Optimization of Process Parameters for Absorption of Milk by *Makhana*

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Makhana a popped kernel of gorgon nut (*Euryale ferox*), is widely used for preparation of its *kheer* and pudding. Milk absorption behaviour of *makhana* particles is an important factor that needs to be maximized for preparation of good quality *makhana kheer*. Various parameters, like size of *makhana* particles, milk temperature and its total solid content; moisture content of *makhana* and absorption time were optimized. Response surface methodology was adopted to optimize various parameters. The optimum levels of various parameters for maximum absorption of milk by *makhana* were medium size *makhana* particles (between 0.85 and 1.4 mm) with moisture content of 5.2%, milk temperature 77°C, milk total solids 28.5% and absorption time 112 sec.

Keywords : Gorgon nut (*Euryale ferox*), *Makhana*, *Kheer*, Pudding, Milk absorption.

Makhana is a popped kernel of gorgon nut (*Euryale ferox*) cultivated in stagnant fresh water pools of the Northern and North-eastern States of India. Its wild populations are also found in China, Japan and North America (Jha et al. 1991). Gorgon nut is characterized by its hard seed coat (shell), black colour and round shape with diameter, ranging from 4.5 mm to 14.5 mm (Jha and Prasad 1996).

The edible part of the nut is its starchy kernel, which is taken out by popping. Popping is the process of creating super-heated water vapour within the conditioned nut. Heating and suddenly releasing the pressure (by breaking the shell) cause a volumetric expansion of the kernel. The expanded kernel is referred as *makhana*.

Makhana on an average contains (g/100g) 12.8 moisture, 76.9 carbohydrates, 9.7 proteins, 0.1 fat, 0.5 total minerals, 0.02 calcium, 0.9 phosphorus and 0.0014 iron (Gopalan et al. 1987). Though *makhana* is used in the preparation of a variety of food items, its *kheer* and pudding are widely accepted delicious dishes (Jha and Prasad 1993). In the absence of standard process for the preparation of *makhana kheer*, it is presently prepared in households, resulting in quality variations of the product with respect to particle size, texture, viscosity, other physico-chemical and sensory attributes. The objective of the present paper was to standardize the process parameters such as *makhana* particle size, moisture, milk total solids (TS) and time of absorption (soaking duration) of milk for preparing *makhana kheer* of uniform quality.

Materials and Methods

Design of experiments : The experiment was designed as per the Central Composite Rotatable Design (CCRD) of response surface methodology (RSM) (Cochran and Cox 1980). This design was preferred, as it required fewer measurements to obtain useful and valid information without affecting "precision". In the design of experiments "coded values" of variables used were defined as below and presented in Tables 1 and 2.

$$\text{Coded value} = \frac{\text{Natural value-Base level (level 0)}}{\text{Interval of variation}} \quad \ldots (1)$$

The relationships between the coded and natural values in RSM design for experiment are as follows:

$$X_1 = \frac{(T_m - 60)}{20} \qquad X_2 = \frac{(T_s - 30)}{7}$$

$$X_3 = \frac{\theta - 90}{30} \qquad X_4 = \frac{(M_c - 7)}{3}$$

where T_m, T_s, θ and M_c = milk temperature, milk total solids, duration of absorption (soaking in milk) and moisture content of *makhana*, respectively.

The ranges of variables were selected on the basis of a preliminary study of the traditional method and whole *makhana* samples were excluded from the experiment, because they remained almost uncooked in a short period of soaking in milk.

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TABLE 1. LEVELS, CODES AND INTERVALS OF VARIATIONS OF INDEPENDENT VARIABLES

Independent variables	Codes	Levels					Internal of variation
		−2	−1	0	+1	+2	
Milk temperature, °C	X_1	20	40	60	80	100	20
Milk total solids, %	X_2	16	23	30	37	44	7
Milk absorption time, sec	X_3	30	60	90	120	150	30
Moisture content of *makhana*, % wet basis	X_4	1	4	7	10	13	3

TABLE 2. PLAN OF EXPERIMENT FOR STUDY OF MILK ABSORPTION CHARACTERISTICS OF *MAKHANA* (SECOND ORDER DESIGN IN FOUR VARIABLES)

