Optimization of process variables for instant preparation of *neyyappam* like product and extension of shelf-life

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Abstract *Neyyappam* is a very popular traditional sweet of Kerala and Tamilnadu. It is prepared from soaked rice (10–12 h) and had limited shelf-life. Response surface methodology was adopted to optimize, the levels of variables i.e. jaggery, baking powder and holding time based on quantity of rice with selected responses i.e. hardness, bulk density and overall acceptability. All the three variables i.e. jaggery, baking powder and holding time had negative effect ($p \leq 0.05$) on hardness at linear level. Bulk density was also negatively affected ($p \leq 0.05$) by jaggery and baking powder. However, the overall acceptability was positively affected ($p \leq 0.05$) by jaggery and baking powder. It was recommended to use jaggery 90 g/100 g rice flour, baking powder 1.5 g/100 g rice flour and holding time of 13.0 min. Optimized samples were packed in polyethylene pouches, heat treated in a cabinet dryer at 90°C for 2 h, stored under ambient temperature conditions along with control and monitored for peroxide value, free fatty acids and thiobarbutyric acid values as well as microbiological, sensory and textural changes. It was observed that control samples had visible yeast and mould growth at 3rd day, while the treated ones remained highly acceptable up to 30 days with respect to chemical, textural, sensory and microbiological changes.

Keywords *Neyyappam* · Sodium bi-carbonate · Texture · Bulk density

In a manner similar to fermentation of wheat dough for making bread, rice batter along with blackgram is leavened by natural fermentation and dishes such as *idali* and *dosa* are prepared. Traditional method i.e. soaking and fermentation takes more than 20 h, however, with the help of chemical leavening agent the product can be prepared in shorter time (15–20 min) with the same taste and texture. Chemically leavened *idali* (Susheelamma and Rao 1974) and *dhokla* (Mahajan and Chattopadhyay 2000) have been prepared by using sodium bicarbonate and glucono-delta-lactone, respectively. Mahajan and Chattopadhyay (2000) reported that good quality *dhokla* can be prepared with rice semolina, bengal gram semolina and black gram flour in the ratio of 45:45:10, sodium bicarbonate (1.86 g/100 g dry mix), citric acid (1.28 g/100 g dry mix), sugar (4%) with soaking dry mix at room temperature for 15 min followed by steaming for 20 min.

Neyyappam is fried sweet product made from rice flour, jaggery and coconut milk and a popular traditional sweet dish of Kerala and Tamilnadu. It is commonly prepared by deep-frying the batter in hemi-spherical shape pan and is brown or dark brown in colour. Traditionally, the rice is soaked overnight (10–12 h), ground to paste, fermented (4–6 h) and then batter is deep-fried in hemispherical shape pan. Traditional method takes almost 16–18 h for preparation and shelf-life of the product is also limited up to only 2–3 days at ambient temperature conditions. There is a need to reduce the preparation time of this popular sweet dish and to optimize the processing variables as well as to extend its shelf-life under ambient conditions. Therefore, the present study was planned with objectives, to reduce the processing time for the preparation of *neyyappam* like

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product using chemical leavening agent as well as extension of its shelf-life at ambient conditions.

Material and methods

Rice flour, jaggery, cardamom powder, maida flour, citric acid, baking powder, dry ginger powder and sunflower oil were procured from local market.

Experimental design Central composite rotatable design was used for selecting combination levels of variables in each experiment. The variables used were, level of jaggery (70–90 g), baking powder (0.50–1.50) per 100 g rice flour and holding time 10–20 min. The actual values of the variables for each experiment along with the measured response value have been presented in Table 1. For the analysis of experimental design by the RSM, it was assumed that n -mathematical functions, f_k ($k=1, 2, \dots, n$), Y_k in terms of m independent processing factors X_i ($i=1, 2, \dots, m$) existed for each response variable.

$$Y_k = f_k(X_1, X_2, \dots, X_m)$$

In this case $n = 3$ and $m = 3$

$$Y_k = b_{k0} + \sum_{i=1}^3 b_{kii} X_i + \sum_{i=1}^3 b_{kii} X_i^2 + \sum_{i=1}^3 \sum_{j=i+1}^3 b_{kij} X_i X_j$$

The function was assumed to be approximated by a second order polynomial where, b_{k0} is the value of the fitted response at the centre point of the design i.e. point (0,0) and b_{ki} , b_{kii} and b_{kij} are the linear, quadratic and interactive regression terms, respectively.

