

RESEARCH ARTICLE

Application of Taguchi orthogonal array design to optimize microencapsulation of zinc by spray-drying

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Abstract: Zinc is an essential trace element for the body to maintain normal health and perform important functions. As zinc is not stored in the body, it needs to be replenished through our daily diet. Microencapsulation technique can be used to encapsulate the functional ingredient such as zinc by masking its metallic taste and enhancing its bioavailability. Among the microencapsulation techniques, spray-drying is the most economical and scalable method. Zinc was microencapsulated by spray-drying using maltodextrin, HI-CAP[®] 100 and WPI along with gum Arabic as wall materials. The spray-drying conditions were optimized using Taguchi L₁₈ orthogonal array design using encapsulation efficiency and bulk density as response factors. The influence of wall materials, wall material to zinc ratio and inlet air temperature was evaluated. Microcapsules prepared with HI-CAP[®] 100 in the ratio of 20:1 at 185°C showed maximum encapsulation efficiency, whereas microcapsules with HI-CAP[®] 100 in the ratio of 10:1 at 185°C had maximum bulk density. The microencapsulated zinc powder had bulk density of 437.40-541.20 kg/m³ and encapsulation efficiency of 76.86-92.65%. Validation experiments confirmed that Taguchi orthogonal array design was successful in optimizing microencapsulation of zinc. Microencapsulated zinc can be fortified in various delivery systems such as milk and milk products.

Keywords: Microencapsulation, Optimization, Spray-drying, Taguchi orthogonal array, Zinc

Introduction

Minerals are the vital inorganic nutrients required by human body for its normal functioning and growth. Among these, zinc is an essential trace element needed by the body to maintain normal health and perform vital functions such as cell growth, wound healing, immune system function, bone mineralization, blood clotting, cognitive functions and intellectual development (Maret and Sandstead 2006). Zinc is a cofactor to more than 300 enzymes and a powerful therapeutic tool to manage a long list of illness (Polekkad et al. 2021). As zinc is not stored in the body, it needs to be replenished through our daily diet. Zinc deficiency in humans is known to be a major malnutritional problem. According to International Zinc Association (IZA), nearly 1.9 billion people are suffering worldwide due to zinc deficiency (International Zinc Association 2018). Foods that are rich source of zinc include chicken, oysters, tofu, beef, pork, lentils, nuts, hemp seeds, yoghurt, oatmeal and mushrooms. With most of the vegetarian diet consisting of whole grains and plant proteins, the vegan populations are under the risk of zinc deficiency. Though some of the vegetarian foods are rich in zinc, its absorption from composite foods is a problem due to the presence of phytate (Oberleas and Harland 1981). To alleviate the problem of zinc deficiency, it could be fortified in suitable food systems. However, zinc cannot be added directly to food as it has a bitter, unpleasant and metallic after-taste, which is not liked by the consumers (Öner et al. 1988).

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Microencapsulation is a delivery technology that can be used to encapsulate and mask the metallic taste of functional ingredients such as zinc (Ré 1998). In addition, microencapsulation also helps in controlled-release of bioactives, which enables their better absorption (Singh et al. 2010). In food systems, encapsulation by physical methods is mostly preferred for protection and effective delivery of nutrients (Madene et al. 2006). Studies demonstrated that microencapsulation of minerals was useful in preventing the deteriorative reactions in food matrix,

since the minerals could not act as catalysts (Gupta et al. 2015). Microencapsulation should ensure the reduction of reaction of minerals with other ingredients, its proper release and dietary uptake. The stability of microencapsulated mineral depends on the reaction between wall and core materials (Gharsallaoui et al. 2007). Therefore, depending on the stability, bioavailability and compatibility with the selected technique, wall materials required for encapsulation are selected (Augustin and Sanguansri 2008).

