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# Influence of encapsulating materials on spray-dried palm oil-based powder

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**Abstract**
The present study was carried out to study the development of palm oil-based spray dried fat powder using three different encapsulating wall materials namely TSLP (Sodium caseinate + Lactose), TSMP (Sodium caseinate + Maltodextrin), and TSHAP (Sodium caseinate + Hi-Cap + Acacia gum). Results revealed that TSHAP has the lowest moisture content (6.66%) and water activity (0.591%) after 60 days of storage at the room temperature. Further, higher surface oil values (nearly 30% of total oil) were obtained for all three combinations of wall material. The peroxide value of encapsulated palm oil increased with an increase in storage period but was found to be less than unencapsulated oil. Bulk density ranged from 0.2974 to 0.342 g/cm$^3$ for TSLP and TSHAP, respectively whereas, tapped density was found to range from 0.3875 to 0.4675 g/cm$^3$. These developments can find relevant applications in the bakery and dairy industry.

**Keywords:** Microencapsulation, spray drying, surface oil, peroxide value, palm oil

## 1. Introduction
A global trend is a growing demand for vegetable oil, while palm oil (PO) makes a major contribution to the production of edible oils globally. PO is fully free from the genetically modified organism (GMO), producing up to ten times as much oil as other oilseed plants per unit area. PO represented 32% of global production in fats and oils in 2012 and overtook soybean oil as the world's leading vegetable oils.[1] The palm tree (*Elais guineensis*) is a tropical plant native to many western African countries, mainly used by local people for cooking and other purposes. Large-scale palm trees are cultivated in tropical regions, Malaysia and Indonesia represent 86% of the world production, as are the leading PO producers; Colombia, Nigeria, Papa Guinea, Ivory Coast, India, Thailand and Brazil are other countries that produce PO.[2] The application of PO in the food industry has grown exponentially during the past few decades owing to the fragrance, texture, and neutral taste that the end products guarantee. Palm olein (low melting liquid, 65-75%) and palm stearin (high melting solids, 30-35%) are the two major fractions of PO. Different applications of PO fractions in the food industry include palm olein as frying oil due to its high smoke point (230 °C) and in margarine; whereas palm stearin is used as hydrogenated oils and shortenings as butter replacements. For the preparation of mayonnaise double-fractioned palm olein is used.[1] PO is usually present in the following food products: candies, cakes, chocolate, chips, confectionary fats,oatmeal, margarine, cookies, baked goods, cooking oil, crackers, frozen meals (pies, pizza,pancakes, potatoes), doughnuts, ice cream, industrial frying fats, cheese analogues, instant noodles, supplements/vitamins, etc.[2]
Microencapsulation seeks to safeguard delicate food particles, decrease nutritional losses, extend the applications of susceptible material, attach certain food particles to other food products, mask flavours and aromas, and alter the food product condition from fluid to solid for easy use. Microencapsulation protection may also deter radiation or oxidative degradation and delay the evaporation of volatile compounds.[3] The advantages of microencapsulation include the preservation of active compound features, prolongation of shelf life, masking of bad flavor or scent, ease of handling, control, improved visibility, and better taste and colour.[4] Biopolymers may be used in food, chemical biomedical, pharmaceutical and waste treatment industries as matrices for microencapsulation, immobilisation, or controlled release of many active compounds.
Spray drying has been used in the food industry for decades and it is one of the oldest encapsulation techniques (since the 1930s) in the making, using gum arabic as an encapsulating agent, of initial encapsulated flavours.[5,6] The spray-drying method is versatile,

