrans* Houtt.), an evergreen tree spice is distributed from India and South-East Asia to North Australia and the Pacific Islands. Nutmeg, and its derivative nutmeg oleoresin, extracted by solvent extraction are being used across the world as inevitable flavors in the preparation of various food formulations. These have also made forays as aromatic adjuncts, into the composition of numerous medicines and cosmetics products.

Microencapsulation is the technique by which the preserved (active) material is entrapped within a protective (carrier) material. Apart from protecting and preserving the flavor compounds from light/temperature/moisture-induced reactions and/or oxidation, encapsulation is beneficial to retain aroma in a food product during storage, to protect the flavor from undesirable interactions with food, to minimize flavor–flavor interactions, to facilitate dosing, and to allow a controlled release (Reineccius, 1991). Among the various encapsulation methods of flavors that have been proposed, spray drying is the most common technique for the microencapsulation of spice oleoresins because it ensures rapid drying, is economical and flexible and produces powdery spherical particles of good quality (Gouin, 2004; Reineccius, 2004; Bhandari, 2010).

There have been studies to evaluate the potential of maltodextrin combination with four types of wall materials (gum arabic, whey protein concentrate and two types of modified starches), as alternative materials for microencapsulation of flaxseed oil by spray drying. The feed emulsions were characterized for stability, viscosity and droplet size, while the microcapsules were characterized for encapsulation efficiency, moisture content, particle size, bulk density, morphology and oxidative stability (Helena *et al*., 2013). Frascareli *et al.* (2012) studied the influence of total solid content and oil concentration with respect to total solids and inlet air temperature on the microencapsulation of coffee oil by spray drying, using gum arabic as wall material. Encapsulation efficiency, oil retention, moisture content and

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hygroscopicity were analyzed as responses. Gac oil encapsulation, using spray drying was carried out by Tuyen *et al.* (2014). The experiments designed for this study aimed to optimize wall material concentration and oil load in terms of the encapsulation efficiency, moisture content, and total color difference between the infeed and reconstituted emulsions to obtain high encapsulation efficiencies and to minimize moisture content and color difference.

Though there have been studies reported on the various parameters that affect the retention of the volatile active ingredients, the outcome of such studies have indicated contradicting results neither has any generalization been arrived at in many cases. It has been reported that the optimized values differ for each material encapsulated. Although many works on microencapsulation of oleoresin matrix by spray drying can be found in the literature, none of them reports the microencapsulation of nutmeg oleoresin. This is an interesting issue that deserves to be studied, since it can represent a promising alternative for the food, ingredient and cosmetic industries. Therefore it may be concluded that while attempting to encapsulate nutmeg oleoresin, it is essential to optimize the main process parameters that pave way for an efficient encapsulation of the same. In general, the process parameters which have been stated as influencing the volatiles retention during the process and which perchance can be controlled are: type of capsule carrier material, concentration (flavor load) and nature of the volatile to be retained, dryer inlet air temperature and capsule morphology (Re, 1998).

Previous studies on the carrier (wall) material most adapted for oleoresins indicated that gum arabic is the standard of excellence as flavor encapsulating material (Leahy *et al*., 1983). However exorbitant costs, impurities and variations in quality and the problems associated with its supply are its limitations. The proportion of the gum arabic in excess of its role as an emulsifier could perhaps be performed as effectively by a less expensive ingredient. Maltodextrin is a viable option as it is not only a good matrix former but also provides protection against oxidation, permits increased solid content with low viscosity and is economical, besides other advantages. The proportion of the oleoresin that could be efficiently entrapped (flavor load) in the carrier matrix is important. Though the highest possible flavor load is advantageous, because less wall materials are needed, this may result in lower encapsulation efficiency and surface oil content of the powder.

The present work investigates the effect of the factors that might affect the encapsulation efficiency such as proportions of maltodextrin (MD) and gum arabic (GA) as carrier material, and the percentage of nutmeg oleoresin (flavor load) in the carrier solution. Based on the effect of the blend ratios and flavour loads on the dependant variables, the blend ratio and flavour load combination which presents optimum encapsulated powder characteristics were selected and reported.

