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DEVELOPMENT OF A COMPOSITE DATE-MILK BEVERAGE USING RESPONSE SURFACE METHODOLOGY

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ABSTRACT

This study was aimed to develop a milk-based date beverage (MDB) that combined the nutrient properties of milk with dates (*Phoenix dactylifera* L.). Since the addition of dates to milk resulted in the curdling of milk, therefore various types and levels of stabilizing salts were screened. Amongst the different stabilizing salt evaluated, trisodium citrate @ 0.15% (w/w of milk) was found most suitable. Different levels of date extract and sugar, prepared in milk (3.0% fat and 8.5% MSNF), were used to optimize the most acceptable DMB as per response surface methodology using two factor central composite rotatable design comprising of 13 experiments. The levels of date extract and sugar ranged from 13 to 17 and 3 to 7% (w/w of milk), respectively. The suggested solution from RSM analysis for DMB was the date extract level of 15.0% and sugar level of 5.6%. The developed product had 21.65% total solids, 2.70% fat, 3.08% protein, 15.08% carbohydrates and 0.79% ash. The study revealed that an acceptable quality of DMB can be manufactured by the addition of date.

Keywords: Beverage, date extract, milk, response surface methodology

INTRODUCTION

There is a stiff competition amongst the manufacturers to develop the new varieties of milk beverage to attract the consumers amid increase in the demand of liquid milk products. These days consumers are more health- and quality-conscious so willingly pay premium prices for the innovative products. Moreover, milk *per se* has certain limitations like allergenicity, lactose intolerance, cholesterol, saturated fat content, etc. (Lucey, 2015). With recent advances in dairy technology, several dairy-derived ingredients are used in conjunction with other food ingredients to manufacture the 'value-added' products that have balanced nutrition and exhibit superior functionalities as well as 'wellness' in food application (Krupa and Patel, 2011). Therefore, new varieties of milk beverages are developed targeting the palatability and health concerns of consumers. Milk beverages are considered among the most functional and nutritional foods (Bangar, 2011).

Fruits and their products are rich source of vitamins, essential minerals, digestible carbohydrates and organic acids that stimulate appetite, help in proper digestion and impart specific food flavour. The date fruit (*Phoenix dactylifera* L.) is recognized for its high nutritive value, immense flavour, high calorific food value and rich dietary fiber contents (Chandra *et al.*, 1992). Dates are rich sources of selenium, copper, potassium and magnesium; and the consumption of 100 g dates provides over 15% of the daily recommended daily allowance (RDA)/adequate

intake (AI) of these minerals. Dates possess high carbohydrate content (44-88%), and have 0.2-0.5% fats, 2.3-5.6% protein, 6.4-11.5% dietary fiber and appreciable amounts of vitamins (Al-Shahib and Marshall, 2003). The moderate concentrations of manganese, iron, phosphorus and calcium provide over 7% of daily RDA/AI (Al-Farsi and Lee, 2008). In comparison to other dry fruits (plums, apricot, figs, raisins and peaches), the USDA national nutrient database has reported that 100 g fruits contain on average of 0.8 µg Se, 0.3 mg Cu, 864 mg K and 43 mg Mg (USDA, 2007). The selenium is a coenzyme for antioxidant enzyme glutathione peroxidase; and has a role in the protection of body tissues against oxidative stress, maintenance of defenses against infection, and modulation of growth and development (NIM, 2000). The high levels of selenium in dates could be used to promote and market this crop in producing countries as well as in other countries. However, the high level of selenium is also a cause of concern as the concentration present in dates (0.31 mg 100 g^{-1}) is close to the toxic level (0.85 mg) (Al-Farsi and Lee, 2008). The total dietary fiber content in dates and the recommended daily intake of total dietary fiber [25 g day^{-1}] (Marlett *et al.,* 2002) reveal that the dates could be a good source of dietary fiber as 100 g dates provide 32% of the recommended daily intake of dietary fiber.

