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Phenolics from potato peel and its extraction intensification using response surface methodology and genetic algorithm approach

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ABSTRACT

Potato peels are zero-value waste generated during potato processing. They are a concentrated source of phytochemicals. The extraction of phenolics from this waste may add valuable products for the growing food industry due to their antioxidant and antimicrobial properties. In this study, phenolics were extracted from potato peel by using ultrasound assisted extraction process with a combination of sonication time (5-25 min), ethanol concentration (0-100%) and extraction temperature (25-65°C) as independent variables using central composite design (CCD). The process parameters were optimized by applying response surface methodology (RSM) and a genetic algorithm (GA) approach. The extraction parameters were optimized for maximum total phenol content (TPC). Using the genetic algorithm approach, the optimized conditions for maximum recovery of total phenols (621.27 mg/ 100g dwb) from potato peel are 22.032 min sonication time at 61.818°C in 99.99% ethanol. Ultrasound-assisted extraction was highly efficient compared to conventional solvent extraction, increasing the extraction yield by 2.84 fold. Optimized conditions and the predicted model provide a brilliant opportunity for the food industry with great health advantages.

Keywords Phenolics, Potato peel, Ultrasound, Genetic algorithm, Response surface methodology

INTRODUCTION

Polyphenols are naturally occurring secondary metabolites available commonly in fruits, vegetables, cereals and beverages. Across various plant species, more than 8000 polyphenolic compounds have been identified (Beckman, 4). Predominantly, phenolic compounds exist as conjugated forms in nature. However in higher plants, low molecular weight phenols are present as glycosides or esters. In food, phenolic compounds may be involved in flavour modification, imparting bitterness, odour, colour and providing oxidative stability (Ezekiel *et al.,* 6). They regulate the defense mechanism in plants over aggression by pathogens and ultraviolet radiation. Recent studies suggest that long term consumption of phenolic rich diet may give protection against cardio-vascular diseases, cancer, diabetes, neurodegenerative diseases and osteoporosis (Pandey and Rizvi, 14).

Valorization of food waste is one of the important components in sustainability research. Food processing industries produce huge amounts of waste in the form of rag, seeds, peel, pomace etc. Potato processing industry forms a major chunk of food processing sector wherein ready-to-eat preparations such as chips, French fries and mashed potatoes are being developed. During processing operations, about 15-40% potato peel is produced depending upon the process operations (Schieber *et al.,* 16). Such huge quantum of peel generated creates disposal problems leading to environmental hazards. The peel can be pretreated for change in its functional characteristics (Azizi *et al.,* 3) and maybe used for fibre enrichment in fabricated foods (Azizi *et al.,* 2). Upgrading of peel waste into value added products has created interest among the processors on the natural compounds which can be extracted and utilized for food and pharma purposes (Liguori *et al.,* 10).

Potato peel is a rich source of bioactive compounds including antioxidants, dietary fibres, minerals, pigments and vitamins which have apoptic, anti-inflammatory and chemo-preventive property (Joshi *et al.,* 8; Azizi *et al.,* 3). The phenolic compounds in the peel are the major bioactive principles that impart disease resistance to the plant and when extracted can be used for their antimicrobial and antioxidative properties. Conventional processes for extraction of bioactives usually results in low yield. Ultrasonic waves in the range of 20-1000 kHz can be utilized for escalating the efficiency of extraction of useful compounds. The propogation of these waves through food matrices via rarefaction and compression creates a negative pressure in the solvent leading to the formation of microscopically small bubbles (Ojha *et al.,* 13). Collapsing of these

