

Research Article

Functional and Oxidative Quality Characterization of Spray-Dried Omega-3-Enriched Milk Powder

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In the present study, fish oil (FO) and wall material were supplemented to milk to produce spray-dried powder (SDP). Furthermore, the mandate of the study was to enlighten the effect of spray-drying (SD) operating conditions on functional and oxidative quality of produced SDP samples. Purposefully, the cow milk was supplemented with 3% FO as omega-enriched source of eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) for development of milk and FO blends (MFOBs). The lecithin was used as an emulsifier and maltodextrin was supplemented as the wall material (WM) in the MFOBs. Initially, the FO, milk fat (MF), and MFOB samples were characterized for EPA, DHA, and peroxide value (PV) before the SD. The SD of MFOB samples was carried out to produce SDP samples by using a mini spray dryer. Central composite design (CCD) with face-centered rotation was used to optimize SD independent conditions such as inlet air temperature (IAT), pump speed (PS), maltodextrin percentage (MD), and needle speed (NS) in the ranges of 160–200°C, 3–9 mL/min, 10–30%, and 5–9 s, respectively. The encapsulation efficiency (EE) ranged between 89.30 and 81.57%. The EPA and DHA retentions were in the ranges of 2.19–1.87 g/100 g and 3.20–2.75 g/100 g, respectively. The highest results for responses were observed on the following conditions: IAT was 160°C, PS was 9 mL/min, MD was 30%, and NS was 9 s, respectively; the minimum values of response factors were obtained on the following conditions: IAT was 200°C, PS was 3 mL/min, MD was 10%, and NS was 5 s, respectively. The percent losses of EPA and DHA were noted in the range of 2–18%. The IAT was observed as main factor for FA reduction in SDP samples. The SDP samples were stable, and low rate of peroxide values was noted. Overall, spray drying can be potentially used to incorporate the essential fatty acids in milk to produce stable SDP for food applications.

1. Introduction

Omega-3 (ω -3) fatty acids (FAs) are a significant member of biologically active ingredients belonging to a group of polyunsaturated fatty acids (PUFAs). These have double bonds, where the first double bond is always located at the 3rd carbon atom from the methyl group [1]. FAs like ω -3 PUFAs, especially eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA), show numerous beneficial positive impacts in boosting the human health [2]. The available previous literature supports the applications of PUFAs to regulate the chronic diseases [3]. DHA is the key constituent

to impart significant contributions in development of fetal brain, neurons, sharpness of vision in infants, metabolisms of lipids, and cognitive support. DHA in combination with EPA provides the best results for preventing atherosclerosis, Alzheimer's, rheumatoid arthritis, dementia, and other diseases [4]. Recently published studies have emphasized the importance of utilization of very-long-chain (LC) ω -3 FAs as they play a key role in sharpening learning abilities at childhood and their behavior [5] and also decreased the burden of psychiatric sickness in young age adults [6]. The daily recommended consumption of EPA and DHA ranged from 250 mg to 1000 mg for normal adults and preferably

higher intake requirements are suggested for both pregnant and lactating females [7]. Generally, the daily consumption of ω -3 FAs in developing countries is very much low or below the recommended intake range [8, 9].

The sources of long-chain ω -3 FAs are very limited including marine-fisheries depending upon access and affordability [10]. Worldwide dietary intakes of ω -3 FAs are reported as only 20% of world's population obtained 250 mg/day of seafood's ω -3 PUFAs [11]. According to "World Health Organization" (WHO) and "Food and Agriculture Organization" (FAO), the fish consumption per serving supplies almost 200–500 mg of EPA and DHA [12]. Other factors that can affect the intake of EPA and DHA are availability, time, and composition of food [13]. So, time is needed to raise the awareness regarding beneficial impacts of EPA and DHA supply among the discerning consumers [4]. Extracted fish oil (FO) has been considered as one of the best sources for the EPA and DHA and can be supplemented into food products [14–16]. One known matrix/carrier medium for provision of ω -3 FAs in human biological system is milk. Milk fat (MF) is uniformly present in micelles which can effectively enhance the surface areas to carry out the bioactive components [17, 18]. Milk is regularly consumed as food in the diet of infants, children, teenager, pregnant women, adults, and the elderly [19]. Milk and milk-based dairy products are consumed on a daily basis and they can contribute as an excellent way of ω -3 FAs provision in the forms of functional milk, powders, and dairy products [20]. The consumption of EPA- and DHA-enriched food can ultimately reduce the burden of cardiovascular diseases (CVD) and mortality rate [21].

Omega FAs as EPA and DHA have very high susceptibility to autocatalytic lipid oxidation due to presence of free radicals specifically when molecular oxygen is active [22]. Protection of ω -3 PUFAs using spray drying (SD) technique has showed significant results to reduce the oxidative deterioration of these FAs and this technique allows food handlers and technologists to apply these FAs in several food systems [1]. SD is a common practice being applied in the food processing industries and more specifically in the dairy industry to produce stable milk powder. SD also helps to minimize water content to lowest level and reduces oxidation and this process and mechanism give numerous advantages and options for product development [23]. Milk is highly perishable food and application of SD extends its shelf life. Milk powders are mostly used in the food industry because of their nutritional, physical, and functional properties [24]. Noteworthy, SD has economic importance at industrial scale to develop spray dried whey powder, instant coffee, and soups [25–27]. In this regard, projective techniques have been seen as a fast way to assess information about new products and are mainly used for answering the questions regarding new products [28]. Moreover, SD has an advantage to produce products with quality and safety [29]. However, the concept of producing the spray dried powder (SDP) enriched with EPA and DHA is an alternative route for supplementing these important essential FAs in human diet [19, 30]. SDP may have better storage ability compared to conventional milk fortified with omega FAs. The SD

technique may help to maintain the availability of these bioactive omega FAs throughout the year for all age groups and also supports the economic loss by reducing different processing steps from packaging to delivery [24, 31–34]. The main mandate of the present study was to develop the SDP using optimized SD operating conditions and evaluate the quality of SDP for encapsulation efficiency, omega FAs loss, and oxidative stability.

2. Materials and Methods

2.1. Procurement of the Raw Material. The substrate material (milk) was collected from commercial farm. The collected milk samples (MS) were kept at 4°C. Meanwhile, the fish meal (*Catla catla*) was purchased from local fish market. Lecithin and maltodextrin (MD) were purchased from chemical supplier (Punjab, Pakistan) which were utilized as emulsifier and wall material (WM) for SD process, respectively. Analytical grade reagents and standards were purchased from Merck (Merck KGaA, Darmstadt, Germany) and Sigma-Aldrich (Tokyo, Japan).

2.2. Milk Fat (MF) Extraction. MF was drawn by following the method detailed by Feng et al. [35] with some modifications. 20 mL MS was placed within a 50 mL conical plastic tube and the tube was placed in machine and centrifugation was done at 12,000 rpm for 30 min at 4°C. An aliquot of 1.0 g weight from the formed fat-cake layer was taken to 1.5 mL micro tube and further it was left at 20–25°C for almost 30 min till the melting point followed by centrifugation by micro centrifugal machine, where rpm was set at 13000 and time was 20 min. Separation of MS into 3 different layers was completed where top most was lipid, middle one was mix of protein/fat/other water insoluble solids, and last layer was water.

2.3. Fatty Acids (FAs) Analysis of MF. The extracted lipid was 40 mg and it was trans-esterified following the procedure of transmethylation [36]. First, 40 mg of sample (oil) was taken and 2 mL of hexane was then added. Further, 40 mL of methyl-acetate was added, and this mixture was vortexed. After this, the methylation process was done by adding 40 mL mixture prepared as 1.75 mL methanol + 0.4 mL of 5.4 mol/L sodium methylate. Again, the mixture was vortexed and left for 10 min to complete the reaction. Then, there was addition of termination reagent 60 mL volume prepared as 1 g oxalic acid and 30 mL diethyl ether. The centrifugation was performed at 2400 rpm for 5 min. The temperature was 5°C. The process produced the prominent layer of hexane. This aliquot of hexane layer was taken and stored at –20°C. Fatty acid methyl esters were quantified using a Gas Chromatograph fitted with a capillary column (30 m L × 0.25 mm ID × 0.25 μm film thickness). Conditions were set as injector temperature of 230°C, column-oven temperature of 180°C to 210°C, detector temperature of 250°C, detector used FID (Flame Ionization Detector), carrier gas used oxygen free nitrogen, and flow rate was 1 mL/min. The volume of fatty acid oil was 0.1–0.2 μL.

