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Functionalization of ash gourd: Infusion of citrus peel polyphenols through vacuum impregnation

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ABSTRACT

Citrus peels have high flavonoids known for potent pharmaceutical and food applications. The present study explores the infusion of citrus peel polyphenols (CPP) into ash gourd (*Benincasa hispida)* through vacuum impregnation (VI). The intent was to functionalize ash gourd for diversifying its food use. Vacuum infused ash gourd (VIAG) cubes were evaluated for physicochemical parameters, total phenolic content (TPC), total flavonoid content (TFC), antioxidant activity (AOX), and sensory quality. The VI process was optimized through Box-Behnken Design (BBD) of response surface methodology (RSM), considering blanching time, vacuum pressure and vacuum time as independent variables, and TPC, TFC and AOX as dependent variables. At optimized conditions of blanching pre-treatment (2.21 min), vacuum pressure (432.31 mbar) and time (28.18 min), there was ~300% increase in TPC and AOX. Peleg model validated the mass transfer kinetics for TPC. Sensory evaluation through descriptive analysis revealed no perceived bitterness in VIAG. Overall, it seems that VI is a promising tool for functionalization of ash gourd with bioactives.

1. Introduction

Citrus peel is a promising source of functional ingredients including flavonoids, dietary fiber, pectin, essential oils, protein, and pigments (Wedamulla et al., 2022). Overwhelming scientific research has established health promoting effects of citrus peel against degenerative diseases, such as cardiovascular disease, type-2 diabetes, cancer, and hypertension (Liu et al., 2021; Park & Shin, 2021). High antioxidant, antimicrobial, anticancer, anti-inflammatory activities of citrus peel polyphenols (CPP) have found wider applications in food, pharmaceutical, packaging, and cosmetic industries (Liu et al., 2021). Extraction and instability of CPP is a major challenge in its commercial utilization. Plethora of publications report extraction of polyphenolics from citrus peel using different non-conventional methods and their antioxidant potential in food (Klangpetch et al., 2016; Nishad et al., 2018; Nishad, Saha, Dubey, et al., 2019,b; Liu et al., 2021; Nishad et al., 2021). In the present study CPP extract was used for enhancing the functionality of ash gourd using vacuum impregnation (VI).

VI is one of the widely used non-destructive technologies which has significant applications in developing phytochemical enriched foods, where low pressure treatment is given for easing infusion of functional ingredients (Mujica-Paz et al., 2003; Moraga et al., 2009). VI is driven by pressure gradients to modify the composition of porous foods in a simple and rapid way, upholding the ideas of food safety, fortification and shelf-life enhancement (Panayampadan et al., 2022). Various minerals (calcium, iron, zinc), sugars, natural colorants, anthocyanins, phenolics and anti-browning agents have been successfully infused into several food matrices (de Lima et al., 2016; Diamante et al., 2014; Erihemu et al., 2015; Joshi et al., 2016; Moraga et al., 2009; Perez-Cabrera et al., 2011; Song et al., 2017). The efficiency of VI depends on many intrinsic and extrinsic parameters such as porosity of raw material, solution characteristics, intensity of vacuum pressure, time of exposure to negative pressure, and restoration time (de Lima et al., 2016; Fito et al., 1996; Panayampadan et al., 2022). Thus, optimization of VI should take into consideration the food matrix and other process variables.

Ash gourd (*Benincasa hispida*) is commonly utilized in processing industry to make confectionery and sweet items. The process involves a series of pre-treatment such as blanching, pricking, etc., followed by

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long osmotic dehydration. The low porosity (~13%) of ash gourd enhances the impregnation time, making the process uneconomic (Banjongsinsiri et al., 2004; Mandal et al., 2017). Mandal et al. (2017) reported the combined effect of ultrasonic pre-treatment and osmotic drying for increasing mass exchange and reducing drying time in ash gourd.

The main aim of the study was to evaluate the effect of blanching time, vacuum pressure and vacuum time on infusion efficiency of CPP in ash gourd. The process was optimized using response surface methodology (RSM), where Box Behnken Design (BBD) was applied to achieve maximum infusion of phenolics, flavonoids, and antioxidant activity in ash gourd. RSM is a statistical tool for optimization of complex processes with reduced number of experimental trials to study the effect of multiple factors and their interactions. The data obtained from RSM optimization was then validated using three kinetic models viz. Fick's model, Fito and Chiralt's model, and Peleg's model.

2. Materials and methods

2.1. Chemicals

The analytical grade solvents and chemicals from Sigma-Aldrich (St. Louis, MO, USA) and Merck (Darmstadt, Germany) were used for experimentation and analysis.

2.2. Material and sample preparation

For peel, grapefruit (*Citrus paradisi cv. Redblush*) were harvested from main orchard of Division of Fruits and Horticultural Technology, IARI New Delhi (Latitude 28.3823° N, Longitude 77.0927° E, Altitude 228.61 msl). Ash gourd fruits (cultivar unknown) were procured from local market, washed and hand peeled using stainless steel knife. The peeled slices were cut into cubes of $1 \times 1x1\ cm^3$ and dipped into cold water.

2.3. Extraction and characterization of CPP

Two step enzyme-assisted extraction as described by Nishad, Saha, Dubey, et al. (2019) was followed for extraction of CPP (Fig. 1). Peels were dried at a temperature of 60 °C for 48 h and ground to fine powder (500 μm particle size). For extraction, 0.5 g of peel powder was mixed with 0.9% *Viscozyme* L. in 20 mL of 0.2 M sodium acetate buffer (pH 4.8) and incubated for 5 h at 60 °C. The viscous mass obtained after enzyme inactivation (at 90 °C) was centrifuged for polyphenolic extract and residues left after decanting supernatant were washed with 70% ethanol. A second extraction was performed with the residue obtained from the first extraction and all the collected supernatants were mixed and stored at 4 °C for further analysis. Total phenolic content (TPC) (Singleton et al., 1999), total flavonoid content (TFC) (Zhishen et al., 1999), antioxidant activity using CUPRAC (cupric reducing antioxidant capacity) (Apak et al., 2004), and individual phenolic compounds using HPLC were analyzed for the characterization of the extract (Table 1).

2.4. Vacuum impregnation (VI) process

Preliminary impregnation trials were conducted at atmospheric pressure for 1 h, suggesting no significant infusion of phenols. Thus, VI process was used to facilitate CPP infusion. Pre-weighed cubes immersed in CPP (1:10) were placed inside a closed chamber and subjected to varying vacuum pressure and vacuum time (Table 2) followed by 30 min of restoration of atmospheric pressure. After treatment, sample was taken out from the solution, surface dried using tissue paper, and weighed. Ash gourd cubes obtained before and after VI were characterized for total soluble solids (TSS), pH, acidity (AOAC, 2007 method), moisture content (AOAC, 2007 method), TPC, TFC, and CUPRAC.

