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Utilization of response surface methodology for development and optimization of effervescent tablets from kokum (*Garcinia indica* Choisy) fruit

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Abstract
In this study, the objective was to develop and optimize effervescent tablets made from kokum fruit using response surface methodology, with the intention of creating a fizzy drink. Sodium bicarbonate and citric acid were chosen as independent variables. The tablets were prepared through dry granulation, and various parameters such as tablet friability, pH of the effervescent solution, effervescent time, and palatability were evaluated as dependent variables. The optimized formulation consisted of 300 mg of kokum fruit powder, 239.36 mg of sodium bicarbonate, and 100 mg of citric acid, resulting in a high-quality effervescent kokum fruit drink. The experimental values of the optimal formulation closely matched the predicted values for friability (0.090 and 0.10±0.006 %), pH (7.911 and 7.62±0.03), effervescence time (144.590 and 135±3.00 s), and palatability score (5.838 and 7.03±0.06). This study demonstrates the significant potential for future development of effervescent kokum tablets and their potential for commercialization.

Keywords: Kokum, response surface methodology, effervescent tablet, fizzy drink

1. Introduction
Kokum (*Garcinia indica* Choisy) is a valuable tree species found in Western Ghats of India. Among the 36 reported species of the *Garcinia* genus in India, *G. indica* stands out as the most significant. Kokum possesses exceptional potential as a spice, colorant, and medicinal plant. Recognized by the National Medicinal Plant Board (NMPB) as an important species for promotion and development, *G. indica* holds great promise (Ananthakrishnan and Rameshkumar, 2017) [1].

The chemical composition of kokum fruit makes it a promising raw material for industrial utilization. It has high moisture content (80%) and contains protein (1%), tannin (1.7%), pectin (0.9%), total sugars (4.1%), and fat (1.4%). Notably, kokum fruit exhibits the highest concentration of anthocyanins (2.4 g/100 g) compared to other natural sources (Nayak *et al.*, 2010) [10]. The presence of (-)-Hydroxycitric acid [(-)-HCA] in kokum leaves and rinds contributes to its antioxidant properties. Kokum rind contains approximately 20-30% of (-)-HCA on a dry weight basis (Swami *et al.*, 2014) [15]. Hydroxycitric acid consumption has been linked to reduced appetite, inhibition of fat synthesis, decreased food intake, and weight loss (Jena *et al.*, 2002) [8]. It is commonly used in curries to provide a sweet-tangy taste and acidic flavor as a substitute for tamarind, as well as for preparing syrups (Jayaprakasha and Sakariah, 2000) [7]. The amsol, when strained in water, is boiled to make a soup called solkhadi. Despite the nutritional and therapeutic value of kokum, the range of products traditionally derived from its fruit rind remains limited. Therefore, there is a need to explore the untapped potential of kokum fruits to create new processed products with high therapeutic value. There is a growing global demand for innovative food products that are nutritious and offer delicate flavours.

Presently, synthetic carbonated beverages dominate the market, providing no nutritional value to consumers. Fruit-based beverages, on the other hand, offer both nutrition and health benefits. However, their short shelf life due to high moisture content poses a challenge. Fruit powders, on the other hand, present several advantages over liquid counterparts. They are less bulky but highly hygroscopic, requiring careful handling. Alternatively, fruit powders can be transformed into "fruit drink tablets" through compaction. Food tableting, a relatively recent technique gaining attention in the food industry, involves creating solid unit doses containing one or more active ingredients. Tablets are the most popular and widely used form of drug

administration and can be prepared from fruit powders, granules, pellets, or multiple film-coated units. Fruit powders are typically obtained through drying techniques such as tray, spray, freeze, drum, foam mat, and vacuum freeze-drying. The processing of fruit powders into effervescent tablets offers numerous advantages over regular fruit powder tablets, including enhanced physical and chemical stability, an elegant appearance, rapid dissolution, controlled release of active ingredients, and convenience during storage, transportation, and consumption (Yusof *et al.*, 2011) [17].

