

## Optimize the process for encapsulated spray dried Lemon juice powder to enhance the Vitamin C

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### Abstract

In this study, the parameters were optimized for spray-dried method of fruit juice powder that is encapsulated to increase the bioavailability of vitamin C content. Fruits like lemon were selected to produce the fruit juice powder which provides a high content of vitamin C. Maltodextrin is a carrier material that is used in spray drying techniques at different levels of concentration like 15%, 20% and 25% to coat the fruit juices, reduce stickiness, and produce large quantities of fruit juice powders. In this method, the inlet temperatures ranged from 160 °C to 190 °C while outlet temperatures between 90 °C to 110 °C. Response Surface Methodology (RSM) and Artificial Neural Network (ANN) techniques were used to standardize the method for encapsulating the spray dried fruits juice powder. The physicochemical properties of spray-dried fruit juice powders were analyzed to enhance the nutritional content as well as shelf life. The yield of spray-dried lemon juice powder ranged from 121.56 g to 221.72 g, depending on the maltodextrin concentration. Spray-dried lemon juice powder contains 4.7 % to 5.7 % of moisture, 0.2141 aw to 0.2795 aw of water activity, 14.28% to 17.64 % of hygroscopicity, 91.23 g/100g to 93.26 g/100g of carbohydrates, 138.32 mg/100g to 192.43 mg/100g of vitamin C, 96.54 (%RSA) to 128.62(%RSA) of antioxidant activity respectively. The spray dried fruits juice powders were standardized by using RSM and ANN.

**Keywords:** RSM; ANN; Spray dryer; Lemon juice powder; Vitamin C

### 1. Introduction

According to recent research, in food industry, fruit juices in powder are most acceptable products among the consumers. The fruit juice powders were applied in many products due to its advantages like low cost, storage and transportation, reduced weight of the volume, long shelf life and also nutrient retention. Lemon juice powder was processed by using different drying techniques such as spray dryer, freeze dryer and vacuum dryer. Fresh fruits have high moisture content, highly perishable, having a limited shelf life. In order to extend the shelf life, processing and preservation methods, such as the drying of fruit pieces or fruit juice, are more convenient for end users (Stavra *et al.*, 2022). Drying of fruit juice produces a stable, easy handling form of the juice that reconstitutes rapidly to a good quality product resembling the original juice as close as possible. Dried juice products are used mainly as convenience foods and have a long storage life at ordinary temperatures (Mishra *et al.*, 2015).

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Drying of fruit juices with high sugar content had technical difficulties due to their hygroscopicity and thermoplasticity at high temperatures and humidity. For this reason, addition of maltodextrin and gums as well as other substances such as pectins, calcium silicate, and carboxy-methyl cellulose, has been used in the production of juice powder (Mishra *et al.*, 2015).

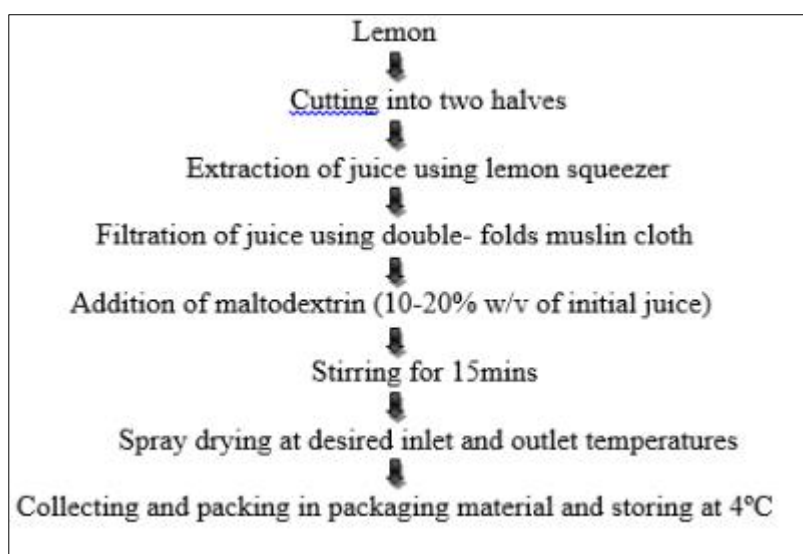
Spray-drying is one of the most widely used in the food industry due to its low cost and flexibility. This technology consists of drying a liquid mixture composed of the core and the wall material which is atomized inside the spray dryer. The water in the mixture is immediately evaporated when the drops comes in contact with the drying gas introduced into the equipment. The resulting microparticles (1 to 100  $\mu\text{m}$ ) are collected at the end of the dryer. Encapsulation is employed to protect chemically sensitive bioactive compounds from degradation due to adverse environmental conditions and is also used to control the release of the encapsulate (Fang *et al.*, 2012). Among the different techniques used for the encapsulation of bioactive compounds, spray-drying is widely used in the food industry due to its rapidity and low cost (Ray *et al.*, 2016). However, spray-drying conditions for the encapsulation of polyphenols must be optimized in order to avoid accelerated degradation (Ballesteros *et al.*, 2017).