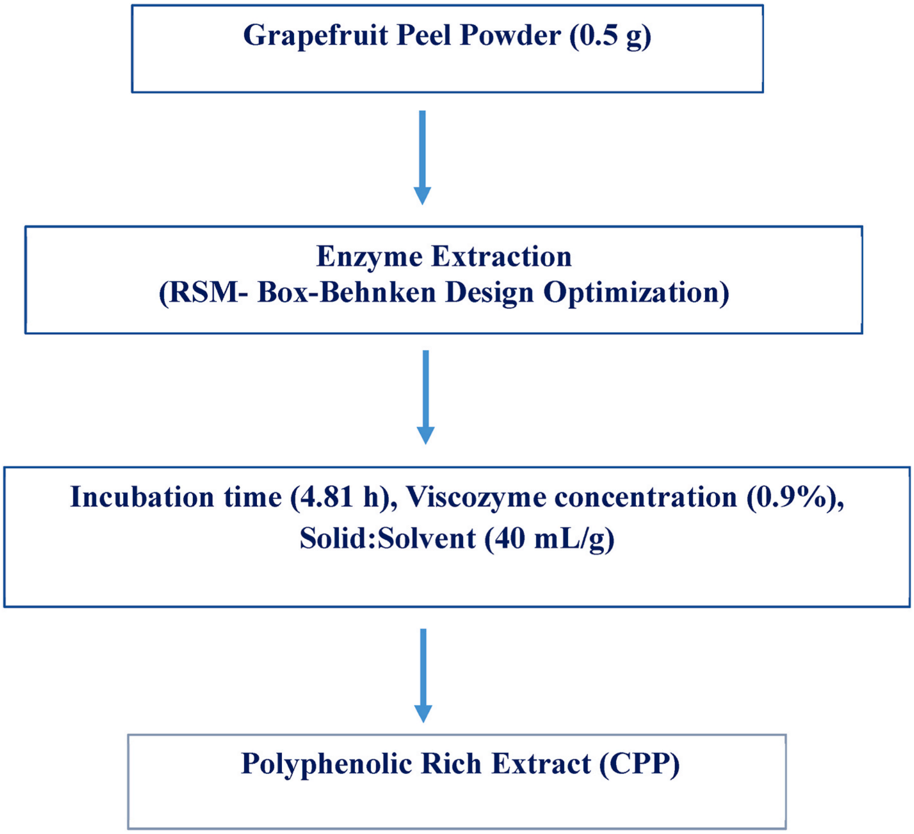

Fig. 1. Enzyme extraction of grapefruit peel polyphenols.

Table 1
Characterization of grapefruit peel extract and ash gourd.

S. No.	Parameters Grapefruit peel extract	Value
1.	TPC (mg GAE/100 g db)	3170.35 ± 173.16
2.	TFC (mg QE/100 g db)	329.89 ± 37.81
3.	AOX (μmol TE/g db)	137.76 ± 36.59
4.	Phenolic compounds (ppm)[a]	Naringin (6975.25) > Trimethoxy benzoic acid (3052.85) > Ferulic acid (278.18) > Epicatechin (198.04) > Caffeic acid (145.76) > Vanillic acid (114.03) > Chlorogenic acid (79.57)

		Ash gourd		
		Raw	**Blanched**	**VIAG**
1.	Moisture content (%)	94.67 ± 1.03	95.33 ± 0.89^{s}	95.82 ± 1.5^{s}
2.	TSS	4.03 ± 0.04	3.83 ± 0.03^{s}	4.56 ± 0.02^{s}
3.	pH	6.6 ± 0.1	6.43 ± 0.1^{ns}	6.25 ± 0.1^{s}
4.	Acidity (%)	0.085 ± 0.001	0.09 ± 0.001^{ns}	0.1 ± 0.002^{s}
5.	TPC (mg GAE/100g wb)	10.51 ± 0.73	10.35 ± 0.57^{ns}	44.08 ± 0.9^{s}
6.	TFC (μg QE/g wb)	12.30 ± 0.006	11.82 ± 0.003^{s}	28.70 ± 1.2^{s}
7.	AOX (μmol TE/100g wb)	29.82 ± 0.97	28.67 ± 1.43^{ns}	120.98 ± 3.4^{s}

Data are expressed as means ± standard error of triplicate samples.
TPC, total phenolic content; TFC, total flavonoid content; AOX, antioxidant activity; VIAG, vacuum infused ash gourd.
GAE, Gallic acid equivalents; QE, Quercetin equivalent; TE, Trolox equivalent; db, dry basis; wb, wet basis.
s, significant; ns, not significant difference from the raw ash gourd.
[a] Phenolic compounds in the extract in decreasing order of their amount.

Table 2
Box–Behnken Design with the experimental values for vacuum impregnation.

Run	X1 – blanching time (min)	X2 – vacuum press. (mbar)	X3 – vacuum time (min)	TPC (mg GAE/ 100g wb)	TFC (μg QE/g wb)	CUPRAC (μmol TE/ 100g wb)
1	1	350	25	23.99	21.76	64.53
2	2	350	15	28.32	25.06	79.9
3	2	350	35	36.21	26.92	99.7
4	3	350	25	37.95	27.58	102.6
5	1	450	15	26.63	23.11	74.9
6	1	450	35	30.12	25.92	87.8
7	2	450	25	45.06	29.53	120.5
8	2	450	25	43.05	29.13	125.8
9	2	450	25	41.13	29.75	128.7
10	2	450	25	42.52	29.64	130.8
11	2	450	25	40.65	30.93	121
12	3	450	15	30.45	26.04	90.4
13	3	450	35	33.95	26.24	95.64
14	1	550	25	21.89	19.99	56.6
15	2	550	15	26.89	24.56	76.4
16	2	550	35	33.49	26.16	93.8
17	3	550	25	25.55	23.04	69.3
SEM				0.07	0.11	0.25

GAE, Gallic acid equivalents; QE, Quercetin equivalent; TE, Trolox equivalent.
TPC, total phenolic content; TFC, total flavonoid content; CUPRAC- cupric reducing antioxidant capacity.
Wb, wet basis, SEM, standard error mean.