2. Material and Methods

2.1 Kokum fruit powder

Ripened kokum fruits were collected from Horticultural farm, Terekanahalli, Department of Horticulture, Government of Karnataka, Sirsi, Uttara Kannada district, Karnataka. Fruits were washed thoroughly in clean water to remove dirt and then surface dried. Later, the fruits were cut in to two halves and seeds were separated. The rind was dried in electric tray drier at 55 °C to constant moisture. The dried rind was pulverised to obtain fine powder.

2.2 Experimental design

The experiment was laid out using Response Surface Methodology (RSM). Design expert 8.0.6 software was used to develop the effervescent formulation and to evaluate the effect of independent variables on dependent variables. Independent variables namely, sodium bi carbonate and citric acid and dependent variables such as friability of tablet, pH of effervescent solution, effervescent time and palatability were taken in to account in the experimental design.

2.3 Preparation of kokum fruit effervescent tablets

Tablets were prepared by using specified quantity of kokum fruit powder, effervescent combination and other additives (Table 1). Known weight of powdered ingredients *viz.*, kokum fruit powder, citric acid, sodium bicarbonate, sucrose and sodium starch glycolate passed through sieve no. 60, collected in mortar and homogenized. Cumin extract, black pepper extract and freshly prepared starch paste (10% w/v) were added to the homogenised powder blend and kneaded to impart adhesiveness to the powder blend. The dough was passed through sieve no. 10 and then dried in the hot air oven at 50 °C for about 6h to retain with 5% w/v moisture. The dried granules were milled and passed through sieve no. 18 *versus* sieve no.44. The granules passed through sieve no.18 and retained over 44 were collected. About 10 percent of the fines (passed through sieve no. 44) were also added to the above granules. Finally, the granules were lubricated using magnesium stearate for about five min. Then, lubricated granules were compressed using 16mm flat-faced punches on tablet punching machine. The prepared tablet formulations were packed in PVC vails and stored in stability chamber for further studies

2.4 Pre-compression analysis

2.4.1 Bulk density (g/cc)

The bulk density of the samples was determined using glass cylinder technique. The bulk density was calculated from the ratio of the mass of powder to its volume.

2.4.2 Tapped density (g/cc)

The tapped density of the sample was measured by placing a known quantity of powder sample in a graduated measuring glass cylinder and the tapped volume was measured after the sample was gently tapped 100 times onto a rubber mat. Subsequently, the tapped density was calculated by dividing the weight of the powder by the tapped volume.

2.4.3 Angle of repose (°)

The angle of repose of powder was determined by the funnel method. The angle of repose value < 25 indicates excellent flow, 25-30 good, 30-40 satisfactory and > 40 very poor flow of powder.

2.4.4 Hausner ratio (HR) and Carr index (CI) (%)

The flow property of samples was determined using Carr Index (CI) and the Hausner Ratio (HR). These calculated from the bulk density (Bd) and tapped density (Td) as follows:

$$CI = \left[\frac{Td - Bd}{Td}\right] \times 100$$

$$HR = Td \div Bd$$

2.5 Post-compression analysis

2.5.1 Friability (%)

Tablet strength was measured by its friability. 10 tablets were accurately weighed and placed in a plastic chamber that revolves at 25 rpm, with each revolution dropping the tablets at a distance of six inches. The tablets were dusted and reweighed after 4 min, and the percentage loss in weight of tablet was determined. Compressed tablets with loss less than one percent of their weight are generally considered acceptable

2.5.2 pH of effervescent beverage

pH of effervescent beverage was determined using pH meter (make systronics) by dissolving one gram of effervescent tablet in 100 mL of distilled water.

2.5.3 Effervescence time (s)

One kokum fruit effervescent tablet was placed in 50 mL of distilled water. The time taken for complete dissolution was expressed in seconds.

2.5.4 Palatability

The palatability of kokum fruit effervescent tablet beverages were prepared according to the formulation and assessed on a 9 point hedonic scale by 15 semi-trained judges whose age varied from 23 to 57 years. One kokum fruit effervescent tablet was dissolved in 50 mL water to make a beverage. The critical attributes *viz*., colour and appearance, flavour, taste, after taste, residue left in mouth, fizzing sensation, foaming sensation and overall acceptability were measured. A maximum rating reflected good quality attributes (dislike very much – like very much).

2.5.6 Statistical analysis

The data was analysed by one way analysis of variance

(*ANOVA*) using Design Expert version 8.0.6 software. The level of significance was expressed at $p<0.05$.

