Drying Technology

An International Journal

ISSN: 0737-3937 (Print) 1532-2300 (Online) Journal homepage: https://www.tandfonline.com/loi/ldrt20

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To cite this article: Mamta Thakur, Kirty Pant, Rishi Ravindra Naik & Vikas Nanda (2020): Optimization of spray drying operating conditions for production of functional milk powder encapsulating bee pollen, Drying Technology, DOI: 10.1080/07373937.2020.1720225

To link to this article: https://doi.org/10.1080/07373937.2020.1720225

Published online: 30 Jan 2020.

DRYING TECHNOLOGY
https://doi.org/10.1080/07373937.2020.1720225

Optimization of spray drying operating conditions for production of functional milk powder encapsulating bee pollen

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ABSTRACT
This study was aimed to develop functional milk powder encapsulating bee pollen grains. The manufacturing of functional milk powder was successfully optimized by Box–Behnken design using inlet air temperature (180–200 °C), feed rate (8–10 mL min^{-1}), and bee pollen (5%–15%) as independent variables. The optimum spray drying conditions for obtaining milk powder were 187.58 °C inlet air temperature, 8.94 mL min^{-1} feed flow rate, and 8.04% bee pollen, respectively. The functional milk powder had an amorphous nature, narrow particles' size range, and polyphenolic content of 11.24 mg GAE g^{-1}. Further, scanning electron microscopy showed adequate encapsulation of pollen grains and FT-IR spectroscopy analysis confirmed the presence of polyphenolic compounds.

ARTICLE HISTORY
Received 1 November 2019
Revised 14 January 2020
Accepted 18 January 2020

KEYWORDS
Bee pollen; spray drying; functional milk powder; total phenolics content; encapsulation

1. Introduction

Recently, the bee pollen has been received great attention in the food industry as a natural dietary supplement due to its exceptional quality of healthy nutrients and bioactive compounds.[1–3] Bee pollen contains carbohydrates (13%–55%), proteins (10%–40%), lipids (1%–13%), minerals (2%–6%), and vitamins mainly B-complex, in addition to carotenoids and flavonoids.[3–5] It is composed of essential amino acids, fatty acids particularly α-linolenic acid, palmitic, linoleic acids and oleic acid, and polyphenols such as apigenin, catechins, chrysin, ferulic acid, gallic acid, hydroxycinnamic acid, p-coumaric acid, myricetin, quercetin, rutin.[6–8] Besides this, the bee pollen is also known for its potent biological functions attributed to its anti-inflammatory, antioxidant, anti-mutagenic, antimicrobial, immunomodulatory, and anticancerous properties.[9–11]

Acclaimed for its excellent nutrition and therapeutic properties, currently, bee pollen is available commercially in fresh or dried form and as capsules or dietary supplement worldwide; however, its applications in food remains challenging due to its degradation when exposed to heat.[12] Therefore, the encapsulations of bee pollen could open the gateway for its exploration in food processing. Milk due to its huge intake globally is an ideal medium for bee pollen valorization, and milk proteins have previously been known to encapsulate many bioactive compounds.[13–16] Further, milk being a good source of nutrients, however, lacks particular functional compounds such as phenolic compounds and dietary fiber besides ω-3 fatty acids, folates, niacin, and vitamin C which would be complemented by bee pollen.[2,17,18] Among several encapsulation techniques, the spray drying is the most widely used inexpensive and flexible encapsulation technique in the food and pharmaceutical industries due to reproducibility, retaining the quality and stability of product due to short drying time and ease of scaling up.[19–21] Huggett et al.[22] reported the promising results of curcumin encapsulated in spray-dried milk powders rather than whey protein isolate. The process of spray drying comprises the atomization of feed material against the hot and dried air resulting in the removal of water (solvent) due to highly efficient heat and mass transfer.[23] The spray drying conditions mainly temperature, and feed concentration and rate strongly affect the powder properties.[24,25] Duc et al.[26] showed the reduction in moisture content, bulk density, water holding capacity, and filling rate of spray-dried soymilk powder, while the interstitial air volume, Hausner ratio, porosity, and wettability index were increased on the account of variation in inlet air temperature and feed

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Table 1. Experimental design of independent variables and data of response variables of developed bee pollen-enriched milk powder.

Run(s)	Factor: 1 Inlet air temperature (°C) (x_1)	Factor: 2 Feed flow rate (mL min^{-1}) (x_2)	Factor: 3 Bee pollen (%) (x_3)	Response: 1 Bulk density (gcm^{-3})	Response: 2 Color (Whiteness)	Response: 3 Hygroscopicity (%)	Response: 4 Solubility (%)	Response: 5 Total phenolics content (mg GAEg^{-1})
1	190	10	5	0.49 ± 0.11	83.96 ± 0.75	18.97 ± 0.09	96.51 ± 0.70	11.86 ± 0.16
2	180	9	5	0.52 ± 0.14	86.86 ± 0.80	18.96 ± 0.10	97.21 ± 0.73	12.33 ± 0.18
3	190	9	10	0.41 ± 0.10	82.98 ± 0.77	19.98 ± 0.13	97.05 ± 0.71	11.63 ± 0.14
4	200	10	10	0.43 ± 0.13	80.17 ± 0.72	22.52 ± 0.16	95.64 ± 0.69	10.03 ± 0.13
5	190	10	15	0.41 ± 0.11	80.14 ± 0.76	20.32 ± 0.14	93.38 ± 0.62	9.77 ± 0.11
6	190	9	10	0.44 ± 0.12	84.43 ± 0.74	19.79 ± 0.14	96.87 ± 0.72	11.48 ± 0.15
7	190	9	10	0.42 ± 0.16	82.65 ± 0.71	20.15 ± 0.13	96.71 ± 0.71	11.34 ± 0.14
8	190	8	15	0.34 ± 0.09	78.83 ± 0.79	22.74 ± 0.17	94.26 ± 0.68	10.13 ± 0.12
9	200	8	10	0.40 ± 0.10	79.94 ± 0.67	23.43 ± 0.19	97.45 ± 0.74	10.05 ± 0.10
10	180	9	15	0.44 ± 0.14	84.42 ± 0.74	19.65 ± 0.11	93.57 ± 0.65	10.86 ± 0.11
11	190	8	5	0.48 ± 0.16	83.25 ± 0.72	20.18 ± 0.15	97.33 ± 0.72	11.19 ± 0.13
12	190	9	10	0.4 ± 0.11	83.76 ± 0.74	19.28 ± 0.12	96.42 ± 0.70	11.28 ± 0.15
13	180	10	10	0.49 ± 0.17	85.78 ± 0.81	19.04 ± 0.10	94.79 ± 0.69	11.81 ± 0.16
14	190	9	10	0.43 ± 0.14	83.22 ± 0.75	19.83 ± 0.13	96.69 ± 0.73	11.86 ± 0.15
15	200	9	15	0.31 ± 0.07	78.09 ± 0.64	24.22 ± 0.19	94.18 ± 0.67	8.25 ± 0.09
16	200	9	5	0.47 ± 0.12	84.53 ± 0.80	20.38 ± 0.13	97.82 ± 0.76	10.97 ± 0.11
17	180	8	10	0.47 ± 0.15	84.66 ± 0.79	20.16 ± 0.12	96.83 ± 0.73	11.32 ± 0.14

Values are the average ± standard deviation calculated on three replicates.

concentration. Bansal et al.[27] also showed the significant effect of temperature, feed flow rate, and concentration on moisture content, bulk density, solubility, DPPH scavenging activity, total phenolic, and flavonoid content while optimizing the production of spray-dried honey-based milk powder. Thus, the literature survey clearly showed significant variations in spray drying parameters as per raw materials and desired properties of the end product thus underlining the importance of optimizing drying conditions.