Spray-drying is an economical and scalable method of encapsulation used in the food industry, which transforms the dispersion of liquid feed into dried capsules. It has already been used to microencapsulate nutrients due to its low processing time and high throughput (Rezvanhah et al. 2019). It effectively microencapsulates all active ingredients and reduces their chemical and biological degradation. The properties of spray-dried microencapsulated minerals depend on the type of polymer and its concentration (Oneda and Ré 2003). Spray-dried powders have instantaneous solubility and superior functional and reconstitutive properties. The desired properties of spray-dried powders can be achieved by regulating the process parameters. However, microencapsulation is influenced by spray-drying conditions such as inlet temperature of drying air, the ratio of wall material to active ingredient, flow rate of feed, total solids content in feed, etc. For successful microencapsulation of zinc, appropriate wall material or a combination of wall materials and spray-drying conditions need to be optimized.

The Taguchi orthogonal array optimization is a robust design that considers both the controllable and noise factors. The tools used in the Taguchi design are signal-to-noise (S/N) ratio (measures quality) and orthogonal arrays (accommodate design parameters) (Ghani et al. 2004). This method can be used to optimize the spray-drying conditions for microencapsulation of zinc for production of the microencapsulated zinc powder. To alleviate the problem of zinc deficiency, zinc could be fortified in suitable foods. Milk is considered as a potential vehicle for fortification. Hence, fortification of milk and milk products with zinc is an effective method to alleviate this problem. However, zinc cannot be added directly to milk as it has a bitter, unpleasant and metallic after taste, which is not liked by consumers (Öner et al. 1988).

Thus, the objective of the study was to establish spray-drying as a successful method for encapsulation of zinc and to optimize microencapsulation of zinc using Taguchi orthogonal array design.

Materials and Methods

Zinc sulphate heptahydrate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) was procured from Sisco Research Laboratories Pvt. Ltd. Mumbai, India. Whey protein isolate (WPI) was procured from Nakoda Dairy, Bengaluru, India. HI-CAP® 100 was supplied *on gratis* by Ingredion India

Pvt. Ltd. Mumbai, India. Gum Arabic and all other chemicals used in this study were of analytical grade, and were purchased from HiMedia Laboratories, Mumbai, India.

Preparation of dispersion

Dispersions of zinc sulphate heptahydrate were made using different wall materials such as maltodextrin, HI-CAP® 100 and WPI along with gum Arabic. The wall material to zinc ratio were 10:1 and 20:1, and the total solids (TS) content of the dispersions was maintained at 30.5%. To prepare the dispersions, zinc sulphate was dissolved in double distilled water, and the wall materials were blended using a magnetic stirrer (Model: CMAG HS7, IKA, Staufen, Germany). The dispersions were homogenized using high shear mixer (Model: Unidrive X 1000D, CAT Scientific, Staufen, Germany) at 15,000 rpm for 15 min.

Microencapsulation process

Microencapsulation of zinc was carried out in a co-current flow spray-dryer (Model: LU-222 Advanced, Labultima, Mumbai, India), equipped with two-fluid nozzle atomizer, drying chamber, cyclone separators, hot air system, pre-filters and bag filters. The feed flow rate of 6 mL/min was adjusted by peristaltic pump, and the inlet drying air temperatures were 170, 185 and 200°C. The outlet air temperature was kept at 80-85°C. The aspirator flow rate was maintained at 60 Nm³/h. Hot air was used as heating medium with co-current flow mode and the powder was collected from the chamber and cyclone, and mixed.

Experimental design and optimization

Wall material to zinc ratio, wall material and inlet air temperature were selected as the process factors for microencapsulation. Taguchi L₁₈ (3²×2¹) mixed orthogonal array design was used to optimize the spray-drying conditions using Minitab 17 software (Minitab Inc., Pennsylvania, USA) (Table 1). This method uses a loss function, which is converted into S/N ratio (η). The S/N ratio is the logarithmic function of target value. The targets are maximum encapsulation efficiency and bulk density of the microcapsules as they are generally desirable properties of food powders. Larger-the-better was used because they are important characteristics of microcapsules as given in Equation (1).

$$\eta = \text{SN} = -10 \log \left[\frac{1}{n} \sum_{i=1}^n \frac{1}{X_i^2} \right] \quad (\text{Haq et al. 2008}) \quad (1)$$

where, 'X_i' is the observed value at the ith response and 'n' is the total number of observations in the experiment. ANOVA was run using Minitab 17 software (Minitab Inc., Pennsylvania, USA) to evaluate the effect of individual factors at 5% level of significance. The significance of process factors was assessed by relative

comparison of 'F' values, and the contribution rate of each factor indicating the degree of influence on process performance was also calculated. Once the optimal levels of the process factors were selected, a confirmation experiment was conducted to validate the optimized conditions. For predicting the optimum spray-drying conditions, the estimated S/N ratio ($\hat{\eta}$) was computed using Equation (2).

$$\hat{\eta} = \eta_m + \sum_{i=1}^q (\eta_i - \eta_m) \quad (2)$$

where, ' η_m ' is the mean S/N ratio, ' q ' is the number of significant factors and ' η_i ' is the average S/N ratio corresponding to i^{th} significant factor on j^{th} level.