Preparation of jaggery syrup 45 ml of water was added to 90 g jaggery and kept on flame until it reached at liquid consistency followed by filtered through muslin cloth to remove the impurities and boiled with continuous stirring till 62° brix.

Preparation of batter Ingredients i.e. rice flour 100 g, maida flour 15 g, cardamom powder 0.5 g and dry ginger 2 g were mixed thoroughly in previously prepared jaggery syrup with 45 ml water. Citric acid was also added and the amount varied with the amount of baking powder (exp 1–20, Table 1) and was calculated as: amount of citric acid = 0.688 x baking powder (Mahajan and Chattopadhyay 2000). Baking powder was added just before the frying to avoid the loss of gases. The batter was held at room temperature for different periods (Table 1 exp 1–20).

Frying of batter Batter (40 g) was fried in molded pan using sunflower oil at 160±5 °C for 5 min.

Table 1 Experimental combinations and actual response values

Exp Nr	Jaggery, g/100 g rice flour	Baking powder, g/100 g rice flour	Holding time, min	Hardness, N	Bulk density, g/cc	OAA
1	70.00	0.50	10.00	9.5	0.96	7.4
2	90.00	0.50	10.00	7.9	0.76	8.0
3	70.00	1.50	10.00	8.2	0.88	7.5
4	90.00	1.50	10.00	7.0	0.72	8.0
5	70.00	0.50	20.00	8.3	0.88	7.2
6	90.00	0.50	20.00	6.90*	0.70	8.1
7	70.00	1.50	20.00	8.0	0.84	7.6
8	90.00	1.50	20.00	6.5	0.66	8.3
9	63.18	1.00	15.00	9.1	0.95	7.3
10	96.82	1.00	15.00	6.1	0.54*	8.5**
11	80.00	0.16	15.00	8.1	0.96	7.7
12	80.00	1.84	15.00	6.8	0.60	8.3
13	80.00	1.00	6.59	10.4**	1.04**	7.0*
14	80.00	1.00	23.41	9.1	0.92	7.2
15	80.00	1.00	15.00	8.1	0.83	7.7
16	80.00	1.00	15.00	8.0	0.84	7.6
17	80.00	1.00	15.00	8.0	0.82	7.8
18	80.00	1.00	15.00	8.0	0.83	7.8
19	80.00	1.00	15.00	7.9	0.82	7.8
20	80.00	1.00	15.00	8.1	0.82	7.8

OAA overall acceptability, *minimum, **maximum

Heat treatment Four fried *neyyappams* were packed in polyethylene pouches (75 μ), heated in cabinet tray drier at 90 °C for 2 h (pre trial basis) and stored with control at ambient temperature conditions for further study.

Chemical analysis Moisture, total protein, crude fat and ash were determined using AOAC (1997) methods. The carbohydrate content of the samples was calculated by difference. Changes in chemical parameters in the form of peroxide value and free fatty acids were monitored by methods of AOCS (1990). Thiobarbutyric acid value was estimated by the distillation method of Tarladgis et al. (1960). Microbial profile was determined using the Petri plate method of APHA (1992). All the chemicals used in this investigation were of analytical grade and procured from M/s E-Merck, Mumbai, India.

Texture analysis Firmness of the product was determined using TA plus texture analyzer (Model 01/TALS/LXE/UK LLOYD Instruments, Hampshire, UK). TA plus with 50 N load cell connected with 2.5 in. cutting blade probe (warmer bratzler) was used for the study. The blade probe with test speed of 60 mm/min was allowed to penetrate. The radial centre of the product up to 20 mm diameter resulted in cutting action. The trigger of the test was set to 10 gf with depression limit of 20 mm.

Bulk density The face and edge of the samples were cut and rest of the inner core was considered as cube and weight and volume were measured. Volume of the cubes was calculated as the product of length, breadth and height. Bulk density was calculated as: Bulk density (g/cc)=Weight/Volume.

