

Utilization of dairy byproduct for development of whey ice candy

ABSTRACT:

Background: Whey is by product of dairy industry and contained about 6 % milk solids. It may cause environmental problem if not utilized properly.

Aims: The present study aim to utilize whey in the preparation ice candy enriched with fruit pulp.

Methodology: Whey candy mix was prepared by combination of fruit pulp, sugar and stabilizer in clarified paneer whey followed by homogenization, heating and cooling. Mix was poured in mold and passed through continuous blast freezer to harden. Hardened candies were wrapped in aluminium foil and stored in deep freezer at $-20 \pm 2^{\circ}\text{C}$. The suitability of fruits i.e. kiwi, orange and pineapple in whey based ice candy was determined by organoleptic quality. The level of selected fruit and sugar were optimized using Response Surface Methodology (RSM). The optimized product was evaluated for sensory and physico-chemical qualities. Also the storage stability, consumer acceptability and economical feasibilities of the product were tested.

Results: Amongst the three different fruits, the whey candy prepared by using pineapple fruit pulp was received highest sensory score. The RSM results shows that, increase in pineapple pulp and sugar level resulted in higher sensory score for color and appearance, taste and flavor. Optimized level of pineapple pulp and sugar were 14.23 % and 21.80 %, respectively. This combination obtained 7.7, 7.6 and 7.65 sensory score for color, flavor and overall acceptability respectively whereas, melting rate was 42.19%. The whey candy subjected to study consumer acceptability and it was found that 58% of the consumers rated experimental candy as excellent (score 5) and 20.5 % rated on very good. The whey candy was acceptable up to 60 days at refrigerated storage condition.

Key words: *Whey, Pineapple, Candy, RSM, Byproduct.*

1. INTRODUCTION

Milk is a major diet component worldwide and it is considered natural complete food providing fat, protein, carbohydrate, vitamin, and minerals particularly calcium [1]. Near about 60% of milk produced in world is converted into dried, fermented, acid and enzyme coagulated, heat desiccated, etc.. During the production of acid/ enzyme coagulated milk products like cheese, paneer, channa, casein and also in the

course of production of fermented products like shrikhand, quarg etc., whey is separate out as a by-product. Though it is by-product eventhough on an average it contained 6.5 to 7.0 % Total solids (TS), 0.4 to 0.9 % protein, 0.3 to 0.5 % fat, 4.9 to 5.5 % lactose, 0.6 % ash and 600 ppm calcium [2]. Further it is being viewed as one of the major disposal problems of the dairy industry as it imposes a very high biological oxygen demand value which is being in the range of 30,000 to 50,000 mg/Lit and chemical oxygen demand in the range of 60,000 to 80,000 mg/L. With increased efforts to conserve the food nutrients with available technologies, the loss of milk solids in whey is no longer considered as a waste by-product and its utilization in a human food chain is now being predominantly flavored due to economic opportunities provided by the milk nutrients contained in whey [3]. In India, the production of whey is estimated at about 4.84 million tons per annum, which consists of about 290 million kilograms of valuable milk nutrients [4]. Nowadays, whey and whey components are increasingly incorporated in various dairy and non-dairy foods like beverages, soups, sports drinks, baked goods, frozen dairy goods etc.

Ice cream is a frozen dairy product made by suitable blending and processing of cream and other milk products, together with sugar and flavor, with or without stabilizer or color and with the incorporation of air during the freezing process [5]. Ice cream is a very delicious frozen dessert and ice candy is an attractive form of it which is especially liked by the children [6]. Indian ice cream industry is one of the fastest-growing segments of the dairy or food processing industry. The ice cream industry (including candy) in India generated revenue of more than USD 1.5 billion in 2016 and is projected to generate revenue of approximately USD 3.4 billion by 2021. Currently, its market in India is estimated to be over INR 4,000 crores, and is growing at a rate of 15-20% year-on-year [7].

Recently, there has been a growing interest in the field of functional food and search for their potential to improve the immune system. Natural fruit pulp are believed to enhance natural resistance of the body against infection and their immune modulatory have been reported in several studies but due to their undesirable and sour or unacceptable taste their health benefits cannot be fully utilized, especially in case of children. Therefore, a matrix like candy, with its chilling exciting taste, can help in utilizing the potential therapeutic properties of fruits. Thus producing whey candy by incorporating fruit pulp can fill the gap in the market and make availability of new functional product. Amongst various fruit pulp, pineapple may on of the choice as it contains considerable amount of calcium, potassium, vitamin C, carbohydrates, crude fibre and other minerals that is good for the digestive system and helps in maintaining ideal weight and balanced nutrition [8]. Considering the availability of nutritious whey in throable cost and wide scope for functional cheapest product, the present study was undertaken with a goal to standardize the method for utilization of whey and fruit pulp in preparation of whey candy type frozen products.