In order to evaluate the accuracy of optimization, the confidence intervals (C.I.) for encapsulation efficiency and bulk density were computed using Equations (3) and (4) (Haq et al. 2008).

$$C.I. = \sqrt{F_{\alpha(1, f_c)} V_e \left[\frac{1}{\eta_{eff}} + \frac{1}{R} \right]} \quad (3)$$

Table 1. Taguchi orthogonal array design $L_{18}(3^2 \times 2^1)$.

Trial	Process factors			Designation
	A Wall material to zinc ratio	B Wall material	C Inlet air temperature (°C)	
1	10:1	Maltodextrin	170	A ₁ B ₁ C ₁
2	20:1	Maltodextrin	170	A ₂ B ₁ C ₁
3	10:1	Maltodextrin	185	A ₁ B ₁ C ₂
4	20:1	Maltodextrin	185	A ₂ B ₁ C ₂
5	10:1	Maltodextrin	200	A ₁ B ₁ C ₃
6	20:1	Maltodextrin	200	A ₂ B ₁ C ₃
7	10:1	HI-CAP® 100	170	A ₁ B ₂ C ₁
8	20:1	HI-CAP® 100	170	A ₂ B ₂ C ₁
9	10:1	HI-CAP® 100	185	A ₁ B ₂ C ₂
10	20:1	HI-CAP® 100	185	A ₂ B ₂ C ₂
11	10:1	HI-CAP® 100	200	A ₁ B ₂ C ₃
12	20:1	HI-CAP® 100	200	A ₂ B ₂ C ₃
13	10:1	WPI	170	A ₁ B ₃ C ₁
14	20:1	WPI	170	A ₂ B ₃ C ₁
15	10:1	WPI	185	A ₁ B ₃ C ₂
16	20:1	WPI	185	A ₂ B ₃ C ₂
17	10:1	WPI	200	A ₁ B ₃ C ₃
18	20:1	WPI	200	A ₂ B ₃ C ₃

where, $F_{\alpha(1, f_c)}$ is the 'F' ratio at 95% confidence, ' α ' is the significance level, ' f_c ' is the error degrees of freedom, ' V_e ' is the error variance, ' η_{eff} ' is the effective number of replications, ' R ' is the number of replications.

$$\eta_{eff} = \frac{N}{1 + T_{dof}} \quad (4)$$

where, ' N ' is the total number of experiments and ' T_{dof} ' is the total degrees of freedom of the main factor. The regression models were established between the process and response variables. The error percentage between Taguchi design and regression models was compared.

Analysis of microcapsules

Mineral contents

Exactly 2 g of microencapsulated powder was taken in a pre-weighed silica crucible. The sample was decarbonized on a flame, and transferred to muffle furnace for ignition at 550-600°C for 5 h. Then the sample was cooled and weighed quickly. Exactly 15 mL of 5 N HCl was added into the silica crucible and the contents were boiled for 15 min. The solution obtained was filtered through Whatman No.41 filter paper into a volumetric flask. The volume

was then made up to 100 mL by repeated washing of crucible. The zinc content was estimated in inductively coupled plasma optical emission spectrometer (ICP-OES) (Model: Optima 8000, Perkin Elmer, Shelton, USA) at a wavelength of 206.20 nm using zinc oxide as standard solution. The equipment was operated in radial spectrophotometric view with the sample volume uptake of 1 mL/min.