According to the Ayurveda, the use of certain food combinations is beneficial for health. Date is a good source of iron and provides a practical method of Fe-supplementation during childhood, pregnancy and adolescence for iron deficiency anemia (Fazal *et al.,* 2012). Dates have antioxidant, anti-mutagenic and antimicrobial properties (Ashraf and Hamidi-Esfahani, 2011). Milani and Koocheki (2011) used date syrup to prepare products like halwa-ardeh, low-fat frozen yogurt dessert, and low caloric cakes, thus successfully replaced sugar in the product. Tammam *et al.* (2014) prepared ice cream in which date syrup was used as a substitution of sugar @ 20, 40, 60 and 100%. Salama (2004) evaluated date syrup as a sweetening and flavouring ingredient in ice cream manufacture. Response surface methodology (RSM) is a statistical tool to examine the relationship between the factors and responses. It is used to minimize the number of trials and to provide a multiple regression approach for the optimization of ingredient levels, formulations, and processes in food technology (Demirkesen *et al.,* 2011). The present study was aimed to optimize the interaction of date extract with sugar levels in date milk beverage (DMB) formulation, thereby combine the nutrient properties of milk with dates and establish a DMB manufacturing procedure.

MATERIALS AND METHODS

Fresh raw mixed (cow and buffalo) whole milk was procured from Anubhav Dairy, Anand, Gujarat (India) and standardized to 3.0% fat and 8.5% MSNF (milk solids-not-fat) by mixing the required quantity of skim milk and cream. The average composition of milk was 3.1±0.05% fat and 8.6±0.05% MSNF. Cane sugar (Madhur brand from M/s Shree Renuka Sugars Ltd., Mumbai, Maharashtra, India), deseeded dates cv. 'Fard' (Apis brand from Roorkee Uttarakhand, India) were used in the preparation of DMB. The approximate composition of dates revealed 75% carbohydrates, 2% proteins, 0.03% fats, 53% total sugar and 21% dietary fiber.

Preparation of date extract

Deseeded dates and equal amount of water were taken in a stainless-steel vessel. The dates were boiled in water at 100°C for 2-3 min and then kept in hot water for 1 h. This was followed by grinding the material to a fine paste and straining it through a fine wire mesh to obtain date extract.

Preparation of date milk beverage (DMB)

DMB was prepared using varying levels of date extract and sugar as per the response surface methodology (RSM) using Design Expert 8.0.3 to choose the best combination to get the most acceptable DMB. The levels of date extract ranging from 13 to 17% and sugar 3 to 7% (w/w of

milk) were evaluated. The solutions were optimized by adopting two factor central composite rotatable design (CCRD) consisting a total of 13 experiments. The standardized milk was heated to 60°C prior to homogenization (175 ± 35 kg cm^{-2}). Dates were used as sweetener as it contributes to sweetness and consistency. Deseeded date and equal amount of water were taken in a stainless-steel vessel. The dates were heated in boiling water at 100°C for 2 to 3 min and kept in the same hot water for 1 h followed by grinding of date to fine paste and strained through fine wire mesh to obtain date extract. Date extract @ 15% (w/w of milk), trisodium citrate @ 0.15% and sugar [sucrose] @ 5.6% (w/w of milk) were added and stirred for one min. The milk was blended by heating at 70°C to 5 min. The product (DMB) was filled in 200 mL sterilized glass bottles and sterilized at 121°C for 15 min. The bottles were stored at room temperature (37°C).

Sensory evaluation

The milk beverage was subjected to the sensory evaluation by a committee of 10 members comprising of the college faculty members. The sensory attributes were assessed on the basis of 9-point hedonic scale for colour and appearance, body (mouth feel), taste, sweetness and overall acceptability ranking (Stone and Sidel, 2004). The samples of developed milk beverages were randomly served to the penalists in 50 mL glass beakers marked with random digit codes. Sensory product assessment was carried out in isolated booths, illuminated by incandescent light in sensory evaluation laboratory maintained at 23±2ºC. The panelists were instructed to use lukewarm water as rinsing medium, between the samples, as and when required.