Materials and Methods

Emulsification and spray drying

Nutmeg oleoresin was supplied by Clarity extracts (Kerala, India). The carrier materials used in the current study were analytical grade maltodextrin (DE:20) and gum arabic obtained from Sigma-Aldrich Canada Ltd. (Oakville, Canada). Two hundred and fifty grams of carrier material blends of maltodextrin (MD) and Gum arabic (GA) in the proportions of 0% GA, 20% GA, 40% GA and 60% GA (w/w) were blended and dissolved in 300 ml of distilled water at 60°C by continuous stirring and after complete dispersion, the volume was made up to 500 ml by the addition of distilled water. The prepared 50 percent solid carrier solution (wet basis) was filtered using a muslin cloth, covered and left overnight (12 h) at room temperature, to improve film forming properties (Krishnan *et al.*, 2005a). The resultant 500 ml of carrier solution with 50% carrier solids of different proportions of MD and GA were then fortified with 25, 50 and 75 g of nutmeg oleoresin to obtain a flavor load of 10, 20, 30 percent (w/w) of the wall solids, respectively (Fernandes *et al.*, 2008; Shaikh *et al.*, 2006). One drop of Tween 80 was also added to enhance the emulsifying and film forming properties. The resultant solution was then emulsified in a shear homogenizer for 5 minutes at 3000 rpm until complete dispersion of the oleoresin was attained. The emulsions were then spray dried in a pilot model vertical co-current spray dryer (Goma Engineering Private Ltd., Mumbai, India) with a water evaporating capacity of 2 kg/h equipped with a rotary wheel atomizer operated at 18000 rpm. The feed rate was adjusted to the rate of 2100 ml/h. The inlet air temperature was set at 190 ± 2°C and the out let temperature observed to be 90 ± 5°C. The hot air supplied at the rate of 110 kg/h provided the latent heat of evaporation and the evaporation of the solvent, water, consequently led to the formation of the microcapsules. The microcapsules collected from the collecting chamber were then filled in aluminum foil pouches, sealed air tight, and stored at a room temperature of 28 ± 2°C.

Encapsulated oleoresin characteristics

Moisture content

The moisture content of the spray-dried, microencapsulated nutmeg oleoresin was determined according to the modified toluene distillation method (Anker and Reineccius, 1988). Distillation was carried out for 2 h.

True density

The true volume of a sample was measured using a nitrogen gas-operated multi-pycnometer (Quantachrome Corporation, Boynton Beach, FL, USA). The true density was calculated by dividing the sample weight by the volume, and expressed in g/cm^3.

Cold water solubility

The method proposed by Loksuwan (2007) was used to analyze cold water solubility of spray dried encapsulated powder. One gram of powder was mixed with 100 ml of water using a magnetic stirrer at room temperature for 30 min. A 25 ml aliquot of the supernatant solution was transferred to a 50 ml centrifuge tube and centrifuged at a speed of 15000 rpm for 15 min. The aliquot of the supernatant was then taken in a pre-weighed aluminum moisture dish, evaporated on a steam bath and dried in an oven at 110°C overnight. The cold water solubility was calculated as:

$$\text{Cold water solubility, \%} = \frac{4 \times \text{Grams of solid in supernatant}}{\text{Grams of sample}} \times 100$$

Encapsulation efficiency

Microencapsulation efficiency is defined as the proportion expressed as percentage of oleoresin that cannot be extracted by a suitable solvent from 1 g microcapsules (Rosenberg and Sheu, 1996). The total oleoresin content of the powder sample was estimated by the method of ASTA (1968). Surface oleoresin was determined by a modified method described by Varavinit *et al.* (2001). Fifty milliliters of hexane were added to 10 g of encapsulated powder in a 100 ml flask with a screw cap, and shaken with a vortex mixer for 2 min at ambient temperature, to extract free oleoresin. The solvent mixture was then decanted and filtered through a Whatman No. 1 filter paper. The residual powder was then dried to vaporize all residual solvent at 50°C to a constant weight. The surface oleoresin content was then calculated by the weight difference in the powder, before and after, extraction and washing with hexane. Encapsulation efficiency was then calculated using the formula:

$$\text{Encapsulation efficiency} = \frac{\text{Total oleoresin} - \text{Surface oleoresin}}{\text{Total oleoresin}} \times 100$$

