Effect of Active Packaging and Coating Materials on Quality Parameters of Jaggery Cubes

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Abstract - **Jaggery is one of the ancient sweetening agents known to man and is an integral part of the rural diet in many countries. It's storage is highly influenced due to presence of invert sugars and mineral salt which are hygroscopic in nature. To extend the shelf life of this commodity there is a need of new technologies. Edible coating and active packaging appears one of the good alternatives for this. Edible coating is used as a barrier to minimize water loss while active packaging maintains dry conditions within packages. The independent variables decided for active packaging of jaggery cubes were, moisture absorber (2.64, 4,6,8 and 9.36g), concentration of CMC and HPMC (0.528, 0.8, 1.2, 1.6 and 1.872g/ml) and thickness of high density polyethylene bags (66, 100, 150,200 and 234µm) were taken for research work. The storage study of active packaged edible coated jaggery cubes was conducted for 180 days. The responses via pH, water activity, and reducing sugar were analyzed at different interval. The statistical analysis of each sample was done to check the significance of data. The water activity and reducing sugar decreased with increase moisture absorber, concentration of CMC and HPMC and thickness of HDPE bags. The pH of stored active packaged edible coated jaggery varied from 5.47 to 5.74 and water activity was in range of 0.531 to 0.699 and reducing sugar varied from 1.33 to 2.98 µg/ml. For best shelf life of jaggery. Optimum values of independent variables obtained by compromise optimization of the responses were; 8g moisture absorber, 1.178 g/ml concentration CMC and HPMC and 197µm thickness of HDPE bags for 180days storage.**

Keywords: Jaggery cubes, moisture absorber, HDPE, pH, water activity and Reducing sugar.

1. INTRODUCTION

Gur (Jaggery) is a natural, traditional sweetener made by the concentration of sugarcane juice and is known all over the world [1] in different local names (called as Gur in India, Desi in Pakistan, Panela in Maxico and South America, Jaggery in Burma and African countries, Hakuru in Sri-Lanka, Naam Taan Oi in Thailand) and Khandsari [13]. It is a traditional unrefined non-centrifugal sugar consumed in most of country. Containing all the minerals and vitamins present in sugarcane juice, it is known as healthiest sugar in the world. India is the largest producer and consumer of jaggery. Out of total world production, more than 70% is produced in India [11]. In India, of the 300 Mt of sugarcane produced, 53% is processed into white sugar, 36% into jaggery and khandsari, 3% for chewing as cane juice, and 8% as seed cane [12]. Jaggery and khandsari have withstood competition protecting farmers' interests besides meeting ethnic demands. Processes and equipments have been developed for quality solid, liquid and powder jaggery. Liquid jaggery has been commercialized. The organic clarificants developed help to retain jaggery as organic food. Jaggery is prepared by concentrating the sugarcane juice and it is available in the form of solid blocks and in semi-liquid form. Besides this, the sap collected from some palm trees such as palmyra-palm (*Borassus flabellifer* L.), coconut-palm (*Cocos nucifera* L.), wild datepalm (*Phoenix sylvestris* Roxb.) and sago-palm (*Caryota urens* L.) is used for preparation of jaggery [9]. For ease of handling, packaging and storage, jaggery in granular form is becoming popular. India is world's largest producer of sugar and sugarcane. Sugarcane in India is processed in to sugar, gur and khandsari and undergoes considerable weight reduction during processing. The methods of converting sugarcane and manufacturing sugar, gur and khandsari are different but a great value is added in the manufacturing of these consumable final products. Further it offers employment opportunity to millions of people. It is also known as Gul, gud, Jaggery, Vellum and Bella [1]. Jaggery is far complex than sugar, as it is made up of longer chains of sucrose. Hence, it is digested slower than sugar and releases energy slowly and not spontaneously. This provides energy for a longer time and is not harmful for the body. But this does not certify it fit for consumption by diabetics, because ultimately it is sugar. Jaggery also gathers a considerable amount of ferrous salts (iron) during its preparation, as it is prepared in iron vessels [4]. This iron is also good for health, particularly for those who are anaemic or lack iron. Again, jaggery also contains of traces of mineral salts which are very beneficial for the body. Furthermore, jaggery is very good as a cleansing agent. It cleans lungs, stomach, intestines, oesophagus and respiratory tracts. Gur is known to produce heat and give instant energy to a human body. In many parts of India, there is a tradition of serving a glass of water with Gur to welcome the guests. Gur is also used as a cattle feed, in distillery, medicine manufacturing unit, ayurvedic medicines, ayurvedic sura and ayurvedic health tonics. Recently Gur has also found a place in confectionary items. A usage of Gur is also seen in leather and tobacco industries. Besides, in cement industries and coalmines, Gur is supplied to the workers for in order to protect them from dust allergies as well as at the time of natural calamities, the district administration purchases Gur and distributes it to the victims for various health benefits [2]. The 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: physical, chemical, biological and microbiological. The main problems related to solid jaggery storage are running-off (liquefaction) and deterioration of color during storage [8]. These problems arise due to quick absorption of moisture and microbial attack on jaggery. Jaggery from mature cane recorded less reduction in quality parameters under cold storage compared to the jaggery from immature and over aged cane. Fermentation brought about by yeasts and complex biochemical degradation caused by moulds is the usual forms of microbial deterioration [10]. Therefore, the aim of this study is to evaluate the effect of High Density Polyethylene (HDPE) bags of different sizes, concentrations of hydroxy propyl methyl cellulose (HPMC; E464) and carboxy methyl cellulose (CMC; E466) based edible coating and moisture absorber to improve shelf life of jaggery. The moisture absorber (food grade silica gel) absorbs moisture present in the jaggery cubes inside the package to ensure the quality of product during storage and extends its shelf life. It can also eliminate odor problems and prevent microbial growth in the product. HPMC and CMC based edible coating is known to have desirable barrier properties against moisture, oxygen and gases.