Physicochemical analysis

The fat content of developed milk beverage product was estimated by Gerber method (BIS, 1977). The total solids and ash contents of milk beverage were determined by standard procedure (BIS, 1989) using Mojonnier milk tester (Model-D, Mojonnier Brothers Co., Chicago, USA). The protein content was determined using semi-micro-Kjeldahl's method (Jayaraman, 1981). The ash content of all the milk beverage samples was determined by standard method (BIS, 1989). The pH of milk beverage was measured by an electronic digital pH meter (Mettler Toledo AG, Schwerzenbach, Model CH-8603). The viscosity of milk beverage was measured at 20°C using a 'Brookfield' viscometer (DV II + Pro Viscometer, Model- LVDV- II + P, USA). Sediment value in milk beverage was measured by centrifugation method using calibrated 50 mL centrifuge tubes (Prakash *et al.*, 2010). The weight of centrifuge tube was first noted and milk weighed in them. The samples were placed in a centrifuge machine (Remi) and operated at 3000 rpm for 15 min. After centrifugation, the solid sediments were separated from the supernatant by decantation. The tubes were then placed in an oven at 120°C for 36 h and dry weight of sediments noted. The results were expressed in terms of g sediment 100 g^{-1} milk.

Statistical analysis

The data was analysed using response surface methodology (RSM). To carry out their optimization in the final product formulation, an advanced statistical software program tool 'Design Expert' (Version 8.0.3) was used.

RESULTS AND DISCUSSION

In preliminary trials, it was observed that the addition of date extract as sole ingredient for sweetness resulted in the formation of a viscous product that was disliked by panelists. Also, based on the preliminary sensory evaluation, the replacement of sugar upto 60% gave a good quality ice cream with enhanced nutritive value of the products. Preliminary evaluation showed that the addition of date extract @ <13% did not give the desired date flavour, whereas the concentration higher than 17% resulted in too thick consistency, hence date extract @ 15% (w/w of milk) was

chosen. During preliminary trials for the preparation of DMB, it was found that all the experimental samples coagulated during sterilization. Therefore, it was decided to add stabilizing salts. Preliminary studies were conducted to select the type of stabilizing salts from amongst trisodium citrate and disodium hydrogen orthophosphate. Hence, this part of the study was carried out to select the appropriate type and level of stabilizing salts for use in DMB. To select the optimum blend of stabilizing salts formulation, six batches of DMB *viz*., trisodium citrate @ 0.05, 0.10, 0.15 and 0.20% (w/w of milk) and trisodium citrate: disodium hydrogen orthophosphate [Na_2HPO_4] (1:1) @ 0.1 and 0.2% (w/w of milk) were prepared. These levels were decided based on preliminary trials. Date extract @ 10% and sugar @ 5% were added to the standardized milk. All the experimental samples were subjected to sensory evaluation by 10 panelists. The effect of type and level of stabilizing salts on acceptability of date milk beverage is presented in Table 1. It was found that experimental samples having trisodium citrate @ 0.05 and 0.1% and trisodium citrate: Na_2HPO_4 (1:1) @ 0.1 and 0.2% coagulated during sterilization. Amongst all the blends of emulsifying salts tested, trisodium citrate @ 0.15 and 0.2% did not coagulate during sterilization. So, DMB prepared with trisodium citrate @ 0.15% was selected for use in the latter part of the study.

Table 1: Effect of type and level of stabilizing salts on acceptability of date milk beverage

Stabilizing salts in DMB	Acceptability* of sensory characteristics			
	Flavour	Mouthfeel	Colour and appearance	Overall acceptability
Trisodium citrate [TSC] @ 0.05%	-	-	-	-
TSC @ 0.10%	-	-	-	-
TSC @ 0.15%	+++	+++	+++	+++
TSC @ 0.20%	++	+++	+++	++
TSC: Na_2HPO_4 (1:1) @ 0.1%	-	-	-	-
TSC: Na_2HPO_4 (1:1) @ 0.2%	-	-	-	-

* Highly acceptable (++++); acceptable (+++); moderately acceptable (++); slightly acceptable (+); Unacceptable (-)
TSC = Trisodium citrate; Na_2HPO_4 = disodium monohrdrogen phosphate

Optimization of parameters for date milk beverage

All the 13 batches of DMB were evaluated for their sensory attributes and the results are presented in Table 2. The regression analysis of the suggested models for sensory scores of date milk beverage is presented in Table 3. The results have been explained under the relevant sections *viz*., effect of level of date extract and sugar on flavour, colour and appearance, mouthfeel, sweetness and overall acceptability scores of date milk beverage.