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bubbles generates extreme mechanical forces that rupture the tissue to release the bound compounds. In recent times ultrasound assisted extraction is being widely used to recover valuable phytocompounds from waste as it increases the percentage recovery of targeted compound by more than 20% in comparison with conventional methods (Mourtzinos and Goula 11). Ultrasound application improves efficiency of process in terms of yield, selectivity and quality of phytochemicals. The technology has been applied for by-product utilization such as apple pomace (Kadam *et al.,* 9) and pomegranate peel (Dang *et al.,* 5) for extraction of useful compounds. Phenolic compounds extracted from plant matrices can further be utilized in diverse food products such as beverage and bakery alongwith use as dietary supplements, animal feed and cosmetics. Application of ultrasonication is thus, a promising technology with wide application in food for efficient extraction of the phytoceuticals. Therefore, our study aimed to test the applicability of ultrasound assisted extraction of phenolics from potato peel with optimization of operational parameters using response surface methodology through the use of central composite design and further by genetic algorithm approach.

MATERIALS AND METHODS

Potato (*Solanum tuberosum* L. var. 'Lady Rosetta') peels were procured from Haldiram Foods, Gurugram, Haryana. The same day, peels were washed in clean running water to remove dust and sand particles and dried for 6-7h at 60°C. The dried potato peels were ground to a fine powder in a portable grinder (Prestige Iris 750W) and sieved through 500 μ sieve (BSS No. 30) to get uniform particle size. The peel powder (8.64% moisture content) was packed in airtight polythene bags and stored in cold chamber (10±1°C) until further analysis. Chemicals were obtained from Merck and Sigma Aldrich.

Conventional protocol for extraction of phenolics was followed as described by Sethi *et al.* (17). For this, 1 g of potato peel powder was extracted with different peel to solvent ratio (1:5, 1:10, 1:15 and 1:20) of 80% ethanol overnight under ambient temperature. The mixture was centrifuged at 10000 rpm for 15 min at 4°C. Total phenolic content was quantified in the extract obtained by the method of Sethi *et al.* (17) and expressed as mg GAE/ 100g on dry peel weight basis.

Ultrasound assisted extraction process was carried out in ultrasonic bath (LeelaSonic-138) with working frequency of 33 KHz using three independent variables viz., sonication time, extraction temperature and solvent concentration as mentioned in Table 1. The obtained mixture was centrifuged at 10000 rpm for 15 min at 4°C. The supernatant obtained was evaluated for its total phenolic content (mg GAE/100g) using the Folin-Ciocalteu reagent (FCR) method as described by Sethi *et al.* (17). The absorbance of the reaction mixture was taken at 765 nm using a UV-vis spectrophotometer (Spectra Max M2, Molecular Devices, USA).

All the analyses were taken in three replicates and the data presented here are the mean values. ANOVA (analysis of variance) at 5% level of significance was carried out to find the influence of each factor on the TPC. The first stage optimization was done using second order response surface model based on the data obtained through the use of central composite design (CCD) (Varghese *et al.,* 18). The response surface optimization was carried out using Design Expert Software version 9.0.6.2. The second stage optimization was carried out using Genetic Algorithm (GA) approach which is an artificial intelligence based technique and more robust than classical response surface optimization. For carrying out GA optimization, an exponential form of second order response surface model was considered. The parameters obtained through response surface methodology were considered as the initial value for fitting the exponential model which was used as a functional relationship between TPC and the set of predictors for optimization through GA approach. The exponential model fitting was carried out in SAS (r) Proprietary Software 9.4 (TS1M1).The genetic algorithm approach for optimization was carried out in R version 4.0.3.

RESULTS AND DISCUSSION

In the preliminary part of experiment, the effect of solute solvent ratio (SS ratio) on the yield of

Table 1. Uncoded and coded levels of the independent variables of the extraction process

Independent variable	Symbol	Coded Levels				
		-2	-1	0	+1	+2
Sonication time (min)	X_1	0.000	4.6	11.3	18.0	22.568
Extraction temperature (°C)	X_2	28.182	35.0	45.0	55.0	61.818
Ethanol concentration (%)	X_3	0.000	20.3	50.015	79.73	100.00

TPC was studied. Increasing the ratio from 1:5 to 1:10 and 1:15 resulted in a corresponding increase of the TPC in the extract (99, 106.87, 142.12 mg GAE/100g, respectively) which reduced thereafter at 1: 20 (129.54 mg GAE/100g). The observations are in lieu with the mass transfer principle which shows development of larger concentration gradient at high SS ratio leading to higher phenolic diffusion in the extract (Nayak *et al.,* 12). However, in ratio greater than 1:15, dilution of phenolics was observed leading to significant reduction in TPC. Based upon these results, 1:15 ratio was selected for further study.