2. MATERIALS AND METHODS

2.1 Raw materials

The fresh jaggery cubes of dimensions 1"x1"x1" were made from sugarcane juice of same day harvested sugarcane (variety early 0238) at M/s jaggery making plant located at village- Raipur, Udham Singh Nagar, Uttarakhand using stainless steel frame with dimensions (L x W x H: 14"x10"x1"). HPMC (Hydroxy propyl methyl cellulose) and CMC (Carboxy methyl cellulose). The moisture absorber like Silica gel in the form of sachet pad and packaging materials such as HDPE bags of different thickness.

2.2 Methodology

The active packaging material as amount of moisture absorber (Triple refined super activated white DMF and PCP free silica gel) was calculated and decided on the basis of amount of water transmitted from environment into bags and water vapor present in air inside the bags. So the total amount of water was calculated for 180 days i.e. from 30th December, 2016 to 30th June, 2017 (storage period). A stainless steel frame of dimensions 16 x 9 x 1" (L x W x H) was made from

(Kumoun steel works and general order supplier, Nagla, Pantnagar). Liquid jaggery at ambient temperature was poured into mould for making uniform size 1x1x1"jaggery cubes, after one hour solidified jaggery cubes were separated from frame and kept into carton. The powder form of HPMC and CMC (0.528, 0.8, 1.2, 1.6 and 1.872g/100ml) was taken and mixed with 100 ml distilled water for preparation of edible coating solution. The uncoated jaggery cubes were dipped for 22 to 25 seconds with the help of forceps into the solution and kept them into cleaned aluminum tray for making complex film over the surface of each jaggery cubes.. The coated jaggery cubes were dried into tray dryer (MSW) at 40 ºC for 3-4hours and cooled the material at room temperature [2, 3]. Finally dried coated jaggery cubes of 195±5g were kept into different thickness of HDPE bags with moisture absorber (kept into low density foam) and sealed using hand operated sealing machine (MSW) and stored for a period of 180 days.