2. MATERIALS AND METHODS

2.1 Materials

Fresh buffalo milk was procured from Dairy Farm, RCSM College of Agriculture, Kolhapur (MS). Whereas, pineapple fruit of queen cultivar, kiwi fruit of the hardy variety, orange fruit of Nagpur variety

were procured from the local market of Kolhapur city. Carboxy-methyl cellulose (CMC) of Bijur Scooper Foods Pvt. Ltd. make was used as a stabilizing agent.

2.1 Methodology

2.1.1 Extraction of Fruit Pulp / juice

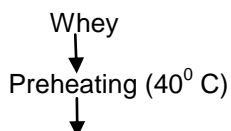
Fresh pineapple and kiwi fruit were washed under running tap water and peeled, cut into small piece and pulp was made as per developed protocol [9]. Whereas, fresh orange fruit was washed, peeled and cut into two halves, juice was extracted manually and filtered through muslin cloth [10].

2.1.2 Preparation of paneer whey

Buffalo milk was standardized to 6.0 % fat and 9.0 % SNF, followed by heating up to 86°C and cooled to 78°C. Then 1.5 % of citric acid solution was poured to till to coagulate casen. Coagulated milk was filtered through two layered muslin cloth to get clear whey [11].

2.1.3 Preparation of whey candy

Whey candy was prepared with certain modification (Fig 1) as reported in literature [12]. Initially whey was preheated at 40°C and allow for clarification through centrifugal separator at 5000 rpm. In clarified whey, fruit pulp and sugar were mixed as per treatment combination. Also CMC as a stabilizer was used @ 0.5 % of whey. The mixture (whey candy mix) was allow to homogenize by passing through double stage homogenizer followed by heating to 74°C and subsequently cooled to 5°C, it was then poured in 80 ml ice candy mold and passed it through contionues blast freezer to harden it. Hardened candies were wrapped in aluminium foil and transferred into deep freezer at - 20 ± 2°C for storage.



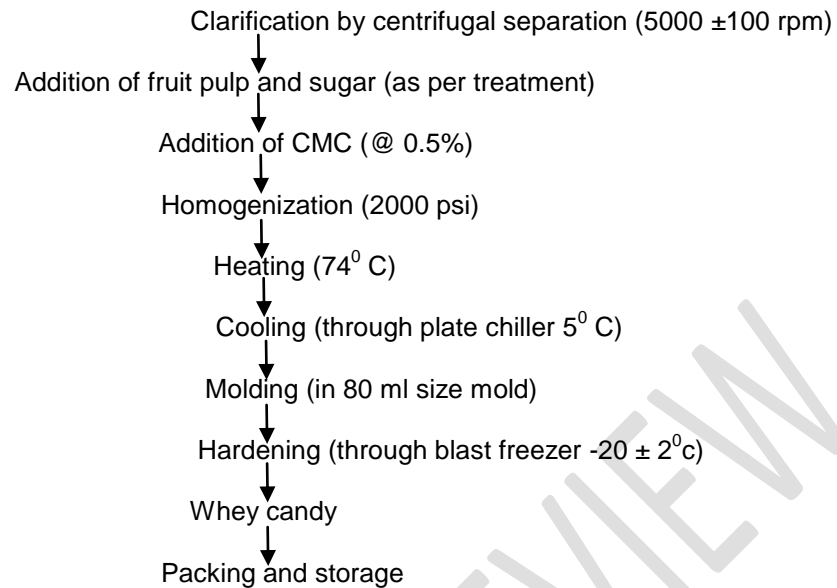


Fig.1 Flow diagram for preparation of whey candy

2.1.4 Process optimization

2.1.4.1 Selection of fruit for whey candy

Three different fruits i.e. pineapple, kiwi and orange were purposely selected on the basis of their nutritional qualities and possibilities of their suitability in whey candy. Each fruit either in pulp or in juice form was incorporated @ 10 % of whey along with 20% sugar and 0.5% CMC. Thus treatments were whey candy without fruit pulp (P_0), whey candy with pineapple pulp (P_1), whey candy with kiwi pulp (P_2) and whey candy with orange juice (P_3). Accordingly one fruit was selected on sensory analysis.

2.1.4.2 Optimization of level of Pineapple fruit pulp and sugar

Central Composite Rotatable Design (CCRD) of Response Surface Methodology (RSM) was used to optimize the level of selected fruit pulp and sugar for two factors as independent variables (www.statease.com). The color and appearance, body and texture, taste and flavor, and melting rate were considered as responses for optimization of experiment. The level of ingredients of the design matrix for the experiment is presented in Table 1.