Optimization seems to be more significant in the food industry since it is essential to produce food products with appropriate physical, chemical and sensory properties. Response Surface Methodology (RSM) and Artificial Neural Network (ANN) have been used widely in the food process optimization and prediction. RSM is a set of experimental configurations and optimization tools that aid in creating a connection between the variables used as input and output. In recent years, the food sector has been engaged in ANN, a mathematical technique used in artificial intelligence for predicting process outcomes. By discovering the relationship between the input and output variables, ANN is able to anticipate complicated and non-linear processes involving many inputs (Ciric and Krajnc 2020).

## 2. Material and methods

### 2.1. Process Standardization for Development of Spray-Dried Lemon Juice Powder

Lemon were purchased from local market and cut into the halves, and the juice was extracted using a lemon squeezer and filtered the juice using a muslin cloth. Maltodextrin was added to the lemon juice at 10%, 15%, 20% and 25% levels. Preliminary trials showed that when the maltodextrin concentration was lower than 15% most of the material stuck to chamber wall, and when the maltodextrin concentration was higher than 25% there was a significant decrease in the free radical scavenging activity of powder. The feed mixtures containing maltodextrin and juice were dried in lab spray dryer. The inlet temperatures ranged between 160°C to 190°C and outlet temperatures ranged between 90°C to 110°C and the feed rate used were 12rpm to 15 rpm. (Kha *et al.*, 2010). Fig 1.



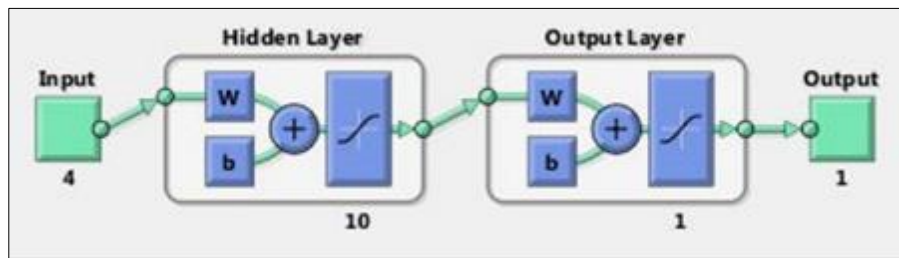
**Figure 1** Processing of spray dried lemon juice powder

## 2.2. Experimental design for optimization of spray drying process

The experimental design of the spray drying process was represented from the central composite design to study the interaction of different variables by Response surface methodology (RSM). The RSM is a widely adopted tool for the quality of optimization processes. The immediate effects of independent variables were inlet temperature, outlet temperature, maltodextrin concentration and feed rate. Four response variables were selected as dependent to represent the important quality attributes of spray dried lemon juice powder, such as water activity (aw), Hygroscopicity (%), colour values (L\*, a\* and b\*) and vitamin C (mg). Response surface methodology was applied to the experimental data using a commercial statistical package (Design Expert, version 13) for the generation of quadratic model that fit the experimental data, to draw the 3D response surface plots and optimization of process variables.

