



Application of Response Surface Methodology to Food Science and Technology: A Review

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Abstract: Response Surface Methodology (RSM) is a statistical technique used to optimize processes and products, by modeling the relationship between input variables and output responses. In Food Science and Technology, RSM is used to optimize Food processing conditions, preservation methods, Quality retention of Food and Food safety interventions. Modeling and optimization is an important task in food processing. It enables one to understand and describe processes which in turn help establish quantified relationship between input and output variables. Modeling and optimization help to make informed decision on a process with the objective of improving efficiency and minimizing cost while maintaining quality. Response surface Methodology (RSM) has been employed in modeling and optimizing several food processing operations including the Application of baking, cooking, roasting, drying, extrusion, fermentation and many others. Moreover, RSM has been extensively used in product formulation and ingredient optimization. This review describes the application of RSM in both Food Science and Technology and Food Processing Modeling and Optimization (FPMO). The steps to be followed, the experimental designs that can be used and the interpretation of response surfaces developed are described. Moreover, selected application of RSM in food process modeling and optimization are reviewed and presented.

Keywords: RSM, Application of RSM, Food Process Modeling, Optimization, Response Surface to Food.

Introduction

Response Surface Methodology (RSM) is a collection of statistical and mathematical techniques useful for Food products developing, improving, and optimizing processes. It also has important applications in the design, development, and formulation of new products as well as in the improvement of existing product designs. The most extensive applications of RSM are in the industrial world, including the food industry, particularly in situations where multiple input variables potentially influence the quality characteristics of the product or the process. RSM has

been extensively used in modeling and optimizing food processing operations and formulation of products. Major food process operations or application like **Drying, Extrusion, Fermentation, Baking and Cooking** operations/application have been modeled and optimized using RSM. Moreover, food product formulations and product design and development has been carried out using RSM. Several experimental designs including factorial designs, central composite design with its variants, D-optimal design, and mixture designs have been used with RSM.

Besides analyzing the effects of the independent variables, RSM generates an empirical model which describes the processes. The term *Response Surface Methodology (RSM)* was derived from the graphical view created after fitting the mathematical model. The objectives of this review were to present a brief historical and theoretical overview of RSM, describe its application in food processes modeling and optimization and product formulation, highlight interpretation of response surfaces and graphical optimization techniques (overlay plots) if possible and review previous works where RSM has been used. Moreover, in this review, the steps to model and optimize food processes and formulations using RSM are presented and different experimental designs used in RSM are also described.

Modeling and optimization of processes including food processes has been done through focusing on the effect of changes in one parameter on a response keeping all other factors constant. This is called one-variable-at-a-time technique. The major limitation of this method is that the interactive effects among the variables are not accounted for and there is a lack of explanation of the complete effect of the factors on the response or an overview of the variables' behavior within the entire experimental space. Moreover, one-variable-at-a-time method increases the number of experimental runs required to conduct the research, which eventually leads to increased cost and time to do the research. In order to address this limitation, optimization studies should be conducted by applying procedures like response surface methodology (RSM) where multiple factors are considered at a time. RSM has been found to be an effective method for food product modeling and optimization.

Improving system performance without increasing cost of production and process time while maintaining the required quality attributes is the main objective of food processing and manufacturing. Finding the optimum processing condition and recipe (formulation) for food products of high quality and high marketability is paramount importance for successful products output. The method used for coming up optimal processing condition and combination of ingredients with the best output is called optimization. Modeling precedes optimization and helps establish a quantitative relationship between independent and dependent/response variables. In the food industry, models are used for exactly the same purpose as in the scientific world. They help practitioners/reviewers and scientists to think about processes that are too complicated to understand in every detail, (Solomon Worku Kidane, 2021).

2.1 Theory and steps in carrying out Response Surface Methodology (RSM)

RSM is a statistical technique used to model and optimize the relationship between a response variable (y) and one or more predictor variable (x). The RSM goal is to find the optimal combination of predictor variables that result in a desired response. Though RSM was developed in the 1950s, its application in food process operations began in 1960s. The RSM's major

advantage is generating large amount of information from a reduced number of experimental runs that are required to evaluate multiple parameters and their interactions. The relationship between the independent variables and the response can be represented by $y = f(x_1, x_2, x_3 \dots x_n) + \epsilon$ where y is the response, f is the unknown function of response, $x_1, x_2, x_3, x_4 \dots x_n$ denote the independent variables and n is the number of independent variables, ϵ is error that represents other sources of variability which is not explained by the mathematical relationship (by the function, f).