Treatment	Coded values of the factors				Natural values of the factors			
No.	X_1	X_2	X_3	X_4	X_1	X_2	X_3	X_4
1	-1	-1	-1	-1	40	23	60	4
2	1	-1	-1	-1	80	23	60	4
3	-1	1	-1	-1	40	37	60	4
4	1	1	-1	-1	80	37	60	4
5	-1	-1	1	-1	40	23	120	4
6	1	-1	1	-1	80	23	120	4
7	-1	1	1	-1	40	37	120	4
8	1	1	1	-1	80	37	120	4
9	-1	-1	-1	1	40	23	60	10
10	1	-1	-1	1	80	23	60	10
11	-1	1	-1	1	40	37	60	10
12	1	1	-1	1	80	37	60	10
13	-1	-1	1	1	40	23	120	10
14	1	-1	1	1	80	23	120	10
15	-1	1	1	1	40	37	120	10
16	1	1	1	1	80	37	120	10
17	-2	0	0	0	20	30	90	7
18	2	0	0	0	100	30	90	7
19	0	-2	0	0	60	16	90	7
20	0	2	0	0	60	44	90	7
21	0	0	-2	0	60	30	30	7
22	0	0	2	0	60	30	150	7
23	0	0	0	-2	60	30	90	1
24	0	0	0	2	60	30	90	13
25	0	0	0	0	60	30	90	7
26	0	0	0	0	60	30	90	7
27	0	0	0	0	60	30	90	7
28	0	0	0	0	60	30	90	7
29	0	0	0	0	60	30	90	7
30	0	0	0	0	60	30	90	7
31	0	0	0	0	60	30	90	7

Sample preparation : *Makhana* samples procured from local market were graded, based on their quality, into three grades namely grades 1, 2 and 3 (Jha and Prasad 1996) and only the best quality, i.e., the grade 1 *makhana* was used in the study. Selected *makhana* was ground in domestic grinder (make - Sumeet, model sp-16), using the blade recommended for dry grinding. The particles obtained were classified as large (particles' size between 4.75 mm to 1.4 mm), medium (particles' size 1.4 mm to 0.85 mm and small (less than 0.85 mm). The moisture contents of the particles were determined by hot air oven method (Hall 1970). The desired moisture levels in the particles were achieved either by drying the sample or by keeping them in a humidity-controlled chamber. The conditioned samples prior to use were stored in desiccators partly filled with appropriate saturated salt solutions so as to equilibrate and maintain in moisture at the desired level.

Milk samples were prepared from buffalo milk standardized to 6% fat and desired levels of total solids, obtained by admixing calculated quantities of condensed milk (6% fat, 44% T.S.) and buffalo milk (6% fat, 9% TS).

Experimentation : Milk absorption characteristic of *makhana* was conducted in a water bath, maintained at various pre-determined temperatures. About 50 ml milk of known TS was taken in a 100 ml beaker kept in the water bath and was stirred continuously. As soon as the milk attained the desired temperature, a known weight of sample of *makhana* particles was dropped into the milk and simultaneously a stopwatch was started. The contents of the beaker were stirred and the sample was allowed to absorb milk for a specific period of time. Milk-*makhana* mixture was then immediately filtered through a pre-soaked muslin cloth. The weight of *makhana* (soaked) was taken and the amount of milk absorbed per unit gram of *makhana* was computed.

Analysis : Milk absorption characteristics for various particles size of *makhana* were compared in terms of amount of milk absorbed per unit mass of each grade of *makhana* particles.

A "Computer program" was developed for multiple-regression analysis and optimization of data of the best *makhana* particle size. The program was run on a personal computer. The developed program had the provision to discard the terms of the regression models having F-values less than 1 that are not significant at any level for any degree of freedom (Myers 1971) and to re-compute the coefficients of the remaining terms. The re-computed coefficients of the model were further subjected to the F-test at 5% level of significance. The characteristic roots λ of the coefficients' matrix and the stationary points of the response surface were also generated and the sign of λ values for maxima/ minima or saddle points of stationary values was studied for the optimum levels of parameters for absorption of milk by *makhana* particles.

Results and Discussion

The amount of milk absorbed was found to be the highest for medium size *makhana* particles, followed by small and large sizes (Table 3). Least amount of milk absorbed by large size particles may be due to its lower total surface area and porosity. In case of small particles, however, lesser absorbance of milk than that of medium, which may be attributed to its lower pore volume. The overall effect of total surface area and pore volume of medium size particle may be falling in between the large and small size particles. It is, thus, inferred from data (Table 3) that the *makhana* ground to medium sized particles are the most appropriate size for maximum absorbance of milk. The regression model for the data of medium size particles, after discarding the terms of F-values less than 1, for amount of milk absorbed (Y, g/g) was found to be as follows :

$$Y = -5.776 + .581T_s + 0.103\theta + 0.402M_c + 0.0005T_mT_s - 0.0113T_s^2 - 0.0005\theta^2 - 0.034M_c^2 \quad .....(2)$$

The adequacy of the developed regression model was tested by analysis of variance (ANOVA) as shown in Table 4.