## 2.3. ANN modelling for prediction of spray drying process

The same data of independent and response variables used for Response Surface Methodology were used for development and testing of the Artificial Neural Network (ANN) model. For ANN modelling, MATLAB (Version R2018b) software was used. (Fan *et al.*, 2017). Fig 2



**Figure 2** Network architecture of ANN models with 4 inputs, 10 neurons and 1 output layer

## 2.4. Physical properties

### 2.4.1. Powder yield (%)

The spray drying yield was estimated as the relationship between the mass of the final product and the mass of the feed mixture and calculated as (León-Martínez *et al.*, 2010):

$$Y = \frac{(W_2 - W_1) - X_{wb}(W_2 - W_1)}{F_v T_s} \times 100$$

where  $Y$  is powder yield (%),  $X_{wb}$  is the moisture content (wb),  $F_v$  is the feed volume,  $T_s$  is the total solid content, and  $W_1$  and  $W_2$  are the weight of the powder bottle before and after spray drying, accordingly.

### 2.4.2. Encapsulation yield

The encapsulation yield (EY) was calculated according to equation (Nunes, Mercadante, 2007).

$$\text{EncapsulationYield}(\%) = \text{MSA} \times \frac{100}{\text{MSB}}$$

MSA - total mass of microcapsules obtained after encapsulation

MSB - total mass of solids before encapsulation

### 2.4.3. Moisture

Moisture content of the lemon juice powders were estimated using vacuum oven method. (Michalska *et al.*, 2017)

### 2.4.4. Calculation

$$\text{Moisture}(\%) = \frac{\text{Loss in weight}}{\text{Weight of the sample}} \times 100 = \frac{W_2 - W_3}{W_2 - W_1} \times 100$$

$W_1$  = Initial weight of empty plate

$W_2$  = Weight of empty plate + sample before drying

$W_3$  = Final weight of empty plate + sample after drying

#### 2.4.5. Hygroscopicity,

Hygroscopicity, degree of caking and dispersibility of spray dried lemon juice powder was estimated. (Si *et al.*, 2016)

The hygroscopicity was calculated using Equation

$$\text{Hygroscopicity}(\%) = \frac{\frac{b}{a} + W_i}{1 + b/a}$$

b (g) is the increase in weight of powder,

a (g) is the amount of powder taken for the measurement, and

$W_i$  (% wb) is the free water present in the powder before measurement.

#### 2.4.6. Determination of colour values

The colour intensity of the spray dried lemon juice powder, in terms of lightness, redness and yellowness indices ( $L^*$ ,  $a^*$ ,  $b^*$ ), was measured by using Hunter Laboratory chromometer (Model : Lovibond RT 100) with the Lovibond RT Colour software (Version 3.0). (Wuttipalakovorn *et al.*, 2009).

The net colour difference ( $\Delta E$ ) was calculated with the equation

$$\Delta E = \sqrt{(L_2^* - L_1^*)^2 + (a_2^* - a_1^*)^2 + (b_2^* - b_1^*)^2}$$

#### 2.4.7. Chemical Properties

Chemical properties like carbohydrate, ascorbic acid, total total phenols, total flavonoids and antioxidant activity were analyzed in the spray dried lemon juice powder. Sadasivam and Manickam (2023).

### 2.5. Statistical analysis

RSM was analyzed by using Design Expert (Version 13.0) software. ANN was analyzed using MATLAB R2013a. RSM and ANN mainly used to standardize the process of spray dried lemon juice powder. Analyzing the physical and chemical properties of spray-dried lemon juice powder using OPSTAT. (Ciric and Krajnc 2020)

## 3. Results and Discussion

### 3.1. RSM model effect of spray drying on response of the lemon juice powder

RSM models for the four response variables statistical summarized ( $R^2$  and ANOVA estimation) were presented in the Table 1. The model fitting using regression analysis exhibited that the models described the relationship between the input and output variables with regression coefficient ( $R^2$  = water activity – 0.93, hygroscopicity – 0.93, colour L- 0.92, a – 0.92, b – 0.90, Vitamin C – 0.94) indicating that the predicted values were well fitted with the actual values in the experimental conditions.