2.1.1 The Modeling and Optimization Procedure using RSM

The modeling and optimization procedure using RSM is normally carried out in stages. Though different steps or stages are reported in literature, all the steps outlined in different literature have similarities or commonalities. The steps in general include:

- i. Identification of independent variables and their levels: Clearly define the problem and objective of the study. Identify the response variable and the predictor variables.
- ii. Selection of the experimental design: Choose an appropriate experimental design, such as Central Composite Design (CCD), Box – Behnken Design (BBD), or Doehlert Design (DD).
- iii. Selection of a regression model and prediction and verification of model equation: Conduct experiments according to the selected design. Measure the response variable and record the data.
- iv. Graphical presentation of the model equation: develop a Mathematical Model, use the data to develop a mathematical model that describes the relationship between the predictor variables and the response variable.
- v. Prediction and determination of optimal operating conditions: Analyze the Model to ensure that it is adequate and accurate. Check for significance, lack of fit, and residual plots.
- vi. Optimize the Response: use the Model to optimize the response variable. This involves finding the combination of predictor variables that result in the desired response.
- vii. Validate the Model: validate the model by conducting additional experiments at the optimal conditions. Compare the predicted response with the actual response.

2.1.2 Identification/Selection of Independent Variables and their Levels

Many factors often affect foods manufacturing processes, both recipe/ingredient related and process parameter related. The independent variables to be studied are selected based on experience, research results obtained from literature or preliminary experiments. If there are too many variables involved, as is the case in most new food products development, screening procedure should be used to identify those that critically influence the responses of interest. Screening designs allow the researcher or reviewers to look at the effects of several variables each of which takes on two levels with less number of runs. Those significant variables are then selected for further optimization. Screening designs like Plackett-Burman (PB) and Saturated Fractional Factorial Designs (SFFD) are commonly used in food processing and formulation. Some specifically designed preliminary experiments are conducted using screening designs and they enable the food researchers to estimate the effect of each factors and to select the most significant and critical variables from the potential variables with minimum experimental efforts.

2.2 Selection of Experimental Design

An important aspect in applying RSM is the design of experiments. After selection of the food quality attributes of interest (response) and identifying the significant independent variables, the next step of statistical food product design and development is to design an appropriate experiment. Some computer packages offer optimal designs based on the special criteria and input from the user. These designs differ from one another with respect to their selection of experimental points, number of runs and blocks. The 3^n factorial, the central composite design (CCD), the Box–Behnken Design (BBD), the D-optimal designs (constrained designs) and mixture designs are commonly used in RSM. The following review introduce the designs briefly.

2.2.1 Full Factorial Design (3^n Factorial Design)

A 3^n factorial design is suitable for supporting the building of a quadratic model, if there are less than four significant variables ($n \leq 4$) selected for modeling in the food systems and chemical processes. A 3^n experimental design supplies 3^n degrees of freedom, in which one is fixed for determining the total average value β_0 (constant term) in the model. The remaining ($3^n - 1$) degrees of freedom then allow estimation and calculation of the effects of each factor, the interactions between and among factors, and the curvature in the system. A 3^n factorial design is constructed by the combination of all the possible test levels of each variable. It can be divided into four subgroups: a 2^n factorial plan with 2^n trials, $2n$ central points of all the surfaces, border middle points and one central point (in practice, this should be repeatedly performed).

A 3^n factorial design suitable for supporting the building of a quadratic model

Runs number	3^3 factorial design			Central Composite Design			Bo –Behnken Desing		
	x_1	x_2	x_3	x_1	x_2	x_3	x_1	x_2	x_3
1.	0	-1	0	-1	-1	1	0	1	-1
2.	1	1	0	-1	-1	-1	0	-1	1
3.	1	0	0	-1	1	-1	-1	0	1
4.	1	-1	1	1	1	1	0	1	1
5.	-1	1	0	1	-1				
6.	1	-1	1						

Table 1: Number of runs for designs with three factors used in RSM.