Modeling of the extraction process for 20 runs was performed according to CCD for the optimization of ultrasound assisted extraction of phenols from 'Lady Rosetta' potato peel (Table 2) taking sonication time, temperature and ethanol concentration as three independent factors. The analysis based on CCD considered a second order response surface model (quadratic polynomial). For three factors, the second order polynomial was:

$$y=\beta_0+\beta_1*A+\beta_1*A+\beta_2*B+\beta_3*C+\beta_{11}*A^2+\beta_{22}*B^2+\beta_{33}*C^2+\beta_{12}*AB+\beta_{13}*AC+\beta_{23}*BC$$

where, the response y = Total phenolic content; A = Sonication time; B = Extraction temperature and C= Ethanol concentration

Initially a second order quadratic model was fitted with the data. It was observed that linear effects of A and C alongwith the quadratic effects of B i.e. B^2 remain significant at 5% level of significance. However, the R^2 value does not remain high enough. Moreover, since the R^2 value was less, thus, a partial third order model by adding AB^2 was fitted again in the given data as follows:

$$y=\beta_0+\beta_1*A+\beta_1*A+\beta_2*B+\beta_3*C+\beta_{11}*A^2+\beta_{22}*B^2+\beta_{33}*C^2+\beta_{12}*AB+\beta_{13}*AC+\beta_{23}*BC+\beta_{122}*AB^2$$

The results of TPC from the potato peel ranged from 306.674 to 531.057 mg GAE/ 100 g db (Table 2). The analysis of variance (ANOVA) of the modified model is shown in Table 3. It was observed that for the given data set the overall model remains significant at 1% level of significance. Beside, total phenolic content was significantly affected by the linear effects of sonication time, extraction temperature and ethanol concentration at 1% level of significance. Among the quadratic model, TPC was significantly affected by the quadratic effects of extraction time at 5% level of significance. Among the interaction effects, TPC was significantly affected by the linear interaction effects of both sonication time and ethanol concentration at 5% level of significance whereas the interaction of linear effect of sonication time and quadratic effect of extraction temperature also affected TPC significantly at 1% level of significance. The R^2 = 0.9557 for the modified model indicated that this model was able to explain 95.57% variability which is quite good. The adjusted R^2 =0.9065 which was also good, indicating that the significant portions of variations explained by the modified model was 90.65%. It is to be noted here that, the adjusted R^2 would increase if only significant variables are included in the model.

Table 2. The CCD matrix and the experimental data for the responses

Run	X_1 Sonication time (min)	X_2 Extraction temperature (°C)	X_3 Ethanol concentration (%)	Total phenolic content (mg GAE/100 g db)
1	18.0	35.0	20.30	306.7
2	18.0	55.0	20.30	367.7
3	11.3	45.0	50.01	431.5
4	11.3	28.2	50.01	394.5
5	11.3	45.0	50.01	458.9
6	22.7	45.0	50.01	442.3
7	11.3	45.0	99.99	506.5
8	4.6	35.0	20.30	377.9
9	11.3	45.0	50.01	425.2
10	11.3	45.0	50.01	430.9
11	4.6	35.0	79.73	474.6
12	11.3	61.8	50.01	452.6
13	11.3	45.0	50.01	429.3
14	4.6	55.0	79.73	487.7
15	11.3	45.0	50.01	434.9
16	11.3	45.0	0.000	287.2
17	4.6	55.0	20.30	421.4
18	18.0	35.0	79.73	420.7
19	18.0	55.0	79.73	531.1
20	0.0	45.0	50.01	313.6