The data generated was analyzed using Design-Expert11 Software and polynomial equation was obtained for each response. Model adequacy was evaluated using F ratio and co-efficient of determination (R^2). The lack of fit was calculated. Model was considered adequate when F-calculated was more than Table F-value and R^2 will be more than 80 per cent [13]. The effect of the variables at linear, quadratic and interactive levels on individual responses was described using 1 and 5 per cent levels of confidence. From the results obtained through sensory evaluation of whey candy with different levels of pineapple fruit pulp and sugar most desirable combination was selected by verification of their sensory qualities.

Table 1: Experimental level of Independent variables in RSM:

Std	Pineapple pulp (A) %	Sugar (B) %
1	10	8
2	16	18
3	8	26
4	16	26
5	8	22
6	16	22
7	12	16
8	12	26
9	12	22
10	12	22
11	12	22
12	12	22
13	12	22

2.1.4.3 Storage study of developed whey candy

The best optimized level of fruit pulp and sugar obtained in whey candy were used for further storage stability studies. For that the whey candy samples were packed in aluminum pouch of 75 ml capacity and stored at $-20 \pm 1^{\circ}\text{C}$ for storage studies. The samples were evaluated after 15 days interval for changes in sensory attributes, chemical constituents, physical properties, sensory attributes and microbial load.

2.1.5 Physico-chemical analysis

Standard methods [AOAC] for analysis was implemented for determining values of moisture, fat, crude protein, total carbohydrates, ash. The pH of the sample was determined using a digital pH meter (Orion 3 star pH bench). Whereas, total acidity of sample was determined by titration with 0.1 N NaOH and the total soluble solids ($^{\circ}\text{Brix}$) in a digital refractometer [14]. Ascorbic acid of sample was estimated by 2, 6 – Dichlorophenolindophenol visual titration method [14]. All analyses were carried out on five batches of whey candy (replicates) with three analyses for each batch (triplicate).

Viscosity of premix was determined with certain modification by using Brookfield viscometer at $21 \pm 1^{\circ}\text{C}$ with the help of spindle no 61 [8]. Flow ability (Sec) of candy mix was determined by time requires to travel 10 ml sample through pipet whereas, solubility index of premix determined by centrifugation method in remi centrifuge at 10000 rpm and melting rate was quantified by placing one candy of each experimental sample on meltdown apparatus and determine the weight of melted ice cream that drip into beaker through the mesh screen (144 mesh per square inch) as function of time [15].

2.1.6 Organoleptic evaluation

Sensory evaluation were carried out by a semi-trained panel of judges from the staff of the Department of technology, Shivaji university Kolhapur, by using 9- point Hedonic scale for body and texture, colour and appearance, taste and flavor, melting resistance and overall acceptability [16]. Samples were served in coded number mold.

2.1.7 Statistical analysis

The level of sugar and pineapple pulp was optimized using Stat-Ease Design Expert 11.0.0 package procured from stat ease Inc., USA (www.statease.com).

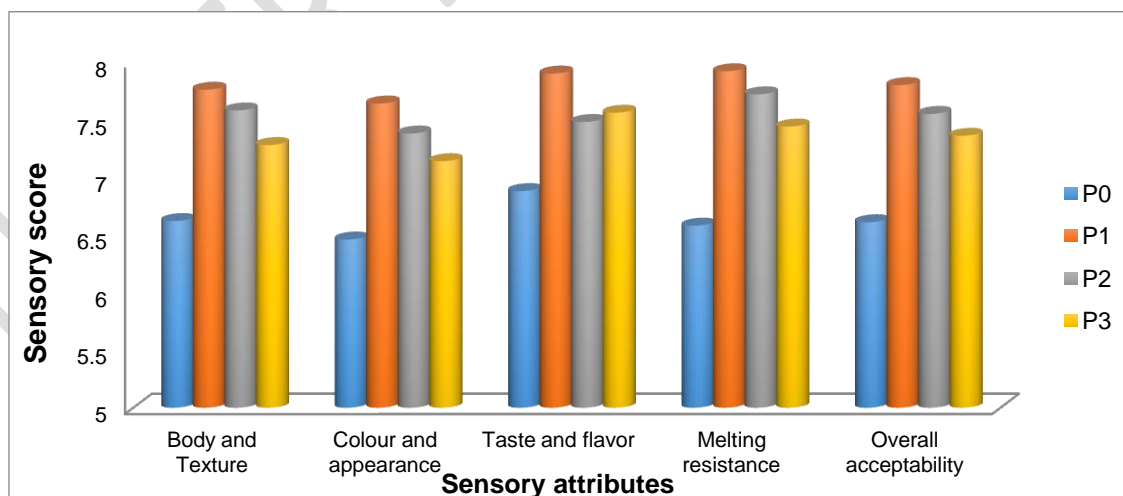
2.1.8 Consumer acceptability study

The experimental whey candy was subjected to consumer acceptance. Consumer acceptance study was conducted for 153 respondents from local area. The hardened whey candy was offered to participants in the size of rectangular shape with an average weight of 80 g and requested to evaluate sample for acceptability of taste and flavor, size, shape, colour, appearance and overall liking using 5- point hedonic scale [5 for excellent and 1 for unsatisfactory]. Further the information was also collected for willingness to purchase whey candy on pre designed questionnaire [17, 18].

