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Optimization of coating materials on jaggery for augmentation of storage quality

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ABSTRACT

Storage of jaggery is highly influenced by surrounding environment due to presence of sugars and mineral salt which are hygroscopic in nature. The investigations were carried out to evaluate the effect of coating materials on jaggery cubes for improvement of shelf life during storage. A new technology is required to prolong the shelf life of this commodity. Edible coating could perform one of the good alternatives as it has the capability to barrier the water loss. Hence jaggery cubes were coated with various ratio of CMC and HPMC concentration in glycerol solution and total concentration of CMC and HPMC (0.66, 1, 1.5, 2, 2.34 g/100ml) were taken into account for the study. The parameters such as moisture content, pH, total viable count (TVC) and reducing sugar were analyzed at an interval of 45 days and all these responses were analyzed statistically using design expert software to determine the significant results. The study determined that moisture content of edible coated jaggery decreased with increase of concentration of glycerol and total concentration of CMC and HPMC while TVC decreased with increase in the ratio of CMC and HPMC. The most favorable regressors of influencing factors obtained by compromise optimization of the responses were; 75:25 ratios of CMC and HPMC, 8 ml/100ml concentration of glycerol and 1.51 gm/100ml of total concentration CMC and HPMC recommended for coating the jaggery before storage to enhance the self-life.

Key words: CMC, HPMC, Jaggery cubes, Moisture content, Reducing sugar, Total viable count

The one major problem associated with jaggery storage is the presence of invert sugars and mineral salts which being hygroscopic in nature absorbs moisture particularly during monsoon season when ambient humidity is high and lead to spoilage. During storage, jaggery, basically suffers from four types of deterioration, viz. physical, chemical, biological and microbiological. The main problems related to solid jaggery storage are running-off (liquefaction) and deterioration of colour during storage (Kunte 1952). These problems are because of absorption of moisture and microbial attack. Rao (1973) found that jaggery from mature cane recorded less reduction in quality parameters under cold storage compared to jaggery from immature and over aged cane. Fermentation brought about by yeasts and complex biochemical degradation caused by moulds is the usual form of microbial deterioration. Moisture uptake resulting from exposure to humid atmosphere either during handling or storage is primarily responsible for most of the storage ills. Physically, it destroys the texture through dissolution and liquefaction. It also dilutes the sugars and lowers the sweetness. Chemically, it promotes inversion of sucrose which inturn leads to loss of texture, structure and body hardness. Moisture gain also encourages microbial infection and degradation. Jaggery also becomes more hygroscopic at higher temperatures (Verma 1985). Drying of jaggery to reduce its initial moisture content is essential for storage (Baboo and Ghosh 1985). Neutralization of sugarcane juice acidity during boiling for preparation of jaggery with calcium carbonate and sodium carbonate resulted in lower absorption of moisture by the jaggery blocks, during storage (Shinde *et al*. 1983). The problem of storage of various food products, especially, jaggery has numerous complications and no standard method of storage for jaggery is available.

Numerous efforts have been made by several researchers to prevent jaggery from moisture and microbial growth and to increase the shelf life of jaggery by wrapping in common packaging materials, in airtight containers, polyethylene and IISR storage bins under ambient and different storage conditions. Although many packaging materials have been studied with the aim of increasing shelf life, no data are yet available on applying edible coating and packaging for a similar purpose. The purpose of the application of edible films or coatings is to prevent the food from moisture and microbial decay, to preserve aroma and flavor. As HPMC is safe for consumption in human beings and declared edible (Burdock 2007) when mixed with CMC (which is soluble

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in either hot or cold water and is compatible with a wide range of other food ingredients including protein, sugar and other hydrocolloids) the moisture resistant properties and mechanical strength of the film will increase (Ghandarzadeh *et al.* 2010). Also in stressed condition this combination does not change its structure and increases the nutritive value of food ingredients. HPMC and CMC based edible coating is known to have desirable barrier properties against moisture, oxygen and gases. Therefore, the purpose of the present study is to evaluate the effect of hydroxy propyl methyl cellulose (HPMC) and carboxy methyl cellulose (CMC) based edible coating to improve shelf life of jaggery.