The optimum point for maximum phenolic extraction from potato peel was observed to be with sonication for 18 min at 48.9°C in 79.73% ethanol concentration. It can be seen that, the optimum point maximizes the total phenolic content and the predicted maximum value is 545.330 mg GAE/ 100g dw with the desirability value as 0.995 which is almost near the maximum possible values of desirability. The optimum value of total phenols may lie between 512.931 to 577.729 mg GAE/ 100g. To validate the adequacy of the models, three trials were performed at the optimized values of the parameters and the results thus obtained were compared with the predicted values of the quadratic model.

The two factor interaction wise three dimensional response surface curves and contour plots for the

Table 3. Analysis of variance (ANOVA) for the experimental results obtained with UAE

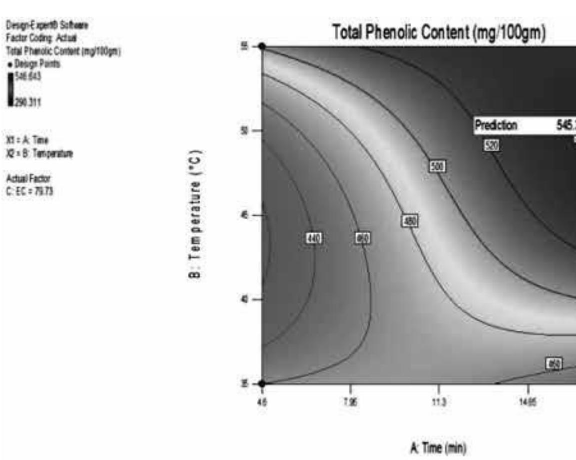
ANOVA for Response Surface Reduced Cubic model						
Analysis of variance table [Partial sum of squares - Type III]						
Source	Sum of Squares	df	Mean Square	F Value	p-value Prob> F	
Model	87039.70	10	8703.97	19.42	< 0.0001	**
A-Time	9230.77	1	9230.77	20.59	0.0014	**
B-Temperature	9030.68	1	9030.68	20.15	0.0015	**
C-EC	58029.00	1	58029.00	129.46	< 0.0001	**
AB	953.50	1	953.50	2.13	0.179	
AC	2927.28	1	2927.28	6.53	0.031	*
BC	303.45	1	303.45	0.68	0.432	
A^2	3191.16	1	3191.16	7.12	0.026	*
B^2	243.96	1	243.96	0.54	0.480	
C^2	313.21	1	313.21	0.70	0.425	
AB^2	11468.84	1	11468.84	25.59	0.001	**
Residual	4034.14	9	448.24			
Cor Total	91073.85	19				

*Significance at 5%, ** significance at 1%

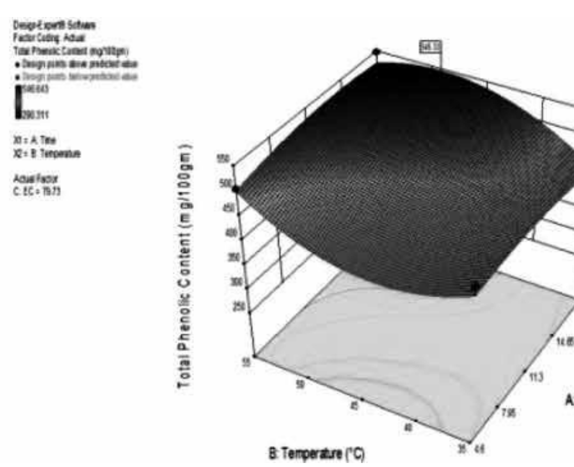
extraction yield of total phenolic content (TPC) are given in Fig 1 (only graph with second order interaction effects are plotted). Figs. 1a and 1b show the effect of sonication time and extraction temperature on the yield of TPC. By increasing sonication time from 0.0319 to 22.568 min and extraction temperature from 28.182 to 55°C, the TPC increased from 440 to 545.33 mg GAE/100 g. However, maximum TPC of 545.33 mg GAE/100 g was observed at 48.9°C with sonication time of 18 min keeping EC at 79.73%. Figs 1a and 1b indicate that keeping ethanol concentration constant at 79.73%, maxima with very high desirability lies towards the higher extreme values of sonication time and higher values of extraction temperature.