3. Results and Discussion

3.1 Pre-compression flow properties

Mixing of effervescent agents with fruit powders significantly increases their bulk density and tapped density. Effervescent agents cause a widening of the particle size, resulting in a wide range of particle size distribution occurring in the fruit powder mixture. Thus, the void space between the large particles is filled by the very small particles, which results in an increased density (Saifullah *et al.*, 2016) [13]. The bulk density and tapped density of pre-formulations had a range from 0.60±0.001 g cc^{-1} to 0.72±0.004 g cc^{-1} and 0.69±0.02 g cc^{-1} to 0.81±0.03 g cc^{-1}, respectively (Table 2).

Flowability is measured by the angle of repose, Carr's index and Hausner's ratio. According to Carr (1965) [2] and Hausner (1967) [4], all powder mixtures should have at least a passable flow characteristic. Angle of repose of nine pre-formulations was ranged from 30.38±0.16 to 37.37±0.14°. The Carr's index and Hausner ratio ranged from 9.55±0.63 percent to 12.27±1.46 percent and 1.03±0.13 to 1.05±0.15, respectively indicating excellent to good flow properties. The Carr index and Hausner's ratio slightly increase upon adding effervescent agents to fruit powder. Addition of fruit powder with an effervescent agent decreases flowability (Saifullah *et al.*, 2016) [13].

3.2 Effect of independent variables on dependent variables of effervescent kokum tablet

3.2.1 Effect of independent variables on friability

Experimental results showed that the friability of all the nine preliminary effervescent kokum tablet formulations was within one percent limit. The friability of preliminary effervescent kokum tablet formulations had a range from 0.08 percent to 0.96 percent (Table 3). Among the formulations F_9 and F_1 recorded the least friability of 0.08 and 0.09 percent, respectively, whereas the highest friability was recorded in F_2 (0.96%) followed by F_4 (0.72%). Friability of tablets was found to be low with formulations having high sodium bicarbonate and low citric acid content. This may be due to binding capacity of sodium bicarbonate (Venkatachalam *et al.* (2017) [16]. Both sodium bicarbonate and low citric acid content have significant effect on friability of effervescent tablet which is evident from polynomial equation. *ANOVA* for friability of effervescent tablet is presented in Table 4 indicated that the model F-value of 3.01 implies the model was not significant relative to the noise. There was 19.65 percent chance that an F-value in this large could occur due to noise. A negative predicted R^2 value (-0.7347) implies that the overall mean may be a better predictor of the response than the current model. In this study, the regression analysis of response was conducted by fitting a suitable quadratic model for response variable (friability) to assess how well the model represented the data. Adequacy of precision value (4.8439) indicates an adequate signal. This model can be used to navigate the design space. The best fit model, in terms of coded factors, is shown as below using a polynomial equation to make prediction about the response.

$$\text{Friability} = 0.5811 + 0.0783\ A + 0.1117\ B + 0.1475\ AB + 0.2183\ A^2 - 0.5017\ B^2 \text{ ------ (1)}$$

3.2.2 Effect of independent variables on pH value

The pH value influences on palatability of the tablet. The pH value for optimum product should be between 5.00 and 7.00 (Jayani *et al.*, 2021) [6]. The pH of effervescent tablet had a range from 6.90 to 10.50. Formulation F_2 recorded lowest pH value of 6.90 followed by F_7 (7.00) and F_1 (7.50) whereas, the highest pH value was recorded in F_8 (10.50) followed by F_6 (10.20) (Table 3). Both sodium bicarbonate and citric acid content have influence on pH value of tablet. The presence of more acids reduces the pH and increases the acidity, while the presence of more base material increases the pH and leads to alkalinity (Sun *et al.*, 2019) [14]. *ANOVA* for pH value of effervescent tablet is presented in Table 5 indicates that the model F-value of 1.25 implies the model is not significant. There is a 38.38 percent chance that an F-value of this large could occur due to noise. A negative Predicted R^2 (-1.7696) implies that the overall mean may be a better predictor of response than the current model. Two factor interaction (2FI) model is suggested to make predictions for the response. The estimated regression coefficients of the 2FI polynomial model are given in Table 5. The best fit model, in terms of coded factors, is shown as below using a polynomial equation to make prediction about the pH value of tablet for optimization.