2.3 Experimental Design

Response surface methodology (RSM) was used for the design and analysis of all experiments for three independent variables at five levels. It also helps to reduce the number of experiments without affecting the accuracy of results and to decide the interactive effects of independent variables on the responses. Central Composite Rotatable Design (CCRD) which is a very efficient design tool for fitting second order mathematical model and was selected for the present study. The total number of design points in (CCRD) is based upon a full fraction ($F = 2^k$) factorial design points [7]. The second order mathematical regression equation is as follows,

$$Y = \beta_o + \sum_{i=1}^{n} \beta_i X_i + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} \beta_{ij} X_i X_j + \sum_{i=1}^{n} \beta_{ii} X_i^2 \qquad ... (1)$$

Where,

Y = Dependent variable and

β_o, β_i, β_{ii}, β_{ij} = regression coefficients,

X_i and X_j = independent variables in coded form (where, i = 1, 2 ...n and j = 1,...n),

Analysis of variance (ANOVA) was used to examine the statistical significance and lack –fit of model. For determining the adequacy of models, using Fisher's F-value, lack of fit test, coefficient of determination (R^2) and R^2 adj. The significance of all the terms in the second order mathematical model was judged statistically by the F value at different significance level.

Table 1 Levels of independent variables in coded and actual form

Independent variables		Coded levels				
Name	Codes	-1.68 (α)	-1	0	+1	+1.68 (α)
		Actual levels				
Moisture Absorber	(X_1,g)	2(2.64)	4	6	8	10 (9.36)
Total Conc. of CMC:HPMC	(X_2,g/100ml)	0.528	0.8	1.2	1.6	2(1.872)
Thickness of HDPE bags	(X_3,μm)	66	100	150	200	234

2.4 Determination of Quality Parameters

2.4.1 pH

The pH value of sample was measured directly by digital pH meter (Triode India Ltd.). Least count of pH meter is 0.01 and can work between temperature ranges of 0-100ºC. The pH probe was calibrated using standard buffer solution (pH 4 and 7) prior to measurement pH of stored active packaged edible coated jaggery sample at 30ºC. One gram of stored active packaged edible coated jaggery cube was mixed with 10 ml distilled water in test tube after properly mixing of sample with distilled water, the probe of digital pH meter was place in test tube and reading of pH was noted. Before the placing of probe in test tube the digital pH meter should be calibrate as reported by [2, 4].

2.4.2 Water activity

Water activity of the sample and relative humidity of the air was in equilibrium then measurement of the headspace, humidity given the water activity of the sample [1]. The crushed jaggery was taken from stored active packaged edible coated jaggery sample and crushed jaggery was placed in sample cup of the water activity meter (Rotronic Pvt. Ltd, Model No. 54858 001) shown in Figure 1. Before placing the sample cup with crushed jaggery inside the water activity meter the bottom surface of the sample cup should be full covered by crushed jaggery then the sample placed inside and in front of sensor of the water activity meter. The reading of water activity, RH and temperature were automatically shown on the screen of the water activity meter.

2.4.3 Reducing sugar

Reducing sugar of stored active packaged edible coated jaggery samples were determined by DNSA method as follows.

Reagents

Solution A: 10 gm of NaOH was added to 1000 ml distilled water.

Solution B: 10 gm of dinitrosalicylic acid and 2 gm of crystal phenol was added to 500 ml of solution A.

Solution C: 0.5 gm of sodium sulphite was added to 1000 ml distilled water.

Solution D: 400 gm of Rochelle salt (Sodium potassium tartrate) was added to 1000 ml distilled water.

One gram stored active packaged edible coated jaggery sample was dissolved in 10 ml of distilled water. The 100 µl of sample 100 µl of distilled water is added and 3 ml of solution B was also added. The contents were placed in a boiling water bath for 15 min. The test tubes were cooled and 1 ml of solution D was added. Colour developed was observed. Immediately after colour development 1 ml of solution C was added. The absorbance was read at 575 nm on spectrophotometer then the reducing sugar was calculated by standard curve method [6].