2.5. Experimental design for vacuum impregnation of CPP in ash gourd

Prior to RSM optimization, OFAT (One-factor-at-a-time) experiments were run to select the range of the process variables. Where, the effect of blanching time (0–3 min), vacuum pressure (150–533.29 mbar) and vacuum time (5–30 min) on total phenolic content (TPC) was investigated (Supplementary Information 1). Selected range of process variables for RSM optimization of VI were: blanching time-1, 2, 3 min; vacuum pressure- 350, 450, 550 mbar; and vacuum time- 15, 25, 35 min. Box–Behnken Design (BBD) was implemented using Design-Expert software (Design-Expert 11), where selected responses were TPC, TFC and CUPRAC (Table 2). The experimental data were fitted to an empirical second-order polynomial regression model (Equation (1)) to predict the optimum conditions of the extraction process.

$$Y = B_0 + \sum_{i=1}^{k} B_i X_i + \sum_{i=1}^{k} B_{ii} X^2 + \sum_{i>j}^{k} B_{ij} X_i X_j + E \tag{1}$$

where, Y represents the response function (TPC, TFC, and CUPRAC); B_0 is a constant coefficient; the linear, quadratic and interactive coefficients are represented by B_i, B_{ii}, and B_{ij} respectively, and X_i and X_j represent the coded independent variables.

According to the analysis of variance, the regression coefficients of individual linear, quadratic and interaction terms were determined. In order to visualize the relationship between the dependent variables (TPC, TFC and CUPRAC) and experimental levels of each factor, and to deduce the optimum conditions, 3-D surface plots were generated from the fitted polynomial equation. To verify the adequacy of the models, additional extraction trials were carried out at the optimal conditions predicted with the RSM and the obtained experimental data were compared with the values predicted by the regression model. The optimized VIAG was also characterized for its physicochemical properties according to the methods mentioned in section 2.1.

2.6. Mass transfer parameters

The VI kinetics of ash gourd cubes were analyzed by calculating the total phenolic gain (ΔM^{TPH}) as described by Rózek et al. (2010) (Equation (2))

$$\Delta M^{TPH} = \frac{M_t . \, x_t^{TPH} - M_0 . \, x_0^{TPH}}{M_0} \tag{2}$$

Where, M is the mass of ash gourd and x is the mass fraction of total phenolics in ash gourd. The subscripts 0 and t indicate initial conditions and conditions at time t of treatment, respectively. The superscripts TPH indicate total phenolics. The mass fraction of total phenolics is expressed as kg/kg on a wet basis.

2.7. Kinetic modeling

The experimental kinetic data was fitted to three mathematical models to estimate the effective diffusivity of the polyphenolics. The three models explain the diffusivity considering different assumptions. Fick's model which is based on the Fick's second law of diffusion (Crank, 1975) considers the effective diffusivity (D_{eff}) as a mean value, and it disregards its fluctuation during the VI process. Considering the experiment as brief process, and taking into account the initial and boundary conditions of the ash gourd cubes, equation (3) was used to calculate the effective diffusivity:

$$W_s = \backslash \left(\frac{8}{\pi^2} \sum_{i=1}^{\infty} \frac{1}{(2i+1)^2} \exp\left(-(2i+1)^2 \pi^2 \, D_{eff,s} \, \frac{t}{4L^2} \right) \right) \tag{3}$$

Where, $D_{eff,s}$ is the effective diffusivity of the TPC, i is the number of series terms, L is the characteristic length (sample half-thickness), t is the time and W_s is the dimensionless solid content.

Fito and Chiralt's model (Fito & Chiralt, 1997) also makes same assumption that D_{eff} is constant during the process. Besides, it takes hydrodynamic mechanism into consideration, which occurs in VI process. Following equation (4), describes the model, depicting the effect of the hydrodynamics and the pseudo-Fickian mechanisms:

$$1 - Y_t^w\Big|_{t=0}^{t=t} = k + 2\left(\frac{D_{eff}t}{\pi L^2}\right)^{0.5} \tag{4}$$

The model proposed by Peleg (1988) and modified by Palou et al. (1994) was employed to fit the data obtained for diffusion of total phenolics during VI. Peleg's equation (Equation (5)) is an empirical model with two parameters initially established to describe sorption isotherms that approach equilibrium asymptotically:

$$\frac{t}{x_t^j - x_0^j} = k_1^j + k_2^j\, t \tag{5}$$

Where x is the mass fraction of each component expressed as kg/kg on a wet basis and t is immersion time. Subscripts 0 and t indicate initial conditions and conditions at time t of treatment and superscript j indicates any of the components transferred. The Peleg rate constant, k_1, relates to the initial rate of mass change of any component and Peleg capacity constant, k_2, relates to the contents at equilibrium. Peleg constants (k_1 and k_2) were obtained by regressing experimental data to Equation (5). The average relative error, ARE, was used to measure fitness of the regression model in describing the data, and calculated as follows:

$$ARE = \frac{100}{n_e}\sum_{i=1}^{n}\frac{|V_e - V_c|}{V_e} \tag{6}$$

Where n_e is the number of experimental data, V_e is the experimental value and V_c is the calculated value.

2.8. Sensory evaluation

To evaluate the consumer acceptability of VIAG cubes, sensory analysis was conducted where blanched untreated ash gourd cubes were served as control. A panel of 50 members having students and faculties was selected for performing affective test. Before conducting the sensory evaluation, the objective, method, and technical aspects of the test were explained to the members. The check-all-that-apply (CATA) questionnaire was used by the panellists to describe the sensory characteristics of the ash gourd with the help of previously identified suitable attributes. The panellists were asked to describe the samples using different terms- color, bright, uniform, citrus aroma, fruity/melon aroma, firm, juicy, dry, sour, bitter, astringent, and bland. The liking for the samples was evaluated on a 9-point hedonic scale, where 9 represents "like extremely" and 1 represents "dislike extremely". Minimum score of 5 was considered as acceptable. The sensory analysis was performed in two sessions: morning and afternoon in accordance to ISO 6658 (1985). Samples were placed on a white plate, coded randomly with three-digit code, and served after equilibration at room temperature (25 ± 2 °C). Panellists were asked to cleanse their palate in between the samples using water.

2.9. Statistical analysis

All vacuum impregnation trials and proximate analyses were conducted in three replicates and the data are presented as mean values. Blanched and vacuum impregnated samples were compared with raw ash gourd with respect to physicochemical and biochemical properties using two-tailed *t*-test. To evaluate the effect of process variables on TPC, TFC, and AOX, analysis of variance (ANOVA) was performed at a significance level of $p < 0.05$ using SAS (9.4) software. For ordinal data from sensory analysis median values were calculated and Wilcoxon signed rank test ($p < 0.05$) was applied to evaluate the significant differences in perceived liking for different attributes.