2.2.2 Central Composite Design (CCD)

The Central Composite Designs (CCD) is the foundation of the RSM and is popularly used to estimate parameters of a full second-degree model in all scientific research areas. One of the advantages of CCD is its efficiency with respect to the smaller number of runs required with each factor having 3 or 5 levels. The other advantage of CCD is that it can be constructed in a sequential program of experimentation by building onto information gathered previously from a 2^n factorial design. If a linear model based on a 2^n factorial design turns out to be insignificant, then some extra trials can be designed, according to the principles of a CCD, to repair the model. All these data will be used to build a quadratic model. This is also known as the build-up principle of the

CCD. Normally, a quadratic model would meet the needs for accuracy in practical product development and process modeling.

2.2.3 Box – Behnken Design (BBD)

The Box–Behnken Design (BBD) comprises a specific subset of the factorial combinations from the 3^n factorial design. These designs are formed by combining 2^n factorials with incomplete block designs. The resulting designs are usually very efficient in terms of the number of required runs, and they are either rotatable or nearly. In addition, in a BBD, the experimental points are situated on a hyper sphere equally distant from the central point. Applying this design is popular in food processes due to its economical design. BBD is appropriate to evaluate interaction between factors and especially to study processes without extreme points (where high levels of factors involved in the process is difficult to implement) such as high temperature and pressure next to each other. Several studies employed BBD to optimize food process operations.

2.2.4 D – Optimal Design (Constrained Designs)

“D-optimal” means that these designs are selected from the list of valid candidate runs that provide as much orthogonality between the columns of the design matrix as possible. D-optimal designs have been used in optimizing food ingredients (D-optimal mixture designs) and process conditions.

The factorial designs are not always applicable for some food processes because of functional or technical restriction. At times there are combinations of some factor levels that are not practically possible to conduct the experiment. Examples of such are combinations of high levels of all the factors or low levels of all the factors in a given experiment. For example, in a roasting operation combining the highest temperature and the longest time may result in a product that is over roasted which is not fit for sensory evaluation whereas the high temperature can be combined with other shorter roasting times. Every trial under factorial design must be performed and the trial number increases rapidly beyond affordable limit when the number of factors increase. On the other hand, though CCD offers a smaller number of trials, it requires the exact setting of the test levels at the defined values and cannot be changed or is not flexible to handle constraints. D-optimal design was developed to overcome these shortcomings or exclude practically unsound scenarios. In D-optimal design, the test level of each variable can be selected flexibly and a variable can be tested at as many levels as the researcher wants. The number of levels of the different factors can be different or same. D-optimal designs are computer-generated.

2.3 Selection of a Regression Model, Prediction and Model Verification

Building a model is one of the most important steps in food process and product design. After the experiments have been conducted and the data collected, the intended model is fitted to the data by using regression analysis least square minimization technique. The two important criteria for selecting a usable and precise model from the alternative equations are: the model with the highest precision for accurate application and the model with the simplest form for easy application. Polynomials have been used extensively in empirical modeling of chemical, biological, and food research systems. They provide a simple curvilinear relationship between a numbers of variables, possess a clearly defined optimum, and use simple computational algorithms by using the least square minimization method for estimation of the model

coefficients in the model. Low-degree polynomials, such as a first-degree polynomial with interaction terms or a quadratic polynomial, are the most appropriate models to adequately describe food processes.

The second order model can be written as the following equations:

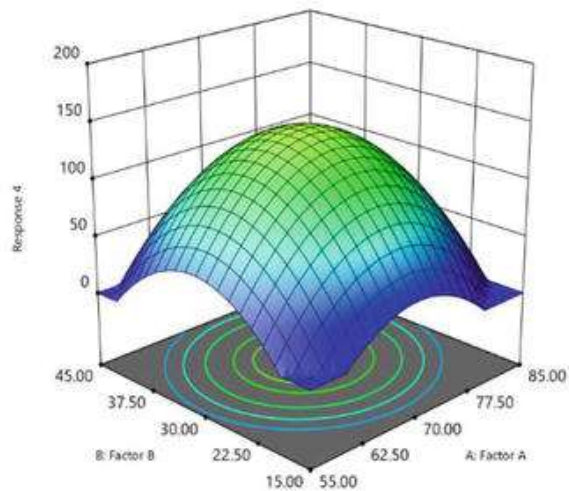
$$Y = \beta_0 + \sum_j = 1n\beta_jX_j + \sum_{j<k} = 2n\beta_{jk}X_jX_k + \sum_j = 1n\beta_{jj}X_j^2$$

where β_0 , β_j , β_{jj} and β_{jk} are regression coefficients for intercept, linear, quadratic and interaction terms respectively and X_j , and X_k , are coded independent variables.