3. RESULTS AND DISCUSSION

Designed investigations were carried out to enhance the shelf life of edible coated jaggery cubes using active packaging. Different levels of independent variables were decided on the basis of preliminary experiments viz moisture absorber (2.64, 4, 6, 8 and 9.36g), concentration of CMC and HPMC (0.528, 0.8, 1.2, 1.6 and 1.872g/100ml) and thickness of high density polyethylene bags (66, 100, 150, 200 and 234µm) and an optimum value of ratio CMC and HPMC (75:25) was recommended to enhance the shelf life of jaggery cubes for 225 days by [2]. The initial moisture content of coated jaggery cube after drying from sample 1 to 20 were (12.2, 12.53, 12.04, 12.94, 12.4, 11.14, 12.1, 11.6, 12.2, 11.9, 12.1, 12.5, 12.2, 11.9, 12.1, 12.5, 12.1, 12.2, 11.91 and 12.11 %, w.b.) in case of control sample it was 12.5% (w.b). Solution of CMC and HPMC was prepared and then coated on the surface of the jaggery cubes by dipping method (20-25 second). After the coating of jaggery cubes these cubes were kept in clean aluminum tray and then dried in tray dryer at 30 ºC for 3 to 4 hours to make the liquid coating solution in form of dry matrix, stiff and hard on the surface of the jaggery cubes, after drying, edible coated jaggery cubes were kept in the different thickness of HDPE bags with the different levels of moisture absorber and sealed by hand operated sealing machine. Storage studies of active packaged edible coated jaggery samples were conducted at different intervals for 180 days. The quality characteristics of stored active packaged edible coated jaggery cubes were evaluated in terms of pH, water activity, and reducing sugar (µg./ml). Statistical analysis was conducted to evaluate the effect of independent variables viz moisture absorber, concentration of CMC and HPMC and thickness of HDPE bags on various quality parameters considered during storage study. The adequacy of the model was tested using coefficient of determination (R^2). Optimization of process parameters was carried out using Design Expert software version 10.

3.1 Effect of independent variables on various responses

The effect of independent variables such as moisture absorbers, concentration of CMC and HPMC and thickness of HDPE bags with their different levels on various responses (quality characteristics) viz pH, water activity, and reducing sugar (µg/100ml) were determined and statistically analyzed.

3.1.1 pH

The sample were analyzed with the help of pH meter at 180 days of storage period the initial pH of active packaged edible coated jaggery cubes of samples 1 to 20 was found in range from 5.4 to 5.56 while in case of control sample it was 5.55. The maximum value of pH was observed to be 5.74 with the combination of moisture absorber ($X_1 = 8g$). concentration of CMC and HPMC ($X_2 = 1.6$ g/100ml) and thickness of HDPE bag ($X_3 = 200$ µm) while the minimum value of pH was found to be 5.47 with the combined effect of moisture absorber ($X_1 = 4$ g), concentration of CMC and HPMC ($X_2 = 0.8$g/100ml) and thickness of HDPE bag ($X_3 = 100$ µm). After 180 days of

storage, pH was found to be slightly increased for almost all samples. The reason behind this nominal increased in pH perhaps could be due to less microbial attack and slightly increased in the value of reducing sugar as compared to control sample kept under ambient condition 5.91. No variation in pH of the sample was found. Similar findings were obtained by [2].