3. Results and discussion

3.1. Characterization of citrus peel polyphenols (CPP) and ash gourd

The phytochemical profile of CPP extract is presented in Table 1. High antioxidant activity (AOX) of the extract is attributed to the presence of different phenolic compounds (naringin, trimethoxy benzoic acid, ferulic acid, epicatechin, caffeic acid, vanillic acid, chlorogenic acid). However, ash gourd presented lower content of phenolics and flavonoids, and low AOX.

Moisture content of ash gourd significantly ($p \leq 0.01$) increased after blanching and VI attributed to water absorption during the treatments. Small variations in total soluble solids are attributed to increase in moisture content and corresponding dilution of the solids (Abalos et al., 2020; Assis et al., 2019). VI significantly increased the soluble solids as a result of immersion of ash gourd in the CPP solution and indicated infusion of polyphenols (Mierzwa et al., 2022). Slight decrease in pH after blanching and VI is due to the release of organic acids after structural disintegration of soluble particles in ash gourd (Bhat & Sharma, 2016), as well due to lower pH of the CPP (pH = 6.01). There was significant increase in TPC, TFC, and AOX in VIAG, suggesting efficient vacuum impregnation of CPP. Similar increase in polyphenols were observed in apple and sweet potato (Abalos et al., 2020; Yılmaz & Ersus Bilek, 2017).

3.2. Efficacy of vacuum impregnation (VI) technique in infusing CPP in ash gourd cubes

Vacuum impregnation (VI) employs low pressure for infusion of bioactive compounds in food matrix. VI is dependent on various process and product parameters, therefore, optimization of these factors is necessary to achieve the desired efficiency. The effects of blanching time (X1), vacuum pressure (X2) and vacuum time (X3) on infusion of CPP were studied. The effect of process variables on the rate of diffusion of CPP into ash gourd was evaluated using model kinetics.

3.2.1. OFAT experiments

OFAT experiments were conducted to select the suitable range of different processing variables viz. blanching time, vacuum pressure and vacuum time, by subjecting the ash gourd cubes under variation of one factor at a time, keeping other factors constant. TPC of infused ash gourd cubes was analyzed for selecting the range of variables.

As shown in supplementary information 1, all the selected variables had a significant effect on the infusion of CPP in ash gourd cubes. Blanching of ash gourd revealed an increase in TPC with increase in blanching time. However, after 2 min of blanching a decrease in the TPC was observed. Sample blanched for 2 min and vacuum impregnated for 15 min at 150 mbar pressure showed maximum phenolic content of 24.01 mg GAE/100 g wb. It indicates that 2 min blanching time was sufficient for increasing porosity of ash gourd cubes for maximum infusion of CPP. These results corroborated with the findings of Tiwari et al. (2018, 2022) who reported the effect of blanching treatment on structural modification, disruption of plant tissues and increase of membrane permeability which accelerate the mass transfer mechanism, thus increasing the cellular uptake of solutes. Significant reduction ($p \leq 0.05$) in TPC during prolonged blanching (>2min) is supported by decrease in CPP uptake by the ash gourd cubes. It is attributed to the solubilisation and degradation of cell wall pectin, causing an irreversible structure loss and cell damage (Priecina et al., 2018; Santarelli et al., 2021). Thus, ash gourd cubes blanched for 2 min were used further for optimizing vacuum pressure and vacuum time.

Experiments with vacuum pressure depicted an increase in TPC (29.99 mg GAE/100 g wb) up to 450 mbar of pressure, beyond which phenolic content started decreasing. 450 mbar pressure was then used for vacuum time optimization experiments. Results with vacuum time showed that 30 min vacuum treatment led to maximum infusion of the

CPP (TPC- 34.31 mg GAE/100 g wb).

This variation in TPC in the infused ash gourd cubes with the increase in vacuum pressure and time is ascribed to the hydrodynamic mechanisms and deformation-relaxation phenomena during VI. VI in porous product occurs in two stages after the product with the impregnating solution is kept in a vacuum chamber. At first, reduced pressures are imposed on the solid-liquid system (vacuum step), allowing the expansion and partial outflow of the gases and native liquid present in the product pores. During the second stage of restoration of atmospheric pressure, the residual gas is compressed and the external liquid flows into the pores as a function of the compression ratio. The existing intercellular spaces containing a gas or liquid phase get impregnated with an external solution (Fito et al., 2001; Zhao & Xie, 2004).

Decrease in phenolic intake in this study at high pressure levels could be attributed to an irreversible deformation of the tissue structure and shrinkage of pores (or collapse), thus reducing the available free volume for impregnation (Andrés, 1995; Fito et al., 1996). Similar findings were reported for apple, papaya, mango, banana, melon, peach, mamey and potato chips (Mújica-Paz et al., 2003; Moreira & Almohaimeed, 2018). Similarly, for long vacuum treatment, loss of phenolics is attributed to the prolonged internal gas expansion and flow out of native liquids from the pores of the ash gourd matrix and, eventually, structural damages (Moreno et al., 2011; Neri et al., 2016). The optimum vacuum period in a VI process is the point necessary to achieve mechanical equilibrium inside the product (equal internal and external applied pressure), with the subsequent outflow of part of the internal gas and the free liquid taken along with it (Hironaka et al., 2014).

On the contrary, there is a reduced outflow of gas and native liquid at low vacuum pressure and less vacuum time which leads to high native liquid concentration inside the cells. This disrupts the concentration gradient inside and outside the product, resulting in low inflow of CPP (Chinprahast et al., 2013).

Thus, based on the OFAT results the range of 1–3 min for blanching time, 350–550 mbar vacuum pressure, and 15–35 min vacuum time were selected for RSM optimization of VI.

3.2.2. *RSM optimization of VI*

For the optimization of the VI process, Box-Behnken Design (BBD) with 3 variables (X_1-blanching time, X_2-vacuum pressure and X_3-vacuum time) and 3 levels (−1, 0, 1) was used. The levels of different variables are presented in Table 2. The responses taken into consideration were total phenolic content (TPC), total flavonoid content (TFC) and antioxidant activity (CUPRAC). Firstly, a suitable model was fitted to data obtained in Table 2 giving a significant model and insignificant lack of fit, followed by validation of the selected model. Fig. 3 represents