offers significant differences in matrix microencapsulation, is adaptable and generates performance particles. Manufacturing costs are smaller than most other microencapsulation techniques. The type of encapsulating agent that should be soluble in water at acceptable levels is a restriction of the spray-drying technology.[7–10] Almost every process of spray drying is done with aqueous formulations in the food industry. The spray drying method usually involves several phases. It consists (1) emulsion sample preparation, (2) emulsion atomization into the thin droplets, (3) contact between hot air and droplets (4) evaporation of droplets, and (5) powder recovery [11]. Spray drying has generally been used to produce encapsulated vegetable oil for food industries [12–14]. The drying parameters (inlet temperature, outlet temperature, size of the nozzle, feed rate, etc.) of spray drying have affected the encapsulation characteristics. Minimized free surface fat powder should be obtained in optimal drying conditions. Low inlet and outlet temperatures were recorded to decrease viscosity and fat diffusiveness. Additionally, the larger size of emulsion droplets and the size of nozzle give a powder with greater particle size having a lesser surface area and greater surface fat [15-17].

Bio-based components like fats, waxes, carbohydrates and plant-derived proteins are the bulk of the components used for microencapsulation in the food sector [18, 19]. The right decision on the wall material is crucial, as it affects the efficiency and reliability of the microcapsule. The wall material should not be receptive to the core; it should be able to protect and preserve the core; must provide maximum insurance in an intense manner against unfavourable conditions; and should have financial practicality [20] A single wall material does not exhibit all the ideal characteristics therefore, a blend of at least two materials is typical practice [21].

Commonly used carbohydrates as encapsulates include maltodextrin, corn syrup solids, starches and acacia gums. These wall materials commonly exhibit poor interfacial characteristics and therefore, require modification or combined with another surface-active material for encapsulating oil-based compounds [22]. Emulsification and amphiphilic characteristics of proteins (specifically sodium caseinate) [23, 24]. tends to offer physical and functional properties needed for lipid encapsulation.

In this study, sodium caseinate in combination with different materials (lactose, maltodextrin and OSA starch and gum acacia) was used for encapsulation of palm oil using spray drying. Combination of lactose and sodium caseinate for encapsulating soybean oil [25], and fats having different melting points [26], has been studied. Maltodextrin has several advantages like low viscosity, high water solubility, mild flavour and economical [27]. Whereas, low emulsifying capacity is the main limitation of maltodextrin. Thus, it is recommended to use in combination with other good emulsifying components to get effective encapsulation to core material [28]. OSA-modified starch has hydrophobic octenyl succinate groups from OSA and hydrophilic hydroxyl groups from the native starch, thereby making it amphiphilic in nature. As a result of its superior emulsifying power, safety, good encapsulating efficiency and low price it has been used widely as an excellent emulsifier [29, 30]. Many studies have recently indicated that using OSA starch alone as both the wall material and emulsifier succeeded in producing encapsulated powder by freeze-drying [31] or spray drying [32] methods.

The aim of present study is to encapsulate palm oil using different wall materials in combination with sodium caseinate using spray drying. The encapsulation efficiency was checked for all the carrier materials and their stability and various analysis were done to investigate their application efficiency.

## 2. Materials and Methods

### 2.1 Raw materials

Palm oil (Raag Gold brand) was bought from the local market. Sodium caseinate bought from Fonterra (New Zealand), Lactose (Leprino Nutrition), Maltodextrin, Hi Cap (OSA Starch), Distilled Monoglycerides (DMG) from Ingredion, India; Acacia gum (Thermo Fisher Scientific India Pvt. Ltd.), Tween 80 (Loba Chemie Pvt. Ltd., India) and tri-Calcium Phosphate (TCP) Thermo Fisher Scientific India Pvt. Ltd., India. All chemicals used were analytical grade.

#### 2.1.1 Emulsion Preparation

Three combinations of encapsulation materials were prepared namely TSLP (Sodium caseinate + Lactose), TSMP (Sodium caseinate +Maltodextrin), and TSHAP (Sodium caseinate + Hi-Cap + Acacia gum). The aqueous phase was prepared by dispersing sodium caseinate and other wall materials in water, adjusting pH to 7.0 with 1N NaOH. The fat phase was prepared by melting the palm oil by heating to about 55°C, adding emulsifiers, and heating to 85 °C and dissolving. The aqueous phase and Fat phase were mixed and added with Tween 80 and TCP followed by homogenization using a high shear homogenizer (Silverson L5M, SILVERSON MACHINES Ltd. England) for the total duration of 45min (15min@7000rpm, 15min@5000rpm, and 15min@7000rpm). After 15 min reduction in rpm was done in order to maintain temperature of emulsion (provides cooling time).