Microstructural characteristics

The morphology and inner structural features of the encapsulated nutmeg oleoresin microcapsules with highest encapsulation efficiency were analyzed by scanning electron microscopy (SEM) (Philips 505, FEG, Eindhoven, The Netherlands). These examinations were carried out using the methods described by Rosenberg and Young (1993). For examining their outer structure, microcapsules were attached to SEM stubs using a two-sided adhesive tape (Ted Pella, Redding, CA) and for analyzing the inner structure; microcapsules were fractured by moving a razor blade perpendicularly through a layer of capsules attached to the specimen holder by a two-sided adhesive tape. Specimens were subsequently coated with gold using a model S150B Edwards sputter gold coater and analyzed using SEM operated at an accelerator voltage of 15 kV.

Statistical analysis

Experiments were replicated three times and mean values reported. Two way Factorial completely randomized design (FCRD) was followed and analysis of variance (ANOVA) was performed employing AGRES statistical software, Version 3.01 (Pascal Intl. software solutions, USA). The treatments and their interactions were compared at $p < 0.05$ level using least significant difference test. Design expert software, Version 8.5.2 (Stat-Ease, Inc., Minneapolis, MN), was used to generate three dimensional response surface plots to study the effect of the predictor variables on the response variables employing general factorial analysis and to fit data to generalized second order polynomial model.

Results and Discussion

Effect of process variables on encapsulated oleoresin

Moisture content

With an increase in flavor load, an insignificant increase in moisture content was observed for encapsulated oleoresin. This finding is consistent with observations of Sankarikutty *et al.* (1988), who found that moisture content remains unaffected by the flavor/carrier ratio. But the moisture content of encapsulated nutmeg oleoresin decreased significantly from 4.90 to 3.32% ($p < 0.05$) when the MD/GA blend proportion was varied from 0 to 60% GA at 20% flavor loading (Figure 1). It may be postulated

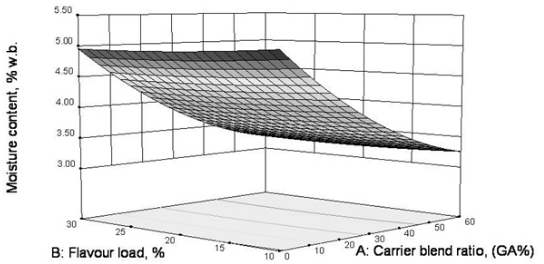


Figure 1. Effect of flavor load and carrier blend proportion on the moisture content of the encapsulated nutmeg oleoresin.

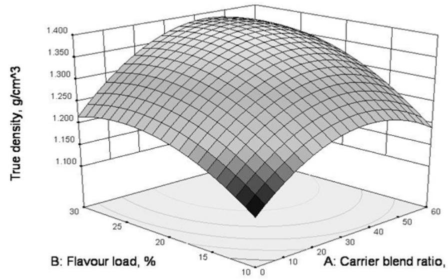


Figure 2. Effect of flavor load and carrier blend proportion on the true density of the encapsulated nutmeg oleoresin.

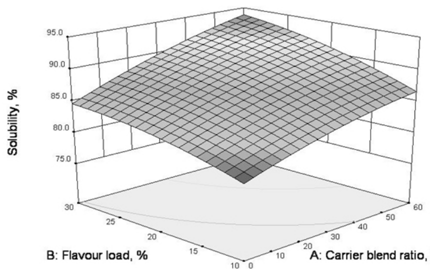


Figure 3. Effect of flavor load and carrier blend proportion on the solubility of the encapsulated nutmeg oleoresin.

that the structure and porosity of the particles could be the parameters that influence their water holding properties during drying (Kneifel *et al*., 1991), as are the drying rate and drier inlet and exit air temperature differentials (Anker and Reineccius, 1988).