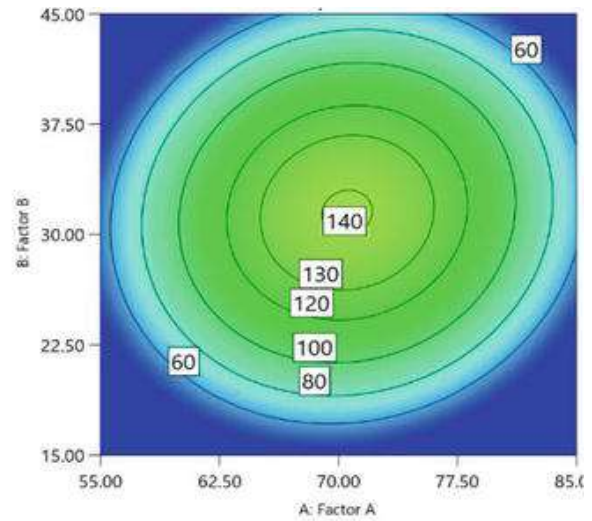
Following the estimation of regression coefficients, the estimated response could be estimated/predicted using the model equation. Moreover, one must check whether the model adequately describes the relationship between the dependent and the response variables, i.e., fits well to the experimental data. Several techniques could be used to check the adequacy of the developed model. These techniques include residual analysis, prediction error sum of squares (PRESS) residuals, and testing of the lack of fit. The overall predictive capability of the model is commonly explained by the coefficient of determination (R^2). However, R^2 alone is not a measure of the model's accuracy. R^2 indicates the percentage of variability in the response explained by the changes in the independent variables.

2.4 Graphical Presentation of the Model Equation

Model building is not the only and ultimate objective of food process and product design. The interest of food product and process designers focus on the effect of different factors on the quality attributes. The question usually is which variables and in which ranges have significant effects on specific quality attributes or response variables. The other question could be under what conditions food should be processed to get a pre-defined quality attribute. Only a significant and precise model can supply reliable and essential information for the food researcher. Generally, two approaches are used to extract this information from the model: the graphical and numerical method. The predictive models are used to generate contours and response surfaces within the experimental range. The response surface plot is the theoretical three-dimensional plot (3D surface) showing the relationship between the response and the independent variables. The two-dimensional display of the surface plot is called contour plot. In the contour plot, lines of constant response are drawn in the plane of the independent variables. It is a two-dimensional screen of the surface plot, in which, ranges of constant dependent variables is drawn in the plane of the independent variables. Indeed, the contour plots improve the researcher's understanding of the shape of a responses surface.

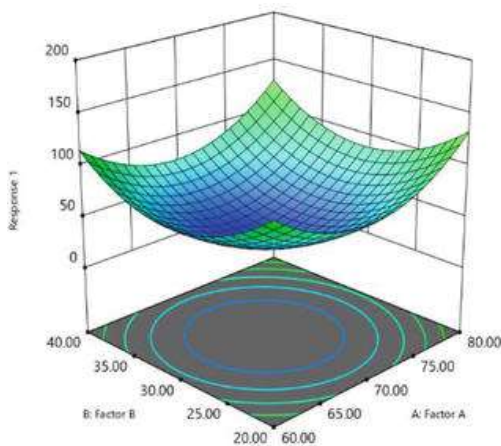


(a)