2.4. Peroxide Value of MF. Extracted MF was subjected to analysis of peroxide value (PV) following the method of Fang et al. [37]. In a 250 mL flask, 0.2 g of extracted oil was weighed and addition of 30 mL acetic acid (3:2 solutions) and 0.5 mL potassium iodide solution was done. After stirring for about 1 minute, 30 mL distilled water was added to the mixture. At that point, an aliquot of 0.5 mL starch indicator (1%) was also added to the mixture, and the resultant solution was titrated against 0.001 N sodium thiosulfate solution until diminishing the purple color. The calculation of peroxide value was performed as follows: $PV = (S-B) \times N \times 1000/W$, where S is the volume of $Na_2S_2O_3$ added to the sample; B is the volume of $Na_2S_2O_3$ of the blank; N is normality of the solution of $Na_2S_2O_3$, while W is the weight of the sample (g) and PV was expressed as active O_2 mEq (milliequivalent) per kg of sample.

2.5. Fish Oil (FO) Extraction. The oil extraction from fish meal was done in departmental innovative lab and extracted FO was counted for use as a source of EPA and DHA for supplementation in milk. The unprocessed fish meal samples were weighed by electronic scales (model Kern 440–35N) for each treatment and solvent extraction procedure was adopted for extraction of oil from fish meal [38]. The fish meal and solvent (hexane) ratio was set as 1:10 ($w:v$). For each treatment, the 100 g of fish meal was weighed in continuous shaker and 1000 mL of solvent was mixed to extract the lipids. The process was carried out at 30°C for 2 hours. After that, the solvent was removed at 50°C in a lab-scale rotary evaporator.

2.6. Fish Oil Purification. The purification of extracted FO was carried out using a series of processes that included different steps such as degumming, neutralization, bleaching, and deodorization [39]. For degumming, the sample of extracted fish oil (100 g) was placed in 500 mL beaker. The sample was heated at 70°C for exactly 1 min in the oven. Then, the aqueous citric acid solution of known amount (3 mL of 3% concentration) was added to the beaker containing heated fish oil. The mixture was gently shaken at 70°C for 1 min. The fish oil was then cooled to 25°C and centrifugation process was carried out at 2500 × g for 10 min to remove the undesirable impurities. The degummed fish oil was neutralized using the sodium hydroxide. For each treatment, the sodium hydroxide (12.6 g of 9.5% NaOH solution) was added to 100 g of degummed fish oil. The mixture was gently heated at 65°C for consecutive 30 min with constant stirring using a magnetic stirrer bar. The neutralized fish oil samples were then cooled to 25°C and were kept undisturbed for 6 hours. After that, the centrifugation process at 2500 × g was carried out for 10 min. This process supported to decant the oil from the precipitated soap. Demineralized water (50 mL) was mixed to each centrifuged sample to wash out any soap residues. This process of demineralized water addition and washing was repeated three times. At the end of neutralization process, the impurities and water contents were removed in the form of separating layer by the application of centrifugation at

2500 × g for 10 min. The bleaching of each neutralized oil sample (100 mL) was performed with 1 g of acid activated earth clay. The operating conditions were set at 100°C for exactly 20 min with constant stirring using a magnetic stirrer bar. The acid activated earth clay and absorbed impurities were removed from bleached fish oil samples using filtration process. This process was completed immediately to avoid chemicals and color of fish oil samples. Finally, the bleached fish oil samples were deodorized with the purpose of removing the free fatty acids, bad odors, and oxidation products.

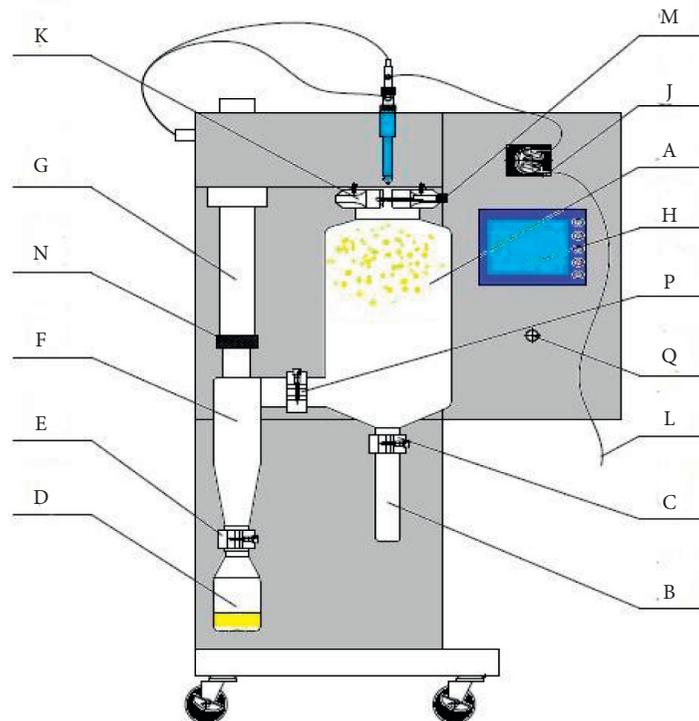
2.7. FO Analysis. FAs analysis and PV of FO was done according to the detailed procedures described in previous sections of FAs and PV analysis of MF.

2.8. Milk and Fish Oil Blend (MFOB) Formation. The FO was added to milk with known volumes for development of milk and FO blends (MFOBs). Each MFOB contained 1000 mL milk and 30 mL FO. Further, the WM was added at specific known concentrations [40, 41]. The MD as WM and lecithin (3%) as the emulsifier were dissolved in milk under continuous magnetic stirring and were kept for cooling at room temperature. The milk was used as the base material for all formulations to make the emulsions. In order to utilize milk proteins as an encapsulating material, the MFOBs were homogenized with a homogenizer at 25000 rpm for 5 min. After that, the samples were allowed to stabilize at room temperature for 60 min.

2.9. MFOBs Analysis. MFOB samples were characterized for FAs and PV according to the detailed methods described in previous sections of FAs and PV analysis of MF.

2.10. Spray Drying (SD). The SD of MFOB and WM was carried out to produce SDP by using mini spray dryer (TPS-15 Lab Spray Dryer, China). The schematic diagram of the lab-scale spray dryer is presented in Figure 1. The evaluation index was based on the performance of ME and the conservation of PUFAs [42]. SD independent variables such as the inlet air temperature (IAT, °C), pump speed (PS, mL/min), maltodextrin percentage (MD, %), and needle speed (NS, s) were optimized using the central composite design (CCD) with face centered rotation as described in Table 1. The SDP samples were collected in collection chamber connected to cyclone separator by locking nut and were directed to collection bottle attached with spray dried chamber. The SDP samples were further analyzed for functional and oxidative quality parameters.

2.11. Encapsulation Efficiency (EE), Fatty Acids, and Peroxide Value (PV). Encapsulation efficiency (EE) was determined according to the method detailed by Santhanam et al. [41]. The surface oil content of SDP samples was determined by adding 50 mL n-hexane and 5 g of powder in a volumetric flask and was stirred for 10 min. The powder and solvent



M	Locking nut	K	Large hoop
J	Peristaltic pump	G	Hot air exhausting pipe
A	Drying chamber	N	Locking nut
H	Touchscreen	F	Cyclone separator
P	3 rd hoop	E	1 st hoop
Q	Power button	D	Collection chamber
L	Feeding tube	B	Collection bottle
C	2 nd hoop		

FIGURE 1: The schematic diagram of the lab-scale spray dryer (TPS-15).

TABLE 1: Coded and actual levels of independent variables for optimization of response factors as determined by central composite design (CCD).