3 RESULTS AND DISCUSSION

3.1 Selection of fruit for whey candy

The three different fruits i.e. pineapple, kiwi and orange were incorporated in whey candy among them pineapple pulp added candy had received significant highest score for body and texture (7.76 ± 0.04), colour and appearance (7.64 ± 0.04), taste and flavor (7.9 ± 0.06), melting resistance (7.92 ± 0.05) and overall acceptability (7.80 ± 0.03) over the other fruits (Figure 2).



P₀ = Whey candy without fruit pulp, P₁ = Whey candy with pineapple pulp, P₂ = whey candy with kiwi pulp and P₃= whey candy with orange juice

Fig 2 Effect of type of fruit on sensory qualities (score*) of whey candy

The panelist commented that pineapple pulp added candy had acceptable which was with light yellow colour, and had vibrant tropical flavour that balance the tastes of sweet and tart. It was noticed that kiwi and orange whey candy and remains in mouth for long time. Judies reported a comment that kiwi whey candy was slightly sour taste and orange whey candy was less sweeter than pineapple whey candy. Bhavsagar *et al.*, [19] also reported that the average sensory score of pineapple whey beverage for colour ranges between 7.5 to 7.9.

3.2 Optimization of level of pineapple pulp and sugar through RSM

On the basis of preliminary trial the range of level of pineapple pulp and sugar was decided and their intermediate level were optimized in this phase of experiment. The maximum colour and appearance score (7.60) was obtained for formulation, which had 12 % pineapple pulp and 22 % sugar. The regression analysis of a data presented in Table 2 reveals that the coefficient of determination (R^2) was 0.8780. The adequate precision was found to be 9.6250, appreciably higher than the minimum desirable (4) for high prediction ability. Further the statistical analysis indicated that the model fitted the data well (model 'F' value 10.07). It can be seen from fig. 3, the increasing sugar level slowly increase in the colour and appearance score whereas, it increased rapidly as the pineapple pulp level increases up to a certain point, beyond that the colour and appearance was decreased. The colour and appearance score of flavored soy ice cream with addition of lemongrass leaf extract was increased at certain point and then decreased [4].

The taste and flavour score of whey candy ranged from 7.1 to 7.7. The maximum score (7.70) was obtained for formulation, which had 12 per cent pineapple pulp and 22 per cent sugar. The minimum score (7.10) was obtained for formulation, which had 8 per cent pineapple pulp and 22 per cent sugar. The regression analysis of a data presented in Table 2 reveals that the coefficient of determination (R^2) was 0.8732. The adequate precision was found to be 8.9047, appreciably higher than the minimum desirable (4) for high prediction ability. Further the statistical analysis indicated that the model fitted the data well (model 'F' value 9.64). The obtained results are also represented in fig 4 which shown that the flavour score increased with increasing level of pineapple pulp and sugar level at certain point but beyond that flavour score declined slowly. The taste and flavor score of whey beverage by incorporating pineapple juice was found to be the best [19].

The graphical representation reflected in fig 5 indicated that as the sugar and pineapple pulp level increased the overall acceptability increased up to certain point and beyond that it start decline, this nature of graph means that at the highest level of pineapple pulp (16 per cent) and sugar (22 per cent) had low overall acceptability score (7.5) and at lowest level of pineapple pulp (8 per cent) and sugar (18 per cent) had also low overall acceptability score (7.25) but at middle level of pineapple pulp (12 per cent) and sugar (22 per cent) had maximum overall acceptability score (7.6). The above finding is supported by the views published in the literature [18].

The melting rate of whey candy ranged from 40.00% to 54.23%. The maximum melting rate (54.23%) was obtained for formulation, which had 8 per cent pineapple pulp and 22 per cent sugar. The regression analysis of a data presented in Table 2 reveals that the coefficient of determination (R^2) was 0.9832. The adequate precision was found to be 23.13, appreciably higher than the minimum desirable (4) for high prediction ability. Further the statistical analysis indicated that the model fitted the data well (model 'F' value 81.91). The obtained results are also represented in fig 6 which shown that the melting rate property increased with increasing level of pineapple pulp and sugar level slowly.