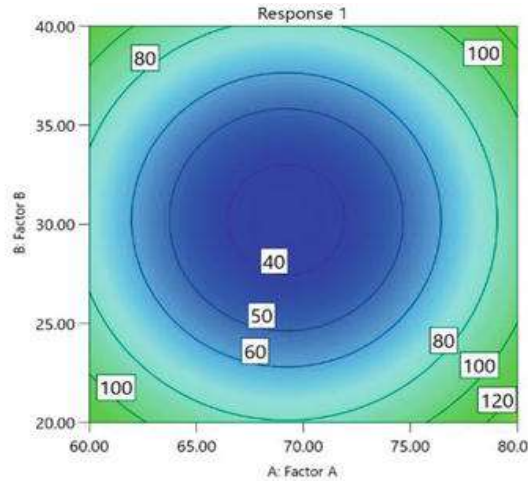


(b)

Table 2.1 Graphical presentation of 3D surface (a) and contour plot (b) where there is a maximum response.



(a)



(b)

Table 2.2 Graphical presentation of 3D surface (a) and contour plot (b) where there is a minimum response.

Proper interpretation of contour plots is an important part of the optimization exercise. When the contour plot displays ellipses or circles, the center of the system refers to a point of maximum or minimum. Sometimes, contour plot may display hyperbolic or parabolic system of the

contours. In this case, the stationary point is called a saddle point and it is neither a maximum nor a minimum point as shown on Figure 6 below. These plots give useful information about the model fitted but they may not represent the true behavior of the system. It is important to keep in mind that the contours or the 3D surfaces represent contours or surfaces of estimated response and the general nature of the system that arises as a result of a fitted model, not the true structure.

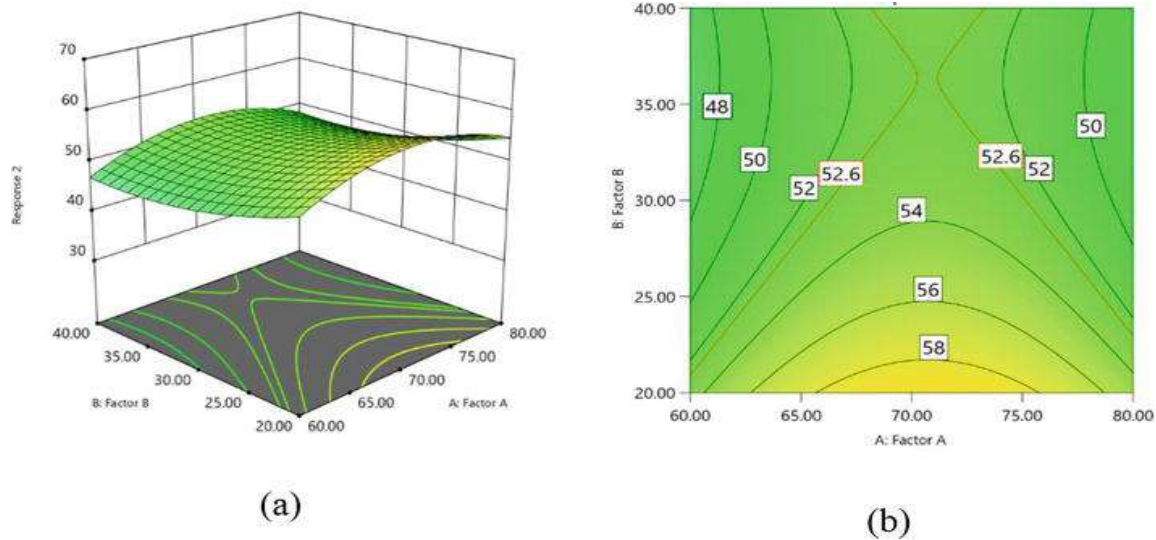


Table 2.3 Graphical presentation of 3D surface (a) and contour plot (b) where there is a saddle point (no maximum and no minimum).

2.5 Prediction and Determination of Optimal Operating Condition

Prediction of food quality attributes enables the researcher to estimate the response variable given the independent variables in the experimental region where no trials have been conducted. Prediction also helps in calculating the possible independent variables for a given response value. Apart from prediction, researchers are also interested in optimization which is an important step in statistical food process and product design. Optimization gives more detailed information about the level combinations of the independent variables that will result in optimum food quality attributes. This information from the optimization is reliable only if the model built is significant and adequately describes the relationship between the independent and the response variables.