Independent variables	Units	Coded levels		
		-1	0	+1
Inlet air temperature (IAT)	°C	160	180	200
Pump speed (PS)	mL/min	3	6	9
Maltodextrin (MD)	%	10	20	30
Needle speed (NS)	s	5	7	9

were separated using filter paper (Whatman No. 1). The SDP residues left on filter paper were further washed with hexane (solvent) of known volume of 20 mL. The solvent was evaporated and dried off using a rotary evaporator. The total oil of SDP was extracted by Soxhlet extraction technique with hexane. Accurately weighed 5 g powder was extracted

by placing in thimble using 180 mL volume of hexane for total time of 8 h to make full extraction of oil. The encapsulation efficiency was assessed using the following formula:

$$EE\% = \left(\frac{TO - SO}{TO} \right) \times 100, \quad (1)$$

where TO is the total oil extracted and SO is the surface oil collected.

Fatty acids and PV of oil samples extracted from SDP were observed as documented in above sections.

2.12. Statistical Analysis. CCD design with face centered rotation was used which was elaborated by quadratic equation for each response parameter. Each treatment of SDP was statistically analyzed for its significant values using software package (MATLAB) as described by Montgomery

[43]. Optimized run was performed in triplicate and the average mean values were reported with standard deviation. Moreover, the sample analysis was done, and the significant variation was determined among means at a probability level of 5%.

3. Results and Discussion

3.1. Characterization of Samples before Spray Drying (SD).

The milk contains high water content and other major ingredients like fat, protein, lactose, and minerals. Although many types of dietary lipids are present, fat present in milk is very much complicated as it is comprised of more than one hundred different types of FAs and numerous triglycerides. Huge varieties of triglycerides and FAs structure enable the possibility to categorize MF into different fractions on the grounds of their melting abilities. Functional characteristics of MF can be increased by converting it into different fractions [44]. The composition of the milk by itself plays a major role in the drying rate and the functional properties of the final product. FAs analysis of MS has shown very low EPA $0.044 \pm 0.01\%$ and DHA $0.006 \pm 0.01\%$ content among the PUFAs group of total FAs present in MF (Table 2). The findings of the present study are supported by the research work of Stergiadis et al. [45] which described that the cow milk has very low percentages of the EPA and DHA as 0.048% and 0.007%, respectively, among PUFAs groups. Mainly, milk FAs have been focused on from research point of view as milk FAs respond quickly and they are sensitive in nature. Moreover, the farming practices also produce their results on FAs composition of milk. Recently, the chemical characteristics and changes in milk FAs during storage are central areas of dairy development and research. Different aspects that affect the variation in milk FAs are energy status, animal breed, lactation time, and time of season and the most important is udder health [46]. Also, the PV of drawn MF is mentioned in Table 2, which indicated that the PV of MF was 0.5 ± 0.02 meqO₂/kg of MF. The PV of MF is in agreement with the results reported by Ajmal et al. [47]. The PV in the FO was found to be 1.75 ± 0.13 meqO₂/kg and this reported value is very near to the value of the FO which was 0.92 meqO₂/kg [48]. FAs of extracted FO were EPA C_{20:5} 7.19 ± 0.51 and DHA C_{22:6} 8.21 ± 0.69 as % of PUFA_s group presented in Table 2. The FO contained DHA 8.30% of PUFAs and EPA content as 7.23% of PUFAs, respectively [49].

3.2. *SD of MFOB.* The MFOB was spray dried to produce SDP by using mini spray dryer (TPS-15 Lab Spray Dryer, China). The treatment plan of operating drying conditions was based upon central composite design (Table 1). In this research work, response surface methodology (RSM) was applied to surface the optimal SD conditions for MFOB samples. The impacts of four independent variables of spray dryer, that is, IAT (160°C, 180°C, and 200°C), PS (3 mL/min, 6 mL/min, and 9 mL/min), MD (10%, 20%, and 30%) and NS (5 s, 7 s, and 9 s), were extensively studied for SD effect on MFOB, their omega FAs composition, and primary

TABLE 2: Characterization of milk fat and fish oil samples.

Sample	EPA (% of PUFAs)	DHA (% of PUFAs)	PV (meqO ₂ /kg)
MF	0.044 ± 0.01	0.006 ± 0.01	0.5 ± 0.02
FO	7.19 ± 0.51	8.21 ± 0.69	1.75 ± 0.13

Mf = milk fat; FO = fish oil; EPA = eicosapentaenoic acid; DHA = docosahexaenoic acid; PUFAs = polyunsaturated fatty acids; PV = peroxide value. The MFOB samples possessed the EPA 2.24 ± 0.16 (g/100 g extracted fat) and DHA 3.33 ± 0.18 (g/100 g extracted fat), while PV was 1.44 ± 0.11 meqO₂/kg.

oxidative stability. The effects of these four factors and their three respective levels were expressed through polynomial regression equations. In the dairy industry, lipid oxidation has always been a major problem. The bioactive substance such as MF can be packed by SD technique within a WM. Emulsion should be stable as it is one of the key properties for SDP making with lower levels of free fat over surface of powder particles. Tan [50] used the sodium caseinate (NaCas) as WM for development of oil-in-water (O/W) emulsions. The SD operating factors such as IAT, drying air (DA) flow rate, atomization pressure, total solid level of the material, viscosity, and nozzles type are key important to observe and understand. On the other side, composition of the milk also contributes as critical factor to the drying process and the functional characteristics of end product. A whole milk is generally described as a colloidal system, where components like fats, proteins, salts, and lactose make a complex system with water present in milk. The MF is a type of emulsion like oil-in-water and is organized in a globular structure, while the proteins exhibit as colloidal components and make micellar forms [51, 52]. After the completion of SD process, the milk is generally collected as a powder in a collection chamber. Powder yield in the cyclone will be higher with increase in IAT and DA flow speed. This process can be explained as when DA flow rate increases, it speeds up the movement of moisture from the droplet into the DA, which obviously results in increased thermal energy and heat transfer coefficients. Interestingly, powder recovery has been observed to decrease with increase in concentration of milk solid particles at constant DA flow rate. Increased viscosity of feed produced difficulty to flow of raw liquid in feeding tube and resultantly atomization of the liquid through the narrow orifice becomes tough. Moreover, the sticky final product is produced when operating conditions are not properly controlled or optimized [53].

3.3. *Optimization of SD Conditions.* Most of the times in RSM models, the nature of the relationship among the independent factors/variables and output responses is not known. However, the very first thing in RSM is to determine an appropriate relationship among dependent and independent variables [54]. RSM was employed to optimize independent variables on the EE and retention of EPA and DHA. The experimental design based on the CCD was used to study the effects of independent factors on defined responses. A typical SD procedure is shown in Figure 1. A total of 30 runs were conducted in triplicate. The average mean of

performed triplicate runs of optimized response was reported as the measured value with the standard deviation. Table 3 shows the conducted runs with the optimized responses.

3.4. Fitting the Proposed Model. Predicted values for response variables were determined through regression model. These values were analyzed to compare values of responses for verification and validity of second-order polynomial responses. The predicted values of response variables were observed in the same range of reported experimental values. The statistical results of quadratic model for response variables have been shown in Table 4. Several indicators were noted such as coefficient of determination (R^2) which ranged between 0.5798 and 0.9022 for response variables. The R^2 value which is near 1 shows that the model is reproducible. Adequate precision ratio tells the level of adequacy of the model and a ratio more than 4 is considered best. The ratio for response variables ranged between 10.937 and 14.972. This value was considered to navigate the design space. The model regression equations comprising both of coded and actual levels using response surface methodology (CCD) have been documented in Table 5.