Keeping in view the previous discussion, the present investigation was conducted for the first time to produce functional milk powder containing encapsulated pollen grains by optimizing the spray drying conditions (temperature, feed rate, and bee pollen concentration). The flexibility of powder form allows the addition of bee pollen as a functional component in processed food products such as yoghurts, spreads, dietary supplements, beverages, bakery and confectionery products, frozen desserts. The optimized process technology can be simply adapted by dairy industries to produce functional milk powder thus enriching the commercial perspective of bee pollen that may aid in improving the economic status of beekeepers, particularly in developing nations.

2. Materials and methods

2.1. Chemicals and reagents

The chemicals including sodium chloride (NaCl), Folin–Ciocalteau reagent, and sodium carbonate (Na2CO3) were obtained from LobaChemie (Mumbai, India), while the reagents such as methanol (HPLC grade) and gallic acid (GA) were purchased from Merck (Darmstadt, Germany).

2.2. Raw materials

Fresh samples of honeybee-collected pollen ($n = 10$) were procured from northern states of India including Haryana (Jhajjar), Punjab (Mansa and Bathinda), and Rajasthan (Sriganganagar, Hanumangarh, Alwar, and Sawai Madhopur) during January to March, 2017 and regarded as uni-floral rapeseed (*Brassica napus*) pollen belonging to the Brassicaceae family which was determined using palynological analysis.[3] Our previous study showed the presence of 15.76% moisture (in dry matter), 44.89% total carbohydrate, 19.62% proteins, 12.38% lipids, 3.24% ash, and 4.13% crude fiber in rapeseed pollen.[3] On the other side, the skim milk was purchased from the local market of Sangrur (Punjab), India, which had following composition: 90.64% moisture content, 0.35% total fats, 3.16% proteins, 0.79% minerals, and 5.06% total carbohydrates.

2.3. Experimental design

The optimization of multiple process parameters and their interaction to develop functional milk powder encapsulating bee pollen were done using Box–Behnken design of response surface methodology (RSM) to minimize the number of unnecessary trials and efforts, save time, and provide necessary data for statistically acceptable results. RSM works on the plan of examinations according to real scientific techniques; evaluates the impacts of components; and ultimately proposes the optimum states of reaction to get the required effect.[28] The three independent variables – inlet air temperature (180–200 °C), feed rate (8–10 mL min^{-1}), and bee pollen amount (5%–15%) were used at three levels (maximum, minimum, and central) after conducting the preliminary trials to select the minimum and maximum values of variables, as mentioned in Table 1. This results in the seventeen sets of experiments including five replicates at the center point.[29] The response variables considered for experiment were bulk density, color (expressed in whiteness), hygroscopicity, solubility, and total phenolics content (TPC) of developed milk powder. The maximum phenolic contents, solubility, and whiteness (color) and minimum hygroscopicity were aimed to result in the development of powder which would be esthetically similar to commercial milk powder, but contain the optimum polyphenol content along with other functional and physical properties. The bulk density was targeted to 0.45 g cm^{-3} because the desired bulk density of spray-dried milk powder ranges from 0.400 to 0.450 g cm^{-3}.[30]

2.4. Sample preparation and spray drying

Ten percent solution of bee pollen (e.g., 5 g in 50 mL, 10 g in 100 mL, and 15 g in 150 mL) was prepared using potable water which was then ultrasonicated for 60 min in an ultrasonicated bath (MSW-269 MAC Macro Scientific Works, Delhi, India). In our preliminary trials, we found that the pollen pellets when ground and mixed with skim milk as such were very difficult to pass the dryer fluid nozzle for a longer time. The nozzle was used to choke within 30 ± 10 min after running the experiment each time. Therefore, bee pollen was treated with ultrasonic waves to break the pollen grains and release the bioactive compounds in extract. The prepared pollen extract was then taken in the respective concentration, as mentioned in experimental design, and mixed with skim milk. The mixture was stirred thoroughly to obtain homogenous milk bee pollen slurry with a total solid content of 14.26 ± 0.12%, 15.71 ± 0.09%, and 17.16 ± 0.10% in samples containing bee pollen extract at 5%, 10%, and 15%, respectively. The viscosity of feed slurry was determined using Brookfield viscometer DV- II + Pro (AMETEK Brookfield, Middleboro, MA, USA) at constant speed of 100 rpm and temperatures of 25 °C with a spindle no S-62 which varied from 12.2 ± 0.05 cP to 14.7 ± 0.08 cP as the solids increased. Two liters of milk slurry was spray dried in each experimental trial.

The laboratory-scale co-current spray dryer (S.M. Scientech, Kolkata, India; water evaporation capacity of 3 L/h) comprised of the food-grade stainless steel body. It was equipped with a pneumatic nozzle (two-fluid nozzle) atomizer (nozzle tip diameter: 0.7 mm and nozzle screw cap diameter: 1.5 mm), air compressor (air flow rate 5 bars), air filter, and blower (at a constant speed of 2000 rpm). Before drying the sample, the spray dryer was run for 20 min by feeding distilled water to obtain the steady-state conditions. After attaining the desired conditions, milk pollen slurry was fed to the drying chamber of spray dryer using peristaltic pump and regulating the feed flow rate (Table 1) by adjusting pump rotation speed. The feed was then atomized using compressed air leading to droplets which were dried in the main chamber by introducing the dried hot air. The inlet air temperature was varied according to the experimental conditions (Table 1). The obtained powder was collected in the pre-weighed and cyclone separator-connected glass jars and allowed to cool. The powder was then packed and analyzed within one week. This amount of powder collected using cyclone-attached glass jar was used to determine the cyclone recovery, while the amount of powder obtained after manual sweeping of chamber walls and cyclone refers to the sweep recovery. Total recovery (cyclone plus sweep recovery) was therefore calculated to determine the spray dryer performance. During the entire process, the average pressure, feed temperature, and relative humidity of ambient air were kept at 101.325 kPa, 25 ± 2 °C, and 70 ± 5% (at 25 ± 2 °C), respectively.