**Table 1** Regression coefficients and ANOVA estimated for response variables of Spray dried lemon juice powder

Factors	Water activity	Hygroscopicity	Colour L*	a*	b*	Vitamin C
Std. Dev.	0.0073	0.192	0.1179	0.0361	0.0839	5.41
Mean	0.2605	16.4	249.01	31.83	8.7	160.17
C.V. %	2.79	1.16	0.0474	0.1135	0.9639	3.38
$R^2$	0.9315	0.9395	0.9228	0.9219	0.9001	0.948
Adjusted $R^2$	0.9206	0.9077	0.9104	0.9093	0.8841	0.8994
Predicted $R^2$	0.8974	0.7119	0.8843	0.8791	0.8444	0.7003

### 3.2. ANN modeling of spray dried lemon juice powder

An artificial neural network was used to standardize the process of spray drying lemon juice powder using six response variables. In the model, the regression coefficients were analyzed. The regression coefficient of water activity is 0.98, hygroscopicity is 0.96, colour L\* is 0.96, a\* is 0.98, b\* is 0.97, and vitamin C is 0.98. (Fig 2)

#### 3.2.1. Comparison of RSM and ANN models

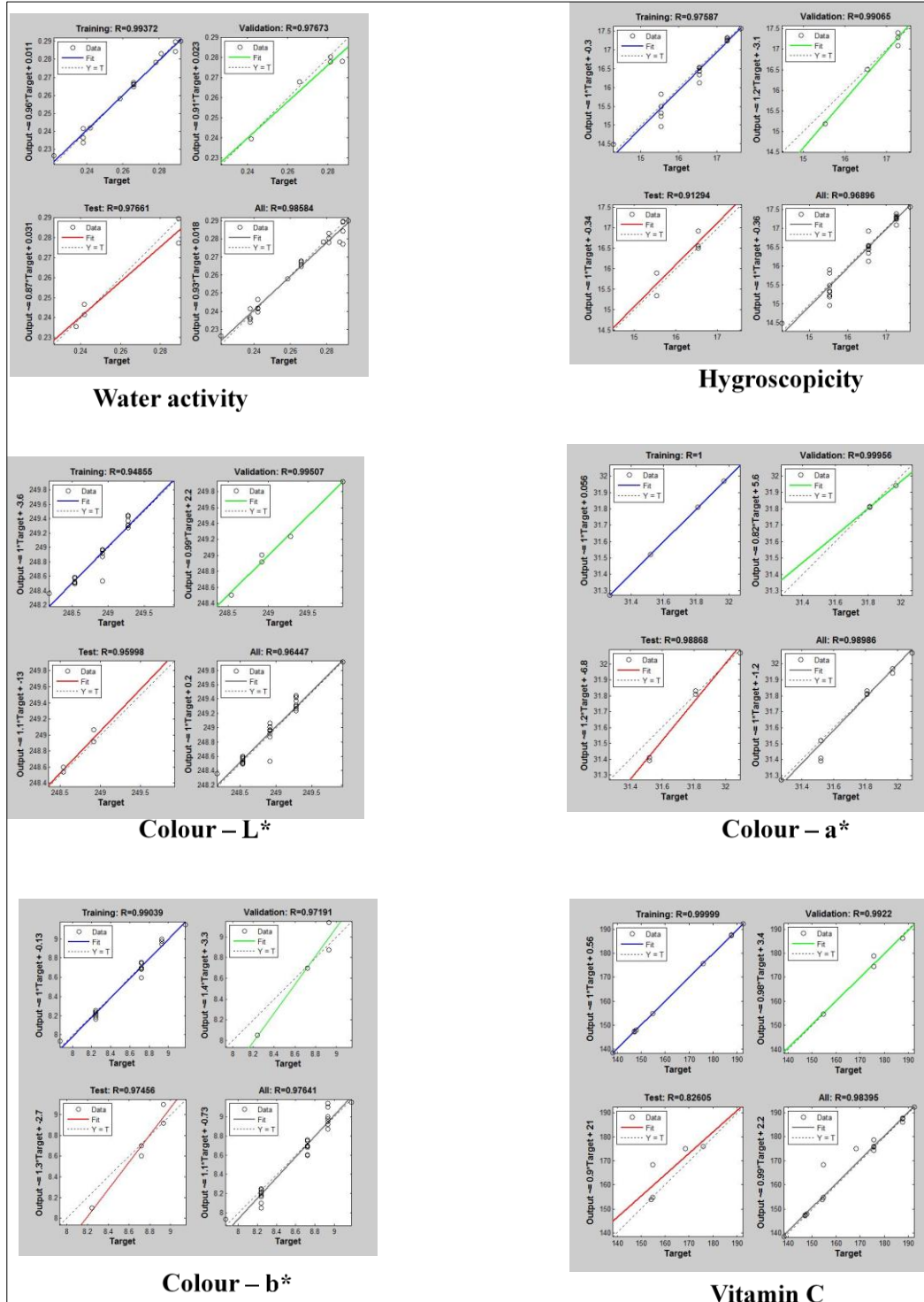


Figure 3 Graphs obtained for training, validation and testing of neural network for prediction of independent variables