#### 2.1.2Microencapsulation by Spray Drying

Spray drying was carried out in a lab spray dryer (Shanghai Pilotech Instrument & Equipment Co., Ltd) with a twin fluid nozzle atomization system. The spray drying operating conditions were as follows: Inlet and outlet temperatures were set to 135±5 °C and 90±5 °C respectively. The flow rate of the pump was maintained at 20 rpm. The finished microcapsules from the product vessel were stored in a Silver Aluminium Foil Pouch at room temperature and further analysis was done at regular interval of 20 days.

### 2.2 Powder Analysis

#### 2.2.1 Moisture Content

The moisture contents (MC) of the microcapsules was determined by an infrared moisture analyser (halogen bulb moisture metre).

#### 2.2.2 Water Activity

Water activity ($a_w$) of all the encapsulated samples was measured by a water activity meter (Aqua lab dew point, 4TE DUO). The sample holder was loaded with samples, and the $a_w$ was measured.

#### 2.2.3 Surface oil

Surface oil measurement was done as per the method used by Aghbashlo *et al.* [33] 15 mL hexane was mixed properly with 2 g dried microcapsules. This mixture was passed through a Whatman no. 1 filter paper. Filter paper with the particles collected was washed with 20 mL hexane three times. The collected solution having the extracted oil was transferred to

an oven (70 °C for 6 h) for vaporizing hexane. The microcapsule's surface oil was calculated by recording the initial and final weights of the solution container. Two times replication of powder analyses was done. Equations (1) and (2) were used to calculate the actual encapsulated oil (AEO), and encapsulation efficiency (EE):

$$\text{AEO(w)} = \text{Total oil} - \text{Surface oil} \quad \text{..... (1)}$$

Total oil is the oil which is added initially in the particle formation mixture and the weight of oil on the surface of the microcapsule is surface oil.

$$\text{EE}(\%) = (\text{AEOw} \div \text{total oil}) \times 100 \quad \text{..... (2)}$$

AEO is the weight of oil inside the microcapsule.

### 2.2.4 Peroxide Value (POV)

For calculating POV in crude palm oil the FAO based method was used [34]. It was calculated by using equation (3):

$$\text{POV}\left(\frac{\text{meq}}{\text{Kg}}\right) = [(\text{S} - \text{B}) \times \text{N} \times 1000] \div \text{W} \quad \text{..... (3)}$$

S and B are the titration reading of sample (mL) and blank (mL) respectively; N is the normality of the standard sodium thiosulphate solution; and weight of the sample (g) is represented by W.

### 2.2.5 Tapped and bulk density

Tapped density ($\rho_T$) and bulk density ($\rho_B$) and were measured by taking a known weight of powder in graduated cylinder. To confirm the removal of any stuck powder to the surface of the cylinder, it was tapped gently. From the cylinder, volume was noted and bulk density was calculated using equation (4):

$$\rho_B = \frac{M}{V} \quad \text{..... (4)}$$

Where V is the volume of the cylinder and M is the mass of the powder.