2.5. Analysis of functional milk powder

2.5.1. Bulk density

The developed milk powder (1 g) was freely transferred to a graduated cylinder (5 mL, readable at 1 mL) which was then tapped repeatedly until the constant volume was obtained.[31] The bulk density was determined using formula (Equation 1):

$$\begin{aligned}&\text{Bulk density}(\text{g cm}^{-3})\\&= \text{Mass of powder (g)}/\text{Volume after tapping }(\text{cm}^3)\end{aligned} \tag{1}$$

2.5.2. Color

The color of powder samples was determined using a colorimeter (Model CR-10, Konica Minolta Sensing Americas, NJ, USA) equipped with D65 illuminant. The color space of $L^*a^*b^*$ was examined where L^* is lightness coordinate, a^* suggests the green-redness coordinate, and b^* indicates the blue-yellowness coordinate. Since the color of milk powder is expressed in white, therefore, the powder whiteness can be calculated from $L^*a^*b^*$ system using the following equation (Equation 2), as reported by Ho et al.[32]:

$$\text{Whiteness} = 100 - \left[(100 - L^*)^2 + a^{*2} + b^{*2}\right]^{1/2} \quad (2)$$

2.5.3. Hygroscopicity

One gram of powder sample was placed over Petri dish in a glass desiccator of saturated NaCl solution (75.29% relative humidity) at 25 °C. The samples were weighed when attained equilibrium, and hygroscopicity was indicated based on gram of adsorbed water per 100 g of dry solids.[33]

2.5.4. Solubility

The solubility was measured by following the procedure of Cano-Chauca et al.[34] wherein 1% powder solution was centrifuged (REMI PR-24, REMI laboratory Instruments, Mumbai, India) for 5 min at 3000 g to obtain 25 mL supernatant in pre-weighed Petri dish. The supernatant was then dried for 5 h at 105 °C to calculate the solubility (%) by the difference in weight.

2.5.5. Total phenolic content (TPC)

TPC of produced milk powder was determined as per the method given by Singleton et al.[35] For sample extraction, 85% methanol (v/v) was used to suspend the powder (sample: solvent as 0.15:1, w/v). The mixture was then ultrasonicated for ½ h and centrifuged to collect the supernatant. The extraction process was then repeated using sample residues, and all supernatants were combined at the end to dry in rota-evaporator at 45 °C; 2 mg mL^{-1} of the prepared extract was added to Folin–Ciocalteau reagent followed by the addition of Na_2CO_3 (7.5%). The final volume was made to 25 mL with distilled water which was then allowed to stand for 2 h at room temperature in the dark before using a UV–Vis double-beam spectrophotometer (HACH DR6000, Loveland, CO, USA) to measure the absorption at 760 nm using methanol as the reference. Gallic acid standards were prepared in different concentrations varying from 0–100 mg L^{-1} to produce a standard calibration curve. TPC was calculated from this curve and presented as mg of gallic acid equivalents (mg GAE) per g of sample extract.

2.6. Analysis of developed powder

2.6.1. Scanning electron microscopy (SEM)

The developed powder was sputter-coated with platinum by depositing on SEM aluminum stubs. The morphological characteristics were observed using a scanning electron microscope (JSM-6510 LV SEM, JEOL Ltd., Tokyo, Japan) with an accelerated voltage of 10 kV and magnification of 1000X.

2.6.2. Particle size distribution (PSD)

0.5 g of powder was mixed in water and filled in a cuvette, and particle size was recorded in five successive trials using a laser light diffraction particle size analyzer (SALD-2300, Shimadzu Corporation, Kyoto Prefecture, Japan) with software SHIMADZU WingSALD II version 3.1.0. The mean, median, and modal of different particles (%) with varying sizes were then noted.

2.6.3. X-ray diffraction (XRD)

The crystalline or amorphous state of powder was determined using an X-ray diffractometer (D8 Advance Brucker, Billerica, MA, USA) with Cu Kα radiation at 40 kV and 40 mA. The samples were analyzed at angles ranging from 10° to 50° (2θ) with a step size of 0.02° and rate of 1 step/s. The diffractograms were collected using BruckerDiffrac.saxs v1.0.

2.6.4. Fourier transform-infrared (FT-IR) spectroscopy

The developed milk powder after mixing with potassium bromide (1:100 w/w) was compacted into the pellet form and analyzed using FT-IR spectrophotometer (Spectrum RX-FTIR, Perkin Elmer, Waltham, MA, USA) at 1 cm^{-1} resolution and 400 to 4000 cm^{-1} scanning frequency.[36]

2.7. Statistical analysis

The Design Expert (version 7.0.0, Stat-Ease, Minneapolis, MN, USA) was employed for analysis of experimental data. Multiple regressions using the least-squares method were used to fit the model to experimental data and represented by a second-order polynomial equation (Equation 3) as follows:

$$Y_k = \beta_{k0} + \sum_{i=1}^{n} \beta_{ki}x_i + \sum_{i=1}^{n} \beta_{kii}x_i^2 + \sum_{i=1}^{n-1}\sum_{j=i+1}^{n} \beta_{kij}x_ix_j \quad (3)$$

where Y_k = response variable (Y_1 = bulk density, Y_2 = color/whiteness, Y_3 = hygroscopicity, Y_4 = solubility, and Y_5 = TPC); x_i = coded process variables (x_1 = inlet air temperature, x_2 = feed rate, and x_3 = bee pollen) where β_{ko} is the value of fitted response at the design center point, i.e., point (0,0,0) and β_{ki}, β_{kii}, and β_{kij} were the linear, quadratic, and cross-product regression coefficients, respectively.

Table 2. ANOVA evaluation of linear, quadratic, and interactive terms for each response variable of spray drying process.