Table 2. Coefficients of Quadratic polynomial model for coded sample

Factor	Response score			Physical Parameter
	Colour & appearance	Taste & Flavour	Overall acceptability	Melting rate
Intercept	7.54	7.62	7.58	42.62
Pineapple pulp(A)	0.1082	0.1509	0.1295	-4.46
Sugar (B)	-0.0125	0.0052	-0.0037	-1.62
AB	-0.0250	0.0250	0.0000	-1.81
A ²	-0.2012	-0.1537	-0.1775	2.32
B ²	-0.0012	-0.0537	-0.0275	3.85
R ²	0.8780	0.8732	0.9015	0.9832
F-value	10.07	9.64	12.82	81.91
Mean	7.42	7.49	7.45	46.42
SD	0.0873	0.0861	0.0743	0.8839
Adequate Precision	9.6250	8.9047	10.6646	21.1302

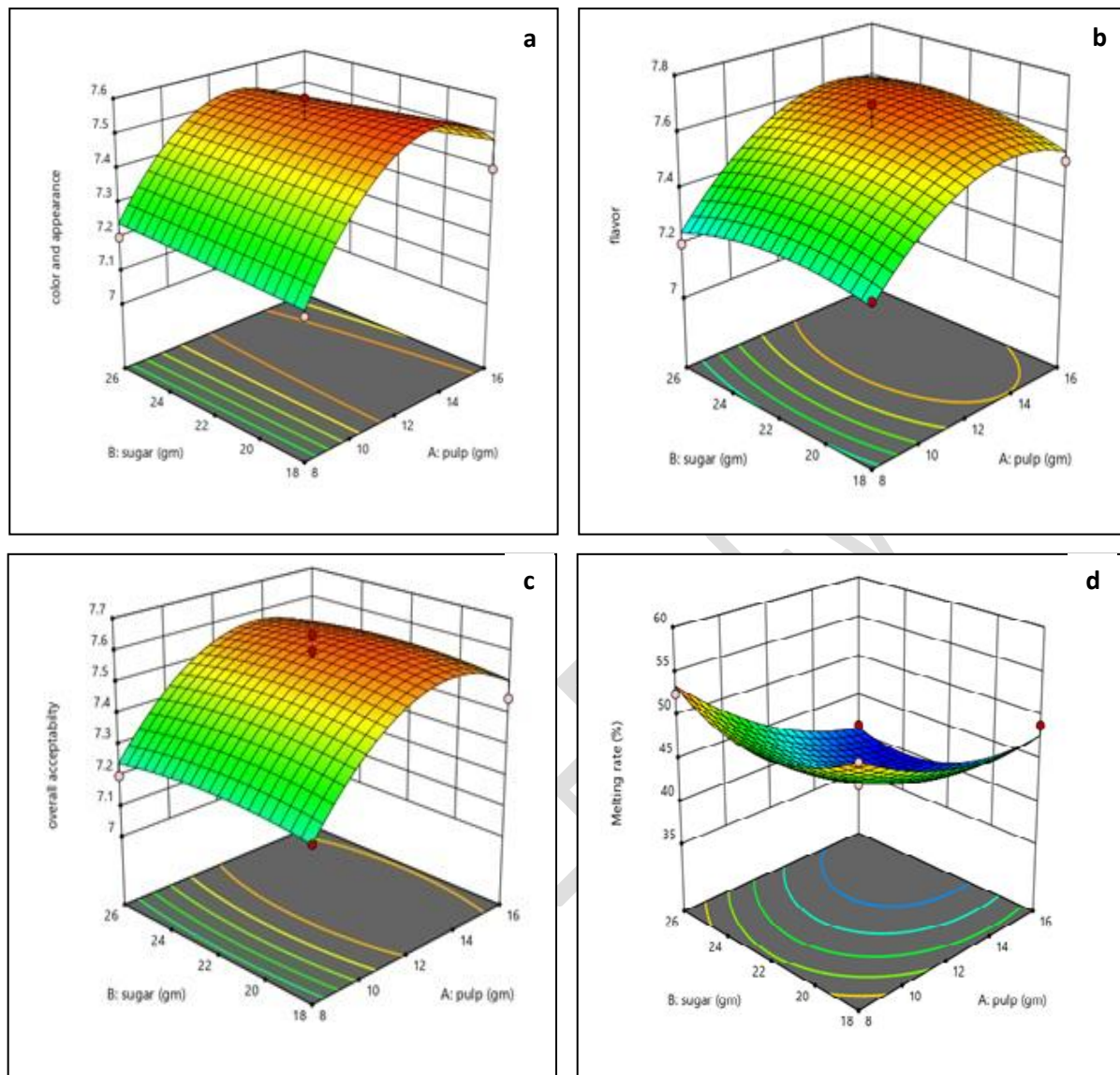


Fig. 3. Effect of Pineapple pulp and sugar on (a) colour and appearance, (b) Flavour, (c) Overall acceptability and (d) Melting rate

3.3 Optimization of independent variables:

The optimization of the independent variable levels was achieved by desirable maximization of the sensory and physical response along the fitted polynomial model by numerical optimization procedure of design expert software. The responses had direct effect on acceptability and quality of whey candy as shown by their respective R^2 values (Table 2). The goals set for two factors and five responses were represented in table 3.