3.5. Encapsulation Efficiency (EE) of SDP. The EE ranged between 89.30 and 81.57%. The highest results for response EE (89.30%) were observed at spray dryer run no. 26, where independent variables (IAT was 160°C, PS was 9 mL/min, MD was 30%, and NS was 9 s) were set to study response. The spray dryer run no. 9 (IAT was 200°C, PS was 3 mL/min, MD was 10%, and NS was 5 s) produced lowest response values of EE as 81.57%. It has been documented that the liquid conversion to powder by SD ensures quality aspects and safety concerns of final powder. However, the uncontrolled processing conditions at extreme levels impart negative affect on SDP, alter the product value, and are responsible for the losses of valuable nutritional components [29]. Thermal dehydration has been reported as the best and cost-effective method that is normally used for food storage [55]. SD is still the one of the best drying methods that have significant potential for the food processing industries [56]. The factors including IAT, PS, WM, and NS were observed to develop the optimized drying conditions. The relationship among these variables for EE is shown in Figure 2. During the SD method, the core material is housed within the WM. The formation of SDP and the properties of WM are critical aspects for the EE and stability properties of SDP [57]. The EE depends significantly on temperature condition. The evaporation was significant at IAT (160°C), which resulted in EE up to 89.30%. It shows that, at that temperature, the drying rate of particles was optimized, and particles were uniformly sprayed in cyclone, and further SDP was collected in collection bottle. Also, significant effect on EE was noted on the increase of wall material concentration and at low temperature. It was observed that higher temperature affected the outer layer formation in powder particles. Aghbashlo et al. [58] documented EE of FO as 81.94% using WM separated from milk and EE was significantly influenced by

temperature of DA in cyclone chamber. Moreover, it was noted that the SD process at optimized conditions did not negatively affect the efficiency of oil encapsulation. The drying phenomena of material at optimized conditions helped in the quick formation of surface/crust of SDP. Furthermore, the rate of crust formation was high, which gave protection against oil leaching from SDP. Similarly, Wu [59] described in detail that the temperature conditions significantly affect wall material. At the high IAT, undesired evaporation occurs and causes the cracks formation on the outermost layer of SDP, which leads towards release of core matter and its deterioration. The same trend was observed in the present study as when IAT was 200°C, EE was lower when compared with EE at other IAT treatments (Table 3). EE also depends on the composition of the samples [60]. Also, wall material at 30% concentration limited the oil diffusion on the particle's surface, which is also considered a contributing factor towards the optimum EE. Aghbashlo et al. [58] used the combination of wall materials for FO encapsulation and observed that the viscosity increased during blend formation and produced the powder with best EE. Other operating independent variables such as NS and PS are also contributing factors. It is noteworthy to mention that the low NS and high PS operating conditions lead to increased liquid flow through atomizer and caused bigger particle size formation. Proper homogenization of WM and oil contents during blend formation also affect the PS and SD process. It has also been noted that the decrease in quantity of oil matter during homogenization of blend leads to speedy crust formation. Furthermore, the semipermeable encapsulate droplets were formed, which was maybe due to WM layer during the SD technique. The quick formation of protecting layer reduced the oil leaching from SDP and ultimately enhanced the EE [61]. PS at 9 mL/min and NS of 9 s in combination with IAT at 160°C relatively were best to achieve quick evaporation and to avoid cracks on wall material and ultimately produced best EE results. Similar type of optimized relationship between NS and PS for best spray drying pattern was discussed by Amaro et al. [62] to achieve highest EE of the resultant samples.

3.6. Eicosapentaenoic Acid and Docosahexaenoic Acid Retention and Losses. Spray drying operating conditions should be optimized to ensure maximum retention of nutritional components in milk and technofunctional aspects of the SDP. The present work was based on studying the effects of independent parameters on the SDP production and selected nutritional composition as response variables of SDP. However, retaining of nutritional ingredients is a key factor that should be considered during processing and preservation of food. When SD technique is applied on milk, the powder recovery and FAs retention are affected based on changing levels in temperature and atomization pressure [53]. The effects of SD parameters related to percent loss of EPA and DHA have been explained in Figures 3 and 4, respectively. The EPA and DHA retentions were in the ranges of 2.19–1.87 g/100 g and 3.20–2.75 g/100 g, respectively, being the highest to lowest among all 30 runs. The highest results for responses are shown at spray dryer run no. 26 where independent variables (IAT was 160°C, PS was 9 mL/

TABLE 3: Effect of spray drying conditions on the response parameters.

Spray drying run	Independent variable combination					Response parameters				
	IAT (°C)	PS (mL/min)	MD (%)	NS (s)	EE (%)	EPA retained (g/100 g extracted fat)	EPA loss (%)	DHA retained (g/100 g extracted fat)	DHA loss (%)	PV (meqO ₂ /kg)
1	200(+1)	3(-1)	10(-1)	9(+1)	81.99 ± 1.78	1.88 ± 0.14	16.07 ± 0.23	2.78 ± 0.15	16.70 ± 0.21	2.10 ± 0.10
2	180(0)	6(0)	20(0)	5(-1)	84.93 ± 1.67	2.03 ± 0.15	9.37 ± 0.28	2.99 ± 0.12	10.38 ± 0.23	1.98 ± 0.13
3	200(+1)	3(-1)	30(+1)	9(+1)	82.33 ± 1.45	1.92 ± 0.15	14.28 ± 0.25	2.81 ± 0.13	15.82 ± 0.19	2.09 ± 0.14
4	160(-1)	9(+1)	10(-1)	5(-1)	84.82 ± 1.54	2.03 ± 0.16	9.37 ± 0.22	2.97 ± 0.15	10.97 ± 0.25	1.93 ± 0.11
5	180(0)	3(-1)	20(0)	7(0)	85.91 ± 1.96	2.07 ± 0.15	7.58 ± 0.24	3.03 ± 0.19	9.20 ± 0.26	1.95 ± 0.12
6. (C1)	180(0)	6(0)	20(0)	7(0)	85.77 ± 1.76	2.09 ± 0.16	6.69 ± 0.26	3.07 ± 0.13	8.02 ± 0.23	1.94 ± 0.10
7	200(+1)	3(-1)	30(+1)	5(-1)	83.37 ± 1.40	1.97 ± 0.14	12.05 ± 0.27	2.89 ± 0.19	13.32 ± 0.25	2.07 ± 0.12
8. (C2)	180(0)	6(0)	20(0)	7(0)	85.93 ± 1.87	2.07 ± 0.14	7.58 ± 0.25	3.03 ± 0.18	9.20 ± 0.28	1.96 ± 0.14
9	200(+1)	3(-1)	10(-1)	5(-1)	81.57 ± 1.78	1.87 ± 0.15	16.51 ± 0.28	2.75 ± 0.15	17.44 ± 0.28	2.20 ± 0.17
10	160(-1)	3(-1)	30(+1)	5(-1)	88.21 ± 1.92	2.17 ± 0.17	3.12 ± 0.19	3.18 ± 0.14	4.79 ± 0.20	1.91 ± 0.12
11	200(+1)	9(+1)	30(+1)	9(+1)	82.04 ± 1.96	1.89 ± 0.13	15.62 ± 0.20	2.80 ± 0.17	15.97 ± 0.23	2.08 ± 0.13
12	180(0)	6(0)	30(+1)	7(0)	88.31 ± 1.56	2.17 ± 0.17	3.12 ± 0.24	3.18 ± 0.12	4.50 ± 0.26	1.99 ± 0.13
13	160(-1)	3(-1)	10(-1)	5(-1)	85.34 ± 1.89	2.04 ± 0.16	8.92 ± 0.27	2.99 ± 0.15	10.35 ± 0.24	1.92 ± 0.15
14	160(-1)	9(+1)	10(-1)	9(+1)	86.79 ± 1.87	2.09 ± 0.17	6.69 ± 0.20	3.09 ± 0.16	7.43 ± 0.25	1.89 ± 0.15
15	160(-1)	9(+1)	30(+1)	5(-1)	86.81 ± 1.84	2.10 ± 0.15	6.25 ± 0.28	3.09 ± 0.17	7.29 ± 0.24	1.90 ± 0.15
16	180(0)	9(+1)	20(0)	7(0)	85.33 ± 1.39	2.09 ± 0.15	6.69 ± 0.22	3.07 ± 0.15	8.02 ± 0.26	1.95 ± 0.11
17. (C3)	180(0)	6(0)	20(0)	7(0)	85.45 ± 1.84	2.06 ± 0.14	8.03 ± 0.24	3.01 ± 0.19	9.64 ± 0.28	1.96 ± 0.11
18. (C4)	180(0)	6(0)	20(0)	7(0)	83.40 ± 1.30	1.99 ± 0.14	11.16 ± 0.20	2.92 ± 0.14	12.44 ± 0.26	1.94 ± 0.14
19. (C5)	180(0)	6(0)	20(0)	7(0)	85.28 ± 1.45	2.08 ± 0.18	7.14 ± 0.27	3.06 ± 0.10	8.17 ± 0.24	1.95 ± 0.10
20	200(+1)	9(+1)	30(+1)	5(-1)	82.76 ± 1.67	1.93 ± 0.16	13.83 ± 0.24	2.85 ± 0.17	14.50 ± 0.27	2.10 ± 0.10
21	160(-1)	6(0)	20(0)	7(0)	87.34 ± 1.47	2.13 ± 0.15	4.91 ± 0.26	3.13 ± 0.15	6.26 ± 0.27	1.90 ± 0.13
22	160(-1)	3(-1)	30(+1)	9(+1)	87.94 ± 1.87	2.14 ± 0.15	4.46 ± 0.26	3.13 ± 0.15	6.11 ± 0.25	1.91 ± 0.11
23	200(+1)	9(+1)	10(-1)	5(-1)	82.21 ± 1.98	1.93 ± 0.15	13.80 ± 0.25	2.82 ± 0.11	15.38 ± 0.24	2.10 ± 0.16
24	160(-1)	3(-1)	10(-1)	9(+1)	84.57 ± 1.76	2.02 ± 0.14	9.82 ± 0.24	2.98 ± 0.12	10.52 ± 0.26	1.89 ± 0.14
25	180(0)	6(0)	20(0)	9(+1)	87.94 ± 1.56	2.14 ± 0.15	4.46 ± 0.27	3.13 ± 0.12	6.11 ± 0.26	1.90 ± 0.14
26	160(-1)	9(+1)	30(+1)	9(+1)	89.30 ± 1.54	2.19 ± 0.11	2.23 ± 0.26	3.20 ± 0.12	3.90 ± 0.23	1.88 ± 0.16
27	200(+1)	9(+1)	10(-1)	9(+1)	83.40 ± 1.43	1.99 ± 0.12	11.16 ± 0.23	2.92 ± 0.14	12.44 ± 0.20	2.09 ± 0.15
28. (C6)	180(0)	6(0)	20(0)	7(0)	85.30 ± 1.93	2.07 ± 0.11	7.58 ± 0.23	3.02 ± 0.18	9.50 ± 0.25	1.95 ± 0.13
29	200(+1)	6(0)	20(0)	7(0)	82.14 ± 1.45	1.92 ± 0.14	14.28 ± 0.21	2.82 ± 0.19	15.53 ± 0.26	2.09 ± 0.12
30	180(0)	6(0)	10(-1)	7(0)	84.10 ± 1.65	2.01 ± 0.14	10.26 ± 0.20	1.98 ± 0.14	10.57 ± 0.25	1.97 ± 0.10