		Bulk density		Color/whiteness		Hygroscopicity		Solubility		Total phenolics content	
Source	df	Sum of squares	p value	Sum of squares	p value	Sum of squares	p value	Sum of squares	p value	Sum of squares	p value
Model	9	0.045	0.0002*	97.50	<0.0001**	41.25	<0.0001**	32.34	0.0001*	16.06	0.0001*
x_1	1	0.012	0.0001*	45.08	<0.0001**	20.29	<0.0001**	0.90	0.0364*	6.16	<0.0001**
x_2	1	2.113	0.0158*	–	–	4.00	0.0007*	3.85	0.0011*	–	–
x_3	1	0.026	<0.0001**	36.64	<0.0001**	8.90	<0.0001**	22.71	<0.0001**	6.73	<0.0001**
$x_1\ x_2$	1	–	–	–	–	–	–	–	–	–	–
$x_1\ x_3$	1	1.600	0.0283*	4.00	0.0114*	2.48	0.0029*	–	–	0.39	0.0455*
$x_2\ x_3$	1	–	–	–	–	–	–	–	–	–	–
x_1^2	1	–	–	–	–	3.16	0.0015*	–	–	0.76	0.0116*
x_2^2	1	–	–	7.68	0.0022*	1.60	0.0088*	0.84	0.0413*	–	–
x_3^2	1	–	–	–	–	–	–	3.64	0.0013*	1.01	0.0058*
Lack of fit		–	0.631^{ns}	–	0.818^{ns}	–	0.370^{ns}	–	0.912^{ns}	–	0.346^{ns}
R^2		0.968	–	0.975	–	0.979	–	0.971	–	0.971	–
C.V. (%)		3.36	–	0.71	–	1.71	–	0.38	–	2.35	–

x_1: Inlet air temperature; x_2: Feed flow rate; x_3: Bee pollen.
ns – Non-significant.
*Significant at $p < 0.05$.
**Significant at $p < 0.0001$.

The test of statistical significance was conducted on total error criteria at confidence level 95%. Analysis of variance (ANOVA) was used to validate the significant terms ($p < 0.05$) and models thus eliminating the non-significant terms (Table 2). Adequacy of the developed model was evaluated using R^2, Adj-R^2, Pred-R^2, Adeq. Precision, PRESS (predicted residual error sum of squares), lack of fit, and C.V. [$R^2 > 0.95$; Pre-$R^2 > 0.7$; (Adj-R^2 − Pred-R^2) < 0.2; Adeq. Precision > 4; max. PRESS; lack of fit > 0.1; C.V. < 10].

3. Results and discussion

The total recovery was determined for each trial which ranged from 65.38 ± 1.04 to 79.08 ± 1.25% wherein the sweep recovery was varying from 11.40 ± 0.33% to 15.62 ± 0.79%.

3.1. Bulk density

The determination of bulk density for a powder system is essential to regulate the processing, storage, packaging, and distribution conditions in the food industry. It includes the volume and pores, either closed or open, of powders. The bulk density reduces with the increased levels of fat and porosity due to more entrapped air inside the powder structure, whereas the smooth and uniform powder structure increases the bulk density values.[30]

The bulk density of developed powders was in the range of 0.31 to 0.52 g cm^{-3} (Table 1) which falls within the limit for food powders 0.300–0.800 g cm^{-3}.[31] The effect of independent variables on bulk density is shown in Figure 1a–c. The ANOVA analysis showed that the linear effects of temperature (x_1), feed flow rate (x_2), *and pollen* (x_3) as well as the interaction effect of inlet air temperature and bee pollen (x_1, x_3) were significant for bulk density (Table 2). It is shown that bulk density of the developed powder decreased with an increase in inlet air temperature (Figure 1a). This might be due to the quick evaporation of water from the surface of powder particles which would form a dried layer (crust) on the surface and water vapor is produced inside the particle due to high temperature. This resulted in the generation of high pressure within the particle which would cause the steam-leaving pores inside particle thus decreasing the bulk density. Tonon et al.[37] and Chegini and Ghobadian[38] also investigated the effect of inlet air temperature during spray drying and recorded the reduction in bulk density of açai (Euterpeoleraceae Mart.) and orange powder, respectively, with an increase in temperature due to higher tendency of particles to become hollow. Mishra et al.[39] also reported the decrease in bulk density of aonla juice powder due to the higher inlet air temperature. Likewise, Bansal et al.[27] and Suhag and Nanda[40] reported a decrease in bulk density of spray-dried milk and honey powders with the increased inlet air temperature and addition rate of the carrier agent due to reduced residual moisture content and higher total solids of feed.

The higher feed flow rate (x_2) exhibited the slight positive effect on bulk density which might be due to the poor exposure of feed droplet to the inlet air temperature. This would not remove the moisture adequately and produce the moist powder particles which caused higher density. The higher concentration of bee pollen may enhance the feed viscosity (up to 14.7 ± 0.08 cP from 12.2 ± 0.05 cP) thereby increasing the particle size and reducing the bulk density.

3.2. Color or whiteness

Color of milk powder is an important factor and usually determines the degree of drying and scorching the powder. Since the rapeseed bee pollen was yellow in color, therefore color evaluated in terms of whiteness is a significant factor in developing milk powder which is otherwise white or creamish white in color. In this study, whiteness of produced powder varied from 78.09 to 86.86 (Table 1) which decreased significantly on increasing the inlet air temperature (x_1) and bee pollen (x_3); however, the feed flow rate (x_2) caused slight but non-significant increase in whiteness (Figure 1d–f). The high *temperature might induce the Maillard reaction which is a result of the reaction of amino* group of amino acids and the carbonyl group of reducing sugars found in the feed droplets of bee pollen milk slurry.[41] Saha et al.[42] and Ruchita et al.[43] found the decrease in L^* value of groundnut (78.27–89.23) and soy (80.05 to 90.50) milk powder, respectively, on increasing the inlet air temperature. Likewise, bifidus milk powder was also dark in color when dried at higher temperature.[43] The interaction effect of higher temperature (x_1) and bee pollen (x_3) resulted in the reduced whiteness of developed powder due to higher degree of Maillard reaction. The increasing bee pollen amount in milk made the slurry more yellowish and also enhanced the amount of reducing sugars and amino acids in the slurry which may enhance the degree of non-enzymatic reactions during drying process. On the other hand, the particles are dried in lesser time at higher feed rate resulting in short exposure of particles to high temperature.[44] Therefore, the powder prepared under the higher feed rate was whiter in color, whereas the lower feed rate increases the exposure time leading to the dark color powder. Maskat et al.[45] reported the decrease in brightness of roselle powder at lower feed rates during the spray drying process.

3.3. Hygroscopicity

Hygroscopicity refers to the potential of a substance to absorb moisture when kept under the relative high humid surroundings. It is essential to the shelf-life studies during storage that determines the stability of product quality because of increase in the water content of product thus finally creating the favorable conditions for microbial growth and biochemical reaction.[46] It is highly dependent on the inherent product compositions and amount of drying aids. A powder is considered good if it exhibits the low hygroscopicity, moisture levels, degree of caking, and high solubility.[47]

The hygroscopicity of developed powders was in the range of 18.96% to 24.22% (Table 1), and the effect of process variables on powder hygroscopicity is shown in Figures 1g–i. The inlet air temperature (x_1), feed flow rate (x_2), and bee pollen (x_3) significantly affected the hygroscopicity of milk powder. The hygroscopicity was decreased with an increase in feed flow rate due to lesser time for evaporation of moisture and with the reduction in inlet temperature which could be explained by increasing moisture content with lower inlet air temperature. The similar trend of reduced hygroscopicity values with increased feed flow rate and decreased temperature was also observed by Tonon et al.[37] during the production of spray-dried açai powder. Further, bee pollen increased the hygroscopicity of end product due to the increase in lower-molecular weight sugars which turn the pollen-rich milk powder more hygroscopic. Further, the interaction of bee pollen and higher inlet air temperature significantly raised the powder hygroscopicity due to (i) increased moisture gradient between powder particles and atmosphere; and (ii) increased linkages between the hydrogen of water molecules and free aldehyde or ketone group of reducing sugars in amorphous regions. The developed milk powder exhibited slightly lower levels of hygroscopicity compared to nutritionally rich honey powder (20.11%–27.28%) prepared by Suhag and Nanda[40] but had higher hygroscopic nature than spray-dried beetroot juice powder (14.46–20.68) as reported by Bazaria et al.[48]. The hygroscopicity of spray-dried camel's and cow's milk powder varied from 18.8% to 21.26% which is almost similar to this study.[49] Therefore, the experimental results of this investigation are quite acceptable because hygroscopicity should be as low as possible.