Table 3. Goals set for constraints to optimize the technology

Constraints	Goals	Lower limit	Upper limit	Importance
Pulp	in range	8	16	3
Sugar	in range	18	26	3
Color and appearance	target = 7.6	7	8	3
Taste and flavour	target = 7.7	7	8	3
Overall acceptability	target = 7.6	7	8	3
Melting rate (%)	target = 41	40	45	3

The predicted solution generated by Design Expert 11.0.0 package had contained pineapple pulp 14.22 % and sugar 21.80%. The validation of the prediction was done by actual observations recorded for sensory score of whey candy prepared by addition of pineapple pulp and sugar @ 14.22 % and 21.80 %, respectively. The predicted and actual response values (obtained after making the product using the optimum level of ingredients) were almost similar. Hence, 14.22 % pineapple pulp and 21.80 % sugar level was recommended. Optimized whey candy had 7.80, 7.65, 7.72 score for body and texture, colour and appearance, overall acceptability, and 41.33 % melting rate respectively (Table 4).

Table 4. Predicted and actual sensory score of suggested formulation by Design Expert 11.0.0 package

Ingredients (%)	Score	Sensory parameters			Physical parameters Melting rate (%)
		Body & texture	Taste & flavor	Overall acceptability	
Pineapple pulp 14.22	Predicted	7.70	7.60	7.65	41.00
Sugar 21.80	Actual	7.80 ± 0.05	7.65 ± 0.04	7.72 ± 0.04	41.33 ± 0.16

Data are expressed as mean ± standard error of five experiments (n=5)

Physico-chemical analysis of optimized level of whey candy

The optimized product was analyzed for various physico-chemical characteristics and values were presented in below table 5.

Table 5. Physical analysis of pineapple whey candy

Sr. No	Parameters	Values
1	Viscosity (cp)	59.2±0.08
2	Flow ability (sec)	3.12±0.03
3	Solubility Index (ml)	2.1±0.1
3	Melting rate (%)	41.97±0.11
4	Density	1.08±0.04

Data are expressed as mean ± SE (n=5)

From data from the table 5 indicated that whey candy mix before hardening had 59.2 cp viscosity, 3.12 sec, flow ability, 2.1 ml solubility index. Also hardened whey candy had 41.97 % melting rate and 1.08 density. The results concluded that pineapple pulp concentration might be affect on the physical properties of whey candy.

Table 6. Chemical properties of whey candy

Sr. No	Parameters	Values
1	Moisture (%)	71.31±0.08
2	Fat (%)	0.2±0.03
3	Protein (%)	0.32±0.05
4	Carbohydrate	27.81±0.04
5	Ash (%)	0.46±0.04
6	Total Solids (%)	28.69±0.10
7	Acidity	0.27±0.02
8	pH	4.92±0.01
9	Vit. C (mg/100g)	2.18±0.05
10	T.S.S. (Brix)	28-29

From the above table 6, it was observed that whey candy had 0.32% protein, 0.2% fat, 4.92 pH, 2.18 mg/100g Vit C and contained 0.46, 0.27, 27.81, and 28.69 per cent ash, acidity, carbohydrate and total solid respectively. The effect of addition of fruit on physico-chemical attributes of ice cream were also studied [21] and reported that the dry matter content, viscosity of ice cream increased with the addition of green banana flour whereas addition of the dietary fibre affected significantly fat and acidity values. Also

the effect of addition of mango fruit pulp on physico-chemical properties of ice cream were observed [6] and concluded that the mango pulp significantly affected on vit C, protein, fat and TSS value.

3.4 Effect of storage on sensory attributes (score) of whey candy

It can be seen from fig 7 that the body and texture score decreases as the storage period increases. Decline in body and texture scores during storage may be attributed to the changes occurring in the whey candy or might be because of increasing the ice crystals in the product and contribute to the improper texture to the product. As storage period increased the score of colour and appearance was decreases in whey candy. The color and appearance score of sample was best up to 60 day and these changes might be due to chemical changes in product. The addition pineapple pulp was found to improve the flavour scores of the whey candy, but sensory evaluation reveals that with advancing storage period, a gradual deterioration in flavour score was observed. The data revealed that the whey candy with addition of pineapple pulp has better melting resistance up to 45 days while the melting resistance score decreased significantly during storage. The data indicated that the overall acceptability of control sample was significantly lowered as increase in storage period. Decreased in sensorial attributes of ice cream during storage period due to changes in chemical parameters [21].