$$\text{pH} = 8.91 + 0.7500\ A + 0.0000\ B - 0.9000\ AB \text{ ------ (2)}$$

3.2.3 Effect of independent variables on effervescent time

Dissolution in water is an important property for using tablets as ready-to-serve juices and refreshment drinks. From the consumer point of view, rapid, easy, and complete dissolution is an indication of good quality food tablet (Marabi *et al.*, 2007) [9]. The time taken for complete effervescence or dissolution in distilled water was in the range from 36 seconds to 294 seconds. The minimum time taken for complete effervescence or dissolution was recorded in F_7 (36 s) followed by F_9 (47 s) (Table 3). Interaction effect of citric acid and sodium bicarbonate significantly influences the effervescence time of tablet. Increase in acid causes faster reaction between acid and carbonic compound resulting in quick dissolution (Hla and Khaing (2014) [5]. The dissolution time decreased with an increase in the concentration of effervescent agent (Ong *et al*., 2014) [14]

ANOVA on time taken by effervescent tablet for complete effervescence or dissolution in distilled water is presented in Table 36 indicates that the model F-value of 5.32 implies the model was not significant relative to the noise. There is a 9.98 percent chance that an F-value of this large could occur due to noise. The model term A (Sodium bicarbonate) was found to be significant as the *p* value was less than 0.05 (0.0307). A negative predicted R^2 value (-0.1468) implies that the overall mean may be a better predictor of response than the current model. The quadratic model is suggested to predict the response. Adequacy of precision (5.6487) indicates an adequate signal. This model can be used to navigate the design space. The estimated regression coefficients of the quadratic polynomial model are given in Table 6. The best fit model, in terms of coded factors, is shown as below using a polynomial equation to make prediction about the effervescence time of tablet for optimization.

$$\text{Effervescence time} = 77.31 - 81.67\ A - 3.82\ B + 33.75\ AB + 106.03\ A^2 - 45.52\ B^2 \text{ ------- (3)}$$

3.2.4 Effect of independent variables on palatability

Palatability of effervescent tablet is an overall acceptance of product by sensory characters. The palatability score for effervescent beverage was ranged from 4.00 to 7.00. The formulations F_5 and F_9 recorded least score of 4.00 for palatability followed by F_6 (4.25) whereas, maximum score was recorded for F_2 (7.00) followed by F_7 and F_8 (6.90 each) (Table 3). The decrease in sodium bicarbonate content and increase in citric acid content would increase the score for palatability. Thus the concentrations of sodium bicarbonate and citric acid have bearing on palatability of effervescent tablet (Cornelia and Oktavia, 2021) [3]. *ANOVA* on palatability of beverage from effervescent kokum tablet is presented in Table 7 indicates that the model F-value of 11.88 and p-value (0.0082) implies the model was found to be significant. There is only a 0.82 percent chance that an F-value this large could occur due to noise. Model term A (Sodium bicarbonate) was found to be significant. The predicted R^2 value of 0.5192 was not as close to the adjusted R^2 value of 0.7311 i.*e.*, the difference was more than 0.2. This may indicate a large block effect. The linear model was suggested to predict the response. Adequacy of precision (8.718) indicates an adequate signal. This model could be used to navigate the design space. The estimated regression coefficients of the linear polynomial model are given in Table 7. The best fit model, in terms of coded factors, is shown as below using a polynomial equation to make prediction about the response *i.e.*, palatability of tablet for optimization of the formulation.

Palatability = 5.46 - 1.40 A + 0.4667 B ------- (4)

3.3 Check points and desirability for optimization of formulation

The aim of this investigation was to optimize the formulation with minimum friability, pH and effervescence time and maximum palatability. The desirability function was generated after limiting the preferred goal of parameters and responses to obtain highly desired formulation (Table 8). Accordingly, the predicted optimum formulation with highest desirability value (0.660) was obtained as 239.36 mg sodium bicarbonate and 100 mg citric acid and thus selected for numerical optimization.