The interaction of sonication time and ethanol concentration on the recovery of TPC is depicted in Figs. 1c and 1d. The recovery reached its maximum of 545.33 mg GAE/100 g at 79.73 % ethanol concentration and 18 min sonication time keeping extraction temperature at 48.9°C which was 2.84 fold higher than the conventional method Figs 1c and 1d indicate that keeping extraction temperature constant at 48.908°C, maxima with very high desirability lied towards the higher extreme values of both time and ethanol concentration.

Figs. 1e and 1f show the interaction effect of ethanol concentration and extraction temperature on TPC yield at constant sonication time. TPC increased from 300 to 545.33 mg GAE/100 g when ethanol



1a



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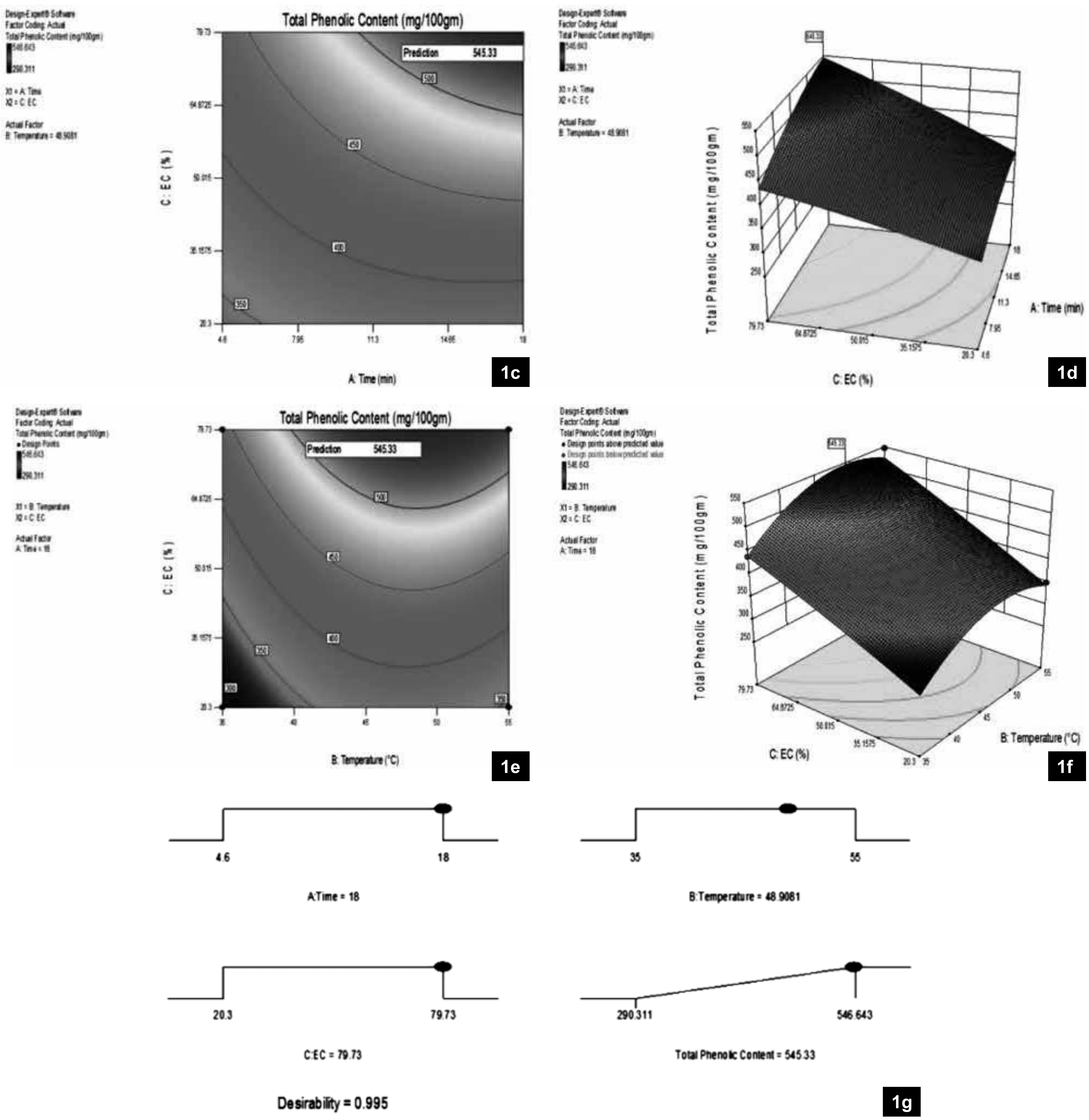