MATERIALS AND METHODS

Fresh jaggery cubes of dimension 1" ×1" × 1" were moulded using the wooden frame (developed to obtain the jaggery cubes of desired dimension), using the juice from sugarcane varieties HD 2684 and HD 2824 at the jaggery manufacturing plant situated at Bhurarani village, Rudrapur, Distt. Udham Singh Nagar, Uttarakhand. Moulded jaggery cubes were packed under hygienic condition in the carton at room temperature for storage study. The edible coating materials as HPMC (Hydroxy propyl methyl cellulose), CMC (Carboxy methyl cellulose) and glycerol.

Preliminary experiments were carried on the basis of reviews cited on different concentration of HPMC, CMC and Glycerol in different edible materials. On the basis of results obtained in preliminary experiments, the variables and its levels were decided for conducting 20 experiments of edible coated jaggery.

As per the experimental design and results shown in Table 1, HPMC and CMC were makeup to 100 ml in distilled water and mix thoroughly to form uniform solution using magnetic. The solution was then heated at 70ºC in hot water bath for 25- 30 min to provide functionality to edible film. The hot solution was then cooled down in cold water bath for 30 min. Then the fixed amount of glycerol was added to solution as plasticizer and mix thoroughly using magnetic stirrer for 5 to 10 min. Prepared solution was heated to form a complex matrix at 45-50ºC for 10 min and cooled to room temperature. Fresh Jaggery cubes were coated using dipping method. The samples were dipped in the prepared edible solution for 25 sec and then placed in aluminum tray for few seconds so that the coating becomes uniform. The samples then placed in cabinet dryer for drying at 40˚C for 3-4 hr reported by Banarjee and Chen (1995).

Jaggery (5 g) with few drops of absolute alcohol was dried to constant weight at 70 °C in a hot-air oven (Mandal *et al.* 2006).

The pH of jaggery samples was measured directly by digital pH meter (Triode India Ltd.) at 30ºC (Chand *et al.* 2013).

Table 1 Experimental result of quality parameters for edible coated jaggery cubes during storage

Expt. No	Coded regressors			MC (%)	pH	TVC (cfu/g)	RS (µgm/ml)
	Ratio of CMC:HPMC (X_1)	Concentration of glycerol (X_2, ml/100ml)	Total concentration CMC and HPMC (X_3, g/100ml)				
1	-1(25:75)	-1(4)	-1(1)	10.93*	5.99	4400	4.4
2	1(75:25)	-1 (4)	-1(1)	11.14	5.92	3280	3.5
3	-1(25:75)	1(8)	-1(1)	11.21	5.9	5160**	3.99
4	1(75:25)	1(8)	-1(1)	11.2	5.98	3440	3.85
5	-1(25:75)	-1(4)	1(2)	11.19	5.97	3160	3.32
6	1(75:25)	-1(4)	1(2)	11.21	5.55*	4400	4.01
7	-1(25:75)	1(8)	1(2)	11.32**	5.96	3280	4.02
8	1(75:25)	1(8)	1(2)	11.11	5.97	3440	4.75
9	-1.68(8:92)	0(6)	0(1.5)	11.12	5.97	4000	4.55
10	1.68(92:8)	0(6)	0(1.5)	11.14	5.55	3440	4.75**
11	0(50:50)	-1.68(2.64)	0(1.5)	11.1	6.11	3040	3.04
12	0(50:50)	1.68(9.36)	0(1.5)	11.11	6.11**	3040*	3.01*
13	0(50:50)	0(6)	-1.68(0.66)	11.12	5.91	4000	3.37
14	0(50:50)	0(6)	1.68(2.34)	11.32	5.92	3520	3.11
15	0(50:50)	0(6)	0(1.5)	11.21	6.01	3160	3.21
16	0(50:50)	0(6)	0(1.5)	11.23	6.02	3160	3.11
17	0(50:50)	0(6)	0(1.5)	11.22	6.04	3040	3.08
18	0(50:50)	0(6)	0(1.5)	11.21	6.01	3160	3.11
19	0(50:50)	0(6)	0(1.5)	11.21	6.01	3040	3.12
20	0(50:50)	0(6)	0(1.5)	11.2	5.99	3040	3.1