Table 2: Experimental design matrix for sensory score of date milk beverage

Run order	Level of date extract (% w/w of milk)	Level of sugar (% w/w of milk)	Flavour score	Color and appearance score	Mouthfeel score	Sweetness score	Overall acceptability score
1	17.00	3.00	7.15	7.79	7.25	7.18	7.21
2	15.00	5.00	8.53	8.57	8.52	8.57	8.52
3	15.00	5.00	8.51	8.63	8.52	8.52	8.64
4	15.00	5.00	8.63	8.47	8.41	8.51	8.61
5	17.83	5.00	8.20	8.10	7.91	7.90	8.2
6	13.00	3.00	7.32	7.15	7.28	7.20	7.35
7	13.00	7.00	8.00	8.2	8.15	8.17	8.01
8	15.00	7.83	7.90	8.21	8.15	8.39	7.95
9	17.00	7.00	8.11	8.15	8.23	8.31	8.13
10	12.17	5.00	8.10	8.2	8.31	8.15	8.2
11	15.00	5.00	8.51	8.55	8.51	8.56	8.51
12	15.00	2.10	7.20	7.1	7.4	7.3	7.3
13	15.00	5.00	8.54	8.64	8.43	8.52	8.55

Effect of level of date extract and sugar on sensory score of date milk beverage

As shown in Table 3, the calculated F values were more than the Table F values at 5% level of significance which indicated the significance of the model terms. Further, the coefficient of determination (R^2) which reflects the proportion of variability in data were > 0.8 in all the studied parameters and was considered as evidence of good fitness of regression equations to the raw experimental date. Larger R^2 values suggest a better fit of the quadratic model. Therefore, it can be concluded that the regression model were suitable for predicting the effect of independent variables on the response variables. The model F values for flavour, colour and appearance, mouthfeel, sweetness and overall acceptability were 29.5, 41.9, 1.2, 40.1 and 23.0, respectively. The adequate precision value (APV) measures the signal to noise ratio. The APV for sensory characteristics were > 4 (Table 3), which support the suitability of the model to navigate the design.

Table 3: P values and partial coefficients of regression equation of suggested models for sensory score of DMB

Parameters		Flavour[1]	Colour & acceptibility[1]	Mouthfeel[1]	Sweetness[1]	Overall acceptability[1]
Intercept		8.54	8.57	8.48	8.50	8.57
Linear level	A	0.0102	0.0561	-0.0645	-0.0292	-0.0025
	B	0.3287*	0.3725*	0.3638*	0.4552*	0.3124*
Interactive effect	AB	0.0700	-0.1725**	0.0275	0.0400	0.0650
Quadratic level	A^2	-0.2482*	-0.2310*	-0.2377**	-0.4935*	-0.2424*
	B^2	-0.5482*	-0.4785*	-0.4052*	-0.5835*	-0.5299*
R^2		0.9547	0.9677	0.9154	0.9662	0.9426
Model F-value		29.47	41.94	15.15	40.05	23.00
APV		15.60	18.11	10.77	16.42	13.76
Suggested model		Quadratic	Quadratic	Quadratic	Quadratic	Quadratic

*Significant 1% level (P <0.01); **Significant at 5% level (P<0.05); R2 = Coefficient of determination; APV = Adequate precision value; A and B refer to the levels of date extract and sugar (% w/w of milk); [1]Sensory score on 9-point hedonic scale