Compacted volume ($V_T$) was noted after tapping the cylinder manually 300 times (by hand tapping). For calculating $\rho_T$ equation (5) was used:

$$\rho_T = \frac{M}{V_T} \quad \text{..... (5)}$$

### 2.2.6 Carr's index and Hausner ratio

Flow characteristics of powder are indicated by Carr's index (C) and Hausner ratio (H) which are calculated from tapped and bulk density. C and H reference values are given by Turchiuli *et al.* [15] and for this study the values were obtained by using equations (6) and (7):

$$\text{H} = \frac{\rho_T}{\rho_B} \quad \text{..... (6)}$$

$$\text{C} = \left(\frac{\rho_T - \rho_B}{\rho_T} \times 100\right) \quad \text{..... (7)}$$

## 3. Results and Discussion

For a good wall material, the most important requirement is its capacity to retain and seal the entire material of core inside its structure during processing and storage. If it does not meet this criterion, it results in excessive losses of core in the process. It will also result in a substantial amount of oil/fat on microcapsule surface. This surface oil is liable to be subjected to the action of atmospheric oxygen. These factors will then affect other characteristics of the microcapsules [35].

### 3.1 Moisture content and Water activity

The two major parameters, which determine the end quality and shelf life of powders are MC and $a_w$. Higher $a_w$ favours lump formation and fungal growth [33]. MC of TSHAP was lowest from the Day 0 to Day 60 when stored at room temperature ranging from 3.824 to 6.664% dry basis (Fig. 1a). Whereas, TSLP and TSMP ranged from 4.43-7.315% dry basis and 4.396-7.115% dry basis respectively. $a_w$ of TSHAP was also found to be lowest at Day 60 (0.591%) as compared to TSLP (0.6191%) and TSMP (0.6408%) (Fig.1b). Since $a_w$ was <0.60 for TSHAP, it was unfavourable for the microbial growth [36].

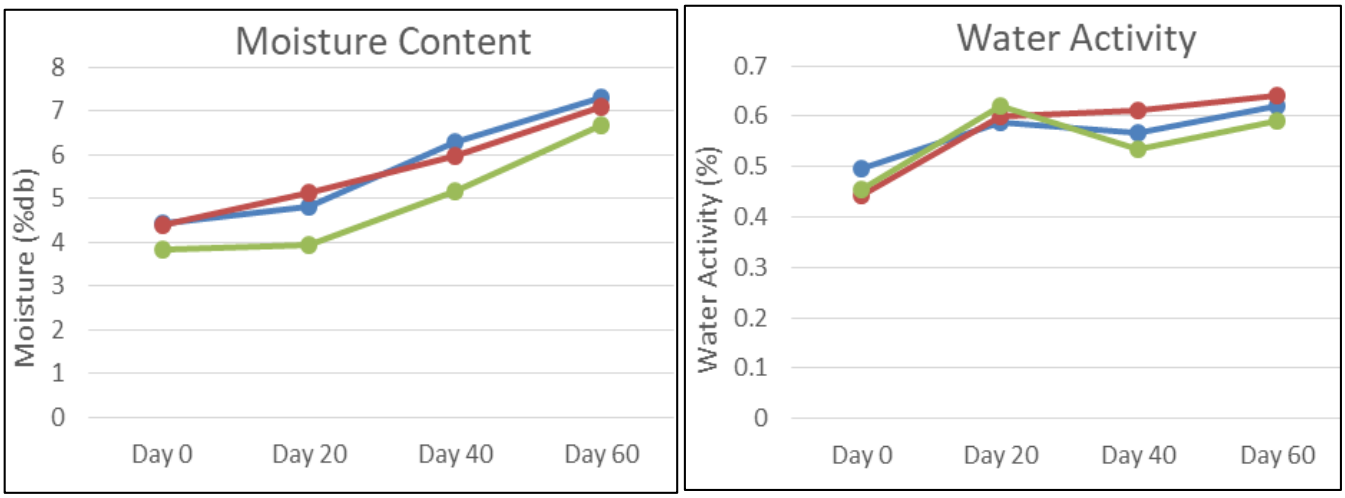


**Fig 1(a)** Moisture content, (b) Water activity of encapsulated and control samples

### 3.2 Surface Oil

High surface oil (about 30% of total oil) (Fig. 2a) was found in all the samples along with low encapsulation efficiency (about 50% of total oil) (Fig. 2b), which can be attributed to the larger emulsion droplets. Higher encapsulation efficiencies (87.0% to 92.1%) were obtained as a result of lower droplet mean diameters in a study by Sharif *et al.* [32] According to Jafari *et al.*, [37] the best method of