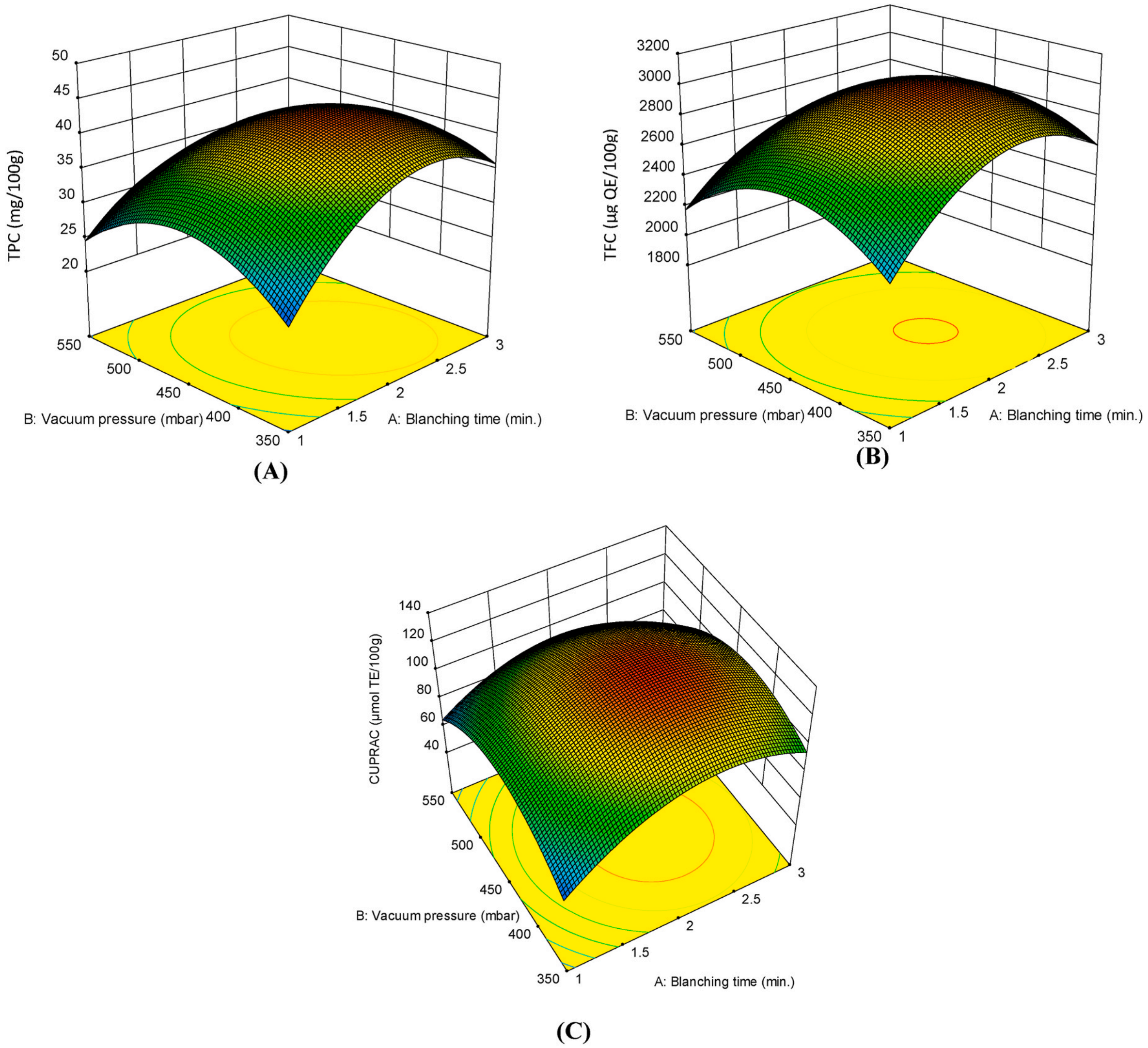

Fig. 2. Response surface analysis for the vacuum impregnation with respect to vacuum pressure and blanching time for (A) total phenolic content (TPC); (B) total flavonoid content (TFC) and (C) cupric reducing antioxidant capacity (CUPRAC).

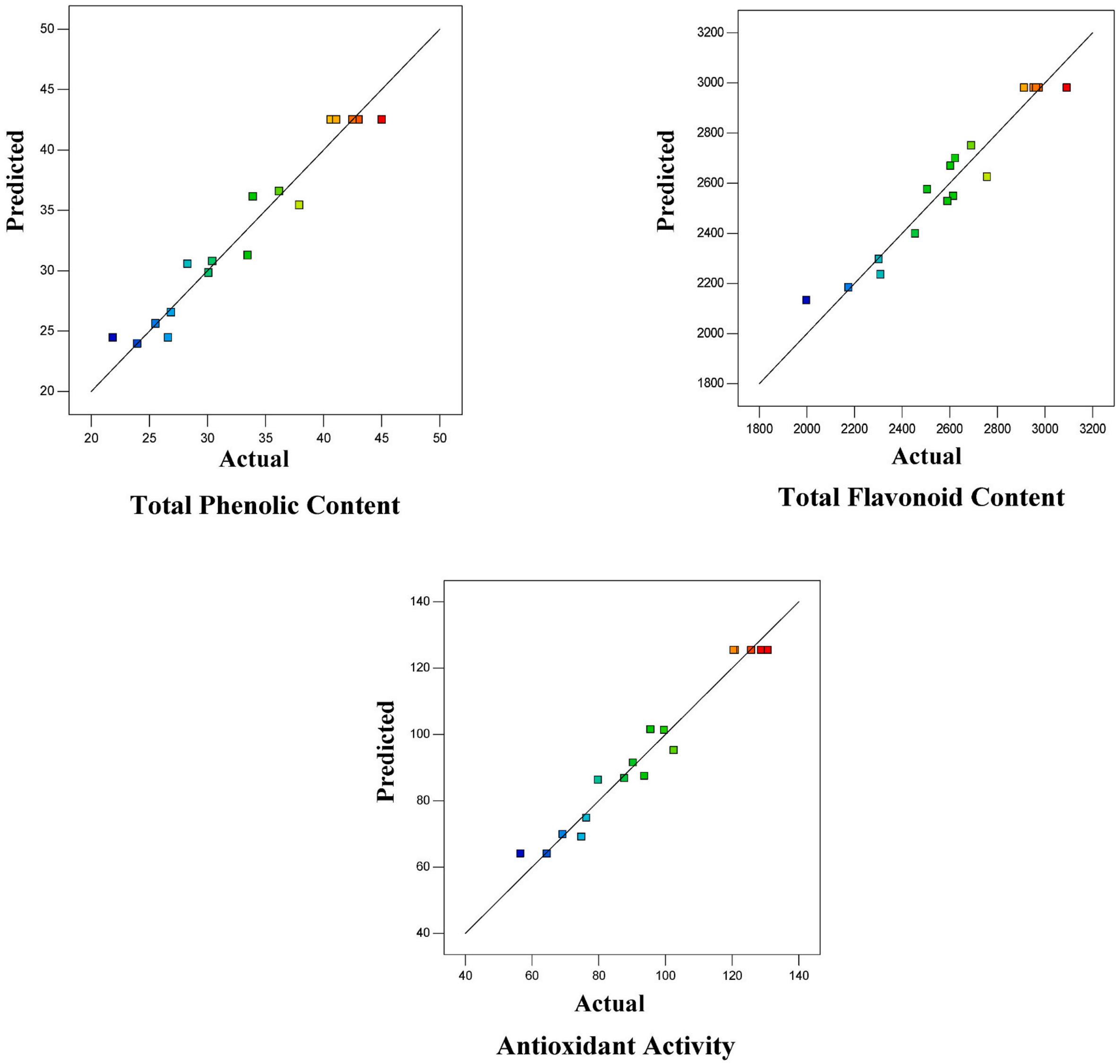

Fig. 3. Comparison between experimental and predicted values obtained from Box Behnken Design.