Fig. 1. Response surface analysis for the total phenolic content (TPC) from potato peel with ultrasound assisted extraction (UAE) with respect to sonication time and extraction temperature (1a and 1b); sonication time and ethanol concentration (1c and 1d); extraction temperature and ethanol concentration (1e and 1f); Optimum combination plot with desirability (1g).

concentration increased from 20.3 to 79.735 % and extraction temperature was raised from 35 to 55°C. The recovery reached its maximum of 545.33 mg GAE/100 g at 79.73 % ethanol concentration and 48.9°C keeping sonication time at 18 min. Figs 1e and 1f indicate that keeping sonication time constant at 18 min, maxima with very high desirability lies towards the higher values of extraction temperature and extreme values of ethanol concentration. Genetic Algorithms (GA) are mathematical models inspired by the famous Charles Darwin's idea of natural selection. The natural selection preserves only the fittest individuals, over the different generations. The advantage of this technique over others is that it allows the best solution to emerge from the best of prior solutions. The idea of GA is to combine the different solutions generation after generation to extract the best information from each one. That way it creates new and more fitted individuals. The GA approach has been effectively used in optimization problems. Recently GA approach based on the results of response surface modeling has been carried out by Sarkar *et al.* (15) for production of inulin from yam bean tubers by ultrasonic assisted extraction. GA algorithm produces random solution in the first generations if no seed values (starting solutions) are provided. Best solutions, with least or most return value based on the nature of optimization are picked on which genetic operators are applied to produce a new solution as part of the second generation. GA produces more unique random solutions in the second generation. This process continues until the most optimal solution is reached or the generation hard limit is reached.

For performing the GA optimization, an exponential form of non-linear second order response surface model based on the data collected through the use of CCD by treating TPC as response variable and sonication time, extraction temperature and ethanol concentration as predictors was used. The non-linear model was initialized by taking the estimated value of the parameters obtained based on the second order response surface model fitted earlier in the present investigation. The non-linear model fitting is an iterative procedure which requires initialization of model parameters. The non-linear model fitting was done through iterative procedure using Gauss-Newton method of non-linear least square. The functional form of non-linear partial third order response surface model is:

$$y = \exp\left(\beta_0 + \beta_1 * A + \beta_1 * A + \beta_2 * B + \beta_3 * C + \beta_{11} * A^2 + \beta_{22} * B^2 + \beta_{33} * C^2 + \beta_{12} * AB + \beta_{13} * AC + \beta_{23} * BC\right)$$

where, A = Sonication time; B = Extraction temperature; C = Ethanol concentration

The convergence criteria satisfied after 5 iterations. The model remains highly significant at 1% level of significance. The fitted equation is as follows:

$$TPC = \exp(5.8240 - 0.00690*A - 0.00487*B + 0.00483*C - 0.00078*A^2 + 0.000061*B^2 - 0.00003*C^2 + 0.000370*A*B + 0.000184*A*C + 0.000021*B*C)$$