C1–C6 = central points of spray drying runs; IAT = inlet air temperature; PS = pump speed; MD = maltodextrin; NS = needle speed; EE = encapsulation efficiency; EPA = eicosapentaenoic acid; DHA = docosahexaenoic acid; PV = peroxide value.

min, MD was 30%, and NS was 9 s) were set to study responses. The maximum results related to EPA and DHA retention were 2.19 g/100 g and 3.20 g/100 g, respectively. The spray dryer run no. 9 (IAT was 200°C, PS was 3 mL/min, MD was 10%, and NS was 5 s) produced the lowest values of EPA and DHA retained as 1.87 g/100 g and 2.75 g/100 g, respectively. The overall percent losses of EPA and DHA were examined in the range of 2–18%. The IAT was observed as main factor for FAs losses. These findings are further supported by De Oliveira et al.'s [29] study in which they reported the impact of SD conditions on milk FAs and noted that the low drying period and IAT were nonsignificant for thermal degradation. Furthermore, in another study, Lavanya et al. [63] documented the effects of SD conditions on EPA (13.64%) and DHA (32.46%) content in FO, which were decreased to 12.48% (EPA) and 30.58% (DHA) after the SD, respectively.

3.7. Oxidative Stability of SDP. The key focus of SDP development was making the protective layer around the core material (FO). This WM layer may protect the core material

from external undesirable conditions and lipid oxidation. Previous studies reported that the SD of FO enhanced its stability. Among the SD variables, the heat is considered as key factor that can enhance chances of lipids oxidation [64]. Further, the PV is determined to estimate the primary oxidation components [65]. The PVs of all SD runs are detailed in Table 3. Lowest PV was noticed at run no. 26 which was 1.88 meqO₂/kg and highest PV of 2.20 meqO₂/kg was observed at run no. 9. The mutual interaction impact of SD conditions on PV of SDP samples has been presented in Figure 5. Santhanam et al. [41] produced dried powder from fish oil and milk blend using sodium caseinate as WM by SD process. The PVs of produced SDP samples were within the range of 3–10 meqO₂/kg at room temperature during the storage interval of 0–32 days. Aghbashlo et al. [58] noted the PV of FO as 5 meqO₂/kg which was further increased to 7 meqO₂/kg after SD. One of the known reasons for this increase in PV was the heating factor. Serfert et al. [66] reported their findings related to SD procedure and its effects on lipids. They discussed that the PV of lipids increased with rise in IAT and produced more peroxides which

TABLE 4: Analysis of variance (ANOVA) of the predicted second-order polynomial model for spray drying conditions' impact on response parameters.

Source of variation	df	Response parameters								
		EE (%)		EPA retained (g/100 g extracted fat)		DHA retained (g/100 g extracted fat)		PV (meqO ₂ /kg)		
		Mean square	p value	Mean square	p value	Mean square	p value	Mean square	p value	
Model	14	8.74**	0.0001	0.0155**	<0.0001	0.0609 ^{NS}	0.2306	0.0148**	<0.0001	
Linear effects	A-IAT	1	85.85**	<0.0001	0.1440**	<0.0001	0.2990*	0.0167	0.1780**	<0.0001
	B-PS	1	0.2763 ^{NS}	0.6183	0.0014 ^{NS}	0.3566	0.0041 ^{NS}	0.7582	0.0008 ^{NS}	0.2253
	C-MD	1	14.72**	0.0021	0.0214**	0.0022	0.1901*	0.0485	0.0014 ^{NS}	0.1124
	D-NS	1	2.19 ^{NS}	0.1724	0.0020 ^{NS}	0.2765	0.0053 ^{NS}	0.7239	0.0044**	0.0099
Interaction effects	AB	1	0.0163 ^{NS}	0.9034	0.0002 ^{NS}	0.7105	0.0005 ^{NS}	0.9132	0.0002 ^{NS}	0.5126
	AC	1	5.53*	0.0379	0.0090*	0.0301	0.0150 ^{NS}	0.5552	0.0009 ^{NS}	0.1997
	AD	1	0.7966 ^{NS}	0.4012	0.0009 ^{NS}	0.4610	0.0018 ^{NS}	0.8370	0.0000 ^{NS}	0.8261
	BC	1	1.37 ^{NS}	0.2742	0.0064 ^{NS}	0.0619	0.0086 ^{NS}	0.6552	0.0004 ^{NS}	0.3853
	BD	1	2.71 ^{NS}	0.1316	0.0042 ^{NS}	0.1220	0.0095 ^{NS}	0.6380	0.0000 ^{NS}	0.8261
	CD	1	0.3452 ^{NS}	0.5780	0.0012 ^{NS}	0.3913	0.0060 ^{NS}	0.7080	0.0016 ^{NS}	0.0939
Quadratic effects	A ²	1	4.03 ^{NS}	0.0711	0.0094*	0.0272	0.0073 ^{NS}	0.6804	0.0045**	0.0088
	B ²	1	0.3485 ^{NS}	0.5762	0.0001 ^{NS}	0.8337	0.0424 ^{NS}	0.3263	0.0000 ^{NS}	0.8233
	C ²	1	0.1234 ^{NS}	0.7385	0.0001 ^{NS}	0.8501	0.3031*	0.0161	0.0019 ^{NS}	0.0725
	D ²	1	0.5206 ^{NS}	0.4956	1.794E-07 ^{NS}	0.9916	0.0493 ^{NS}	0.2912	0.0004 ^{NS}	0.3586
Residual	15	1.07	—	0.0016	—	0.0412	—	0.0005	—	
Lack of fit	10	1.18 ^{NS}	0.3677	0.0017 ^{NS}	0.3923	0.0604**	0.0018	0.0007*	0.0132	
Pure error	5	0.8352	—	0.0013	—	0.0029	—	0.0001	—	
Cor. total	29	—	—	—	—	—	—	—	—	

** Significant at 0.05 level; IAT=inlet air temperature; PS=pump speed; MD=maltodextrin; NS=needle speed; EE=encapsulation efficiency; EPA=eicosapentaenoic acid; DHA=docosahexaenoic acid; PV=peroxide value.