To determine the significance, as suggested by Myers (1971), coefficients of each term of the model were subjected to F-test at 5% probability level with 1 and 23 degrees of freedom. Moisture content of *makhana* (M_c), interactions of milk temperature (T_m) and its total solids (T_s) and square of moisture content (M_c^2) were statistically not significant at 5% level. The effect of milk total solids (T_s) and absorbance time (θ) and their squires were, however, found to be significant.

The optimum levels of absorption parameters were first determined on the basis of the signs of λ values (characteristic roots of coefficients' matrix) and subsequently confirmed by drawing response surfaces. The positive and negative signs of λ values indicated, respectively the minima and maxima at the corresponding stationary points (points of either maxima or minima) of independent variables (Myers 1971). The negative sign of the λ values (Table 4) indicated that the milk absorption would be maximum at the stationary points: milk temperature (T_m) = 77.4°C, milk total solids (T_s)=28.5%, absorption

TABLE 3. MILK ABSORPTION CHARACTERISTICS OF VARIOUS PARTICLE SIZES OF *MAKHANA*

Treatment No.	Milk temp, °C	Milk TS, %	Absorption time, sec	*Makhana* m.c. %, w.b.	Amount of milk absorbed, g/g: Large#	Medium	Small
1	40	23	60	4	5.05	9.29	6.16
2	80	23	60	4	5.59	10.40	6.50
3	40	37	60	4	4.84	7.73	5.63
4	80	37	60	4	5.13	8.13	7.28
5	40	23	120	4	5.16	10.72	6.82
6	80	23	120	4	6.32	11.17	8.06
7	40	37	120	4	5.20	8.41	6.15
8	80	37	120	4	6.17	9.02	7.91
9	40	23	60	10	4.86	8.15	4.91
10	80	23	60	10	4.97	8.98	5.84
11	40	37	60	10	4.01	7.03	4.75
12	80	37	60	10	4.68	7.97	6.11
13	40	23	120	10	5.00	8.69	6.63
14	80	23	120	10	5.01	9.73	7.03
15	40	37	120	10	4.77	7.52	6.12
16	80	37	120	10	5.30	8.52	7.75
17	20	30	90	7	4.79	6.30	5.99
18	100	30	90	7	4.95	7.61	7.53
19	60	16	90	7	4.86	8.00	6.39
20	60	44	90	7	1.90	3.22	2.64
21	60	30	30	7	3.25	4.23	3.51
22	60	30	150	7	4.97	7.95	6.67
23	60	30	90	7	4.85	6.86	6.53
24	60	30	90	1	2.27	6.32	5.18
25	60	30	90	13	5.98	9.02	8.62
26	60	30	90	7	6.02	9.28	8.61*

#4.75 mm> large>1, 1.4 mm, 1.4 mm ≥ medium ≥ 0.85 mm, small < 0.85 mm

*Mean of six replications

TABLE 4. ANALYSIS OF VARIANCE FOR ABSORPTION OF MILK BY MEDIUM SIZE PARTICLES OF *MAKHANA*

Source of variation	Reduction in sum of squares	Degree of freedom	Mean sum of squares	F-value computed
T_s	6.360	1	6.360	4.73*
θ	7.391	1	7.391	5.497*
M_c	1.827	1	1.827	1.359ns
$T_m \times T_s$	2.436	1	2.436	1.812ns
T_s^2	8.857	1	8.857	6.588*
θ^2	5.404	1	5.404	4.019ns
M_c^2	2.755	1	2.755	2.049ns
Error	30.924	23	1.344	
	Stationary points		**λ values**	
T_m	77.387		–0.00038	
T_s	28.522		–0.00068	
θ	112.298		–0.01180	
M_c	5.189		–0.0372	

F-value at 5% (1,23) = 4.28

* significant at 5% level

ns : not significant at 5% level

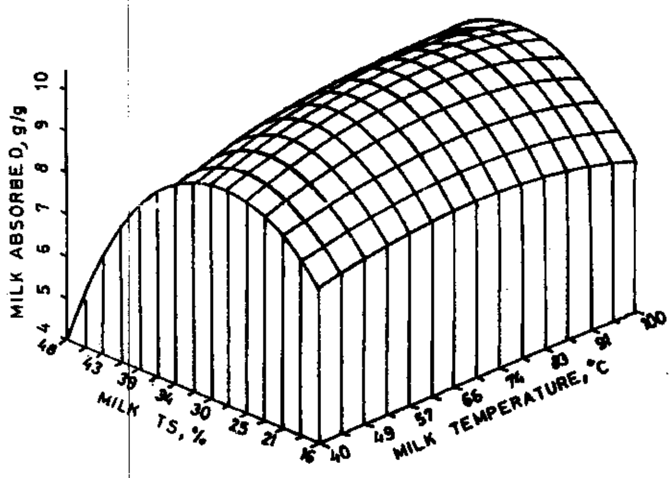

Fig. 1. Response surface of milk absorbed by medium size *makhana* particle at moisture content 5.2% (w.b.) and absorption time 112.3 sec