emulsification to obtain minimum amount of unencapsulated oil at the particle surface with optimum encapsulation efficiency is microfluidization. Ultrasound was also found as a good emulsification method, whereas Silverson rotor-stator emulsification wasn't found to be good equipment for preparing in feed emulsions. Droplet size of the final product was very important as smaller oil droplets entrap more easily inside the wall material, and the emulsion remains highly intact during atomization and drying. During atomization of emulsion, larger the oil droplets greater is the breakup inside the spray drying chamber [38]. Breakdown of emulsion results in a rise in surface oil, thereby reducing the efficiency of encapsulation [39]. Further, a high surface oil might be attributed to comings of internal oil from the microcapsules to it's the surface with hexane during surface oil estimation. As reported by Jafari *et al*. [37] porous particles with dents, cracks and fractures was revealed after washing with petroleum ether which might be responsible for high unencapsulated oils at the surface.

During storage study, surface oil of all the samples increased leading to further decrease in encapsulation efficiency. This increase maybe due to leaching out of encapsulated oil through deep dents and cracks on the external surface of powder particles. While no substantial variation could be seen between the microsphere's encapsulation efficiencies, the TSMP powder had lowest surface oil content as compared to TSLP and TSHAP powder, which shows the protection provided to the fat droplets by the wall material. An enhanced knowledge regarding external structure can be obtained from SEM studies of the encapsulated powder.