TABLE 5: Coded and actual regression equations for response parameters after spray drying process.

Response parameter	Regression form	Regression equation
EE	Coded	$R1 = + 85.59 - 2.18A + 0.1239B + 0.9044C + 0.3489D - 0.0319AB - 0.5881AC - 0.2231AD - 0.2931BC + 0.4119BD - 0.1469CD - 1.25A^2 - 0.3668B^2 + 0.2182C^2 + 0.4482D^2$
	Actual	$R1 = - 11.63130 + 1.11393IAT + 0.051123PS + 0.642490MD - 0.655353NS - 0.000080IAT*PS - 0.002941IAT*MD - 0.005578IAT*NS - 0.001466PS*MD + 0.010297PS*NS - 0.007344MD*NS - 0.003117IAT^2 - 0.000917PS^2 + 0.002182MD^2 + 0.112061NS^2$
EPA retention	Coded	$R2 = + 2.07 - 0.0894A + 0.0089B + 0.0344C + 0.0106D + 0.0038AB - 0.0238AC - 0.0075AD - 0.0200BC + 0.0163BD - 0.0087CD - 0.0603A^2 - 0.0053B^2 + 0.0047C^2 - 0.0003D^2$
	Actual	$R2 = - 2.75637 + 0.053077IAT - 0.001034PS + 0.029987MD + 0.032449NS + 9.37500E-06IAT*PS - 0.000119IAT*MD - 0.000188IAT*NS - 0.000100PS*MD + 0.000406PS*NS - 0.000438MD*NS - 0.000151IAT^2 - 0.000013PS^2 + 0.000047MD^2 - 0.000066NS^2$
DHA retention	Coded	$R3 = + 2.97 - 0.1289A + 0.0150B + 0.1028C + 0.0172D + 0.0056AB - 0.0306AC - 0.0106AD - 0.0231BC + 0.0244BD - 0.0194CD + 0.0530A^2 + 0.1280B^2 - 0.3420C^2 + 0.1380D^2$
	Actual	$R3 = + 8.11788 - 0.049769IAT - 0.029331PS + 0.186054MD - 0.431515NS + 0.000014IAT*PS - 0.000153IAT*MD - 0.000266IAT*NS - 0.000116PS*MD + 0.000609PS*NS - 0.000969MD*NS + 0.000132IAT^2 + 0.000320PS^2 - 0.003420MD^2 + 0.034496NS^2$
PV	Coded	$R4 = + 1.95 + 0.0994A - 0.0067B - 0.0089C - 0.0156D - 0.0037AB - 0.0075AC - 0.0012AD + 0.0050BC + 0.0013BD + 0.0100CD + 0.0418A^2 - 0.0032B^2 + 0.0268C^2 - 0.0132D^2$
	Actual	$R4 = + 4.32177 - 0.031342IAT + 0.001267PS - 0.009376MD + 0.032650NS - 9.37500E-06IAT*PS - 0.000037IAT*MD - 0.000031IAT*NS + 0.000025PS*MD + 0.000031PS*NS + 0.000500MD*NS + 0.000105IAT^2 - 7.89474E-06PS^2 + 0.000268MD^2 - 0.003289NS^2$

A: IAT=inlet air temperature; B: PS=pump speed; C: MD=maltodextrin; D: NS=needle speed. EE=encapsulation efficiency; EPA=eicosapentaenoic acid; DHA=docosahexaenoic acid; PV=peroxide value.

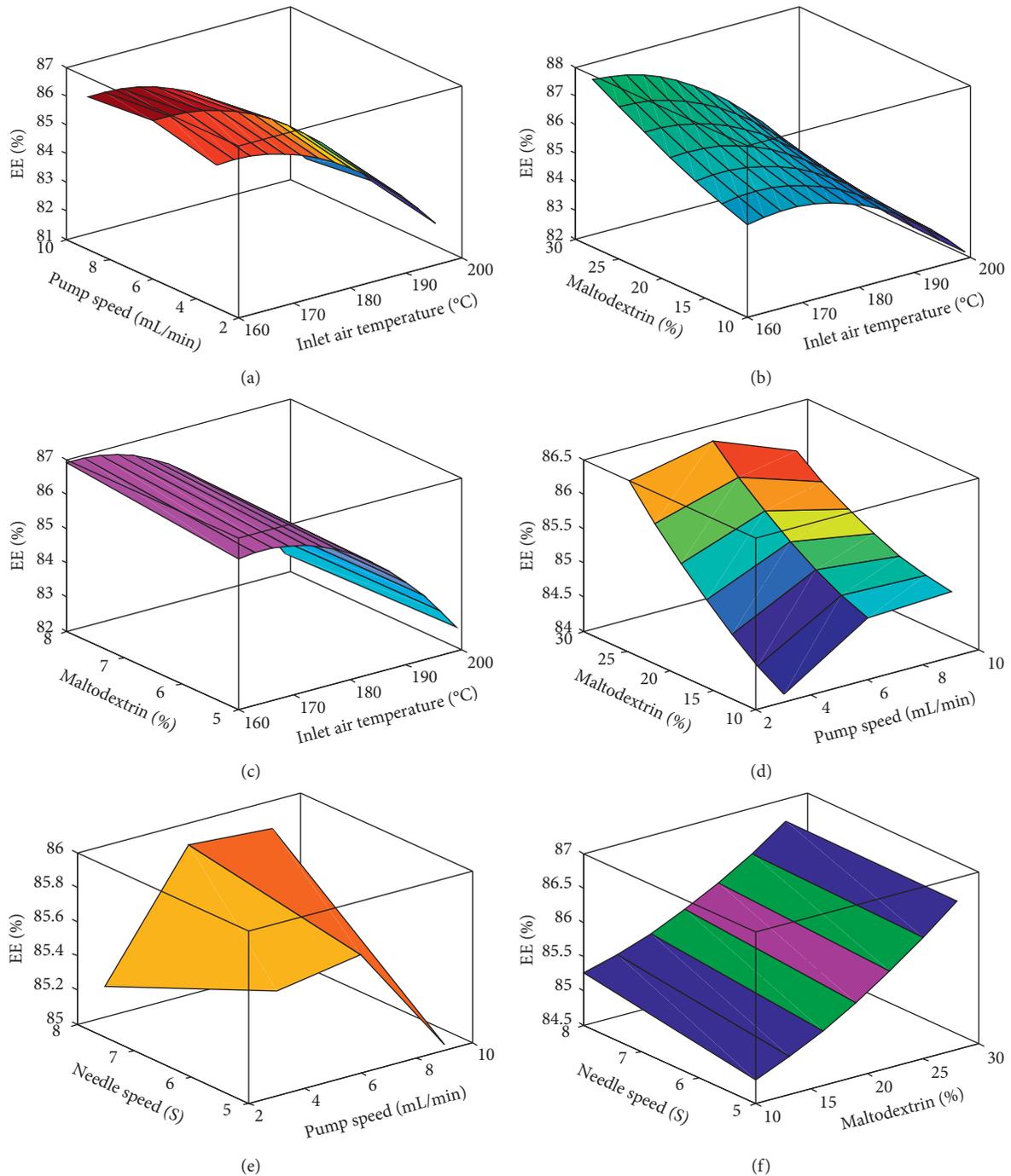


FIGURE 2: The interaction impact of spray drying parameters on encapsulation efficiency.