3.4 Verification of model for optimization of formulation

The predicted optimal formulation was prepared and analysed for the responses (Table 9). The experimental values of most desired optimal formulation was found to be very close to the predicted values pertaining to friability (0.090 and 0.10±0.006 %), pH (7.911 and 7.62±0.03), effervescence time (144.590 and 135±3.00 s) and palatability score (5.838 and 7.03±0.06) which indicates that the response surface methodology can be used for optimization of formulation (Rajamma *et al.*, 2014) [12].

Table 1: Formulations of kokum fruit effervescent tablets

Ingredients	Formulations								
	F_1	**F_2**	**F_3**	**F_4**	**F_5**	**F_6**	**F_7**	**F_8**	**F_9**
Kokum powder (mg)	300	300	300	300	300	300	300	300	300
Sucrose (mg)	216	216	216	216	216	216	216	216	216
Sodium bicarbonate (mg)	200	200	300	400	300	400	300	200	400
Citric acid (mg)	100	150	150	150	100	150	200	200	100
Starch paste (mg)	60	60	60	60	60	60	60	60	60
Cumin extract (drops)	1	1	1	1	1	1	1	1	1
Black pepper extract (drops)	1	1	1	1	1	1	1	1	1
Magnesium stearate (mg)	24	24	24	24	24	24	24	24	24
Sodium starch glycolate (mg)	250	250	250	250	250	250	250	250	250

Table 2: Pre-compression flow properties of kokum fruit effervescent tablet formulations mixture

Formulation	Bulk density (g/cc)	Tapped density (g/cc)	Angle of repose (°)	Carr's Index (%)	Hausner ratio	Flow ability
F_1	0.64±0.01	0.72±0.01	36.38±0.60	10.90±0.32	1.05±0.13	Good /free flow
F_2	0.72±0.04	0.81±0.03	34.71±0.51	10.47±1.25	1.03±0.13	Good /free flow
F_3	0.60±0.01	0.69±0.02	30.38±0.16	12.27±1.46	1.05±0.15	Good /free flow
F_4	0.67±0.01	0.76±0.01	31.85±0.28	10.90±0.95	1.05±0.13	Good /free flow
F_5	0.63±0.02	0.71±0.03	31.47±0.03	11.64±0.11	1.05±0.14	Good /free flow
F_6	0.66±0.03	0.73±0.04	36.39±0.55	9.55±0.63	1.04±0.12	Excellent
F_7	0.71±0.02	0.79±0.02	33.88±0.60	10.97±0.62	1.05±0.13	Good /free flow
F_8	0.68±0.02	0.76±0.03	37.37±0.14	10.77±1.50	1.05±0.13	Good /free flow
F_9	0.68±0.02	0.76±0.02	34.31±0.26	10.91±1.54	1.04±0.14	Good /free flow

Values are mean of three replications ± Standard Deviation

Table 3: Effect of independent variables on dependent variables of kokum fruit effervescent tablet formulations

Formulations	Independent variables		Dependent variables			
	Sodium bicarbonate (mg)	Citric acid (mg)	Friability (%)	pH	Effervescent time (s)	Palatability (out of 9.0)
F_1	200	100	0.09	7.50	253	6.50
F_2	200	150	0.96	6.90	294	7.00
F_3	300	150	0.42	9.50	103	5.80
F_4	400	150	0.72	9.60	103	3.75
F_5	300	100	0.14	9.40	109	4.00
F_6	400	100	0.11	10.20	64	4.25
F_7	300	200	0.18	7.00	36	6.90
F_8	200	200	0.11	10.50	157	6.90
F_9	400	200	0.08	9.60	47	4.00

Table 4: *ANOVA* for friability of effervescent kokum tablet

Variance source	Sum of squares	Degree of freedom	Mean square	F value	p-value
Model	0.7973	5	0.1595	3.01	0.1965
A (Sodium bicarbonate)	0.0368	1	0.0368	0.69	0.4655
B (Citric acid)	0.0748	1	0.0748	1.41	0.3200
AB	0.0870	1	0.0870	1.64	0.2899
A^2	0.0953	1	0.0953	1.80	0.2721
B^2	0.5033	1	0.5033	9.51	0.0540
Residual	0.1588	3	0.0529		
Cor Total	0.9562	8			
Std. Dev.	0.2301				
Mean	0.3922				
C.V.%	58.66				
R^2	0.8339				
Adjusted R^2	0.5571				
Predicted R^2	-0.7347				
Adeq Precision	4.8439				
PRESS	1.66				