3.1.1.1. Statistical analysis of pH

The regression model (Eqn 1) was checked using a numerical method employing the coefficient of determination (R^2), pred-R^2, adj-R^2 and then calculated as shown in equation (2). The second order regression model was developed with the values of R^2 of (0.9841), adj-R^2 (0.9697) and pred-R^2 (0.9220) for pH. Model was found highly significant (P<0.01). Therefore, second order regression equation was considered to be an adequate for describing the effect of independent variables on pH of stored active packaged edible coated jaggery cubes. At linear as well as quadratic level, it was observed that all moisture absorber, concentration of CMC and HPMC and thickness of HDPE had highly effect on pH at 1% level of significance while no significant effect was found at interactive level. A second order polynomial equation

(Eqn.2) was developed which represents an empirical relationship between the response and independent variables in actual form. The predictive equation for pH is given below:

$$pH = 5.57 + 0.050X_1 + 0.047X_2 + 0.054X_3 - 0.005X_1X_2$$
$$- 0.005X_1X_3 -$$
$$0.010X_2X_3 + 0.019X_1^2 + 0.022X_2^2 + 0.020X_3^2$$
$$\dots (2)$$

X1, X2, and X_3 are the moisture absorber, concentration of CMC and HPMC and thickness of HDPE bags, respectively. Eqn. 2 indicates that, with increase in the levels of the variables linearly, pH was increased at 180 days of storage of the samples. Quadratic terms also indicate increasing the effect on pH. The Fig 1 depicts that the effect of thickness of high density polyethylene bags on pH at optimum conditions of moisture absorber (8g) and concentration of carboxy methyl cellulose and hydoxy propyl methyl cellulose (1.178g/ml) of stored active packaged jaggery cubes. Slightly changed the pH from (5.61, 66µm) to (5.60, 80µm) after that it gradually increased upto 5.74 with the increased the values of thickness of HDPE bags for reduction of microbial growth in the sample
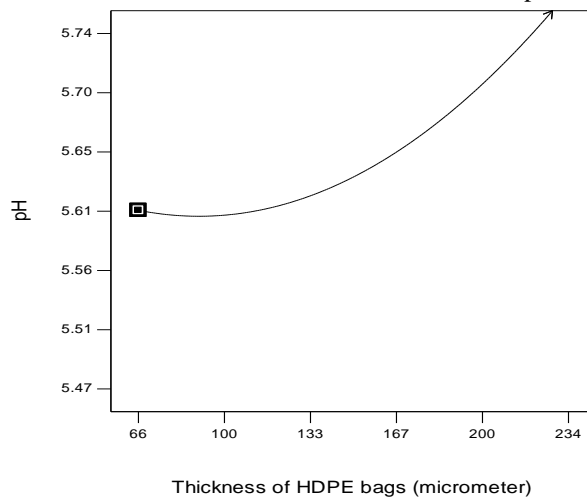

Fig 1 Effect of thickness of HDPE bags on pH at optimum points (8g and 1.178g/ml)

3.1.2 Water activity

The water activity of store jaggery was calculated at 180 days of storage period. It observed that maximum value of water activity was obtained to be 0.699 with the combination of moisture absorber (X_1 = 4, 6g). concentration of CMC and HPMC (X_2 = 0.8, 1.2g/100ml) and thickness of HDPE bag (X_3 = 100, 66 µm) while the minimum value of water activity was found to be 0.531 with the combined effect of moisture absorber (X_1 = 8 g), concentration of CMC and HPMC (X_2 = 1.6g/100ml) and thickness of HDPE bag (X_3 = 200 µm). At 180 days, the water activity of active packaged edible coated jaggery cubes of samples 1 to 20 was found in range from 0.531 to 0.699 while in case of control sample it was 0.962. The water activity was found to be nominal decreased after 180 days of storage time in active packaged edible coated jaggery cubes for all samples, while the water activity for control sample (kept under ambient conditions) was found to

be increased from 0.688 at zero days to 0.962 at 180 days during the whole storage study. The reason behind this was the absence of moisture absorber, barrier of concentration of CMC and HPMC edible film and thickness of HDPE bag. Because of absence of all independent variables with control sample it was found that all environmental factors like water vapor present in air (RH), temperature of environment and microbes presents in the environment were easily affected the control jaggery cubes finally control sample became harder an unacceptable in nature.