increases the speed of oxidation process. The findings of the present study related to PV and temperature relationship are much closer to Lavanya et al.'s [63] study in which they spray-dried the FO at IAT in the range of 100–160°C and PVs of SDP samples were within the range of 1.9 to 3.8 meqO₂/kg. The temperature was significantly responsible for these changes. In another work, PV of pure FO was 1.05 meqO₂/kg before SD and after SD process, and it was observed as 2.10–4.06 meqO₂/kg. Higher IAT range in SD process was considered responsible for increase of oxidation level [67]. Li

et al. [68] also observed the formation of aldehydes and ketones in milk powder at production day and during storage period. These short chain volatile compounds gradually increased at high operating IAT conditions and ultimately cause off-flavors and off-odors. The PV of powdered milk produced by SD ranged from 0.143 to 0.367 meqO₂/kg [68]. Santhanam et al. [41] observed the variation in the PV of dried SO preparation, which was maybe due to the oxidative reactions and uncontrolled factors like oxygen, light, and heat. The formation of pores

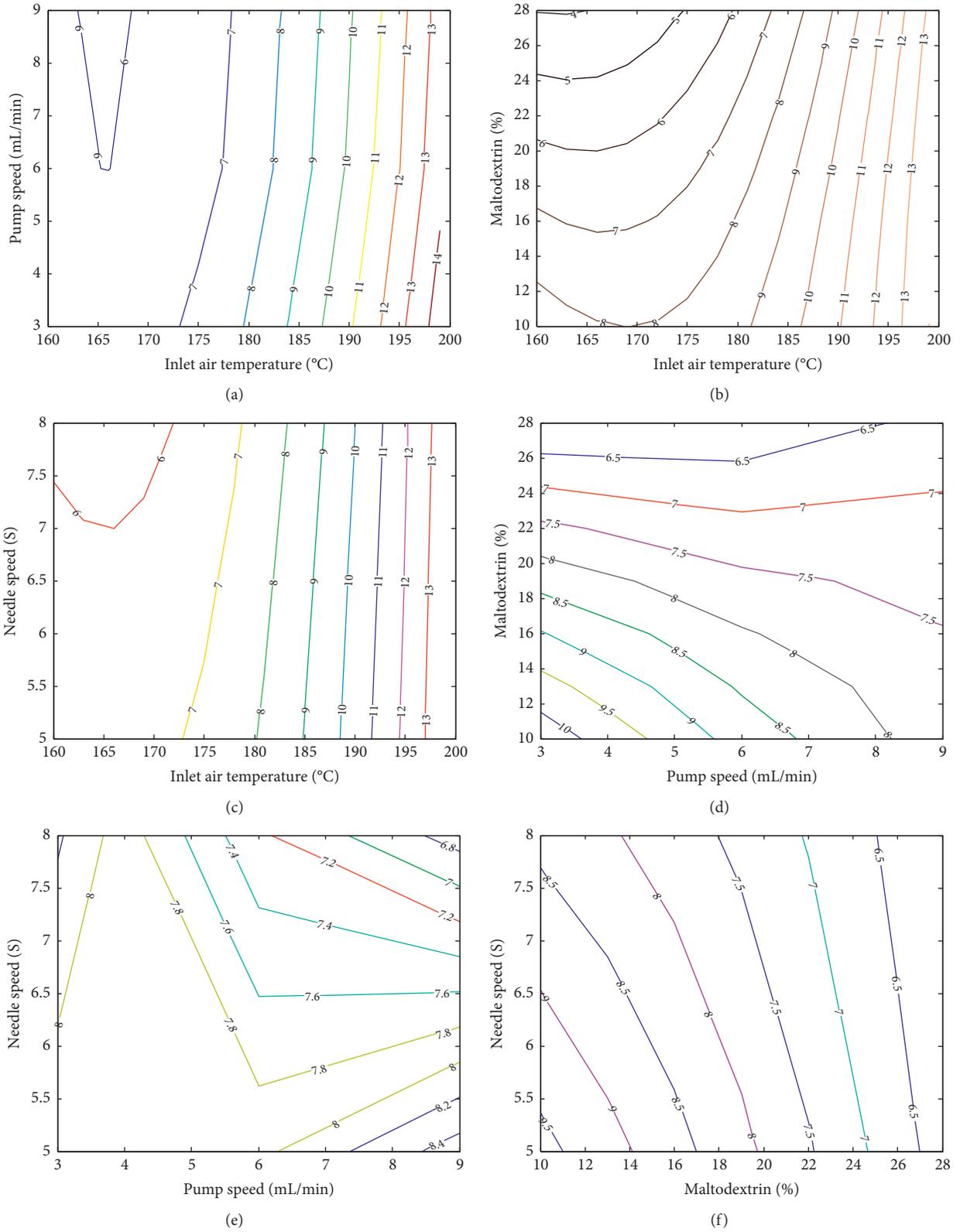


FIGURE 3: The mutual interaction impact of spray drying conditions on percent EPA loss.

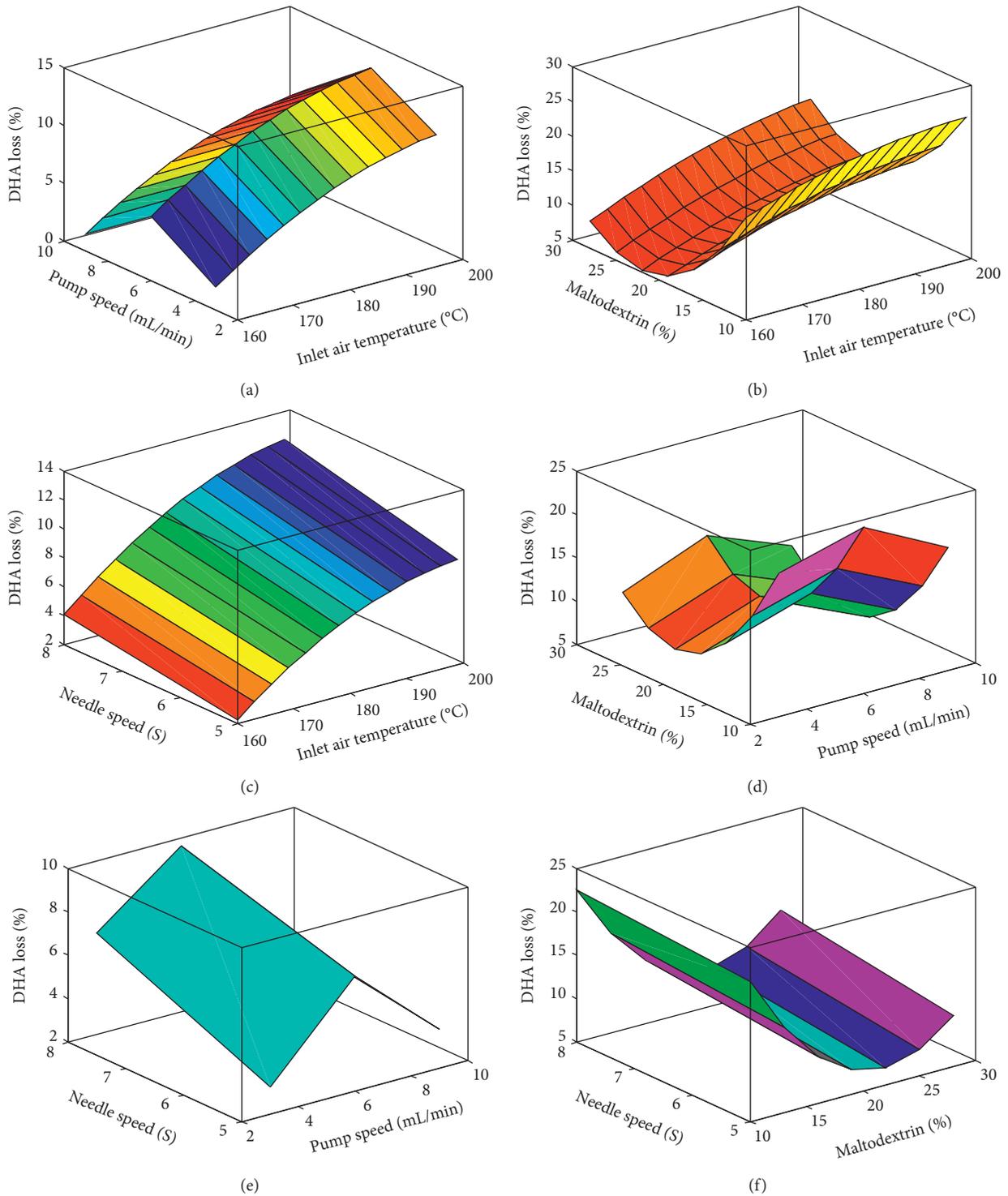
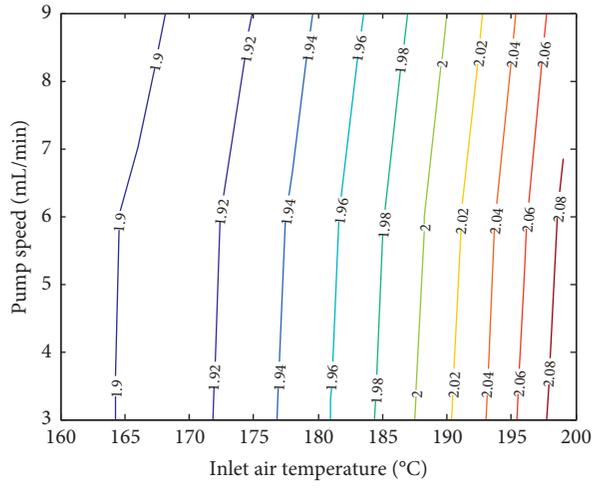