Table 2 Coefficient of second order polynomial regression models with significance level

Coefficient	Hardness	Bulk density	OAA
β_0	8.03	0.83	7.75
β_1	-0.79*	-0.10*	0.35*
β_2	-0.37*	-0.059*	0.11*
β_3	-0.37*	-0.032	0.047
β_{11}	-0.25*	-0.036*	0.078*
β_{22}	-0.30*	-0.024*	0.096*
β_{33}	0.51*	0.047*	-0.20*
β_{12}	0.038	0.005	-0.037
β_{13}	-0.012	0.000	0.063
β_{23}	0.19	0.005	0.063
R^2	0.9528	0.8999	0.9670
F-value	8.29	9.99	32.53

*significant ($p \leq 0.05$), OAA overall acceptability

Sensory analysis Before carrying sensory analysis, stored *neyyappams* were warmed (inpack) in microwave oven for 40s. Sensory evaluation of the sample was conducted to access the acceptability of the product by a semi-trained panel of 10 judges from the laboratory staff. The attributes

Fig. 1 Contour plots showing the effect of variables on hardness of *neyyappam*

considered during the study were colour, aroma, taste, texture and overall acceptability. Panelists used 9-point Hedonic scale to assign numerical score to each attribute (Larmond 1977).

Analysis of data Multiple regression analysis was used to fit the model, represented by an equation, to the experimental data. Maximization and minimization of the polynomials thus fitted was done by numeric techniques, using

the numerical optimization technique given in the software package (Design expert (R) software version 7.0, 2006, Minneapolis, MN, USA). The mapping of the fitted response was achieved using software. The contour plots for the models were plotted as a function of the two variables, while keeping other one at optimum level. The storage data were analysed using the analysis of variance technique (Snedecor and Cochran 1968) using the factorial completely randomized design (CRD).

Fig. 2 Contour plots showing the effect of variables on bulk density of neyyappam

Fig. 3 Contour plots showing the effect of variables on OAA of neyyappam

Table 3 Analysis of variance data for the over all effect of process variables

Processing variables	F - value		
	Hardness	Bulk density	OAA
Total individual effect			
Jaggery, X_1	24.66*	13.94*	45.65*
Baking powder, X_2	9.26*	4.67*	18.65*
Holding time, X_3	15.52*	3.72*	17.95*
Combined effect of all variables at			
Linear	8.29*	11.54*	9.02*
Interactive	0.17	0.018	0.32
Quadratic	23.26*	7.15*	31.61*

*significant ($p \leq 0.05$)

Results and discussion

Locally purchased rice flour had moisture $11.5 \pm 0.12\%$, Protein $6.5 \pm 0.15\%$, fat $0.6 \pm 0.15\%$, ash $0.5 \pm 0.08\%$ and carbohydrate $80.9 \pm 0.11\%$. The actual combinations of variables along with their measured response values have been presented in Table 1. The hardness varies from 6.90–10.4 N, bulk density, 0.54–1.04 and overall acceptability, 7.0–8.5.

Diagnostic checking of fitted models All main, linear, quadratic and interactive effects were calculated for each model (Table 2). The adequacy of the models was tested using F-ratio and coefficient of determination (R^2). The models were considered adequate when the calculated F-value was more than table F-value (5% level) and R^2 value was more than 80% (Henika 1982, Khuri and Cornell 1987). The R^2 value was higher than 80% and calculated F-value was also more than the table F-value (5% level) for all the responses, indicating the adequacy of the models. Therefore, all three responses were selected for further interpretation.

Effect of variables on

Hardness Table 2 revealed that all the three variables had negative effect ($p \leq 0.05$) on hardness of *neyyappam* at linear level. Jaggery and baking powder had negative effect

($p \leq 0.05$), while holding time had positive effect ($p \leq 0.05$) at quadratic level. Mohamed and Hamid (1998) also observed positive linear correlation of sugar content with volume expansion and tenderness of chemically leavened rice cake ($r^2 = 0.99, 0.92$ respectively). No significant effect was observed at interactive level. The contour plots showing the relationship of hardness with independent variables have been shown in Fig. 1. Hardness value decreased with increase in the level of baking powder and jaggery, while a unit change in jaggery brought less change in hardness value as compared to unit change in baking powder. Similarly, a unit change in holding time brought more reduction in hardness than unit change in jaggery. Contour between holding time and baking powder were symmetrical hyperbols. Similar values of hardness can be obtained at various combinations of minimum and maximum levels of the independent variables.