Encapsulation efficiency

Encapsulation efficiency of microcapsules was evaluated by the method reported by Abbasi and Azari (2011) with slight modifications. Exactly 30.5 g of microcapsules was added to 100 mL deionized water. Then 10 mL of this solution was taken in a cellulose membrane bag (MW cut-off 12,400 Da), and it was immersed in deionized water with gentle agitation, where non-encapsulated zinc was leached out. After 6 h, the contents were taken from the bag and the zinc content was estimated using ICP-OES as described above. Encapsulation efficiency of microcapsules was calculated using Equation (5).

$$\text{Encapsulation efficiency} = \frac{\text{Bound zinc}}{\text{Total zinc}} \times 100 \quad (5)$$

Bulk density

Bulk density of the powder prepared under various conditions was determined through ASTM D7481-09 (2009). Exactly 100 g of sample was taken in a graduated cylinder and weighed. The

cylinder was mildly tapped thrice, and the volume was measured (Equation 6).

$$\text{Bulk density (kg/m}^3\text{)} = \frac{\text{Weight of microencapsules}}{\text{Apparent volume}} \quad (6)$$

Results and Discussion

Optimization of spray-drying conditions

Taguchi orthogonal array design was used to optimize the spray-drying conditions for achieving maximum encapsulation efficiency and bulk density of zinc microcapsules. The encapsulation efficiency and bulk density of microcapsules were determined experimentally for 18 combinations listed in the design (Table 2). It is evident that the encapsulation efficiency ranged between 76.86 and 92.65%, while the bulk density lied in the range of 390.0-541.2 kg/m³. It was found that microencapsulation efficiency was highly impacted ($p < 0.001$) by factors such as type of wall materials, ratio of wall material to core material and inlet air temperature. The encapsulation efficiency was most strongly impacted by the ratio of wall material used, with the mean values for 10:1 and 20:1 ratios being 82.15% and 86.53%, respectively. The type of wall material and temperature also influenced the encapsulation efficiency, although to a lesser extent than the ratio of wall material used. According to Hogan et al. (2001), the ratio of core to wall material had greater impact on the microencapsulation efficiency of powders. The authors concluded that a higher proportion of wall material can increase the rate of formation and thickness of the semi-permeable

Table 2. Taguchi orthogonal experimental design responses with S/N

Designation	Encapsulation efficiency (%)	Bulk density (kg/m ³)	S/N ratio for encapsulation efficiency (dB)	S/N ratio for bulk density (dB)
A ₁ B ₁ C ₁	76.86	502.3	38.04	54.02
A ₂ B ₁ C ₁	84.37	476.7	38.52	53.56
A ₁ B ₁ C ₂	82.80	536.4	38.36	54.59
A ₂ B ₁ C ₂	88.00	490.1	38.89	53.80
A ₁ B ₁ C ₃	79.42	515.8	38.11	54.25
A ₂ B ₁ C ₃	86.54	487.3	38.74	53.75
A ₁ B ₂ C ₁	85.00	515.4	38.59	54.24
A ₂ B ₂ C ₁	87.73	485.6	38.86	53.73
A ₁ B ₂ C ₂	87.60	541.2	38.85	54.67
A ₂ B ₂ C ₂	92.65	505.8	39.34	54.08
A ₁ B ₂ C ₃	86.83	524.1	38.77	54.39
A ₂ B ₂ C ₃	88.90	492.4	38.98	53.85
A ₁ B ₃ C ₁	79.83	412.7	37.71	52.31
A ₂ B ₃ C ₁	83.62	390.0	38.24	51.82
A ₁ B ₃ C ₂	82.60	437.4	38.15	52.82
A ₂ B ₃ C ₂	87.90	408.6	38.58	52.22
A ₁ B ₃ C ₃	80.20	417.2	37.97	52.40
A ₂ B ₃ C ₃	84.07	398.7	38.49	52.01