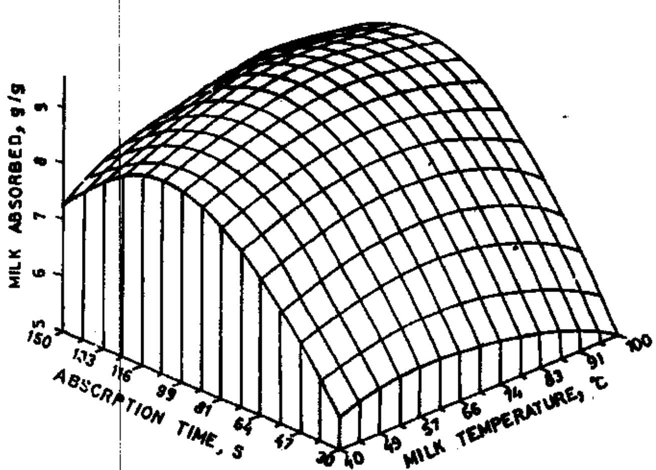

Fig. 2. Response surface of milk absorbed by medium size *makhana* particle at moisture content 5.2% (w.b.) and absorption milk TS 28.5%

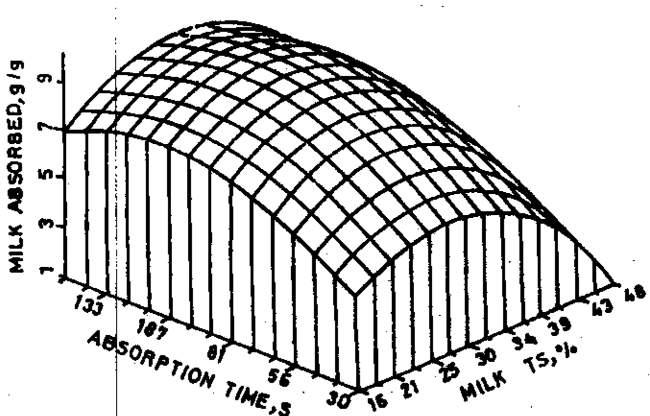

Fig. 3. Response surface of milk absorbed by medium size *makhana* particle at moisture content 5.2% (w.b.) and milk temperature 77.4°C

time (θ)=112.3 sec and moisture content of *makhana* (M_c)=5.2%. These stationary points were further confirmed by drawing the response surfaces between two variables keeping others at stationary points, for milk absorption by medium size *makhana* particles (Fig. 1-3). The amount of milk absorbed increased with decrease in % TS contents of milk and became almost constant between 30 and 34%. Above this TS level, the amount of milk absorbed, declined for the temperatures studied (Fig. 1). Fig 1 also indicted an increasing trend in milk absorption with respect to increase in temperature up to 77°C, beyond which there was no appreciable increase. The increase in absorption of milk with increase may be attributed to reduction in viscosity of milk. Absorption of milk increased initially with increase in TS of milk at a particular temperature. This happened probably due to the combined effect of absorption and adherence of milk solids with *makhana* particles that increased with increase in TS. At higher TS levels, above 30-35%, the milk became so viscous and sticky that it virtually blocked the pores and hardly permitted the milk to seep into the voids of *makhana* particles and that probably resulted in the reduced absorption of milk at higher TS.

The amount of milk absorbed by *makhana* particles increased with increase in duration of time up to 112 sec and milk temperature 77°C. Beyond this time (112 sec), the curves flattened before showing slight decreasing trend (Fig. 2). The interaction of milk TS and duration of absorption also showed that the curves were flat beyond 112 sec and 28.5% TS (Fig. 3). These findings may be attributed to the fact that the *makhana* particles got saturated with milk at 77°C in 112 sec, whereas, beyond this time and temperature, the *makhana* particles were disintegrated due to over saturation and cooking, which reduced the milk holding capacity of the same.

The optimum levels of various parameters for maximum absorption of milk by *makhana* particles may be summarized as particles size medium (0.85 - 1.4 mm), moisture content of *makhana particles* 5.2%, milk temperature 77°C, milk TS 28.5%, and absorption time 112 sec.

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