Regression coefficients of RSM for spray dried lemon juice powder were mentioned in Table 1. compare the RSM with ANN: the ANN regression coefficients have a higher value than the RSM regression coefficients. In two models, the regression coefficients for optimizing the process of spray drying lemon juice powder were analyzed.

### 3.3. Proximate composition of lemon juice

**Table 2** Proximate composition of lemon juice

S.No	Parameters	Lemon Juice
1	Protein (g)	1.2
2	Fat (g)	0.9
3	Fibre (g)	1.52
4	Carbohydrate (g)	10.61
5	Vitamin C(mg)	60.17
6	Iron (mg)	0.26
7	Sodium (mg)	4.8
8	Magnesium (mg)	19.03
9	Phosphorus (mg)	10.64
10	Potassium (mg)	270.48
11	Calcium (mg)	70.62

Lemon juice had 1.2 g protein, 0.9 g fat, 1.52 g fiber, 10.61 g carbohydrate, 60.17 mg vitamin C, 0.26 mg Iron, 4.8 mg sodium, 19.03 mg magnesium, 10.64 mg phosphorus, 270.48 mg potassium, and 70.62 mg calcium. Table 2

### 3.4. Physical properties of lemon juice powder

**Table 3** Physical properties of lemon juice powder

S. No	Parameters	15% MD - Spray dried lemon juice powder	20% MD - Spray dried lemon juice powder	25%MD - Spray dried lemon juice powder	C.D	SE (d)
1.	Yield	121.56 + 3.149	173.34 + 2.346	221.72 + 0.804	8.165	3.273
2.	Encapsulation yield (%)	76.21 + 0.111	82.73 + 1.894	85.44 + 1.421	4.828	1.936
3.	Moisture (%)	5.7 + 0.012	5.1 + 0.101	4.7 + 0.026	0.214	0.086
4.	Water activity (aw)	0.2795 + 0.032	0.2376 + 0.003	0.2141 + 0.003	0.010	0.004
5.	Color values L*	249.12 + 1.360	253.82 + 1.983	258.63 + 6.459	NS	NS
	a*	31.63 + 0.037	31.55 + 0.624	31.34 + 0.749	NS	NS
	b*	9.25 + 0.110	7.29 + 0.146	7.87 + 0.184	0.528	0.212
6.	Color difference ΔE	7.688 + 0.086	25.37 + 0.265	30.21 + 0.708	1.549	0.621
7.	Bulk density (g/ml)	0.56 + 0.009	0.51 + 0.006	0.48 + 0.012	0.033	0.013
8.	Hygroscopicity (%)	17.64 + 0.158	15.10 + 0.307	14.28 + 0.119	0.743	0.298
10.	Water solubility index (%)	97.20 + 1.906	97.51 + 1.014	98.47 + 0.512	NS	NS

\*MD - Maltodextrin, NS - Non significant. Value indicates mean of five replicates ± standard deviation.

Yield of spray-dried lemon juice powder increased from 121.56 g to 221.72 g due to the concentration of maltodextrin increased, as did the yield of fruit juice powders. The encapsulation yield of spray dried lemon juice powders increased from 76.21% to 85.44%. The moisture content decreased from 5.7% to 4.7% in the spray dried lemon juice powder. The 25% maltodextrin mixed with spray-dried lemon juice powder has a minimum amount of water activity is 0.2141 (aw) when compared to other concentrations. The 15% maltodextrin mixed with spray-dried lemon juice powder has a maximum amount of water activity of 0.2795(aw). The color difference is maximum in the 25% maltodextrin mixed spray dried lemon juice powder and minimum in the 15% maltodextrin mixed spray dried lemon juice powder. The bulk density of spray-dried lemon juice powder ranged from 0.48 g/mL to 0.56 g/mL. The hygroscopicity is minimum in the 25% maltodextrin mixed with spray dried lemon juice powder and maximum in the 15% maltodextrin mixed with spray dried lemon juice powder, at 14.28% and 17.64%, respectively. The water solubility index is highest in the 25% maltodextrin mixed with spray-dried lemon juice powder at around 98.47% and lowest in the 15% maltodextrin mixed with spray-dried lemon juice powder at 97.20%. When maltodextrin concentration increased, the solubility also increased. Table 3