Bulk density Bulk density was negatively affected ($p \leq 0.05$) by the jaggery and baking powder at linear as well as quadratic level. Mahajan and Chattopadhyay (2000) also reported increase in the amount of sodium bi-carbonate with citric acid decreased the bulk density and hardness of *dhokla*. Hardness is the peak force during the first bite in the force deformation diagram and could be positively related with bulk density. However, holding time had positive effect ($p \leq 0.05$) at quadratic level. No interactive effect was observed by the variables on bulk density also. Contour plots showing the effect of variables on bulk density have been shown in Fig. 2. An increase in the level of baking powder and jaggery brought a decrease in bulk density, while the effect of baking powder was more than jaggery due to its leavening nature.

Overall acceptability score Overall acceptability was positively affected ($p \leq 0.05$) by the jaggery and baking powder at linear and quadratic level, while holding time had negative effect ($p \leq 0.05$) at quadratic level, whereas, here also no significant ($p \leq 0.05$) interactive effect was observed. Figure 3 represented the changes in overall acceptability score with respect to baking powder, jaggery level and holding time. A unit change in baking powder

Table 4 Constraints criteria for optimization, solution along with predicted and actual response values

Variables	Goal	Lower limit	Upper limit	Importance	Predicted Value	Actual value
Jaggery, g/100 g rice flour	In range	70	90	3	90.0	–
Baking powder, g/100 g rice flour	In range	0.5	1.5	4	1.5	–
Holding time, min	Minimize	10	20	3	12.7	–
Hardness, N	Minimize	6.1	10.4	5	8.6	8.5 ± 0.12
Bulk density, g/cc	In range	0.54	1.04	3	0.63	0.64 ± 0.04
Overall acceptability	Maximize	7	8.5	4	8.2	8.3 ± 0.14

Table 5 Changes in hardness, bulk density and sensory quality of *neyyappam* during storage under ambient conditions

Storage period, days	Hardness, N	Bulk density, g/cc	Colour	Aroma	Taste	Texture	OAA
0	8.6±0.24 ^a	0.69±0.052 ^a	8.0±0.11 ^a	8.0±0.19 ^a	8.8±0.14 ^a	8.2±0.33 ^a	8.3±0.15 ^a
10	10.2±0.25 ^b	0.84±0.061 ^b	7.9±0.15 ^{ab}	7.7±0.18 ^b	8.8±0.12 ^a	8.2±0.67 ^a	8.2±0.12 ^a
20	10.3±0.22 ^b	0.86±0.042 ^{bc}	7.8±0.14 ^{bc}	7.5±0.14 ^c	8.4±0.15 ^b	8.0±0.84 ^b	7.9±0.15 ^b
30	11.4±0.25 ^c	0.88±0.054 ^c	7.7±0.17 ^c	7.5±0.19 ^c	8.5±0.14 ^b	8.1±0.78 ^b	8.0±0.14 ^b

OAA overall acceptability

Values expressed are means ($n=3$)±standard deviation for hardness and bulk density and $n=10$ for sensory parameters. Means in the same column with different superscripts were significantly ($p\leq 0.05$) different

brought more increase in OAA score than the jaggery, whereas when moving in the direction of holding time, a unit change brought least changes compared to baking powder and jaggery level.

The following equations were developed for the hardness, bulk density and overall acceptability using significant ($p\leq 0.05$) model terms:

$$\begin{aligned}\text{Hardness} &= 8.03 - 0.79X_1 - 0.37X_2 - 0.37X_3 - 0.25X_{11} - 0.30X_{22} + 0.51X_{33} \quad R^2 = 0.95 \\ \text{Bulk density} &= 0.83 - 0.10X_1 - 0.059X_2 - 0.036X_{11} - 0.024X_{22} + 0.047X_{33} \quad R^2 = 0.90 \\ \text{OAA} &= 7.75 + 0.35X_1 + 0.11X_2 + 0.078X_{11} + 0.096X_{22} - 0.20X_{33} \quad R^2 = 0.97\end{aligned}$$

Analysis of variance When a model had been selected, an analysis of variance was calculated to assess how well the model represented the data. It is clear from Table 3 that all the independent variables affected all the responses significantly ($p\leq 0.05$). Combined effect at linear level was more as compared to quadratic level, however no interactive effect was observed on responses. The overall effect of jaggery was more followed by baking powder and holding time in the selected range.