3.4. Solubility

Solubility is important to determine the behavior of powder in aqueous solutions, particularly during reconstitution. The mechanism followed during the solubility process include the following: (1) wettability of powder, (2) disintegration of powders into primary particles, (3) material discharge from particles into the aqueous phase and consistent destruction of surface layer simultaneously until the complete particle breakdown, and (4) thorough dissolution of all materials.[50] The solubility of powder is associated with drying conditions as well as physical characteristics like total solids, viscosity of liquid feed.[51]

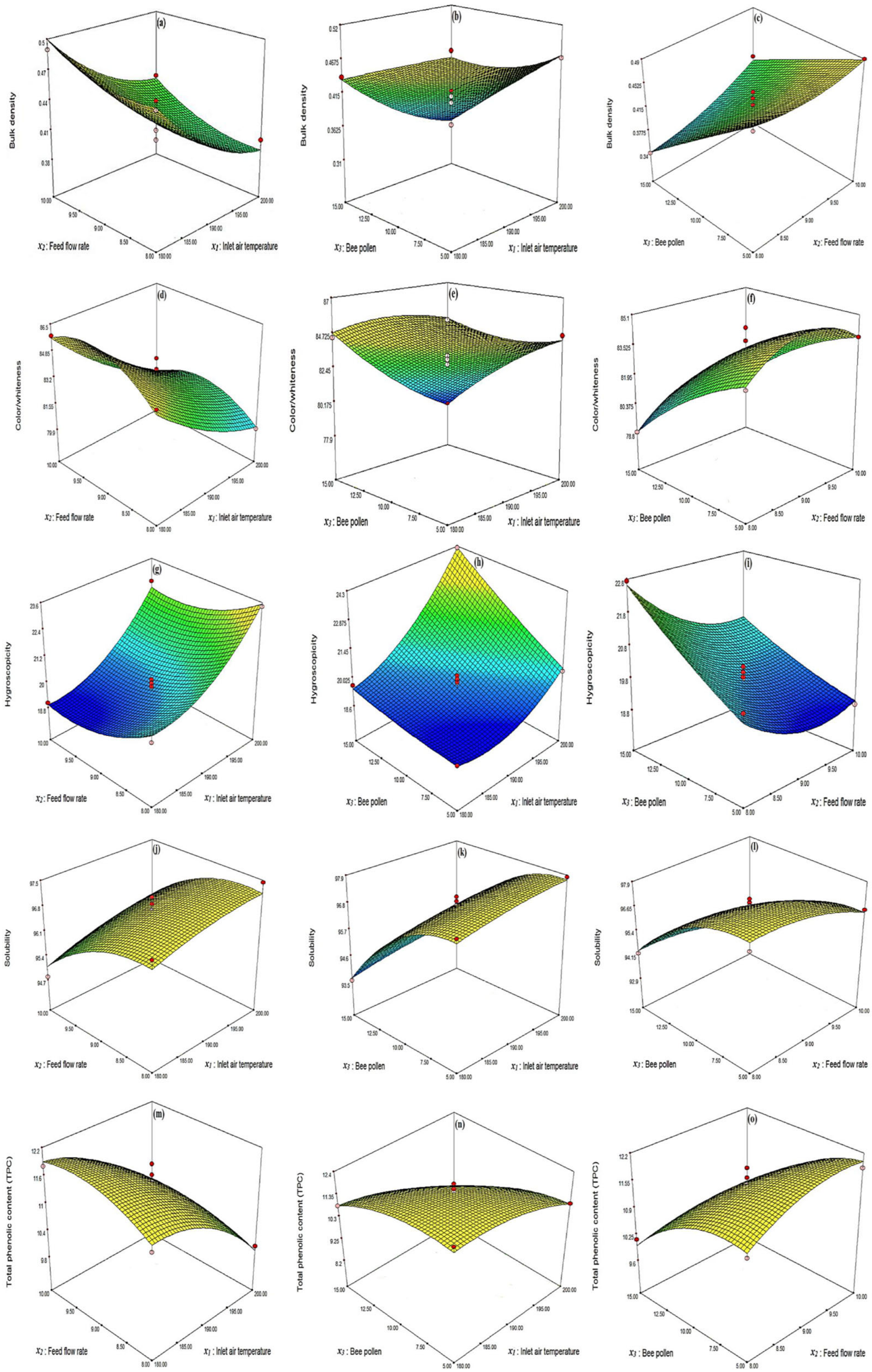


Figure 1. Effect of inlet air temperature, feed flow rate, and bee pollen on bulk density (a,b,c), color/whiteness (d,e,f), hygroscopicity (g,h,i), solubility (j,k,l), and total phenolics content (m,n,o) of developed bee pollen-enriched milk powder.

The solubility of bee pollen-enriched milk powder was in the range from 93.385 to 97.82% (Table 1). The inlet air temperature (x_1) had a little positive effect on solubility, whereas the feed flow rate (x_2) and bee pollen amount (x_3) had significant ($p < 0.05$) negative linear effect on solubility (Figure 1j–l).

Table 3. Experimental range, predicted values, and actual values of responses.

Responses	Experimental range	Predicted value	Actual value*
Bulk density (gcm^{-3})	0.31–0.52	0.45	0.43 ± 0.06
Color/whiteness	78.09–86.86	84.65	84.27 ± 0.15
Hygroscopicity (%)	18.96–24.22	19.19	20.05 ± 0.11
Solubility (%)	93.38–97.82	97.22	97.78 ± 0.23
Total phenolics content (mg $GAEg^{-1}$)	8.25–12.33	11.95	11.24 ± 0.10

*Values are the average ± standard deviation calculated on three replicates.