Since the model was highly significant, the fitted model was used for genetic algorithm for optimization for finding optimal solution. The fitted non-linear second order response surface model as given above was considered as objective function. After 1000 iterations (Fig 2, Table 4), optimization results are summarized as follows:

Predicted values of the experiments were validated in laboratory and results are presented

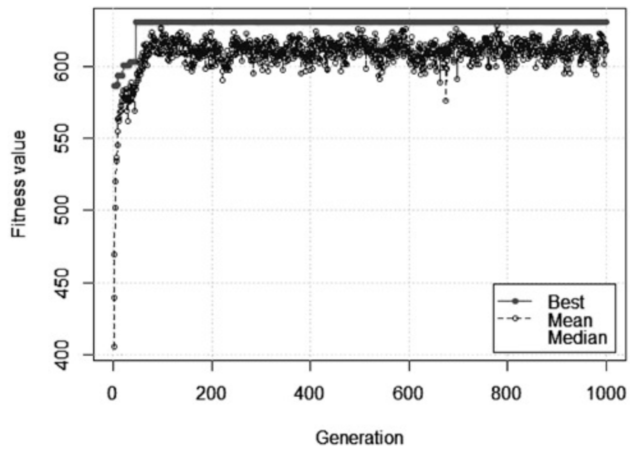

Fig. 2. Iteration results obtained through GA approach

Table 4. Genetic algorithm settings for maximizing phenolic yield

GA settings	
Type	real-valued
Population size	50
Number of generations	1000
Elitism	2
Crossover probability	0.8
Mutation probability	0.1
Search domain	X1 X2 X3 lower 0.000 28.200 0.000 upper 22.568 61.818 99.990
GA results	
Iterations	1000
Fitness function value	631.127 (maximum value for TPC)
Solution	X1 X2 X3 22.032 61.817 99.989

in Table 5. The improved yield of TPC in ultrasound assisted extraction is in agreement with earlier studies on extraction of polyphenols (Garcia-Castello *et al.,* 7). They reported a 50% higher extraction of naringin from grapefruit using UAE. High extraction yield of TPC in comparison with conventional extraction could be attributed to the interaction of ultrasound with the powdered potato peel that promotes the penetration of solvent into the tissue leading to higher extraction efficiency. Altemimi *et al.* (1) demonstrated cell damage to ultrasound treated matrix as observed under electron microscopy.

The study indicates that potato peel can be considered as a good source of phenolics. The results revealed that ultrasound assisted extraction is an effective technique for increasing the yield of

Table 5. Comparison between optimized conditions predicted by RSM and GA

Approach	Total phenolic content (mg GAE/100g)	
	Predicted	Experimental*
UAE_{RSM}	545.33	515.65
UAE_{GA}	631.12	621.27

*Average of triplicate analysis

total phenols from potato peels in comparison with conventional method. Central composite design was an efficient statistical tool to determine the optimum extraction processing conditions of phenolic compounds from potato peel using modified partial third order model. Using genetic algorithm approach the optimized conditions for maximum recovery of total phenols (621.27 mg/ 100g) from potato peel are 22.032 min sonication time at 61.818°C in 99.99% ethanol. Validation by genetic algorithm approach was much closer to the predicted value in comparison to response surface methodology. Hence, genetic algorithm approach is recommended technique for conducting optimization studies. Further, from the view of industrial utilization, this research could be the basis for using green technology for higher recovery of value added compounds from the waste residues. The technology can help in reduction of processing time and higher recovery of targeted compound in comparison to the conventional process.

AUTHORS' CONTRIBUTIONS

Investigation, Writing Original Draft (RHR), Conceptualization, Methodology, Supervision, Review & Editing of Final Manuscript (SS), Statistical analysis, Software, Review & Editing of Final Manuscript (AB), Statistical analysis (EV), Review & Editing of Final Manuscript (AJ).

DECLARATION

Authors declare no conflict of interest.

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