* ,**represent minimum and maximum values of responses

One gram of sample was taken by scrapping off surface of stored edible coated jaggery cube. Serial dilutions were made by mixing the powdered sample and thoroughly agitating it with 10 ml of autoclaved distilled water in a test tube. The suspension was further used for serial dilution by withdrawing 1 ml from the tube and adding it to 9 ml of the sterile distilled water used as blank. Dilutions were made up to ten times for each sample. The sterile petriplates (90 mm) were poured with 20 ml of media. On solidification of media, one ml of each diluted sample was withdrawn aseptically from dilutions and placed on the surface of agar petriplate in triplicate. Then samples were spread by the spreader and plates were incubated at 28˚C for 2-3 days. The number of Colony Forming Units per gram (CFU/gm) was also determined for each jaggery samples. Each petri plate was divided into four equal parts and then the colonies were counted in a single part and then multiplied by 4, this gives the total number of colonies in a single petri plate (APHA 1992).

$$\text{CFU} = \frac{\text{Number of colonies} \times \text{dilution factor}}{\text{Volume of sample}}$$

Reducing sugar was determined by using DNS method as Solution A: added 10 g of NaOH to 1000 ml distilled water. Solution B: added 10 g of dinitrosalicylic acid and 2 g of crystal phenol to 500 ml solution A. Solution C: added 0.5 g of sodium sulphite to 1000 ml distilled water and solution D: added 400 g of Rochelle salt (sodium potassium tartarate) to 1000 ml distilled water. Stored edible coated jaggery sample 1.0 g was dissolved in 10 ml of distilled water. Added 100 µl of sample to 100 ml of distilled water and 3 ml of solution B. The contents placed in a boiling water bath for 15 min, cooled the test tubes and add 1 ml of solution D. Here obtained colour development after it added 1 ml of solution C and finally read the absorbance at 575 nm on spectrophotometer (Gusakov *et al*. 2011 and Chand *et al*. 2013).

Data analysis was done using Central Composite Rotatable Design of Response Surface Methodology (Design Expert 8.0.6 Software). It also helps to reduce the number of experiments without affecting the accuracy of results and to decide the interactive effects of influencing factors on the response. CCRD is a very efficient design tool for fitting second order model. The values of the influencing factors were coded as the axial point α [(2k/4)] in the range of (+1.68 and -1.68). The number of design points in (CCRD) is based upon a full fraction (F = 2k) factorial design points (Khuri and Cornell 1987). Experiments were also carried at centre point. The design is rotatable which means that all the points in the design area are at equal distance from the centre.

RESULTS AND DISCUSSION

Jaggery samples were stored in room temperature from January to August, 2013. In the preliminary experiment it was found that as humidity increases the texture of jaggery and taste of jaggery changed. It was also found that initially up to 30-35 days, no change in the jaggery sample was observed hence the investigation was carried out at an interval of 45 days, for 225 days**.** The initial moisture content of jaggery cube was 12.02 % (wb). Quality characteristics of stored edible coated jaggery cubes were determined in terms of moisture content (%), pH, total viable count (TVC, cfu/gm) and reducing sugar (µgm/ml).

Effect of influencing factors on moisture content

As shown in Table 1 it was observed that all the influencing factors considered during the study had significant effect on moisture content. Samples stored under ambient condition at 225th day of storage period, maximum moisture content was observed to be 11.32% (wb) for the experiment no 7, having ratio of CMC and HPMC (25:75), concentration of glycerol (8 ml/100ml) and total concentration of CMC and HPMC (2 gm/100ml) and minimum moisture content was found to be 10.93% (wb) for experiment no 1, having ratio of CMC and HPMC (8:92), concentration of glycerol (4ml/100ml) and total concentration of CMC and HPMC (1gm/100ml). Initially the samples (at 0 periods) had moisture content 12% (wb) and this moisture content was found to be decreased after 225th day of storage period. The reason behind this nominal decrease could be due to the fact that the samples having coating of CMC, HPMC and glycerol were subjected to drying and due to coating materials and drying, moisture content could not be absorbed by the samples. Coatings done with CMC, HPMC and glycerol could act as a moisture barrier (Bifani *et al.* 2007) and hence moisture could not be increased. While the moisture content for control samples (kept under ambient condition at o storage period) was found to be increased (15.59% (wb)) at 109th day of storage period. Hence, the samples were discarded. All the influencing factors (X_1, X_2 and X_3) were statistically analyzed and found highly significant (P<0.01) while the effect of factor X_2 on moisture content found highest due to high F_{cal}value (21.25)> F_{tab}5.99 and then is followed by factors X_3 and X_1.