During drying, the temperature of particle when moisture is between 10% and 30% controls the solubility of powders. At higher temperatures, powders with low bulk density and moisture are usually produced which possess higher surface area. This would be beneficial in the rapid and quick solubilization of particles.[52] Singh et al.[53] and Patil et al.[54] reported the slight increase in solubility of *jamun* (*Syzygium cumini* L.) pulp and guava powder, respectively, with an increase in inlet air temperature. The solubility of developed powder is slightly greater compared to honey-based milk powder (96.64%).[27]

Further, the solubility of spray-dried bee pollen rich milk powder was comparatively less than commercially available milk powder due to the hydrophobic compounds of bee pollen which impair its solubility in water. Moreover, the unfolding of β-lactoglobulin followed by its combination with casein is another reason for powder insolubility. Further, the ultrasonication treatment disintegrates the pollen into minute pollen grains which were uniformly coated by milk powder, therefore resulting in more solubility than the bee pollen alone.[36]

3.5. Total phenolic content

The values of TPC in developed bee pollen-enriched milk powder varied from 8.25 to 12.33 mg GAE g^{-1} with an average of 10.95 mg GAE g^{-1}. The powder developed from 5% and 15% pollen had average TPC values of 11.59 and 9.75 mg GAE g^{-1}, respectively. On the other hand, the TPC of fresh rapeseed bee pollen was 12.84 mg GAE g^{-1} which had been decreased by 14.72% (on average) in developed powder. The TPC values decreased with the increasing temperature and pollen amount, but feed rate did not exhibit any significant effect (Figure 1m–o). The interaction effect of temperature and pollen was also significant ($p < 0.05$) using the regression model having R^2 value of 0.9719. The TPC values of bee pollen-enriched milk powder are higher than honey powder (0.61–0.63 mg GAE g^{-1}) and honey-based milk powder (2.39 mg GAE g^{-1}).[27,40]

The decrease of TPC in developed powder compared to fresh pollen can be explained based on following reasons. Firstly, the bee pollen was ultrasonicated before spray drying which resulted in the weakening of exine layer; thus, the membrane-bound compounds (polyphenols) are released into the extract. The polyphenolic compounds were then exposed to the higher temperature for a short time during which the loss in phenolic compounds occurred.[54,55] Secondly, the spray drying process coated the pollen particles with milk protein which might hinder the availability of polyphenolic compounds, thereby lowering the TPC value. When pollen was used in a higher amount, the nutraceutical components were encapsulated inadequately with the milk proteins offering a poor protection effect resulting in losses. Further, the dilution effect could be another factor for reduced TPC level because the concentration of polyphenolic compounds (present in bee pollen only) would decrease when pollen grains (5%–15%) were mixed with skim milk. However, the dry matter of milk will act as wall material resulting in maintaining the required functional properties in the final product such as better solubility, stability, convenience, controlled release, and increased shelf-life.[56] Huggett et al.[22] reported the efficient encapsulation of curcumin in milk powders showing better results than the whey protein isolate.

3.6. Optimization of process variables and model validation

The process of developing spray-dried bee pollen-enriched milk powder was successfully optimized using second-order polynomial models of response surface method using Design expert software. During optimization, the independent process variables (inlet air temperature, feed flow rate, and bee pollen) were kept within the rage and according to statistical analysis, the optimized conditions were obtained at 187.58 °C inlet air temperature, 8.94 mL min^{-1} feed flow rate, and 8.04% bee pollen with the desirability of 0.89. For model validation, two liters of pollen milk slurry was spray dried under the optimized process variables. The resulting powder having moisture content and water activity of 3.86% and 0.221, respectively, was examined for response values which

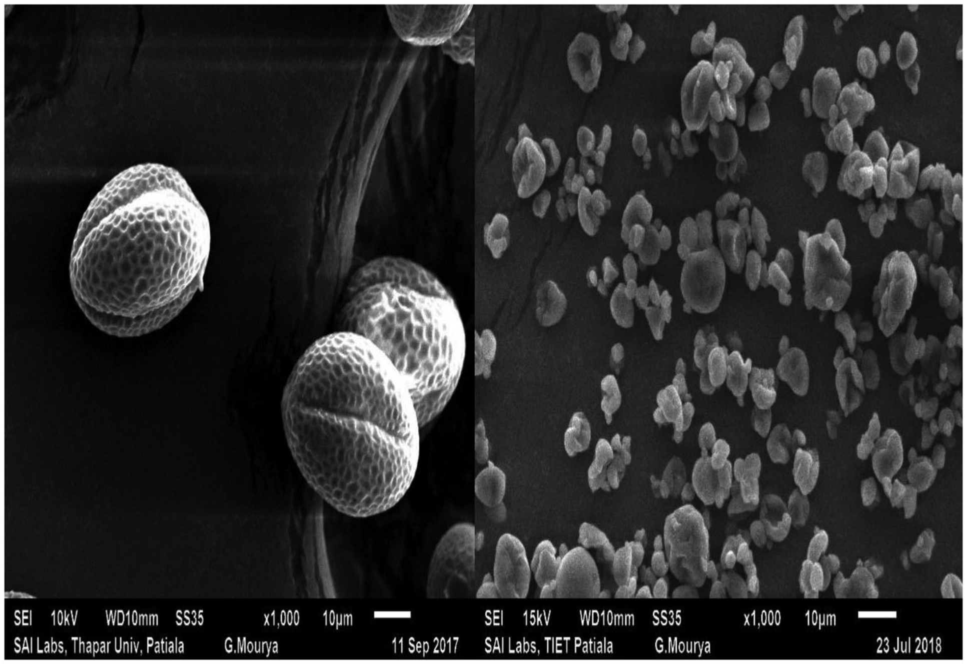


Figure 2. Scanning electron micrographs of (a) fresh rapeseed bee pollen and (b) optimized spray-dried milk powder encapsulating bee pollen.

exhibited sound closeness to the predicted values (Table 3) thus validating the model to optimize the development of bee pollen-enriched milk powder.

3.7. Analysis of optimized spray-dried bee pollen-enriched milk powder

3.7.1. Scanning electron microscopy

The SEM images of optimized milk powder showed the complete coating of rapeseed bee pollen by milk which differentiated the developed powder from its fresh counterpart (Figure 2a,b). The encapsulation of bee pollen might hinder the release of polyphenolic compounds resulting in the reduction of TPC in developed powder compared to fresh bee pollen. The powder particles exhibited the smooth, wrinkled, and folded surface having a nearly spherical shape. In this study, there were also smaller dried milk particles containing dents or vacuoles on the surface which are similar to the spray-dried cow milk and camel milk powder.[32,57,58] The moisture of particles, when exposed to higher temperature of spray drying, is evaporated very quickly and a dried skin is developed on the surface, resulting in vacuole due to the low gas permeability of the surrounding skin.[58] Fyfe et al.[59] also reported the large vacuoles and wrinkles on the surface of spray-dried skim milk powder due to lower inlet temperature which resulted in collapsed shriveled powder particles with higher bulk density compared to totally smooth spherical particles dried at higher temperatures having lower bulk density.