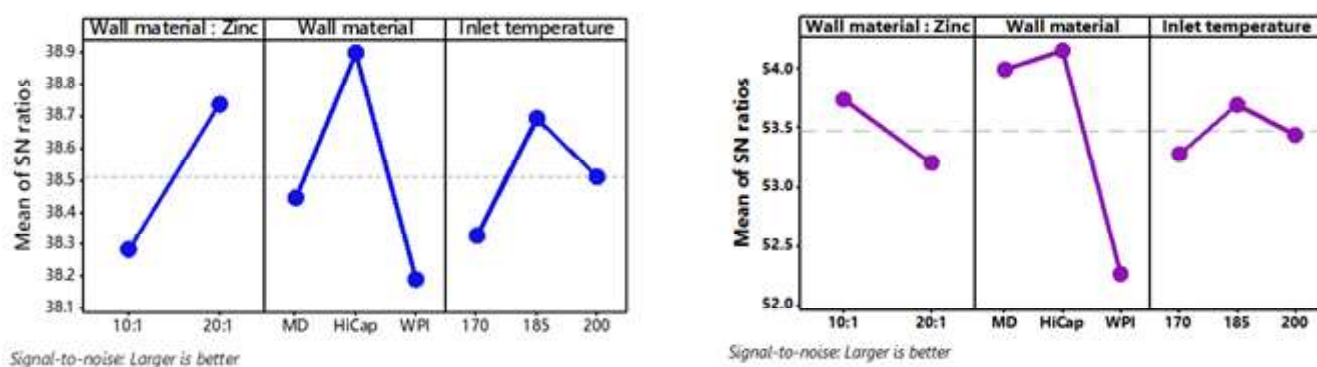


Fig. 1 Effect of parameters on mean S/N ratio for (a) encapsulation efficiency and (b) bulk density

membrane surrounding the core material, leading to increased encapsulation of the active ingredient inside the microcapsules. This finding is in consistent with the results on encapsulation efficiency, which demonstrated that increasing the ratio of wall material resulted in higher encapsulation efficiency.

High ratios of wall materials are often used in microencapsulation processes. For instance, Turasan et al. (2015) employed wall to core material ratios of 40:1, 20:1, and 10:1 for encapsulating rosemary oil in whey protein concentrate and maltodextrin through spray-drying. The authors observed that the best wall to core material ratio for achieving high encapsulation efficiency was 20:1. Similarly, Wu et al. (2014) used wall to core material ratios ranging from 5:1 to 25:1 for microencapsulating sulforaphane through spray-drying and determined that the optimal ratio for encapsulation as 20:1.

The highest encapsulation efficiency (mean 88.12%) was observed with HI-CAP[®] 100, whereas WPI exhibited the lowest efficiency (mean 81.24%). These findings are consistent with those of Gupta et al. (2015), who reported microencapsulation efficiency of 91.58% for iron using 1:10 ratio of wall material. Furthermore, the authors observed that modified starch provided higher encapsulation efficiency compared to maltodextrin-based microcapsules. Notably, HI-CAP[®]100 is a modified starch specifically developed for encapsulation purposes. Additionally, an increase in air temperature was found to increase the efficiency of zinc encapsulation, as it led to faster drying rate of droplets and the formation of particle crust, which locked the core material (zinc) inside the dry matrix (Liu et al. 2016).

The average bulk density of zinc microcapsules produced using maltodextrin, HI-CAP[®] 100, and WPI as wall materials were 536.40, 541.20, and 437.40 kg/m³, respectively. The wall material type had the greatest impact ($p < 0.001$) on bulk density, followed by the ratio of wall material ($p < 0.001$) and drying air temperature ($p < 0.001$). The higher bulk density of HICAP[®] 100-based microcapsules was attributed to their higher molecular weight, ordered and arranged particulates. Conversely, the lower bulk density of WPI-based zinc microcapsules was due to high

occluded air content. The emulsification and membrane forming ability of WPI was believed to lead to ballooning or puffing of the microcapsules during drying, which increased the particle size by increasing the occluded air content and reduced the bulk density (Walton 2000). Samborska et al. (2015) investigated the use of maltodextrin as wall material in microencapsulation of honey by spray drying and determined its impact on bulk density of the dried powder. Results showed that the bulk density ranged between 330 and 550 kg/m³, with a decrease observed as the drying air temperature increased, could be due to the production of larger microcapsules (Tonon et al. 2011). On the other hand, an increase in the wall material ratio resulted in reduction of bulk density, indicating that zinc had higher density compared to the three wall materials used. As bulk density is a crucial factor in packaging, transportation, marketing, and storage, HI-CAP[®] 100 based zinc microcapsules would be the optimal choice.