True density

For a flavor loading of 20%, true density of the microcapsules increased significantly from 1.218 to 1.410 g/cm^3 ($p < 0.05$) with increase in MD/GA proportion in the blend until 40% GA was reached, following which, the true density was found to decrease to 1.310 g/cm^3 with increase in GA content in the blend (Figure 2). With the increase of GA content in the blend, the apparent viscosity of the emulsion increased. Having a high viscosity leads to a slower drying process. It is therefore probable that a stiff encasement on the surface of the particle was not created rapidly, which led to a greater shrinkage of the wall, and thus the creation of smaller sized powder particles with increased true density until a blend proportion of 40% GA was reached. But with further increase in GA content in the blend, the very high viscosity resulted in very rapid formation of the surface crust which hindered water reaching the surface thus building up internal pressure. More viscous feed causes difficulties in droplet formation due to which irregular (oval, cylindrical) and lighter particles are produced. This not only reduces the true density of the product but also causes losses of volatiles and a reduction in encapsulation efficiency.

Table 1. Models for estimation of encapsulated powder characteristics

Parameter	Model	R^2	Lack of fit	Adequate Precision
Moisture content (MC, % w.b)	$MC = 4.06 - 0.01\ C - 0.06\ F + 1.35 \times 10^{-4}\ C^2 + 5.34 \times 10^{-5}\ F^2$	0.91	ns	30.20
True density, (TD, g/cm^3)	$TD = 1.90 + 8.99 \times 10^{-3}\ C + 0.03\ F - 8.61 \times 10^{-5}\ C^2 - 6.649 \times 10^{-4}\ F^2$	0.93	ns	22.16
Solubility, (S, %)	$S = 136.30 + 0.23\ C + 0.59\ F - 1.28 \times 10^{-3} C^2 - 4.74 \times 10^{-3}\ F^2$	0.93	ns	19.24
Encapsulation efficiency, (EE,%)	$EE = -815.58 + 0.32\ C + 1.966\ F - 4.97 \times 10^{-3}\ C^2 - 0.04\ F^2$	0.94	ns	30.17

C- Carrier blend proportion (GA%), F-Flavor load, %; ns - not significant

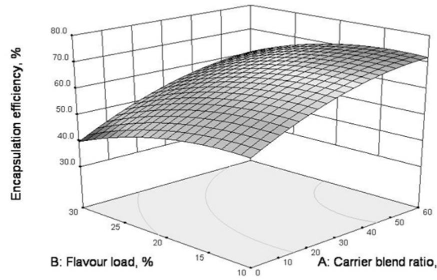


Figure 4. Effect of flavor load and carrier blend proportion on the encapsulation efficiency of the nutmeg oleoresin.

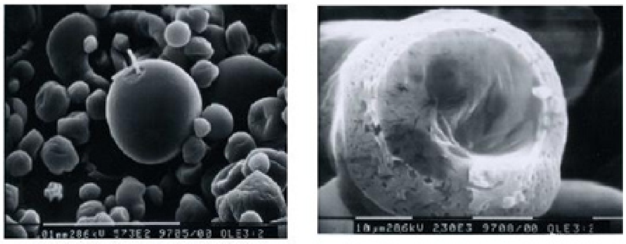
Cold water solubility

Solubility of the encapsulated oleoresin varied in the range of 79.8 to 92.9% with the highest value registered for a blend proportion of 40% GA and flavor loading of 30%. With the increase in flavor load, at all blend proportions, the solubility was found to increase. When flavor load is incremented, there would be only limited or insufficient wall material to produce sufficiently strong structural matrix and only thinner layers of wall material present between encapsulated oleoresin droplets (McNamee *et al*., 1998; Young *et al*., 1993). Similarly the solubility increased from 83.5 to 91.8% when blend proportion was varied from 0 to 40% at 20% flavor load (Figure 3). With increase in GA content in the blend, the viscosity of the emulsion would increase, resulting in formation of small sized particles and an increased surface area contributing to increased solubility.