3.1.2.1. Statistical analysis of water activity

The polynomial regression model (Eqn 1) was checked using a numerical method employing the coefficient of determination (R^2), pred-R^2, adj-R^2 and then calculated as shown in equation (3). The second order regression model was developed with the values of R^2 of (0.9494), adj-R^2 (0.9038) and pred-R^2 (0.5723) for water activity of the

samples. The coefficient of variation as well as standard deviation of water activity were found 2.17% and 0.014 which indicates minimum variability in data fitted in the model. Model was found highly significant (P<0.01). A second order regression equation (Eqn. 3) was developed which represents an empirical relationship between the response and independent variables in actual form. The predictive equation for water activity of active packaged coated jaggery cubes is given below:

$$\text{Water activity} = 0.68 - 0.021X_1 - 0.018X_2 - 0.034X_3 + 0.00675X_1X_2 - 0.013X_1X_3 - 0.017X_2X_3 - 0.017X_1^2 - 0.012X_2^2 - 0.012X_3^2 \qquad \dots (3)$$

Decreases the water activity means decrease the microbial infection on jaggery cube, it was due to the use of moisture absorber in the storage study and by its nature reduces the gaining the moisture from the environment. The Fig 2 shows that the effect of thickness of high density polyethylene bags on water activity at optimum conditions of moisture absorber (8g) and concentration of CMC and HPMC (1.178g/ml) of the samples during storage. The pattern reveals that water activity decreased gradually from (0.687, 66µm) to (0.531, 234 µm) with increase the values of thickness of HDPE bags represent the better storage life of jaggery cubes.
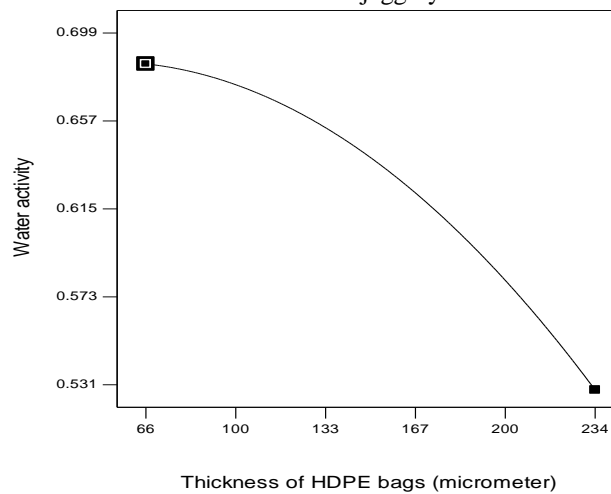

Fig 2 Effect of thickness of HDPE bags on water activity at optimum points (8g and

1.178g/ml)

3.1.3 Reducing sugar
From the experimental data it was observed that storage study of active packaged edible coated jaggery cubes had significant effect on reducing sugar (µg/ml). When samples was stored upto 180 days, the maximum value of reducing sugar was obtained to be 2.98 µg/ml with the combination of moisture absorber (X_1 = 6g). concentration of CMC and HPMC (X_2 = 1.2g/100ml) and thickness of HDPE bag (X_3 = 66 µm) while the minimum value of reducing sugar was found to be 1.33µg/ml with the combined effect of moisture absorber (X_1 = 8 g), concentration of CMC and HPMC (X_2 = 1.6g/100ml) and thickness of HDPE bag (X_3 = 200 µm). At 180 days, the reducing sugar of active packaged edible coated jaggery cubes of samples 1 to 20 was found in range from 1.33 to 2.98 µg/ml while in case of control sample it was 4.04 µg/ml. similar results were found by [2]. Therefore at 180 days of storage period, the reducing sugar of active packaged edible coated jaggery cubes was obtained less than in case of control sample. Due to high moisture absorber and thickness of HDPE bags used in the samples were responsible to stop the inversion of sucrose into glucose and fructose. But in case of control sample, variation was found from zero days to 180 days of storage.