Signal-to-noise (S/N) ratio

Optimization of the process factors was done using S/N ratio, which is indicative of the deviation of the responses from the desired value. The mean S/N ratio for encapsulation efficiency and bulk density were computed as 38.51 and 53.47 dB, respectively.

Analysis of the effect of each process factor (wall material to zinc ratio, type of wall material and inlet air temperature) on encapsulation efficiency and bulk density was performed using S/N ratio responses. By calculating the difference between the highest and the lowest S/N ratio, the delta value was obtained. The process factor with the highest delta value was ranked first (I), and so on. Thus, the optimum levels of process factors for maximum encapsulation efficiency and bulk density of the microcapsules were obtained.

The influence of process conditions on the response factors is graphically illustrated using S/N ratio in Figs. 1 a & b. The best spray-drying process conditions to achieve maximum encapsulation efficiency and bulk density could be visualized from these graphs (Figs. 1 a&b). Accordingly, the levels and S/N

ratio of the process factors yielding maximum encapsulation efficiency were identified as factor A (Level 2, S/N=38.74 dB), factor B (Level 2, S/N=38.90 dB) and factor C (Level 2, S/N=38.69 dB). In real numbers, the optimum spray-drying process conditions for maximizing the microcapsules encapsulation efficiency were wall material to zinc ratio of 20:1, HI-CAP® 100 as wall material and inlet drying air temperature of 185°C. Similarly for maximum bulk density of zinc microcapsules, the optimal conditions were wall material to zinc ratio of 10:1, HI-CAP® 100 as wall material and inlet drying air temperature as 185°C. A comparable result was documented by Pal and Chattacharjee (2018) regarding the encapsulation efficiency of spray-dried marigold flowers enriched with lutein. Additionally, it was observed that the bulk density showed an increasing-decreasing pattern when the temperature of the inlet air used for drying was increased from 170 to 200°C. The highest bulk density was achieved at 185°C. Jafari et al. (2019) employed the Taguchi orthogonal array design to determine the optimal levels of maltodextrin, modified starch, whey protein concentrate, and GA as wall materials for microencapsulating vitamin D₃-enriched whey powder. The results showed that the combination of all wall materials produced the highest yield of powder when the inlet air temperature was set at 170°C. Also, the results were in accordance with Patel et al. (2022), who employed the Taguchi orthogonal array design (L₁₈) to optimize the microencapsulation of curcumin. The results showed that the best conditions for microencapsulation of curcumin were inlet drying air temperature of 185°C, feed rate of 6 mL/min, and HI-CAP® 100 as the wall material. These conditions resulted in moisture content of 4.65%, encapsulation efficiency of 82.42%, and bulk density of 358.40 kg/m³.

Analysis of variance (ANOVA)

ANOVA was done to evaluate the significance of each process factor on response variables. The results of ANOVA are shown in Table 3. All the three factors had statistically significant effect on encapsulation efficiency and bulk density. However, the type

of wall material was observed to be the most significant factor, whereas inlet air temperature of spray-drying was considered as less significant factor to achieve both maximum encapsulation efficiency and bulk density. Moghbeli et al. (2020) used the Taguchi design to optimize the type of drying aid, pH, and inlet air temperature on the moisture content and bulk density of spray-dried powder. The results indicated that temperature had the most significant impact on the responses. Similar results were reported by Patel et al. (2022) for microencapsulation of curcumin.

Regression analysis

The linear and quadratic predictive models for encapsulation efficiency and bulk density are presented in Equations (7-10). The relationship between the observed and predicted encapsulation efficiency and bulk density are shown as Fig. 2. The quadratic relationships between experimental and predicted data were adequate and satisfactory with adj. R² above 0.8, while simple linear relationships were not satisfactory. Thus, the encapsulation efficiency and bulk density of zinc microcapsules were predicted based on the spray-drying conditions.

