

## Evaluation of Traditional Production Process of Rock Candy and Optimization of Sucrose Crystallization (Part 2)

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**Abstract:** This research carried out with take into consideration of traditional process defects and on the basis of the most important effective on process parameters. Selected parameters which used for effluent and rock candy consist of supersaturation at 4 levels (1.3, 1.4, 1.5 and 1.6) and temperature at 3 levels (70, 80, 90°C) with 3 replications. The physiochemical tests consist of pH, color and invert sugar percentage done on all of the samples. According to the results, the rise in supersaturation and temperature has significant effect on all of the effluent and rock candy characteristics ( $P < 0.01$ ) so that color and invert sugar percentage increase and pH decrease as super saturation and temperature are increasing. Furthermore the interaction effect of super saturation and temperature on pH, color and invert sugar percentage of rock candy is meaningful ( $p < 0.01$ ).

**Key words:** Rock candy % Effluent % Supersaturation % Temperature % Invert sugar % Color % pH

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### INTRODUCTION

Arabic writers in the first half of the 9th century described the production of candy sugar [1]. the production of rock candy based on crystallization. The crystallization is separation phenomena that the crystals and mother liquor are separated from each other in the result of cooling or evaporating process [2]. The rock candy is usually produced in the manner of stagnant, whereas the industrial type is manufactured from either still or continuous ways. The letters yield is higher as compared to the former. Nowadays, the coloring and flavoring agents are used in production process and it led to more variety and greatly in demand. The rock candy traditional production is done in many countries; however it is no longer used in developed countries. Due to customer's demand, suitable actions have been performed in different fields such as the more production, variety in products and economical justifications [3].

### MATERIALS AND METHODS

In the second phase the rock candy was manufactured on experimental-scale in the basis of

existing defects in traditional way as well as effective parameters on the production process.

**Experimental Method:** The parameters such as pH, invert sugar percentage, color and sucrose content are very important in first syrup because the quality of final products base on it. This subject has most importance for effluent because it should be used in production process again and again. The water to sugar ratio was determined on the basis of supersaturation. All the physicochemical properties were assigned under twelve different combinations of supersaturation (1.3, 1.4, 1.5 and 1.6) and of temperature (70, 80 and 90°C). The brix of syrup was calculated so that after through dissolving sugar in water and setting its temperature at 70, 80 and 90°C. The mentioned-above supersaturation obtained for entrance syrup into the incubator. In order to incubating and crystallizing was also defined a time period of 24h. If brix correspond with supersaturation next to the achievement of favorite brix, the syrup was poured into tray and thereafter was transferred into the incubator. In this way the experimental samples consist of the rock candy and effluent as final products. The carried out tests on samples consisted of color, pH and invert sugar percentage which performed according to standard's instructions [4,5].

## RESULTS AND DISCUSSION

The effect of super saturation and temperature on various parameters of effluent and rock candy are as follow:

**pH:** One of the products of sucrose hydrolysis is lactic acid which leads to decrease of pH in the produced syrup and effluent and its quantity dependent on preservation temperature besides primary invert sugar percentage. Our results showed that the increase in temperature and supersaturation led to the significant decrease in pH (Fig. 1). The final effluent pH in traditional and experimental ways is 5.21 and 6.67, respectively. Its reason is difference in production method and conditions.

In high supersaturations, sucrose is rapidly hydrolyzed because the sugar complete dissolving needs high temperature and long time. This subject means the loss of pH and invert sugar increase. If such syrup is preserved in high temperature for crystallization, sucrose is faster hydrolyzed and hence, the pH is successively decreased. The produced effluent of this syrup will have the low pH and high invert sugar percentage. High viscosity of effluent and its direct contact with produced rock candy will cause to remain some effluent with rock candy which leads to decrease of rock candy pH. As shown in Fig. 2, at low temperatures and supersaturations

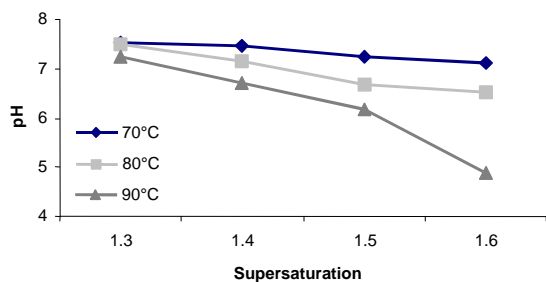


Fig. 1: Variation of the effluent pH with supersaturation at different temperatures

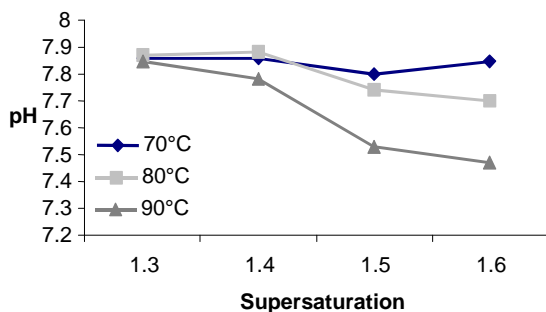


Fig. 2: Variation of the pH of rock candy with supersaturation at different temperatures.