Interpretation of moisture content: Fig 1 shows a contour plot between ratio of CMC and HPMC and concentration of glycerol at optimum point of total concentration of CMC and HPMC. This plot shows a rising ridge surface, i.e. moving away from saddle point moisture content decreased. Fig 2 shows a contour plot between ratio of CMC and HPMC and total concentration of CMC and HPMC at optimum point of concentration of glycerol which was concluded that moisture content increased with decrease in ratio of CMC and HPMC. Fig 3 shows the contour between concentration of glycerol and total concentration of CMC and HPMC at optimum point of ratio of CMC and HPMC. It was found that moisture content decreased with increase in concentration of glycerol from 6.5 to 8 ml/100ml which could be considered appropriate for safe storage of jaggery.

Effect of influencing factors on pH

As shown in Table 1 it was observed that all the

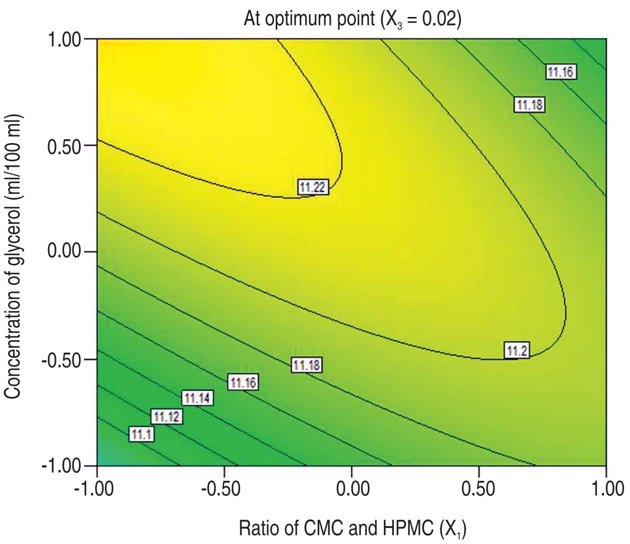

Fig 1 Contour plot of moisture content (%) between ratio of CMC and HPMC and concentration of glycerol

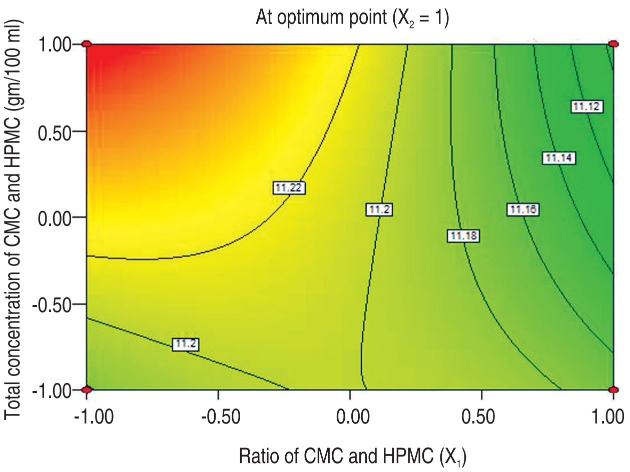

Fig 2 Contour plot of moisture content (%) between ratio of CMC and HPMC and total concentration of CMC and HPMC

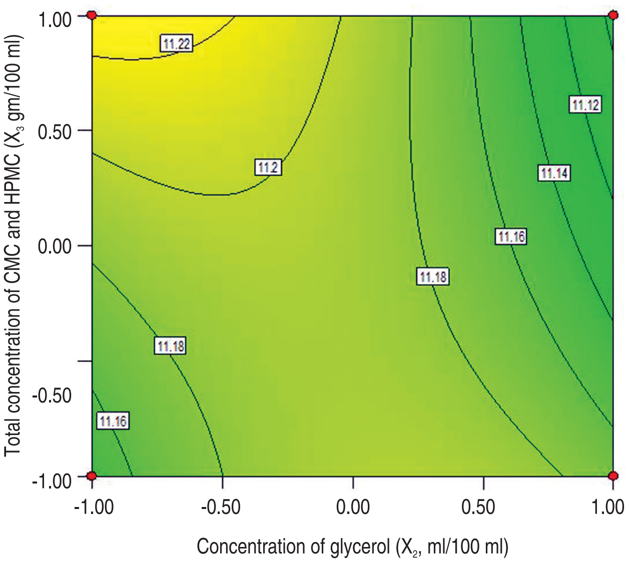

Fig 3 Contour plot of moisture content between concentration of glycerol and total concentration of CMC and HPMC