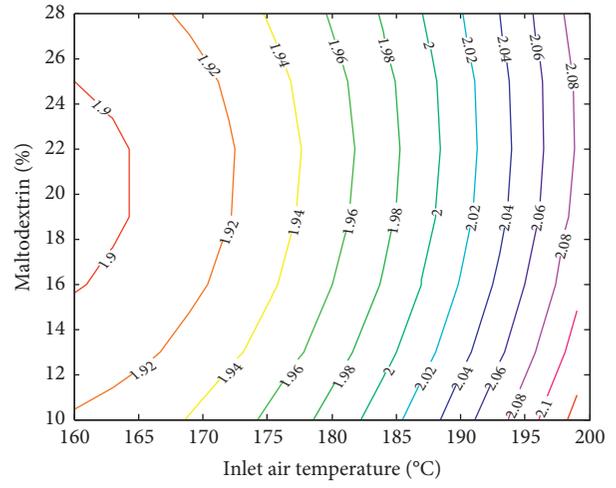
FIGURE 4: The interaction impact of spray drying independent parameters on percent DHA loss.

on surface of SDP supported the oxygen movement and enhanced the oxidation as noted from elevated PVs. Autooxidation of SDP occurs due to changes in independent variable factors [69]. The PS and IAT relationship was noted to be a significant factor to increase or decrease the PV of the SDP samples. It is noteworthy to mention that the higher temperature profile during SD process causes crust cracks,

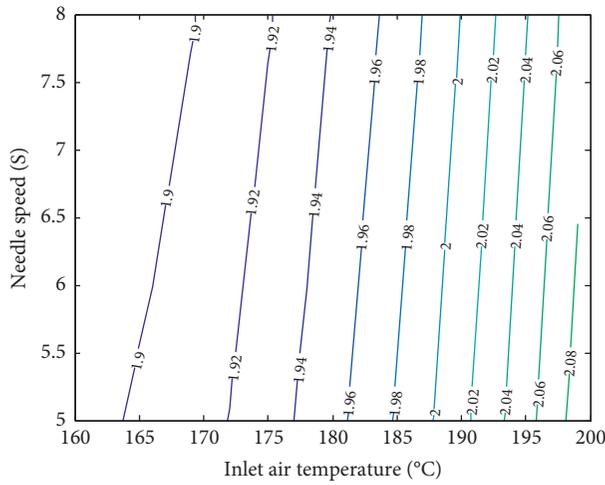
while reduced PS leads to overcooking. Meanwhile, lower NS produced particles with bigger size which is maybe due to more available time for crust formation [70]. PS should be adjusted to avoid too much liquid spray during process. The high rate of PS can cause agglomeration, which may lower powder functional properties [71]. The quick crust formation of SDP leads to the high EE rate and this would



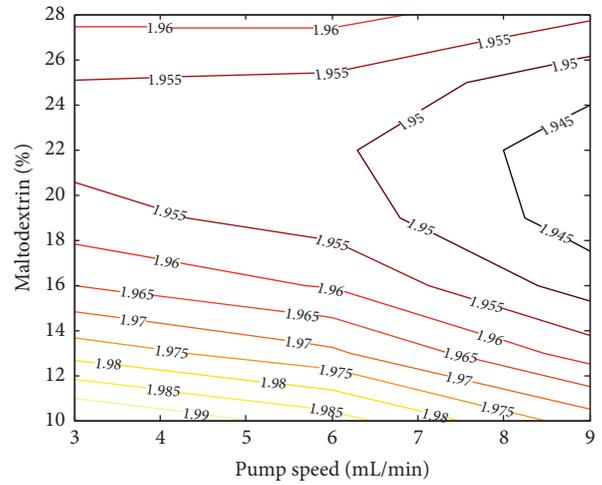
(a)



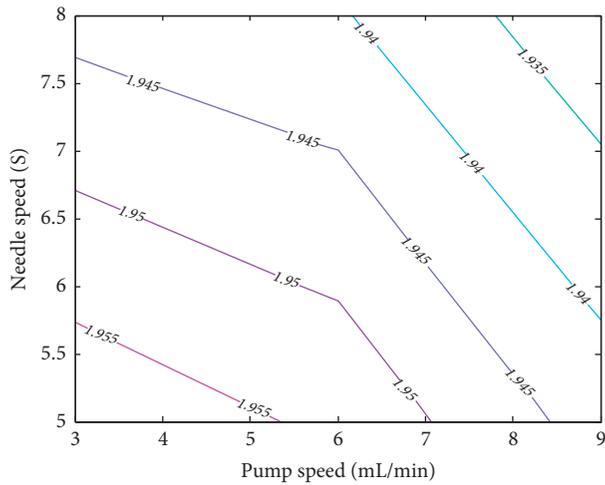
(b)



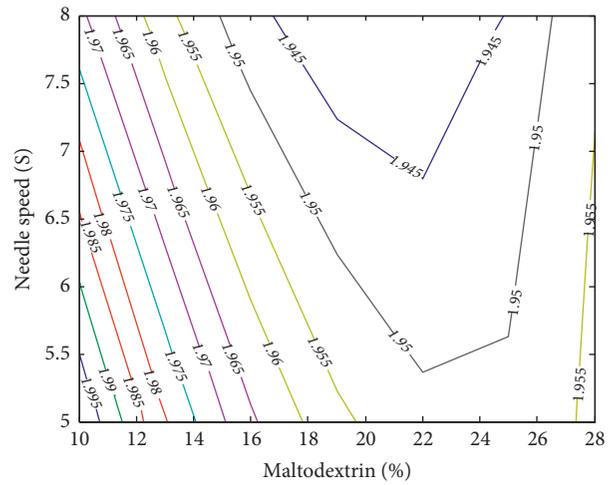
(c)



(d)



(e)



(f)

FIGURE 5: The mutual interaction impact of spray drying conditions on peroxide value of spray dried powder samples.

definitely protect the core from oxidation [58]. Tonon et al. [72] also reported that the high IAT enhanced the PV. Overall, SD is a best-suited process for protection of oil content inside the WM without significant undesirable quality changes.

4. Conclusions

Results obtained during this study indicate that the fish oil can be used to deliver the omega-3 fatty acids in a food system like milk. Moreover, the spray drying technology can be employed to stabilize the EPA- and DHA-enriched fish oil and milk blends. However, the operating variables influenced the performance of spray drying process. The spray drying temperature was noted to be a key parameter for omega-3 fatty acids retention and affecting the oxidative stability of spray dried powder samples. Furthermore, more research would be needed regarding the sensory acceptability to understand the discerning consumers liking and disliking about spray dried products. In future studies, the long-term storage stability of fish oil supplemented spray dried products should also be investigated by using different oxidation indicators.

Data Availability

The data used to support the conclusions of this article are included within the article.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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