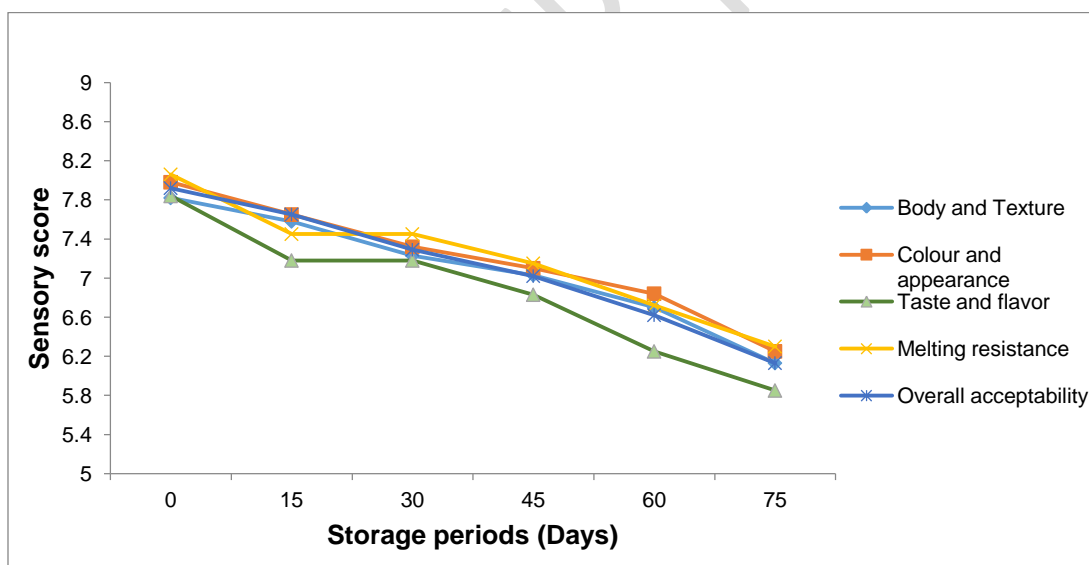


Fig 7. Changes in sensory attributes of prepared whey candy during storage period

3.5 Effect of storage on physico chemical properties of whey candy

From table 7, data revealed that the acidity of whey candy sample increased during storage period. Acidity increased from 0.28 to 0.59 percent. It might be due to inherited acidity present in the pulp and results in the formation of organic acids by degradation of ascorbic acid and utilization of sugars to yield organic acid [22]. As the acidity of whey candy increases the pH of the sample decreases significantly. As the storage period increases there is a gradual decrease in the melting rate of whey candy.

The percent decrease in melting rate ranged from 42.93 to 40.84 percent. The present findings are in accordance with the results published in the literature physicochemical changes in bakery flavoured ice cream during storage period [23].

Table 7. Physico- chemical Changes in chemical properties of whey candy

Chemical parameters	Storage period (Days)					
	0	15	30	45	60	75
Acidity (%)	0.28	0.34	0.40	0.45	0.52	0.59
pH	4.93	4.90	4.84	4.80	4.75	4.71
Melting rate (%)	42.93	42.49	42.17	41.63	41.24	40.84

3.6 Effect of storage on microbial quality of whey candy

Total Plate Count of whey candy in whey candy on decreased gradually from 4.2×10^4 to 3.0×10^4 cfu/ml during 75 days storage period (Table 8). A progressive reduction in the TPC of all samples may be due to destruction of microorganisms at low temperature storage. Decline in microbial count during storage could mainly due to formation of ice crystals that damaged the cell wall leading to lysis of the microorganisms [24]. The YMC and coliform count were not detected in sample up to 75 days because of the sample was stored at refrigerated temperature.

Table 8. Changes in microbial load of whey candy

Microbial parameters	Storage period (Days)					
	0	15	30	45	60	75
Total plate count (cfu/g)	4.2×10^4	4.1×10^4	3.8×10^4	3.6×10^4	3.3×10^4	3.0×10^4
Yeast and mold (cfu/g)	ND	ND	ND	ND	ND	ND
Coliform (cfu/g)	ND	ND	ND	ND	ND	ND

ND= Not detected

3.7 Consumer acceptance and willingness to purchase of whey candy

It was found that 58 % of the consumers rated experimental candy as excellent (5.0) and 20.5 % rated on very good (4.0) (Fig 8). Also it was found that 40.5 percent of consumer shown their willingness to purchase whey candy forthrightly and 25.50 per cent of the consumers will purchase monthly (Fig 9). The flavoured sweetened whey drink was prepared by 4, 5 and 6 per cent mango pulp [18] and recorded highest consumer acceptability as compared to that of control.