Optimization of independent variables For the optimization of jaggery, baking powder and holding time, all the three responses i.e. hardness, bulk density and overall acceptability were selected because these responses had direct effect on the quality of *neyyappam*. These responses were used for the numerical optimization of the variables and the criterion used along with predicted and actual values of the responses have been presented in Table 4. By using the given criterion (Table 4) the solution obtained was, jaggery, 90.0 g/100 g rice flour, baking powder 1.5 g/100 g rice flour and holding time 12.7 (13.0) min. *Neyyappam* were prepared based on solution obtained and responses were measured. The measured responses were very much close to the predicted ones, reconfirming the adequacy of the models. Therefore, the optimized set of condition was recommended for the preparation of *neyyappam*. The optimized samples were also evaluated for proximate composition and values were moisture 22.1±0.42%, protein 4.1±0.22%, fat 11.6±0.35%, ash 1.2±0.05% and carbohydrate 61±0.4% on fresh weight basis.

Storage study The optimized samples were prepared in bulk, packed in polyethylene (75 μ) and heat treated in cabinet tray drier at 90 °C for 2 h. Treated samples along with control (without heat treatment) were stored at ambient

Table 6 Chemical and microbiological changes during storage under ambient conditions

Parameters	Storage period, days			
	0	10	20	30
Moisture, %	22.1±0.15 ^a	21.9±0.14 ^a	21.4±0.14 ^b	21.1±0.12 ^b
Peroxide value, meq O ₂ /kg of fat	6.9±0.24 ^a	8.4±0.22 ^b	9.5±0.25 ^b	10.3±0.24 ^b
Free fatty acid, % oleic acid	1.3±0.24 ^a	1.5±0.24 ^b	1.5±0.24 ^b	1.7±0.24 ^c
Thiobarbituric acid, mg malonaldehyde/kg sample	0.025±0.004 ^a	0.028±0.003 ^a	0.035±0.004 ^b	0.043±0.004 ^c
Total plate count, cfu/ml	5×10 ¹	7×10 ¹	6×10 ¹	9×10 ¹
Yeast and mould, cfu/ml	Nil	Nil	Nil	Nil
Coli forms, cfu/ml	Nil	Nil	Nil	Nil

Values expressed are means ($n=3$)±standard deviation

Means in the same column with different superscripts were significantly ($p\leq 0.05$) different

temperature condition (15–35 °C) and the changes in hardness, bulk density and sensory (Table 5), moisture, peroxide value, free fatty acids, thibarbutyric acids and microbiological (Table 6) were observed up to 30 days. The data related to treated samples for above parameters has been presented, because the control samples had visible fungal growth at 3rd day of storage, therefore the samples were rejected for further analysis. Hardness increased significantly ($p \leq 0.05$) from 8.6 N to 10.3 N after 10 days of storage, thereafter less changes were observed. Bulk density of the samples were initially 0.69 and increased up to 0.84 after 10 days of storage and thereafter no significant ($p \leq 0.05$) change was observed. Among sensory parameters, maximum change in aroma score was observed during storage and probably it was due to changes in peroxide value of lipids, but remained acceptable up to 30 days of storage (Table 5). The overall acceptability score varied between 8.0–8.3 through out the storage period. All the chemical parameters pertaining to peroxide value, free fatty acids and thiobarbutyric acids values increased during storage and it was 10.3 meq/kg fat, 1.7% oleic acid and 0.043 mg of melonaldehyde/kg sample, respectively after 30 days of storage (Table 6). Total plate count was 9×10^1 cfu/ml at the end of 30th day of storage. Yeast and mould and coli forms count was found nil through out the study. Overall the samples were rated very good up to 30 days of storage at ambient temperature conditions.

Conclusion

The instant *neyyappam* can be prepared by using the rice flour 100 g, jaggery 90 g, baking powder 1.5 g, maida 15 g, cardamom powder 0.5 g and dry ginger 2 g, citric acid

1.03 g with holding time of 13 min with same taste and texture as in traditional method. However, it may not have benefits of fermentation as in the case of traditional method. The *neyyappam* can be preserved at least for 30 days by heat treatment in cabinet tray drier at 90 °C for 2 h as compared to control, which had only 2–3 days shelf life at ambient temperature conditions. Stored *neyyappam* can be served in hot condition by just inpack warming in microwave for 40 s. This study will help in reducing the time for preparation of neyyappm and in the commercialization of this product.

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