the similarity between actual and predicted values of TPC, TFC and CUPRAC obtained after 17 runs of RSM optimization.

3.2.2.1. Fitting the response surface models. Based on variance and regression analysis quadratic model was found significant ($p < 0.01$) with an insignificant lack of fit, for all the three responses. The regression coefficients of the model for the response variables, along with the corresponding p-value of lack of fit; R^2 and adj-$R^2$are given in Table 3. The responses were evaluated as a function of linear, quadratic and interaction effects of X_1, X_2, and X_3. Since the model showed lack of fit to be insignificant, the response surfaces were sufficiently explained by the regression equations. High R^2 (>0.9) and adj-R^2 (~0.9) values indicate adequacy of the applied model. Moreover, the low values of coefficient of variation (CV) (<10) represents repeatability within the same treatments or reproducibility within different treatments. Neglecting the non-significant terms ($p \geq 0.1$), the final predicted second-order polynomial equations obtained are given below:

$$Y\ (TPC) = 42.78 + 3.16X_1 - 2.49X_2 - 2.57X_1X_2 - 8.04 \times {}_1^2 - 7.10 \times {}_2^2 - 4.16 \times {}_3^2 \quad (7)$$

$$Y\ (TFC) = 29.90 + 0.12X_1 - 0.098X_2 - 0.35 \times {}_1^2 - 0.32 \times {}_2^2 \quad (8)$$

$$Y\ (CUPRAC) = 125.82 + 8.31X_1 - 6.63X_2 - 26.18 \times {}_1^2 - 25.92 \times {}_2^2 - 11.99 \times {}_3^2 (9)$$

The results of ANOVA indicated the linear effects of blanching time and vacuum pressure ($p < 0.1$) on TPC, TFC and CUPRAC. With respect to quadratic effect, all the three variables showed a significant effect on TPC and CUPRAC ($p < 0.05$), however, for TFC the effect of vacuum time was found non-significant. Interaction effect revealed relation between blanching time and vacuum pressure (X_1X_2) and it was found significant ($p < 0.1$) only for TPC.

Response surface curves depicting the interaction of the two most significant factors, blanching time and vacuum pressure and their effect on TPC, TFC and CUPRAC are shown in Fig. 2 (A-C). Fig. 2 (A) exhibited a significant increase in the TPC with a simultaneous increase in vacuum pressure at a constant blanching time and vice-a-versa. The maximum TPC of 43.42 mg GAE/100 g wb was obtained at 2.22 min blanching time and 429.49 mbar vacuum pressure, and a minimum TPC (24.86 mg GAE/100 g wb) was observed for 1 min blanching time and 350 mbar pressure, suggesting the effect of vacuum pressure and blanching time on infusion of phenols.

Similarly, the interaction of X_1X_2 is shown in Fig. 2 (B), revealing the effect of their variations on TFC. A blanching time of 2.2 min and 432.56 mbar pressure yielded maximum flavonoid (30.16 μg QE/g wb) impregnation into the matrix. The response curves for the CUPRAC

Table 3

Analysis of variance (ANOVA) for the experimental values for vacuum impregnation.

Source	TPC				TFC				CUPRAC			
	Sum of squares	df	*F*-value	*P*-value Prob > F	Sum of squares	df	*F*-value	*P*-value Prob > F	Sum of squares	df	*F*-value	*P*-value Prob > F
Model	824.38	9	14.25	0.001 significant	1.42E+06	9	12.29	0.002 significant	8538.03	9	18.99	0.0004 significant
X1- blanching time	53.22	1	8.28	0.024**	75145.01	1	5.86	0.046**	367.97	1	7.37	0.03**
X2- vacuum press.	33.09	1	5.15	0.058*	51012.48	1	3.98	0.086*	234.35	1	4.69	0.067*
X3- vacuum time	5.97	1	0.93	0.367ns	473.77	1	0.04	0.853ns	71.02	1	1.42	0.272ns
X1X2	26.48	1	4.12	0.082*	19182.25	1	1.5	0.261ns	160.91	1	3.22	0.116ns
X1X3	2.50E-05	1	3.89E-06	0.999ns	17134.81	1	1.34	0.286ns	14.67	1	0.29	0.605ns
X2X3	0.41	1	0.063	0.809ns	161.29	1	0.01	0.914ns	1.44	1	0.029	0.87ns
$X1^2$	271.95	1	42.3	0.0003***	5.24E+05	1	40.86	0.0004***	2886.69	1	57.79	0.0001***
$X2^2$	212.11	1	32.99	0.0007***	4.25E+05	1	33.16	0.0007***	2828.55	1	56.62	0.0001***
$X3^2$	72.76	1	11.32	0.012**	37267.56	1	2.91	0.132ns	605.43	1	12.12	0.0102**
Residual	45	7			89730.21	7			349.68	7		
Lack of fit	32.87	3	3.61	0.123 not significant	71462.46	3	5.22	0.072 not significant	266.1	3	4.25	0.098 not significant
Pure error	12.13	4			18267.75	4			83.57	4		
Cor total	869.39	16			1.51E+06	16			8887.7	16		
R^2			0.95				0.94				0.96	
Adj-R^2			0.88				0.86				0.91	
CV%			7.59				4.32				7.42	
Adequate precision			9.53				9.76				11.32	

df, degrees of freedom.

*Significant at $p < 0.1$; ** Significant at $p < 0.05$; *** Significant at $p < 0.01$; ns: non-significant.

(Fig. 2C), depicted maximum activity (127.05 μmol TE/100 g wb) at 2.2 min and 434.57 mbar pressure. Thus, the steep concave shape of these illustrations at the center signifies the selection of central/middle value for blanching time (~2 min) and vacuum pressure (~450 mbar) to achieve high phenolic infusion.