Changes in encapsulation efficiency

The variation of encapsulation efficiency values of the oleoresin microcapsules with different carrier blend proportions and flavor load are presented in Figure 4. For a given carrier blend proportion, the encapsulation efficiency decreased with increase in flavor load. It was observed that the variation was insignificant with only below 1% decrease when flavor loading increased from 10 to 20%, but a significant decrease was observed ($p < 0.05$) when the loading was increased from 20 to 30%. The general trend of decrease in encapsulation efficiency might have been due to the thinner layers of wall material between encapsulated oleoresin droplets and/or the destabilization of emulsion droplets during the spray drying process. It was reported that higher flavor loads generally result in poorer flavor retention, this result being anticipated since higher loads lead to greater proportions of volatiles close to the drying surface, thereby shortening the diffusion path length to the air-particle interface (Reineccius, 2004). Considering these process parameters, a flavor loading of 20% could be chosen as optimum. This finding is consistent with the other studies that adopted typical flavor loading of 20% and has been reported as optimal for encapsulating wall materials like GA and other carbohydrate derivatives (Bhandari *et al.*, 1992; Rosenberg *et al.*, 1990).

For a given flavor load, as the gum arabic content in the blend increased, the encapsulation efficiency was found to increase significantly until an MD/GA blend proportion of 40% GA was reached and thereafter the efficiency was found to decrease significantly ($p < 0.05$). For 20% flavor loading, the encapsulation efficiency of microcapsules increased from 53.1 to 74.1% when the MD/GA blend proportion was changed from 0 to 40% GA and further increase in GA content to 60% could only reduce the encapsulation efficiency to 68.5%. The increase in encapsulation efficiency with the increase in GA content until 40% GA could be attributed to the better film forming and improved emulsifying/stabilizing properties of the gum. The decrease in efficiency with further increase in GA content in the blend might be due to the high viscosity of the emulsion formed, decrease in water diffusivity and the resultant decrease in solids concentration and film formation. At a given solids content, beyond 40% GA in the carrier blend, the gum arabic could not contribute towards any emulsification or film forming and could only remain as a matrix former. Many reports also suggested the replacement of gum arabic partly by other possible wall materials like maltodextrin and found these blends to be better than gum arabic alone (Kanakdande *et al.*, 2007; Krishnan *et al.*, 2005a; Krishnan *et al.*, 2005b; Shaikh *et al.*, 2006). The polynomial models depicting the influence of the carrier blend percentage and flavor load on encapsulated powder characteristics are presented in Table 1.



(a) (b)

Figure 5. SEM micrographs of encapsuated nutmeg oleoresin produced with MD/GA blend proportion of 40% GA and flavour load of 20% (a) outer surface (b) inner surface.

SEM analysis

The SEM micrographs of the outer and inner structure of the optimally produced encapsulated nutmeg oleoresin powder with an MD/GA carrier blend proportion of 40% GA and flavor loading of 20% are shown in Figure 5 (a) and (b). The outer structure appeared to be spherical and smooth. The absence of surface cracks and pores indicated that the microcapsules had not undergone 'ballooning' which is detrimental to the microcapsule stability. The size of the microcapsules varied between 5 to 30 μm. SEM micrographs also revealed that the encapsulated powder existed as single discrete particles without agglomeration or bridging. Agglomeration of particles indicates high level of surface oil. This could be judged as a proof of efficient encapsulation, better flowability and improved shelf life of the product (Vega and Roos, 2006).

The inner structure revealed that the core material was in the form of small droplets embedded in the wall matrix. The inner structure of the capsules was similar to that reported for spray-dried, encapsulated volatiles and flavors (Krishnan *et al.*, 2005a; Nayak and Rastogi, 2010). All microcapsules formed were of the matrix type, exhibiting a void center where the primary emulsion is embedded as microdroplets within the solid wall matrix. Formation of the central void is related to the expansion of the particles during the latter stages of the drying process.

Conclusions

The proportion of the maltodextrin and gum arabic in the carrier mix and the percentage of nutmeg oleoresin in the carrier solution exhibited significant influence on the microencapsulated oleoresin characteristics such as moisture content, true density, solubility, and encapsulation efficiency. Based on the characteristics studied, a carrier blend proportion of 40% GA (w/w) and a flavor loading of 20% (w/w) were found to be optimum. The obtained microcapsules were spherical with smooth surface without cracks, the size varying from 5-30 µm with a central void and microdrops of oleoresin distributed in the solid matrix.

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