$$\text{Encapsulation efficiency} = 69.30 + 4.38 \times \text{wall material:zinc} - 1.21 \times \text{wall material} + 0.059 \times \text{inlet air temperature} \quad (\text{adj. } R^2 = 0.281) \tag{7}$$

$$\text{Bulk density} = 555 - 29.70 \times \text{wall material:zinc} - 45.33 \times \text{wall material} + 0.293 \times \text{inlet air temperature} \quad (\text{adj. } R^2 = 0.612) \tag{8}$$

$$\text{Encapsulation efficiency} = -355 + 4.38 \times \text{wall material:zinc} + 21.46 \times \text{wall material} + 4.46 \times \text{inlet air temperature} - 5.67 \times \text{wall material}^2 - 0.0119 \times \text{inlet air temperature}^2 \quad (\text{adj. } R^2 = 0.963) \tag{9}$$

$$\text{Bulk density} = -2398 - 39.83 \times \text{wall material:zinc} + 165.67 \times \text{wall material} + 30.55 \times \text{inlet air temperature} - 54.65 \times \text{wall material}^2 - 0.08 \times \text{inlet air temperature}^2 + 5.07 \times \text{wall material:zinc} \times \text{wall material} \quad (\text{adj. } R^2 = 0.993) \tag{10}$$

Table 3. ANOVA for encapsulation efficiency and bulk density

Factor	DOF	SS	MSS	F ratio	Contribution (%)
Encapsulation efficiency					
Wall material : zinc	1	86.417	86.4170	143.82	31.13
Wall material	2	146.027	73.0136	121.52	52.60
Inlet air temperature	2	37.951	18.9756	31.58	13.67
Error	12	7.210	0.6009		2.60
Total	17	277.606			100
Bulk density					
Wall material : zinc	1	3969.4	3969.4	176.03	9.35
Wall material	2	36607.8	18303.9	811.71	86.27
Inlet air temperature	2	1586.6	793.3	35.18	3.74
Error	12	270.6	22.5		0.64
Total	17	42434.4			100