influencing factors considered during the study had significant effect on pH. For the samples, stored under ambient condition at 225th day of storage period, the maximum pH was observed to be 6.11 for the experiment no 12 having ratio of CMC and HPMC (50:50), concentration of glycerol (9.36 ml/100ml) and total concentration of CMC and HPMC (1.5 g/100ml) and minimum pH was found to be 5.55 for experiment no 6 having ratio of CMC and HPMC (75:25), concentration of glycerol (4 ml/100ml) and total concentration of CMC and HPMC (2 gm/100ml). Initially the samples (at 0 period) had pH 6.95 and this pH was found to be decreased after 225th day of storage period for almost every samples. The reason behind this nominal decrease perhaps could be due to the microbial attack on jaggery samples. Due to microbial attack, reducing sugar increased due to which pH of samples decreased while the pH for control samples (kept under ambient condition at o storage period) was found to be decreased (5.37) at 109th day of storage period. Hence, the sample was discarded. All the influencing factors (X_1, X_2 and X_3) were statistically analyzed (ANOVA) out of them factor (X_1) was highly significant (P<0.01) compared to X_2 and X_3(P<0.05) while the effect of factor X_1 on pH was highest due to high F_{cal} value (14.2)> F_{tab}5.99 and followed by factors X_3 and X_2.

Interpretation of pH: At interactive level, Fig 4 shows a falling ridge surface in which the value of pH was decreasing when moving towards the saddle point. From Fig 5 it was found that the value of pH decreased with decrease in ratio of CMC and HPMC and total concentration of CMC and HPMC. Fig 6 concluded that there is increment in total concentration of CMC and HPMC and reduction in concentration of glycerol the pH of coated jaggery samples were decreasing.

Effect of influencing factors on total viable count

The table 1 shows total number of viable count and

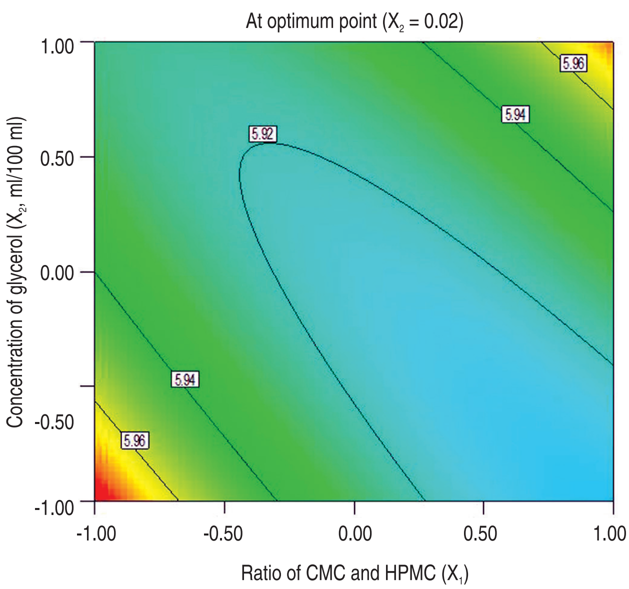

Fig 4 Contour plot of pH between ratio of CMC and HPMC and concentration of glycerol

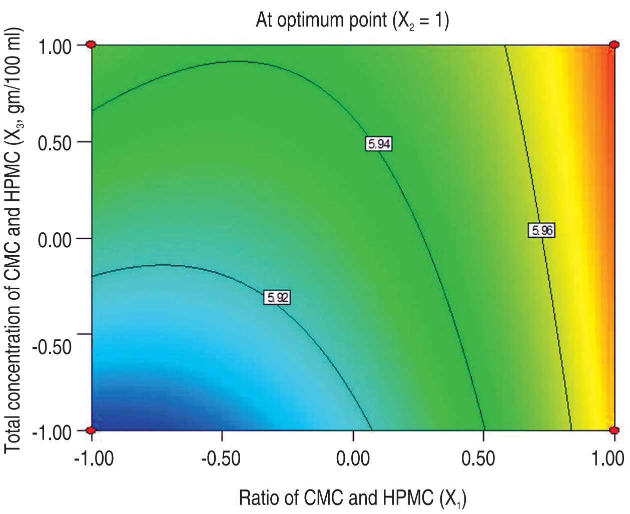

Fig 5 Contour plot of pH between ratio of CMC and HPMC and total concentration of CMC and HPMC