Flavour score

The flavour score of DMB ranged from 7.15 to 8.63 (Table 2). A significant (P<0.01) decrease in flavour score with increase in date extract level was noticed (Table 3). Decrease in flavour score at higher levels of date addition indicated that the product became too sweet and had a pronounced date flavour which was not appreciated by the judges. However, date extract showed a non-significant positive effect (P>0.05) on flavour score, while sugar level had a significant positive effect (P<0.01) on flavour score. The interaction between date extract and sugar levels was non-significant (P>0.05) (Fig. 1a). The addition of dates induced improvement in flavour scores at low levels, whereas increase in sugar levels at higher levels induced decrease in flaovour score. The decrease in flavour score was because of the increase in sweetness since as both dates and sugar contributed to sweetness. The levels of date extract (A^2) and sugar level (B^2) showed a significant (P<0.01) positive effect at quadratic level on flavour score. Gad *et al.* (2010) reported increase in flavour scores in functional yogurt prepared by using a mixture of date palm syrup and skim milk. Their results showed that yogurt enriched with 10% date syrup had increased flavour score as compared to the control. Similarly, Tammam *et al.* (2014) added date syrup as a substitution of sugar in ice cream and observed increase in flavour score when date syrup was replaced with sugar upto 60%, thereafter flavor scores showed decrease. The results of present study are contradictory to Keshtkaran and Mohammadifar (2013) who reported that the date flavoured milk prepared from date syrup (5-25% w/w) and gum tragacanth (0.1-0.3% w/w) decreased the flavour value. Surve *et al.* (2017) prepared milk shake through date incorporation and jaggery and found that the flavour score increased with date pulp level up to 10% and showed decrease beyond this level.

Colour and appearance score

The colour and appearance scores of DMB varied from 7.1 to 8.64 (Table 2). Date extract exhibited a non-significant (P>0.05) positive effect, while sugar level had a significant positive effect (P<0.01) on color and appearance score. Significant (P<0.01) negative effect on colour and appearance score of DMB at quadratic level was also found at extremely higher levels of date extract (A^2) and sugar level (B^2). The interaction effect of date extract and sugar depicted significant