3.7.2. Particle size distribution

Particle size relates to the appearance, flow properties, and reconstitution characteristics. The monomodal distributions of particle size (Figure 3) were obtained for optimized bee pollen-enriched milk powder, indicating that the particles are not agglomerated and possessed almost similar size. This means that the presence of bee pollen did not affect the size of spray-dried particles. The powder particles had a median, mean, and modal diameter as 22.39, 19.62, and 24.13 μm, respectively, while 90% of particles had a diameter below 79.32 μm. According to Schuck and Ouest,[60] the spray-dried milk powders must have particle size in range 10–250 μm which is determined particularly by attributes of spray dryer nozzle. The particle size of optimized milk powder was higher to milk powders containing encapsulated curcumin (14.10–15.20 μm).[61] However, the particle size in the present investigation was higher than the fresh spray-dried skim camel milk powder (3.34 ± 0.08 μm)[32] which indicated the encapsulation of bee pollen grains in this study.

3.7.3. X-ray diffraction

A hump on the XRD curve (Figure 4) of optimized milk powder represented its amorphous structure, and spray drying process is mainly known for producing the amorphous powders.[62] During the process, the

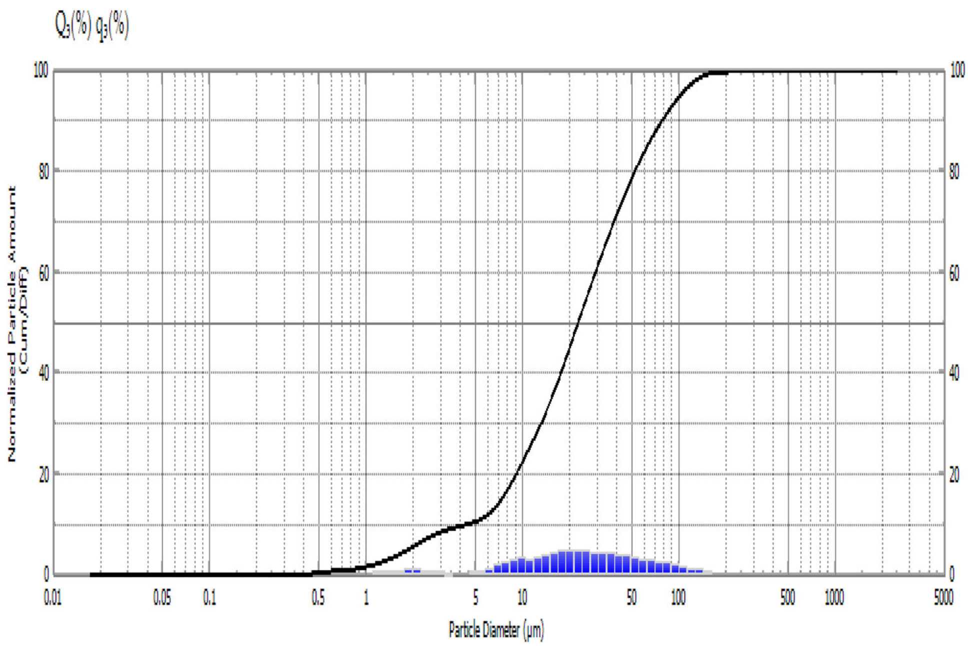


Figure 3. Particle size distribution curve of optimized spray-dried milk powder encapsulating bee pollen.

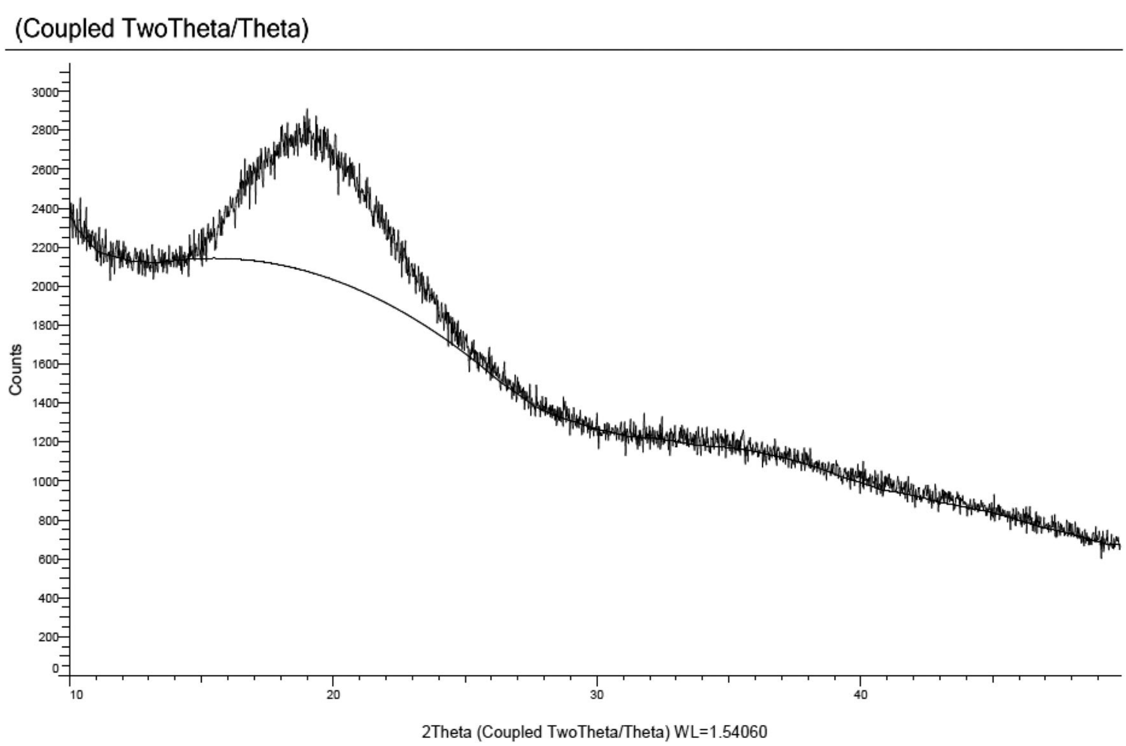
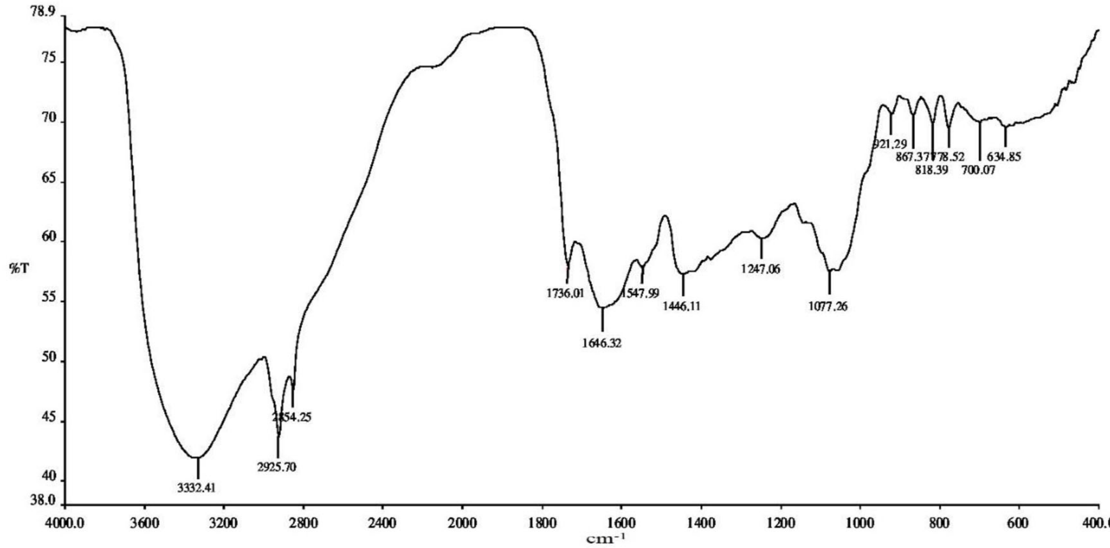