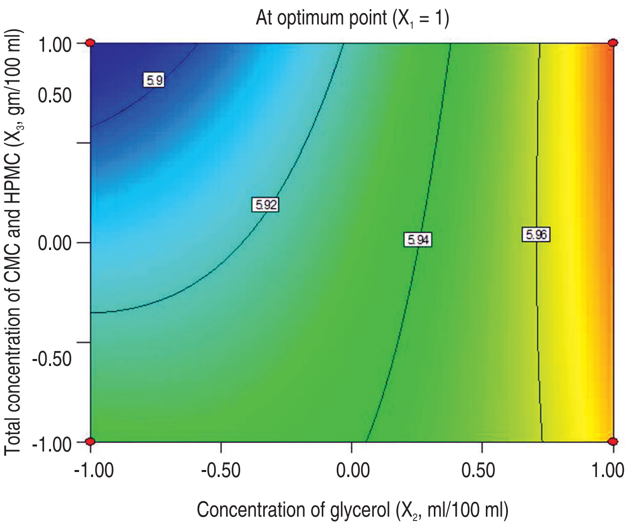

Fig 6 Contour plot of pH between concentration of glycerol and total concentration of CMC and HPMC

observed that all the influencing factors considered during 0the study had significant effect on TVC. For the samples stored under ambient condition at 225th day of storage period, the maximum TVC was observed to be 5.16×10^3 (cfu/gm) for the experiment no 3 having ratio of CMC and HPMC (25:75), concentration of glycerol (8 ml/100ml) and total concentration of CMC and HPMC (1 g/100ml) and minimum TVC was found to be 3.04×10^3 (cfu/gm) for experiment no 12 having ratio of CMC and HPMC (50:50), concentration of glycerol (9.36 ml/100ml) and total concentration of CMC and HPMC (1.5 gm/100ml). Initially the samples (at 45th day) had TVC 1.24×10^3 (cfu/gm) and this TVC was found to be increased after 225th day of storage period for all most all the samples. The reason behind this increase could be due to the storage condition of edible coated jaggery samples as the samples were kept under open conditions without any packaging. But the coating materials, i.e. CMC, HPMC and glycerol upto some extent successfully controlled the microbial attack. After 109th day of storage period, TVC for control sample was found to be 5.843.04×10^3(cfu/gm), which shows that samples without any coating got affected by environmental conditions and got changed. All the influencing factors (X_1, X_2 and X_3) were statistically analyzed (ANOVA); out of them factors ($X_1$and X_3) were highly significant (P<0.01) as compared to X_2 (P<0.05) while the effect of factor X_3 on TVC was highest due to high F_{cal} value (17.15) > F_{tab}5.99 and followed by factors $X_1$and X_2.

Interpretation of total viable count: The Fig 7 shows a contour plot between ratio of CMC and HPMC and concentration of glycerol at optimum point of total concentration of CMC and HPMC decreased the TVC by increasing the regressor of ratio of CMC and HPMC. Fig 8 shows a contour plot between ratio of CMC and HPMC and total concentration of CMC and HPMC from where it can be analyzed that with increase in ratio of CMC and HPMC and total concentration of CMC and HPMC TVC gradually decreased in jaggery samples. Fig 9 shows that a contour plot between concentration of glycerol and total concentration of CMC and HPMC at optimum point of ratio of CMC and HPMC decrease the TVC of Jaggery samples.

Effect of influencing factors on reducing sugar

As shown in Table 1, data of reducing sugar shows that all the influencing factors considered during the study were not showing much significant effect on reducing sugar. For the samples, stored under ambient conditions at 225th day of storage period, the maximum reducing sugar was observed to be 4.75 (µgm/ml) for the experiment no 10 having ratio of CMC and HPMC (100:0), concentration of glycerol (6 ml/100ml) and total concentration of CMC and HPMC (1.5 gm/100ml) and minimum reducing sugar