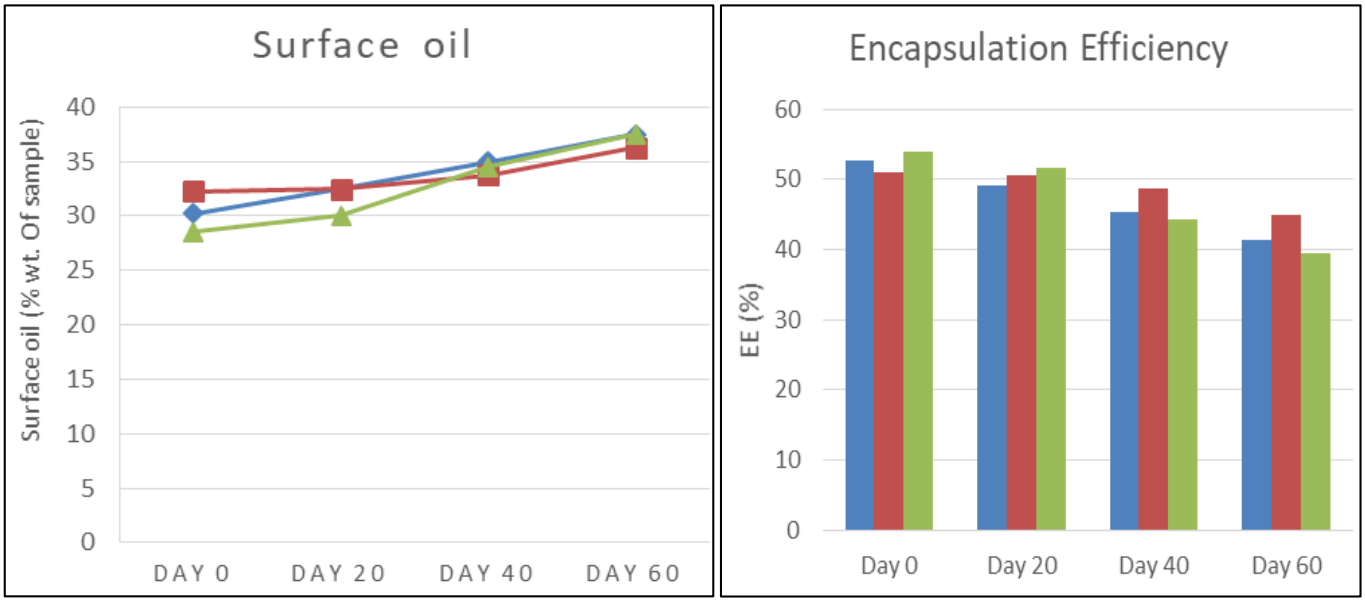


**Fig 2:** (a) Surface oil, (b) encapsulation efficiency of encapsulated and control samples

### 3.3 Peroxide Value

In oxidation of oil, peroxides are the initial products of whose value is used as a standard parameter which tells about the extent of lipid oxidation [40]. Fig 3 indicates that PV (peroxide value) of all samples increased signalling oxidation phase. Although the lipid surface area could be increased by the encapsulation process, allowing for greater exposure oxygen exposure, the formation of peroxide was lower in oil-loaded microcapsules than in encapsulated oil (control). Comparatively the PV of encapsulated oil was much lower than the control. Encapsulated oil PV increased but to a smaller degree from day 0 to day 60 (Fig.3) and displayed a behaviour comparable to control. The initial rise in PV of encapsulated oil was related to the oxidation of the surface oil in contact with the highest temperature, resulting in oxidation of lipids [41].

Highest oxidation stability was observed by microcapsule of sample TSLP (Sodium caseinate + Lactose) which had the lowest PV after 60 days (25meq/kg oil). A possible reason for this result is given by Fäldt and Bergenståhl [26],according to them the protein is the most surface-active element present in the emulsion before drying which accumulate at the air-water interface of the drying droplets. But, the protein is entirely hydrated in the surface film in the emulsion, and the film may shrink due to water loss during drying. If lactose is present in the emulsion, water can be replaced by the lactose upto some degree and after drying keep the protein solubilised, thus minimizing shrinkage. As a result of this, sodium caseinate film stability improves on the powder surface, reducing fat leakage on the powder surface during drying. PV of microcapsule of TSLP was less about 61.98% as compared to control palm oil sample after 60 days storage, indicating the importance of microencapsulation.

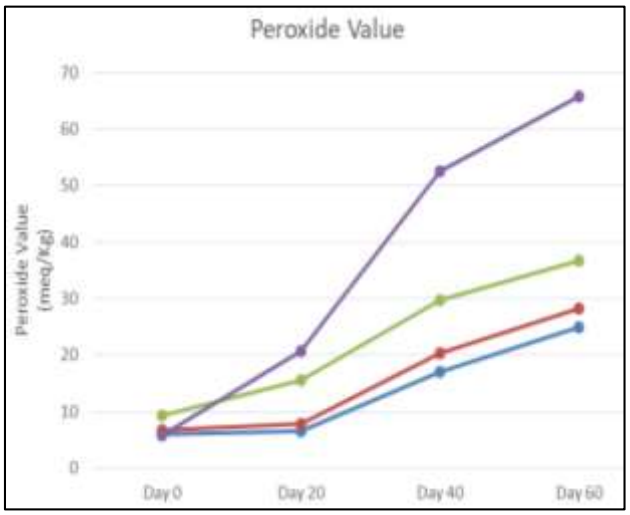


**Fig 3:** Peroxide value of encapsulated and control samples

### 3.4 Tapped and bulk density

Powder's tapped and bulk densities are essential quality variables relevant to packaging and transport. The microcapsules' bulk density values varied between0.2974 (TSLP) to 0.342 g/cm$^3$(TSHAP). Fig 4a shows that the TSLP formulation had the highest tapped density value (0.4675g/cm$^3$) and TSHAP observed the lowest value (0.3875 g/cm$^3$).The present study showed close resemblance to earlier observations on microcapsules of flaxseed oil in the range of 0.289–0.458 g/cm$^3$ [42]. Higher density powders are easy to store and ship by accommodating huge volumes into small containers relative to lower density powders [42].