In food and beverages, the researcher must often deal with multiple quality attributes (physicochemical properties and sensory attributes) as desirable responses. There are several aspects that complicate the process of choosing a best alternative when considering multiple attributes to the decision-making. There are almost no perfect practical decisions where it is possible to get the optimal result for each response or criterion in a single choice. Therefore, for most situations, it is necessary to make trade-offs between the different objectives among the quality attributes. As a result, optimizing based on multiple objectives should provide mechanisms for incorporating the experimenter's priorities. An optimum product may be achieved with different combinations of levels of the variables. The optimal levels of the

independent variables that give the 'best' product can be determined using numerical and graphical techniques.

Food processors and developers usually are interested in optimization of multiple responses simultaneously. A common approach to optimize multiple responses is to use simultaneous optimization technique which makes use of desirability function. The desirability function approach is one of the most widely used methods in industry for the optimization of multiple-response processes. The general approach is first to convert each response y_i into desirability function d_i that varies over the range 0 to 1. If the objective or target T for the response Y is a maximum value, the individual desirability functions are structured.

2.5.1 Graphical optimization

Graphical optimization is preferred when the process variables are few. In graphical optimization the contour plots for each response are superimposed to obtain an overlay plot. Shows an overlay plot for the two responses plots (contour plots) on Figure 7 below. These are contours for which desired values for response 3 ranges from 65 to 68 units and the desired values for response 4 ranges from 98 to 110 units. These ranges of the two responses were judged to be acceptable. If these ranges represent important attributes that must be met by the process, the shaded portion of the overlay plot indicates on Figure 7 that there are a number of combinations of factor A and factor B that result in a satisfactory process and a food product that meets the targeted objectives. The experimenter has the opportunity to visually examine the overlay plot to determine appropriate operating conditions, and select a region that is most desirable given other practical considerations are feasible. According to the overlay plot ranges of factor A and B that give best results are from about 61.5 to 69 and from 22 to 25 units, respectively.

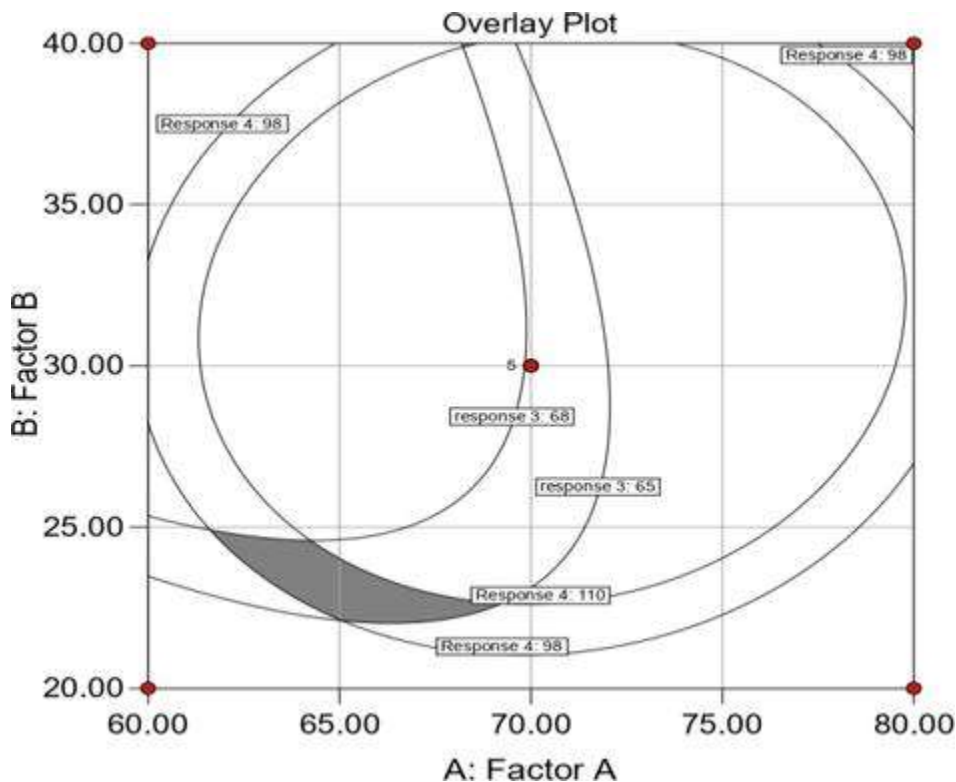


Figure 2.1: Graphical optimization

When there are more than three independent variables, overlaying (superimposing) contour plots becomes difficult because the contour plot is two dimensional, and $n - 2$ of the independent variables have to be held constant to construct the overlay plot. Often a lot of trial and error is required to determine which factors to hold constant and what levels to select to obtain the best view of the surface.