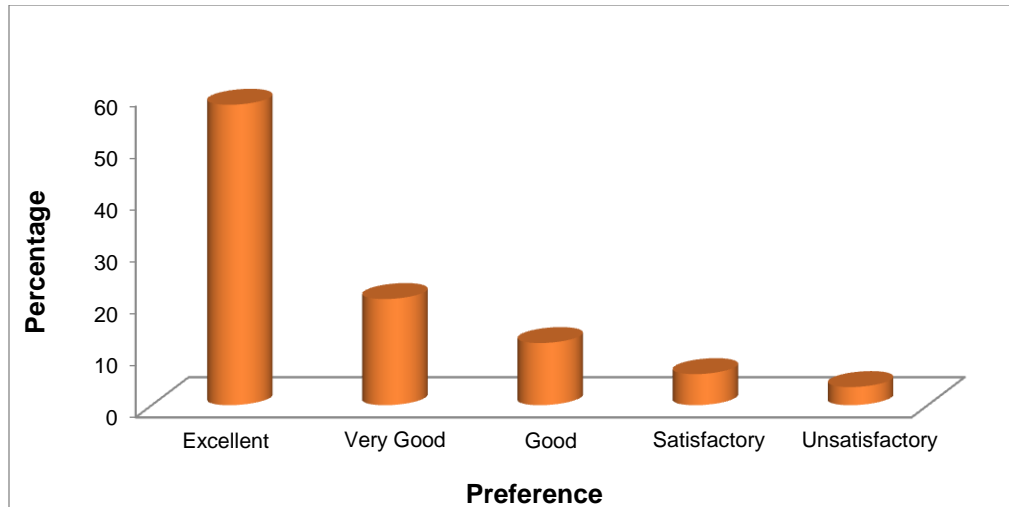


Fig 8. Acceptance of whey candy

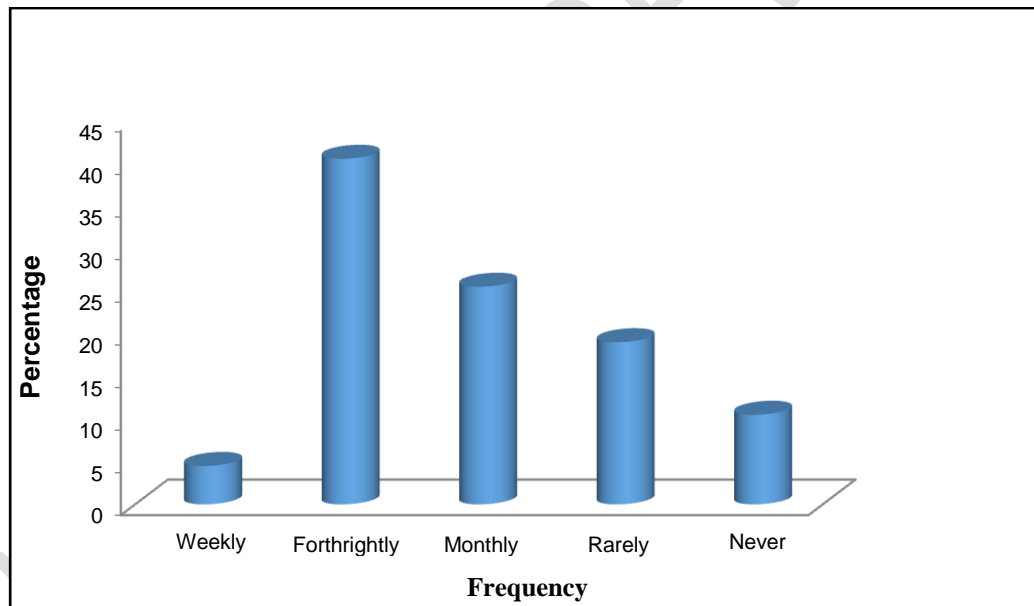


Fig 9. Frequency of purchasing whey candy

4 CONCLUSION:

Use of pineapple fruit over kiwi and orange fruit in the preparation of whey candy was significantly superior. The most sensorial acceptable quality of pineapple pulp added whey candy can be prepared by using 14.22 per cent pineapple pulp and 21.80 per cent sugar. The optimized formulated (14.22 % pineapple pulp and 21.80 % sugar) product had 04.92 pH, 0.27 (% LA) acidity and contained 0.2, 0.32, 27.81, 0.46 and 28.69 per cent fat, protein, carbohydrate, ash, and total solid, respectively.

As the storage period increases acidity, total solids and ash content were increased progressively. Sensory scores of all whey candy samples were found to decrease progressively with

increase in storage period. The microbial analysis i.e. TPC was decreased during storage. Storage study was continued for 75 days and the acceptance of whey candy sample was found in terms of sensory attributes up to 60 days. Consumer study of whey candy was conducted with 153 no of peoples of different age group. Among that 89 no of consumers gave excellent preference to whey candy on sensory basis. The presence of fruit pulp and whey in the current product provides a nutritional edge over conventional products available in the market and is therefore expected to attract sales.

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