### 3.5 Carr's index and Hausner ratio

Carr's index (% compressibility) and Hausner ratio indicates the flowability of powder. Carr's index values were between11.74 (TSHAP) to 36.38 (TSLP) and Hausner ratio between 1.13 (TSHAP) to 1.57 (TSLP) were observed for encapsulated palm oil powder (Fig 4b). The reported values of Hausner ratio for TSHAP lies in the range of 1.12-1.18 which indicates a good flow character and for TSLP it is very poor flow character. Similar results of flow character are obtained in terms of Carr's Index.

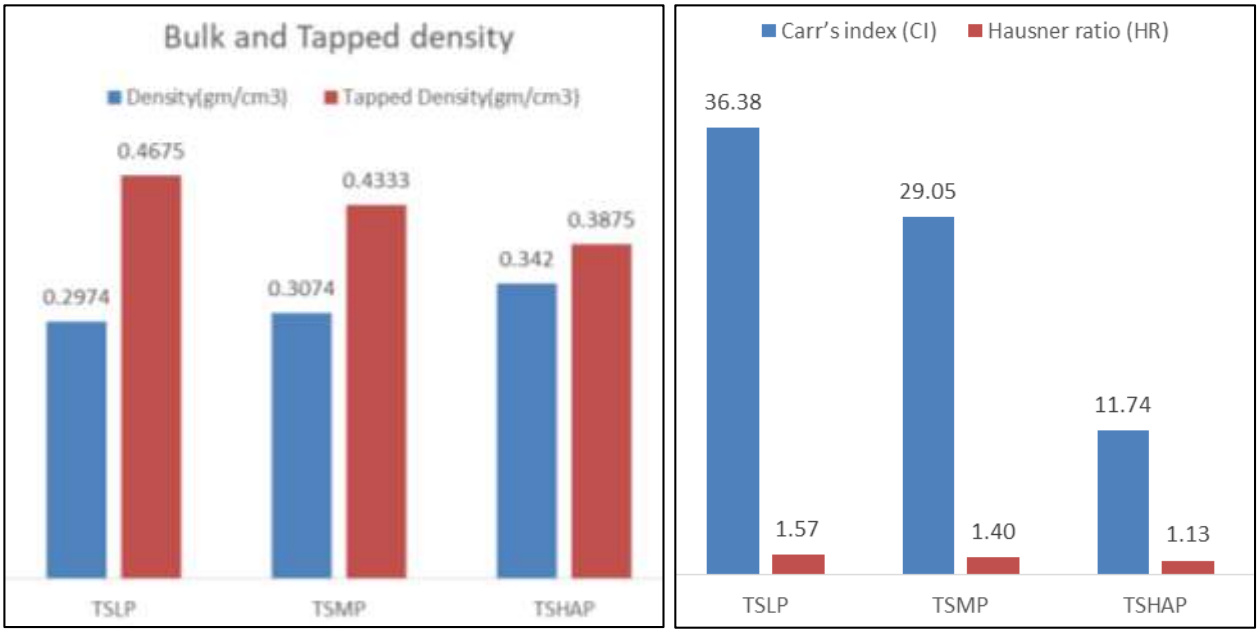


**Fig 4(a):** Bulk and tapped density, (b) Carr's Index and Hausner ratio of encapsulated and control samples

### 4. Acknowledgement

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### 5. Conclusion

Palm oil was successfully microencapsulated with the combination of TSLP (Sodium caseinate + Lactose), TSMP (Sodium caseinate +Maltodextrin), and TSHAP (Sodium caseinate + Hi-Cap + Acacia gum) using spray drying. From storage test and oxidative stability, it can be observed that microencapsulation process can lead to reduction in the lipid oxidation of palm oil. Despite the microspheres having higher surface oil contents, no difference in encapsulation efficiency was observed in the samples. Results show clearly that the method of emulsification influences and determines in various ways the final characteristics of the encapsulated powder including powder particle size, size distributions, emulsion stability, emulsion size, and the surface oil of the spray dried powders. A better emulsification method such as Microfluidization is highly recommended for emulsion preparation.

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