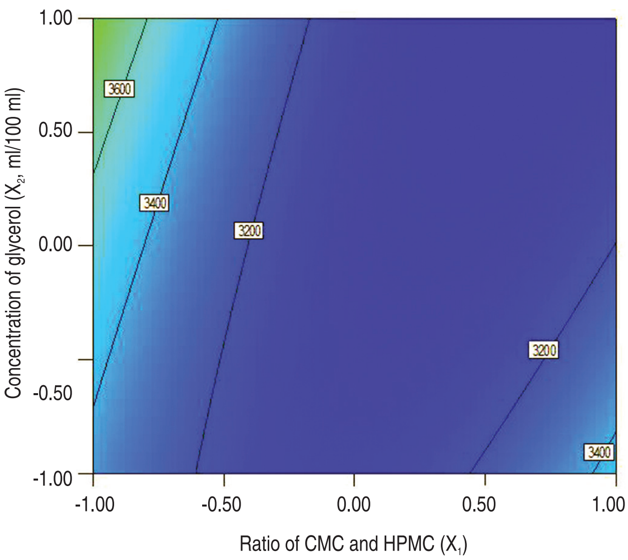

Fig 7 Contour plot of TVC (cfu/gm) between ratio of CMC and HPMC and concentration of glycerol

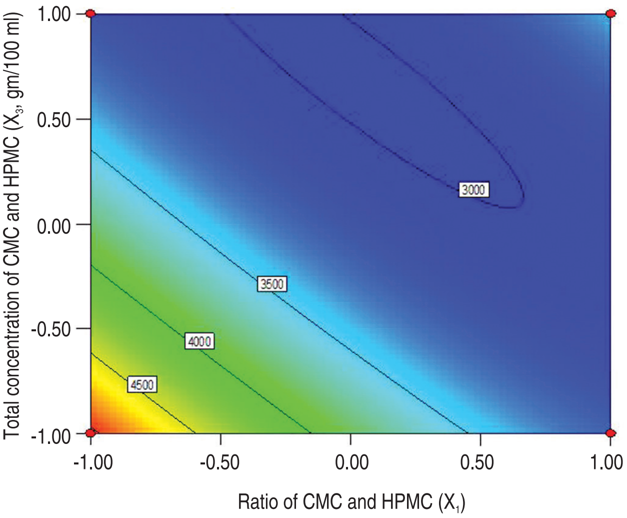

Fig 8 Contour plot of TVC (cfu/gm) between ratio of CMC and HPMC and total concentration of CMC and HPMC

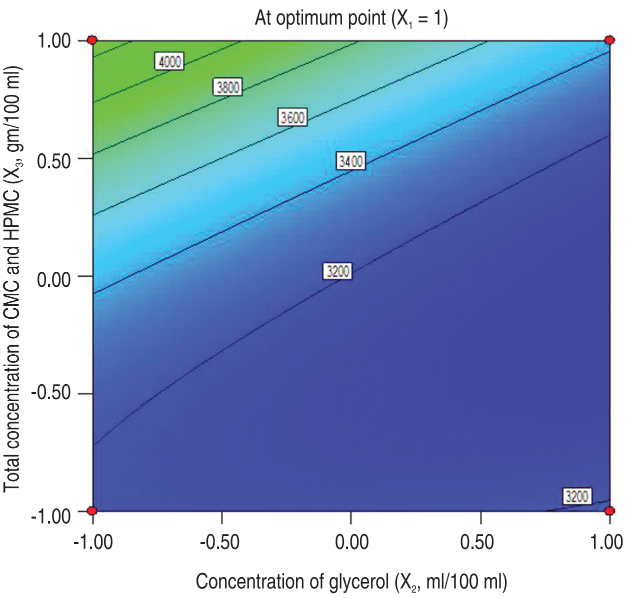

Fig 9 Contour plot of TVC (cfu/gm) between concentration of glycerol and total concentration of CMC and HPMC

was found to be 3.01(µgm/ml) for experiment no 12 having ratio of CMC and HPMC (50:50), concentration of glycerol (9.36 ml/100ml) and total concentration of CMC and HPMC (1.5 gm/100ml). Initially the samples (at 0 period) had reducing sugar 1.263 (µgm/ml) and this reducing sugar was found to be increasing after 225th day of storage period. It could be due to the fact that the samples contain sucrose in high amount and due to moisture content and microbial attack the inversion of sucrose into glucose and fructose takes place. The reducing sugar for control samples (kept under ambient condition at 0 storage period) was found to be increased at 109th day of storage period. Hence the sample was discarded and no further observations were recorded. All the influencing factors (X_1, X_2 and X_3) were statistically analyzed (ANOVA); out of them factor (X_1) was highly significant (P<0.01) as compared to X_3 (P<0.05) while the effect of factor X_1 on reducing sugar was highest due to high F_{cal} value (29.55) > F_{tab}5.99 then factor X_3 (F_{cal} 5.76> F_{tab} 3.48) while no effect of X_2 on reducing sugar.