2.5.2 Numerical Optimization

The numerical method is most universal optimization approach. Though it cannot show overall information about the system, it performs complicated mathematical optimizations and gives specific combinations of levels of the independent variables that gives the best result. The minimum or maximum point of a second order equation is the point where the first derivative of the function is equated to zero.

Food processors and developers usually are interested in optimization of multiple responses simultaneously. A common approach to optimize multiple responses is to use simultaneous optimization technique which makes use of desirability function. The desirability function approach is one of the most widely used methods in industry for the optimization of multiple-response processes. The general approach is first to convert each response into desirability function that varies over the range 0 to 1. If the objective or target T for the response Y is a maximum value, the individual desirability functions are structure.

3.1 Food Application of Response Surface Methodology

3.1.1 Review Case study of Roasting, the Process and Variables

In coffee roasting, operation roasting time and temperature are critical parameters in terms of affecting the quality of roasted beans and the quality of the brewed coffee. In this case study roasting time ranging from 20 min to 40 min and roasting temperature ranging from 160–200°C were used. An experiment was designed using Design-expert. Central Composite Design (CCD) was used with the levels (low, middle, upper and star levels) as indicated in the table below and a total of 13 runs were generated. The response variables studied are acceptability tests in terms of color, aroma, flavor, taste and overall acceptability measured using a 9-point hedonic scale ranging from “1 = Dislike extremely” and “9 = like extremely”.

S/NO.	Factors	Coded Levels				
		-1.414(- ^a)	-1	0	1	+1.414(+ ^a)
1.	Temperature (°C)	151.7	160	180	200	208.28
2.	Time (min)	15.87	20	30	40	44.14

Table 3.1: Actual and Coded Values of Factor Levels.

3.1.2 Data analysis and interpretation of Response Surface

Polynomial equations were fitted to the data and response surfaces were generated for each response variable as presented in the. All the sensory attributes increase with increase in roasting time and temperature followed by a decrease in the sensory attributes of the brewed coffee as roasting time and temperature increased further. Such responses are naturally expected because short roasting time and low roasting temperature result in under-roasted beans which influence

the acceptability scores negatively. Similarly, longer roasting time combined with high roasting temperature may result in over-roasted beans which definitely affect the acceptability score of brewed coffee adversely.

3.2 Selected Applications of Response Surface Methodology (RSM) in the Process Modeling and Optimization

3.2.1 Application of RSM in Optimizing Baked Products

Baking is a method of preparing food that uses dry heat, typically in an oven. Several studies have been conducted to optimize the baking conditions including baking temperature and time and the ingredients used to come up an acceptable product. Some of the studies include the effect of inulin on textural and sensory characteristics of sorghum based high fiber biscuits using response surface methodology, effect of different ingredients on the mixing and fermentation times required, the effect of the interaction of red rice flour and the microbial transglutaminase enzyme in the production of prebiotic gluten-free breads, the effect of hydroxypropylmethyl cellulose, yeast β -glucan, and whey protein levels, the effect of whole oat flour, maltodextrin and isomalt on textural and sensory characteristics of biscuits, optimization of composite flour biscuits, gluten-free bread fortified with soy flour and dry milk, enzymatic treatment using RSM on the quality of bread flour and many others have been reported in literature. These and other studies on baking process used RSM to model and optimize baking processes. Full factorial designs, CCD, BBD were among the experimental designs used.