$p<0.05$ Significant

Table 5: *ANOVA* for pH of effervescent kokum tablet

Variance source	Sum of squares	Degree of freedom	Mean square	F value	p-value
Model	6.61	3	2.20	1.25	0.3838
A	3.37	1	3.37	1.92	0.2246
B	0.00	1	0.00	0.00	1.0000
AB	3.24	1	3.24	1.84	0.2327
Residual	8.79	5	1.76		
Cor Total	15.41	8			
Std. Dev.	1.33				
Mean	8.91				
C.V.%	14.88				
R^2	0.4293				
Adjusted R^2	0.0869				
Predicted R^2	-1.7696				
Adeq Precision	3.7325				
PRESS	42.68				

$p<0.05$ Significant

Table 6: *ANOVA* for effervescence time of effervescent kokum tablet

Variance source	Sum of squares	Degree of freedom	Mean square	F value	p-value
Model	71289.99	5	14258.00	5.32	0.0998
A	40016.67	1	40016.67	14.93	0.0307
B	87.40	1	87.40	0.0326	0.8682
AB	4556.25	1	4556.25	1.70	0.2833
A^2	22486.14	1	22486.14	8.39	0.0627
B^2	4143.53	1	4143.53	1.55	0.3021
Residual	8041.15	3	2680.38		
Cor Total	79331.14	8			
Std. Dev.	51.77				
Mean	117.66				
C.V.%	44.00				
R^2	0.8986				

Adjusted R^2	0.7297				
Predicted R^2	-0.1468				
Adeq Precision	5.6487				
PRESS	90975.67				

$p<0.05$ Significant

Table 7: *ANOVA* for palatability of effervescent kokum tablet

Variance source	Sum of squares	Degree of freedom	Mean square	F value	p-value
Model	13.07	2	6.53	11.88	0.0082
A	11.76	1	11.76	21.38	0.0036
B	1.31	1	1.31	2.38	0.1742
Residual	3.30	6	0.5501		
Cor Total	16.37	8			
Std. Dev.	0.7417				
Mean	5.46				
C.V.%	13.59				
R^2	0.7983				
Adjusted R^2	0.7311				
Predicted R^2	0.5192				
Adeq Precision	8.7185				
PRESS	7.87				

$p<0.05$ Significant

Table 8: Check points and goal for optimization of effervescent kokum tablet formulation

Particulars	Goal	Lower limit	Upper limit
Sodium bicarbonate (mg)	Is in range	200	400
Citric acid (mg)	Is in range	100	200
Friability (%)	Minimize	0.09	0.96
pH	Minimize	6.90	10.50
Effervescence time (s)	Minimize	36	294
Palatability (out of 9.0)	Maximize	3.75	7.00

Table 9: Comparative values of the predicted and observed responses

Formulation	Predicted value	Experimental value*
Friability (%)	0.090	0.10±0.006
pH	7.911	7.62±0.03
Effervescence time (s)	144.590	135±3.00
Palatability (out of 9.0)	5.838	7.03±0.06

*****Values are mean of three replications ± standard deviation

4. Conclusion

Response Surface Methodology (RSM) proved to be a valuable tool for the development and optimization of kokum fruit effervescent tablet formulations. The exploration of response surfaces revealed complex interactions among variables such as friability, pH, effervescence time, and palatability. The palatability of the kokum fruit effervescent fizzy drink was found to be significantly influenced by the content of sodium bicarbonate and citric acid ($p<0.05$). The developed models successfully predicted the friability, pH, effervescence time, and palatability of the effervescent tablets, providing a deeper understanding of the combined effects required to achieve a high-quality drink. The optimal formulation consisted of 300 mg of kokum fruit powder, 239.36 mg of sodium bicarbonate, 100 mg of citric acid, 216 mg of sucrose, 60 mg of starch paste, one drop each of cumin and black pepper extract, 24 mg of magnesium stearate, and 250 mg of sodium starch glycolate, resulting in a good-quality effervescent kokum fruit drink.

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