3.1.3.1 Statistical analysis of reducing sugar
The second order regression model was developed with the values of R^2 of (0.9161), adj-R^2 (0.8405) and pred-R^2 (0.3587) for reducing sugar of the samples. Furthermore, the value of adj-R^2 (0.8405) relatively close to the coefficient of determination (R^2 0.9161) of the model fitted into the experimental data which showing some variability in reducing sugar data. It was observed that the effect of concentration of CMC and HPMC had significant (p<0.05) while thickness of HDPE bags had highly effect on reducing sugar (p<0.01). But no effect of moisture absorber was found on the 1response. It means that reducing sugar had no change due to development of barrier properties in the sample during the storage. A second order regression equation (Eqn. 4) was developed which represents an empirical relationship between the response and independent variables in actual form. The equation in terms of actual factors can be used to make predictions about the response for given levels of each independent variable. The predictive equation for reducing sugar of active packaged coated jaggery cubes is given below:

Reducing sugar (µg/ml)

$$= 1.45 - 0.084X_1 - 0.19X_2 - 0.39X_3 + 0.24X_1X_2 - 0.30X_1X_3 - 0.091X_2X_3 + 0.16X_1^2 + 0.091X_2^2 + 0.41X_3^2 \quad \dots (4)$$

Equation (4) indicates that, with increase in the levels of these variables, reducing sugar was gradually decreased at 180 days storage of the samples. Decreases the reducing sugar means decrease the growth of microbial on jaggery cube, as well as not become conversion of sucrose into fructose shown the best results it was due to the use of different thickness HDPE bags for storage study and stopped the movement of moisture from sample to the environment and vice versa. The Fig 3 reveals the variation in thickness of high density polyethylene bags on reducing sugar at optimum conditions of moisture absorber (8g) and concentration of carboxy methyl cellulose and hydoxy propyl methyl cellulose (1.178g/ml) to analyze their significant effect during storage. The graph shows reducing sugar is gradually decreasing from 2.98 to 1.33µg/ml with increased the values of thickness of high density polyethylene bags from 66 to 200µm due to maintain the best sealing property for the sample and prevent mechanical shock of the product at linear level.
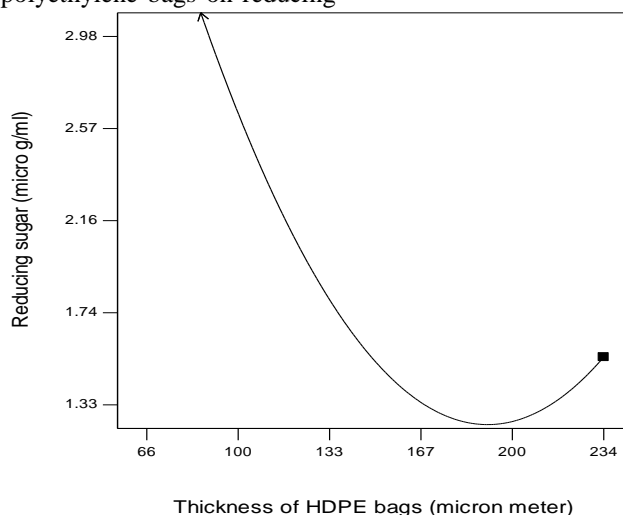

Fig 3 Effect of thickness of HDPE bags at optimum points (8g and 1.178g/ml)

4. CONCLUSION

It was concluded that the effect of active packaging and coating material was significant on the responses. The value of pH slithly increases with increase the values of moisture absorber and thickness of HDPE bags which was in acceptable range. The reducing sugar of coated jaggery cubes decreased gradually with the highest effect of thickness of HDPE bags due to creating best sealing property between products and environment. While, water activity found minimum which inhabit the growth of microorganism in the product. Thus jaggery can be stored in HDPE bags (197µm) with moisture absorber (8 g) for 180 days without any deterioration of product quality.

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