3.2.2 Application of RSM in Optimization of Cooking and Roasting Parameters

Thermal processes like cooking and roasting are commonly used food processing unit operations. Critically important in these operations is finding the optimal combinations of the operating conditions. RSM has been extensively and successfully used to optimize the process parameters. Some of such studies are optimization of cooking protocol for rice bean to improve the efficiency of conventional process, of high-pressure processing of black tiger shrimp (*Penaeus monodon*), process optimization for high-pressure processing of black tiger shrimp (*Penaeus monodon*), development of sensory acceptable, low-salt, shelf-stable frankfurters, optimization of the effect of frying temperature, and frying time on some physicochemical, textural, and sensory properties of wheat chips, optimization of initial water content, saturated steam pressure and processing time for roasted chick pea, optimization of microwave roasting of peanuts, optimization of leavened dough sunflower oil frying process conditions, optimization of roasting time and temperature of coffee beans have been studied are reported. RSM experimental designs and numerical and graphical optimization were used in the studies.

3.3 Application of RSM in Formulation of Products

One important area in food processing is development of new formulation for products using various ingredients. Deciding on the relative proportion of the ingredients calls for application of scientific procedure or experimental design. RSM has been effectively used to optimize ingredients or raw materials. Such studies include wheat dextrin yoghurt formulation, fat-reduced ice cream formulation employing inulin as fat replacer, the effect of Homogenized Infant Foods, optimization of honey, vinegar and tomato powder to make sweet and sour chicken meat spread, optimization of inulin, cocoa powder, and sucrose to develop a dessert made with

soymilk, optimization of a stable palatable oil-in-water emulsion made with soy protein and red guava juice and optimization of soy protein and pink guava juice to develop soy-based desserts. Special designs for formulation studies called mixture designs have been widely used together with RSM to deal with food formulation related problems. Some studies reported are optimization of pasteurized milk with soymilk powder and mulberry leaf tea using user defined mixture design, optimization of diverse chloride salts on the growth parameters of *L. pentosus* using D-optimal mixture design, optimization of natural fermentative medium for selenium-enriched yeast by D-optimal mixture design, optimization of wheat, sprouted mung bean and sorghum composite flour bread using D-optimal mixture design, optimization of blending ration of three different fruits in jam making using augmented simplex mixture design, optimization of sugar, peanut and chocolate using constrained mixture design to develop chocolate peanut spread and formulation of yoghurt using augmented simplex-centroid mixture design, were reported in literature.

3.4 Application in RSM in Drying, Extrusion and Fermentation Process

Some common operations in the food industry like extrusion, drying, fermentation etc. have been successfully modeled and optimized. Development of functional yoghurt via soluble fiber fortification, optimization of ripening temperature, ripening time, the level of rennet on the quality of cheese, bleaching condition on soya-bean oil has been performed using RSM. The effect of stevia and inulin on physicochemical and rheological properties of mango nectar, optimization of high-pressure process to extend shelf life of apple juice, Optimizing the thermal assisted high-pressure process parameters for a sugarcane based mixed beverage, optimization of ultrasonication parameters on chemical and microbiological properties of sour cherry juice has been done using RSM, Some examples of optimization fermentation process using RSM are the effect of fermentation conditions on the phytochemical composition, sensory qualities and antioxidant activity of green tea infusion, the effect of steaming time (20–50 min) and fermentation time (12–48 hr). Drying operations have also been modeled and optimized using RSM which included optimization of drying conditions on the quality of fruit cubes, the effect of spray drying condition on the quality of apricot juice powder the effect of hot air and microwave drying condition.

Extrusion is a versatile operation in the production of wide range of extruded products and extraction. Extrusion conditions like temperature, feed moisture content and screw speed are the dominate parameters and are extensively studies. Studies in extrusion using RSM included optimization extrusion conditions of soybean flour and achda, anti-nutritional factors and protein and starch digestibility of lentil splits optimization of carrot pomace powder, and physical and functional properties of extruded snack foods. The effect of banana peel and rice bran extrusion for value addition has also reported. Optimization of extraction conditions for extraction of olive oil, flavonoids from shallot skin, and phenolic compounds from fruits using RSM have also been reported.

4.1 Conclusion

Response surface methodology (RSM) has been extensively and effectively used to model and optimize food processes. It is important to follow the steps and use proper experimental designs in order to obtain valid results. Modeling and Optimization of both processing conditions and of ingredients in food formulations has been done widely by applying RSM. The advancement in

statistical packages to design experiments and analyze data has contributed immensely in statistical and computer-aided food product design.

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