The change in TPC, TFC and antioxidant activity of the ash gourd cubes with process variables is contributed by structural modifications in ash gourd, and effect of hydrodynamic mechanism and deformation-relaxation phenomenon.

3.2.2.2. Validation of the predicted model. RSM optimization of VI resulted in an optimum processing condition of 2.21 min blanching time, 432.32 mbar vacuum pressure and 28.18 min vacuum time, with a desirability of 1.0 (Table 4). At this process condition, the values of responses predicted were: TPC of 43.53 mg GAE/100 g, TFC 30.20 μg QE/g and CUPRAC value of 127.59 μmol TE/100 g. With the objective of verifying the model conditions, VI was performed at the optimized parameters and a TPC of 44.08 mg GAE/100 g, TFC 28.70 μg QE/g and CUPRAC 120.98 μmol TE/100 g were obtained. The statistical analysis on the data revealed no significant difference between the predicted and experimental values. Thus, VI process was validated as the successful non-conventional impregnation technology for the bioactive compounds in the ash gourd.

3.3. Influence of vacuum impregnation on CPP infusion - model kinetics

All the three process variables showed a positive effect on total phenolic gain (ΔM^{TPC}) in VIAG (Fig. 4), keeping water activity of the impregnated solution constant so that the driving force remains same. A maximum phenolic gain of 30.19 kg/kg was observed in sample which was blanched for 2 min and impregnated with CPP at 450 mbar pressure for 25 min.

Fick's model, Peleg's model, and Fito and Chiralt's model were used for comparing mass transfer kinetics of phenols during VI (Equations (3)–(5)). The effective diffusivities (D_{eff}) of the total phenolics were calculated using above models and results are shown in Table 5. Ash gourd cubes after 2 min blanching and vacuum treatment at 450 mbar pressure for 25 min exhibited higher D_{eff} values. All the models exhibited higher determination coefficient for the TPC kinetics data, showing fitting of the models. The values of the effective diffusion coefficients are analogous to those published by several authors (Correa et al., 2016; Junqueira et al., 2017; Rózek et al., 2010; Souraki et al., 2014). The average relative error (ARE) of 3.67 for Fick's model was highest among three, depicting less suitability for the data obtained in this study. Previous studies have also shown lower fitting capacity for the Fick's diffusive model compared to the other models (Barbosa Júnior et al., 2013; Correa et al., 2010). Fick's model calculates the effective diffusivity as a mean for the entire duration of the VI and does not consider the hydrodynamic process which reduces the quality of the model to fit the data (Ochoa-Martinez, Ramaswamy, & Ayala-Aponte,

Table 4

Optimized conditions for vacuum impregnation.

Blanching time (min)	Vacuum pressure (mbar)	Vacuum time (min)	TPC (mg GAE/100g wb)		TFC (μg QE/g wb)		CUPRAC (μmol TE/100g wb)	
			Experimental	Predicted	Experimental	Predicted	Experimental	Predicted
2.21	432.32	28.18	44.08	43.53	28.70	30.20	120.98	127.59

TPC- total phenolic content.
TFC- total flavonoid content.
CUPRAC- cupric reducing antioxidant capacity.

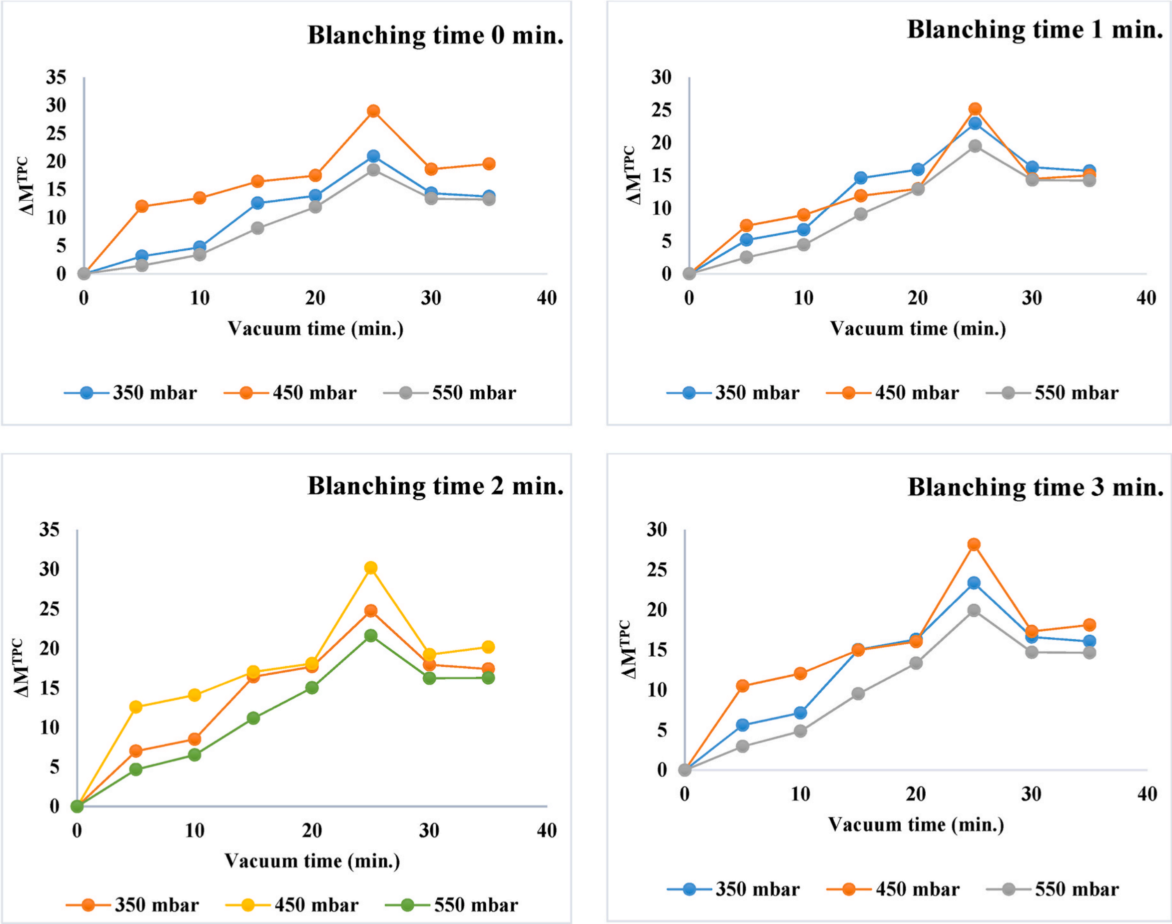

Fig. 4. Total phenolic gain (ΔM^{TPC}) of ash gourd cubes during vacuum impregnation as a function of blanching time, vacuum pressure and vacuum time.