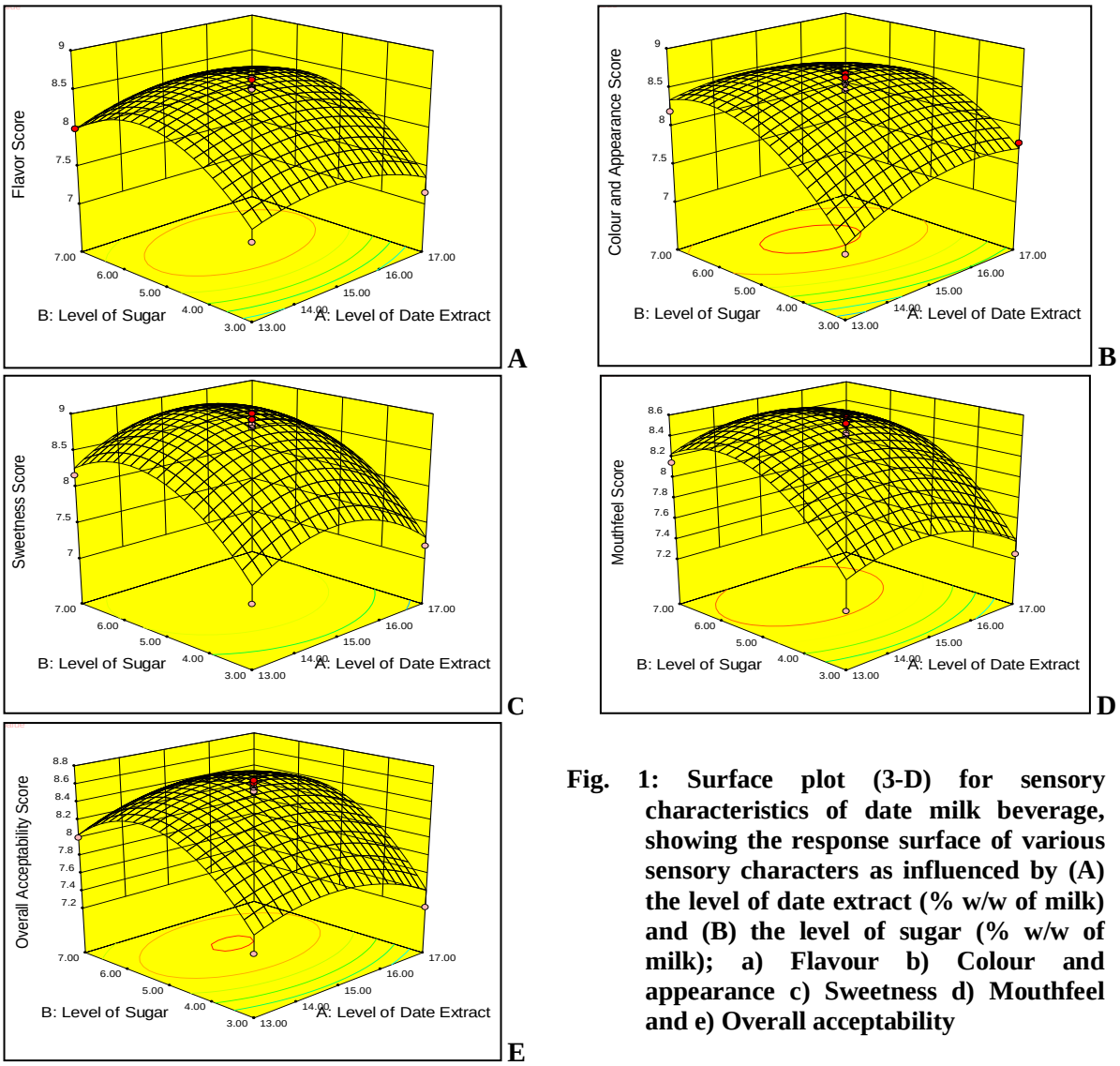

Fig. 1: Surface plot (3-D) for sensory characteristics of date milk beverage, showing the response surface of various sensory characters as influenced by (A) the level of date extract (% w/w of milk) and (B) the level of sugar (% w/w of milk); a) Flavour b) Colour and appearance c) Sweetness d) Mouthfeel and e) Overall acceptability

Table 4: Comparison of predicted v/s actual values of responses selected

Response[1]	P value	Predicted value*	Actual value@	Calculated t-value#	Significance
Flavour	0.9648	8.59	8.47	0.0460	NS
Colour and appearance	0.0665	8.64	8.53	2.0177	NS
Mouthfeel	0.0577	8.55	8.46	2.3414	NS
Sweetness	0.0855	8.58	8.55	2.0557	NS
Overall acceptability	0.0903	8.61	8.56	2.0167	NS

[1]Sensory score on 9-point hedonic scale; *Predicted values of Design Expert 8.0.3 package; @Actual values are average of seven trials of optimized product; #t-values found non-significant at 5% level of significance; NS = Non-significant; Tabulated t-value = 2.447 (Calculated t-value less than tabulated value)

(P<0.05) negative effect on colour and appearance score (Fig. 1b). These results are in agreement with Keshtkaran and Mohammadifar (2013) who reported increase in colour and appearance score of date flavoured milk prepared from date syrup (5 to 25% w/w) and gum tragacanth (0.1 to 0.3% w/w). Surve (2017) prepared milk shake by adding date and jaggery, and found that colour and appearance score increased up to 10% date pulp and subsequently decreased beyond this level. However, our results are in contradiction to Gad *et al.* (2010) wherein the functional yogurt, prepared from a combination of date palm syrup and skim milk, showed that yogurt enriched with 10% date syrup had decreased colour and appearance score than control yoghurt.