Interpretation of reducing sugar: Fig 10 shows a contour plot between ratio of CMC and HPMC and total concentration of CMC and HPMC at optimum point of concentration of glycerol (8 ml/100ml), minimum reducing sugar obtained at centre point (50:50), away from centre the value of reducing sugar increased with increasing the value of regressors of ratio of CMC and HPMC. Fig 11 shows a contour plot between concentration of glycerol and total concentration of CMC and HPMC at optimum point of ratio of CMC and HPMC (75:25), shows that the value reducing sugar is increasing with increase in regressor of concentration of glycerol and total concentration of CMC and HPMC.

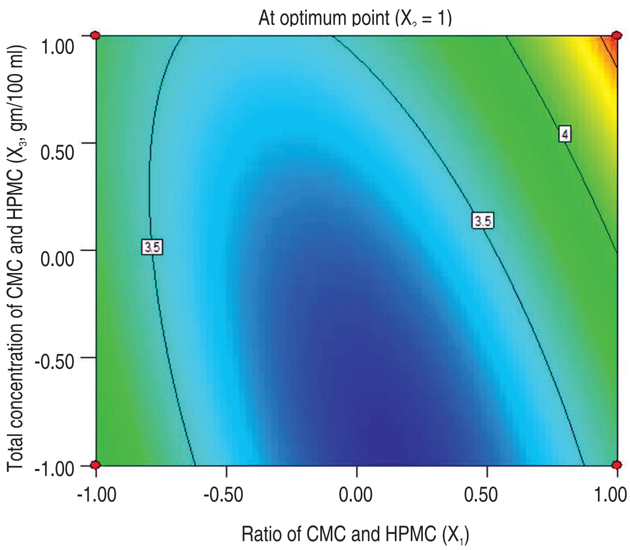

Fig 10 Contour plot of reducing sugar

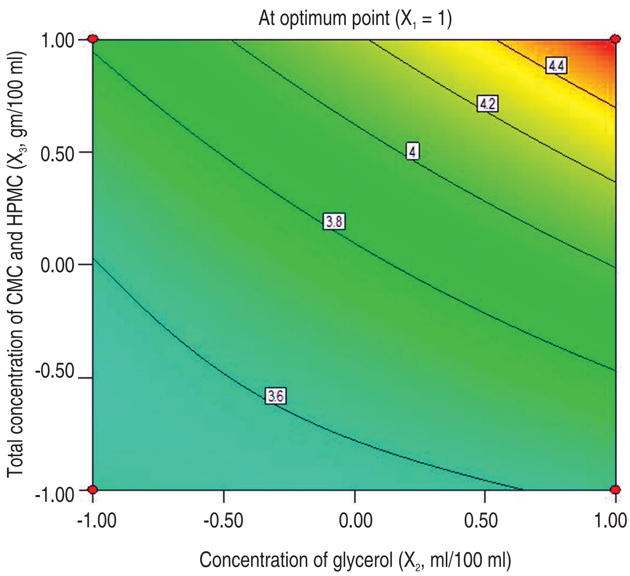

Fig 11 Contour plot of reducing sugar

Table 2 Optimum regress or of influencing factors

Influencing factors	Coded level	Actual level
Ratio of CMC and HPMC (X_i)	1	75:25
Concentration of glycerol (X_2, gm/100 ml)	1	8
Total concentration of CMC and HPMC (X_3, gm/100 ml)	0.02	1.51

Optimization of experimental parameters

The experimental value of quality parameters of edible coated jaggery samples are reported in Table 1. The numerical optimization of all the factors was carried out using Design-Expert 8.1.7.0 statistical software for production of good quality jaggery cubes. The values of these factors obtained and recommended these optimized values for storage purpose of edible coated jaggery given in Table 2 in respect of moisture content, pH, TVC and reducing sugar.

Jaggery storage has an immense problem during summer and rainy seasons due to change in its texture and taste. On the basis of storage qualities viz moisture content, pH, total viable count and reducing sugar, it was observed that moisture content of jaggery cubes decreased due to total concentration of CMC and HPMC and glycerol coating materials. It was also observed that total viable count decreased with increase of ratio of CMC and HPMC. Hence, the edible coating with CMC and HPMC may improve the shelf life of jaggery and retained upto 225 days of storage.

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