Table 5
Diffusivity values calculated using Fick's model, Fito and Chiralt's model and Peleg constants during impregnation.

Models		Vacuum pressure (mbar)		
		350	450	550
Fick's model	**Deff [m^2/s]**	5.61×10^{-8}	4.75×10^{-8}	3.72×10^{-8}
	A	57.36	108.64	39.12
	B	−57.12	−91.80	−40.18
	C	19.39	25.88	14.27
	D	−2.19	−2.41	−1.69
	R^2	0.95	0.96	0.96
	ARE	3.67		
Fito and Chiralt's model	**Deff [m^2/s]**	9.91×10^{-9}	1.19×10^{-8}	8.25×10^{-9}
	R^2	0.92	0.88	0.90
	ARE	2.43		
Peleg constants	**Peleg rate (1/k1, s^{-1})**	0.03	0.06	0.02
	Peleg capacity (1/k2, kg/kg)	36.38	31.15	43.35
	R^2	0.95	0.89	0.95
	ARE	1.28		

Deff-effective diffusivity; R2 – correlation coefficient; ARE-average relative error.

2007). The Peleg constants model showed lowest ARE value and therefore it was selected for studying the mass transfer kinetics for TPC in ash gourd cubes. The correlation obtained between predicted and experimental values of phenolics infusion from Peleg constants is depicted in Fig. 5. The initial rate of phenolic mass transfer at 450 mbar (total phenolic 1/k1 = 0.06/s) was nearly two and three times higher than that obtained at 350 and 550 mbar pressure (total phenolic 1/k1 = 0.03 and 0.02/s) respectively.

3.4. *Sensory evaluation*

The sensory scores for color, bright, uniform, citrus aroma, fruity/melon aroma, firm, juicy, dry, sour, bitter, astringent, and bland demonstrated the degree of liking by the consumers (Table 6). The average scores for color, citrus aroma, fruity/melon aroma, juicy, sour, bitter, and astringent were found significantly different ($p < 0.05$) for control and treated samples.

The sensory values for VIAG were higher for citrus aroma, juiciness, sourness, bitterness, and astringency, which is attributed to infused polyphenolics. Vacuum impregnation in CPP enriched solution had contributed to the improved internal and external moisture characteristics of the ash gourd cubes, hence increased sensory quality. Low scores were given to VIAG for its color, fruity/melon aroma, dryness, and bland flavor. The low preference to VIAG for its color was attributed to the slight change in color from white to creamish white after placing in yellow colored CPP solution during VI. A score of 8 was given to both the samples for brightness, uniformity, and firmness, depicting similar sensory characteristics for these attributes. Overall, panellists described that the sensory quality of the VIAG was on par with the control samples.

4. Conclusion

VI process was successfully employed for infusion of CPP in ash gourd to enhance its functionality. The effective diffusivity of

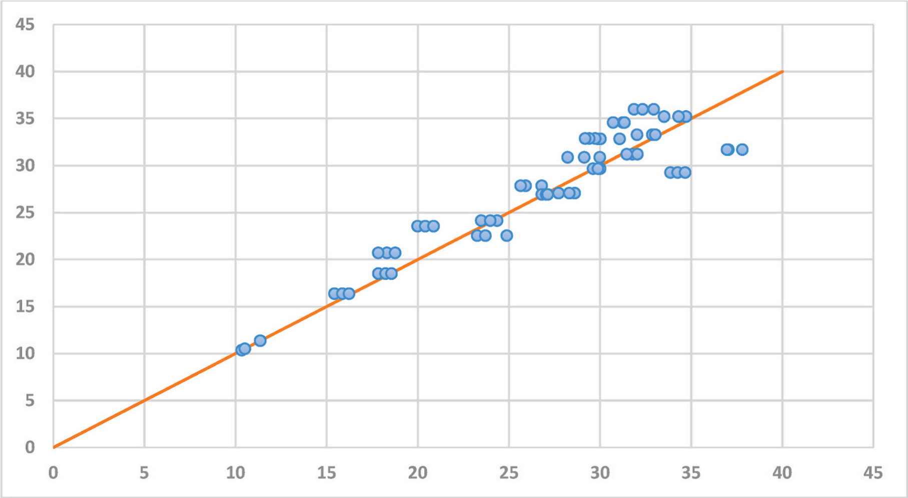

Fig. 5. Correlation of the predicted and experimental data of the vacuum impregnation of total phenolics (Peleg constants).

Table 6
Average values received for different attributes of ash gourd samples and the results from Wilcoxon signed rank test for comparison between the samples.

Attributes	Samples	
	Blanched ash gourd	VIAG
Color*	8	7
Bright	8	8
Uniformity	8	8
Citrus*	3	5
Fruity/Melon*	6	5
Firm	8	8
Juicy*	7	8
Dry	5	4
Sour*	2	3
Bitter*	3	4
Astringent*	3	5
Bland	7	6

VIAG, vacuum infused ash gourd.
*Indicates significant differences between the samples ($p < 0.05$) according to the Wilcoxon signed rank test.
The values in each column are median values of n observations, where $n = 50$.

polyphenols was optimized through RSM. The optimized conditions of blanching for 2.21 min coupled with vacuum impregnation at 432.31 mbar pressure for 28.18 min increased the TPC by ~300%, TFC by ~140%, and AOX by ~300%. The vacuum impregnation was found suitable for infusing polyphenols in ash gourd without affecting the physicochemical and sensory quality. Panellists perceived no significant increase in bitterness in the infused ash gourd. The Peleg constants model was found suitable in explaining mass transfer kinetics of phenolics in ash gourd. Results suggest that VI is a promising tool for impregnation of bioactive in foods to produce a functional product.

CRediT authorship contribution statement

Jyoti Nishad: Conceptualization, Investigation, Data curation, Writing –original draft, preparation. Alka Joshi: Methodology, Formal analysis, Writing – original draft. Shruti Sethi: Resources, Methodology, Investigation. Shalini G. Rudra: Resources, Methodology, Formal analysis. Eldho Varghese: Formal analysis, Software. Nishant Shankhwar: Writing - review & editing, Visualization. Arti Bhatia: Writing - review & editing. Vivek Saurabh: Writing - review & editing. Charanjit Kaur: Conceptualization, Methodology, Supervision, Writing – review & editing.

Declaration of competing interest

"The authors confirm that they have no conflicts of interest with respect to the work described in this manuscript."

Data availability

No data was used for the research described in the article.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fbio.2022.102095.

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