*Bold values indicate the most influential factor

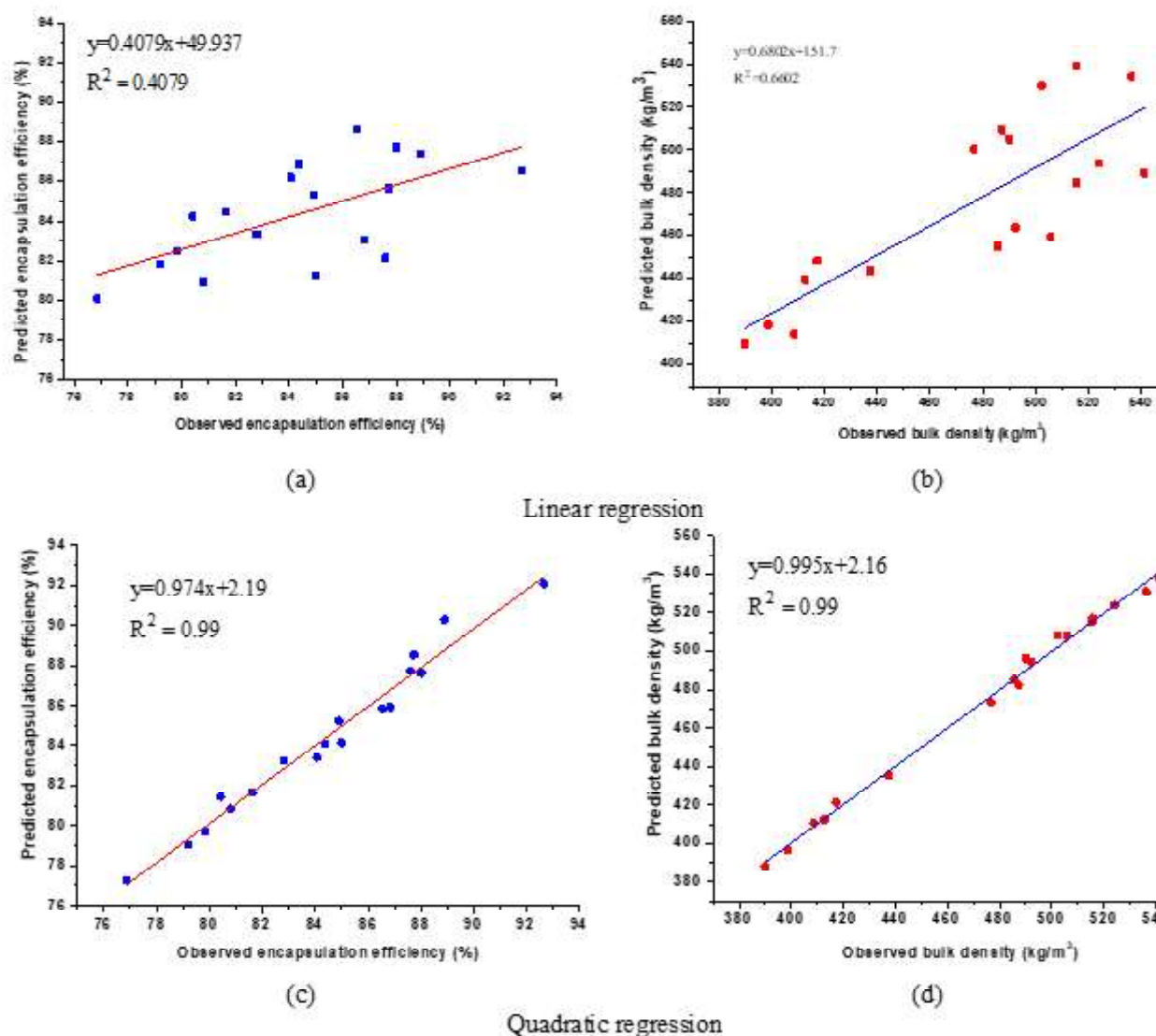


Fig. 2. Relationship between experimental and predicted values of encapsulation efficiency and bulk density using linear (a and b) and quadratic regression (c and d)

Prediction of optimum encapsulation efficiency and bulk density

For the prediction of optimum encapsulation efficiency and bulk density, Equations (11) and (12) were used.

$$Encapsulation\ efficiency_{opt} = \bar{A}_2 + \bar{B}_2 + \bar{C}_2 - 2\mu \tag{11}$$

$$Bulk\ density_{opt} = \bar{A}_1 + \bar{B}_2 + \bar{C}_2 - 2\mu \tag{12}$$

The confidence intervals of encapsulation efficiency and bulk density were calculated using Equations (3) and (4) as ± 1.38 and ± 8.44 , respectively. The experimental values of both responses

were within the limits of confidence interval at significance level of 0.05.

Validation

For validation, experiments were repeated thrice at the optimum and random levels and the predicted values from Taguchi design and predictive regression models were compared (Table 4). As the error obtained was less than 20% (acceptable limits), the selection of levels of spray-drying conditions (independent factors) was appropriate.

Conclusions

Spray-drying was found successful for microencapsulation of zinc with encapsulation efficiency of 92% and bulk density of

Table 4. Predicted values and confirmation test results by Taguchi method and regression analysis

Level	Expt.	Taguchi method		Linear regression		Quadratic regression	
		Pred.	Error (%)	Pred.	Error (%)	Pred.	Error (%)
Encapsulation efficiency							
A ₂ B ₂ C ₂ (Optimum)	92.43	92.10	0.36	86.53	6.38	92.09	0.66
A ₁ B ₁ C ₁ (Random)	79.77	79.70	0.09	82.48	3.40	79.70	0.39
Bulk density							
A ₁ B ₂ C ₂ (Optimum)	539.20	537.97	0.23	489.17	9.28	537.87	0.25
A ₂ B ₁ C ₁ (Random)	474.65	476.07	0.30	500.40	5.43	473.52	0.24

541 kg/m³. Taguchi orthogonal array technique was used to optimize the spray-drying conditions for microencapsulation of zinc. The optimized conditions for maximum encapsulation efficiency were Hicap-100 as wall material, 20:1 as wall material: zinc ratio and inlet air temperature of 185°C, whereas optimized conditions for maximum bulk density were HiCap-100 as wall material, 10:1 as wall material: zinc ratio and inlet air temperature of 185°C. The influence of wall materials, wall material to zinc ratio and inlet air temperature was evaluated. It was established that Taguchi orthogonal array design is a successful method for the optimization of microencapsulation of zinc and HI-CAP® 100 was found to be a suitable wall material for encapsulation. Microencapsulated zinc can be fortified in various delivery systems such as milk and milk products.

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