Mouthfeel score

The mouthfeel scores of DMB indicated variation from 7.25 to 8.52 (out of 9) [Table 2]. The level of date extract showed non-significant (P>0.05) negative effect on mouthfeel score. The rate of sugar addition showed significant (P<0.01) positive effect on mouthfeel score. The interaction effect between date extract and sugar (AB) showed non-significant (P>0.05) positive effect on mouthfeel score (Fig. d). The addition of dates and sugar induced an increase in mouthfeel scores up to a certain level, whereas at higher levels of sugar and dates, the mouthfeel scores decreased. The decrease in mouthfeel score was because of increase in viscosity since dates are rich in fiber. The results are in agreement with Surve *et al.* (2017) who prepared milk shake by incorporation of date and jaggery. The results are, however, in contrast to Jridi *et al.* (2015) who reported beneficial effects of date syrup addition on the textural properties of dairy dessert.

Sweetness score

The sweetness score of DMB varied from 7.18 to 8.56 (Table 3). The level of date extract showed non-significant (P>0.05) negative effect, whereas sugar depicted significant (P<0.01) positive effect on sweetness score at linear level. The interaction between date extract and sugar (AB) showed non-significant (P>0.05) positive effect on sweetness score (Fig. c). Also, at higher levels of date extract (A^2) showed significant (P<0.01) negative effect on sweetness score at quadratic level. The higher level of sugar (B^2) showed significant (P<0.01) negative effect on sweetness score. Gad *et al*. (2010) prepared functional yogurt from combination of date palm syrup and skim milk and found yogurt enriched with 10% date syrup had an increased sweetness score than control yoghurt.

Overall acceptability score

The overall acceptability score of DMB varied from 7.21 to 8.64 (Table 2). The levels of date extract depicted non-significant (P>0.05) negative effect, whereas sugar showed significant (P<0.01) positive effect on overall acceptability score at linear level. Also, the higher levels of date extract (A^2) showed non-significant (P>0.05) positive effect, whereas higher level of sugar (B^2) showed significant (P<0.01) negative effect on overall acceptability score at quadratic level. The interaction between date extract and sugar (AB) was significant (P<0.01) negative effect (Fig. e). The results are in line with Gad *et al.* (2010) and Surve *et al.* (2017). Shinde *et al.* (2018) reported that the most acceptable quality flavoured milk shake could be prepared by using date pulp at the rate of 7.5% of the buffalo skim milk.

Table 5: Proximate composition of date milk beverage

Parameters	Dairy milk beverage
Total solid (%)	21.65 ± 0.16
Fat (%)	2.70 ± 0.15
Protein (%)	3.08 ± 0.18
Carbohydrate (%)	15.08 ± 0.17
Ash (%)	0.79 ± 0.12
pH (at 25°C)	6.12 ± 0.13
Viscosity (cP at 20°C)	6.72±1.09
Sediment value (g 100 g^{-1})	1.13±0.18

Optimization of date milk beverage

The process optimization for the development of DMB was undertaken to determine the best possible combination(s) of various levels of date extract and sugar that would lead to the development of most acceptable product in terms of sensory characteristics. The goals for all sensory responses were set to be maximized. The predicted formulation for optimized DMB from RSM analysis

consisted of date extract @ 15.0% and sugar 5.6% (w/w of milk). The predicted sensory scores of DMB and the calculated t-values are depicted in Table 4. The final product was manufactured by employing the suggested formulation and the results were compared with these predicted values of the criteria/ responses selected for process optimization. The calculated t-values were less than the table values, therefore it was inferred that there were non-significant ($P<0.05$) differences between the predicted and actual values of responses.

DMB was prepared by using the optimized solutions of date extract (@ 15.0% and sugar @ 5.6% (w/w of milk). The chemical composition of developed DMB is given in Table 5**.** The developed product had 21.65% total solid, 2.7% fat, 3.08% protein, 15.08% carbohydrate and 0.79% ash. The pH, viscosity and sediment value of DMB were 6.12, 6.72 cP and 1.13 g 100 g^{-1}. In flavoured milk, the sugar is added @ 8% w/w of milk whereas in this product sugar was added @ 5.6% w/w of milk.

Conclusion: Based on results of present work, the method for the development of date milk beverage using date extract (@ 15% w/w of milk), trisodium citrate @ 0.15% and sugar (5.6% w/w of milk) was standardized.

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