Figure 4. XRD pattern of optimized spray-dried milk powder encapsulating bee pollen.

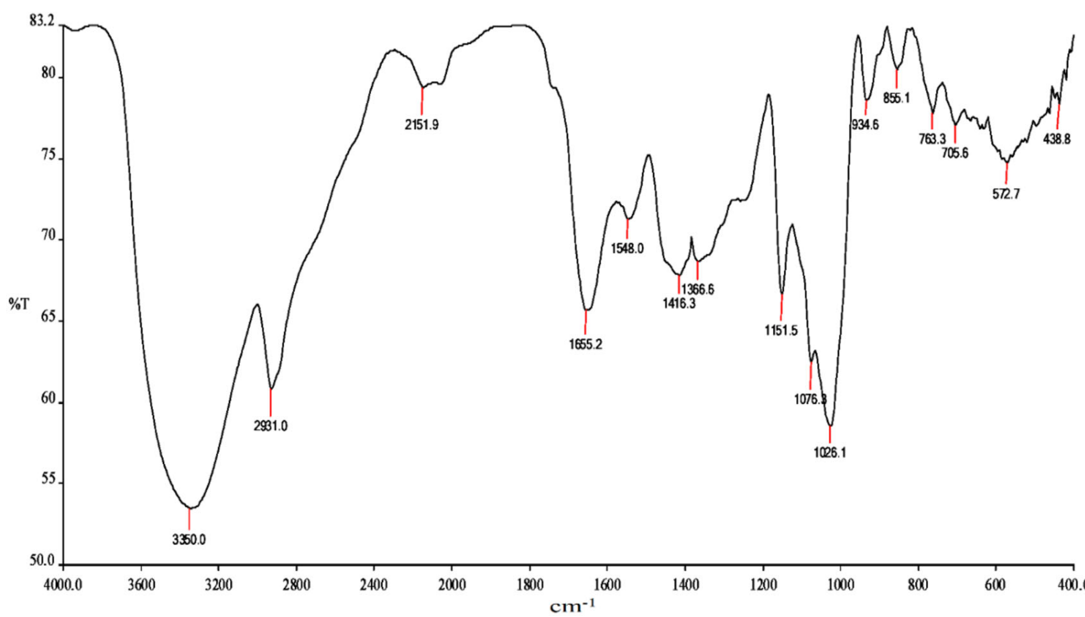
water is flashed off quickly providing no time to solutes for rearrangement in ordered crystalline structures. However, amorphous powders usually have more hygroscopicity.[63]

3.7.4. FT-IR spectroscopy

The FT-IR spectra of fresh rapeseed bee pollen and optimized spray-dried bee pollen milk powder are shown in Figure 5a,b. It is evident that peaks of fresh bee pollen and optimized powder were within a range of wavelengths but difference lied in their intensities. The developed milk powder exhibited the peaks with higher intensities due to the absorption and concentration of nutrients (Figure 5b). Based on the FT-IR spectral bands of whole cow milk powder,[64] it can be concluded that (i) absorption peak at 3350 cm^{-1}



(a)



(b)

Figure 5. FTIR spectra of (a) fresh rapeseed bee pollen and (b) optimized spray-dried milk powder encapsulating bee pollen.

was assigned to N–H stretching vibration of amide A (protein) and the symmetric and antisymmetric O–H stretching mode (H_2O), distinct to carbohydrate and polyphenols mainly; (ii) absorption peaks at 2931 and 2151 cm^{-1} were related to the C–H stretching of carboxylic acids (lipids) and C≡C or C≡N stretching of alkynes or nitriles; (iii) absorption peaks at 1655 and 1548 cm^{-1} were attributed to C = O stretching vibration of amide I (protein), and N–H and C–H bending vibration of amide II (protein), respectively. The peaks between 1700 and 1600 cm^{-1} spectral region were also assigned to the stretching band of carbonyl groups and C ▫ C stretching typically linked with phenolic compounds.[65] In the fingerprint region, the absorption peak at 1416 cm^{-1} was associated with the distinct asymmetric bending of methyl ($-CH_3$) group, while the peak at 1366 cm^{-1} was due to C–H vibrations, commonly found in every organic compound. On the other hand, the peaks at 1151, 1076, and 1026 cm^{-1} were assigned to the C–O, C–C, and C–O–C stretching (attributed by carbohydrates mainly).[66] According to Karimi et al.[67] and Boiani et al.,[68] the particular peaks at 1076 and 1026 cm^{-1} were strictly linked to the disaccharides (lactose) and phosphate groups ($-PO_3^{2}$) covalently bonded to casein. The absorption peak at 934 cm^{-1} was also attributed to carbohydrates, whereas the spectral region from 900 to 800 cm^{-1} showed the C–H, C–O–H, and CH_2 bending and peak at 705 cm^{-1} was assigned to C–O, C–C–O, and O–C–O bending.[63,69]

4. Conclusion

The development of encapsulated bee pollen milk powder was successfully accomplished which has a diverse utilization in different processed food products like breads, biscuits, cakes, ice-creams, yoghurts, smoothies, extruded products like pasta and other bakery, cereals, and confectionery products. However, there is a need to further study the reconstitution properties as well as shelf-life studies of developed functional milk powder for its commercialization.

Acknowledgments

The authors are highly thankful to Mr. Jagtar Singh – the progressive beekeeper and member of National Bee Board (New Delhi, India) for providing the bee pollen samples for the present investigation.

Disclosure statement

The authors declare no conflict of interest.

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