Mishra *et al.*, 2015 concluded that the increase in the concentration of maltodextrin resulted in a decrease in moisture concentration in the finished powder from 4.59 to 3.57% when produced at 175°C. Cai and Corke (2000) and Rodriguez-Hernandez *et al.* (2005) in spray-dried betacyanin pigments and cactus juice powder, inlet temperature influenced the hygroscopicity of the powder positively.

### 3.5. Chemical properties of spray dried lemon juice powder

**Table 4** Chemical properties of spray dried lemon juice powder

S.No	Parameters	15% MD - Spray dried lemon juice powder	20% MD - Spray dried lemon juice powder	25%MD - Spray dried lemon juice powder	C.D	SE(d)
1.	Carbohydrate (g/100g)	91.23+ 0.609	92.15 + 1.966	93.26 + 2.087	NS	NS
2.	Ascorbic acid mg/100g	192.43+ 2.004	161.53 +3.194	138.32 + 2.971	9.778	3.920
3.	Total phenols (mg(GAE)/100g)	96.11+ 0.755	79.73+ 0.830	61.28+ 1.799	4.318	1.731
4.	Total Flavonoids (mg(QUE)/100g)	98.85+ 2.263	87.28+ 1.636	56.34+ 0.600	5.818	2.332
5.	Antioxidant Activity DPPH (RSA %)	128.62+ 0.604	104.73+ 1.088	96.54+ 1.487	3.949	1.583

\*MD - Maltodextrin Value indicates mean of five replicates  $\pm$  standard deviation.

The carbohydrate content of spray-dried lemon juice powders ranged from 91.23 g/100 g to 93.26 g/100 g. The 25% maltodextrin mixed lemon juice powder contains the highest carbohydrate, while the 15% maltodextrin mixed lemon juice powder contains the lowest. The ascorbic acid content in the 25% maltodextrin mixed spray dried lemon juice powder is 192.43 mg/100 g and is 138.32 mg/100 g in the 15% maltodextrin mixed spray dried lemon juice powder. The total phenols content of spray-dried lemon juice powders decreased from 96.11 mg (GAE)/100 g to 61.28 mg (GAE)/100 g. The flavonoid content increased from 56.34 mg (QUE) per 100 g to 98.85 mg (QUE) per 100 g in the spray-dried lemon juice powders. The antioxidant content maximum in the 15% spray-dried lemon juice powders is around 128.62 (RSA%), and the minimum in the 25% spray-dried lemon juice powders is around 96.54 (RSA%). Table 4

Mishra *et al.*, 2015 shows the effect of processing conditions on total phenolic content of spray-dried lemon juice powder. The total phenolic content of the lemon juice powders was reduced when the concentration of the maltodextrin was increased from 10 to 20%. This can be explained to be due to the dilution effect of maltodextrin. The effect of processing conditions of spray drying on DPPH\* scavenging activity of lemon juice powder. DPPH\* scavenging activity of the lemon juice powder was significantly affected by increase in inlet temperature.

## 4. Conclusion

Spray-dried lemon juice powders are in high demand in the food industry. The spray-dried lemon juice powder provide higher amount of vitamin C and other nutrients. RSM and ANN were used to optimize the process of spray-drying lemon

juice powder. When RSM is compared with ANN, the regression coefficients are higher in the ANN process. Among the three concentrations of spray-dried lemon juice powder, 15% maltodextrin mixed with spray-dried powder has the highest nutrient retention.

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## Compliance with ethical standards

### *Disclosure of conflict of interest*

No conflict of interest to be disclosed.

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