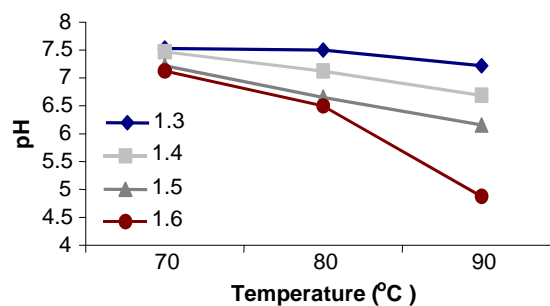


Fig. 3: Variation of the effluent pH with temperature at different supersaturations

the pH of sugar and rock candy is almost equal, but pH loss is observed at high temperatures and supersaturations. The pH of traditional rock candy is lower than that of experimental rock candy. The result showed that the increase in the temperature and supersaturation led to the significant decrease in the pH of effluent and rock candy.

As is stated before, at high supersaturations, heat is not easily transferred and sugar dissolves in water perfectly. Therefore, the through dissolving of sugar needs the higher temperatures. The use of high temperature is also resulted in more inversion of sucrose, so the first syrup will have the more amount of invert sugar and various acids obtained by sucrose hydrolysis. Since the existing of invert sugar in syrup leads to more hydrolysis of sucrose, thus the produced effluent of such syrup has lowest pH values (SS=1.6 and 90°C) (Fig. 3). In traditional and experimental ways, the pH of effluent is 5.21 and 6.67, respectively. At the same brix of first syrup, the pH of traditional and experimental rock candy is also 7.56 and 7.74, respectively. The effect of temperature on the pH of rock candy is significant at three different temperatures (70, 80 and 90°C).

**Invert Sugar Percentage:** In high temperature and supersaturations (90°C and ss=1.5, 1.6) sucrose hydrolysis is intensifying, hence the amount of invert sugar will be excessively increased. Furthermore, prolonged first syrup cooking plays an important role in inversion progress in its produced effluent. The effect of temperature and supersaturation on invert sugar percentage has been shown in Figs. 5 and 6. At the same brix of first syrup, the invert sugar percentage in traditional and experimental productions is 0.591 and 0.072, respectively.

The leaking of effluent in to the rock candy increases its invert sugar percentage. Characteristics of rock candy are influenced by syrup preparation conditions. Hence,

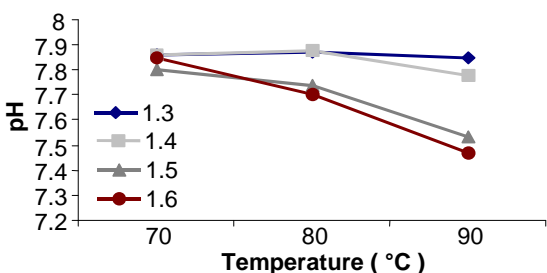


Fig. 4: Variation of the pH of rock candy with temperature at different supersaturations

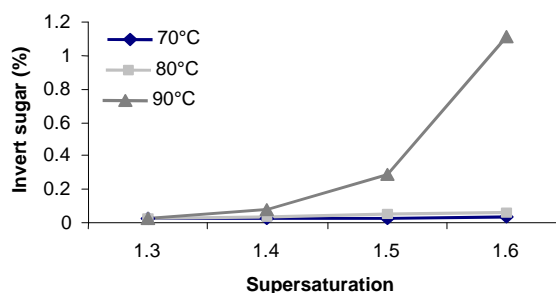


Fig. 7: Variation of the invert sugar percentage of rock candy with supersaturation at different temperatures

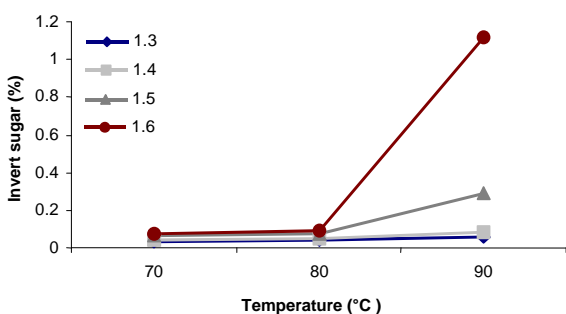


Fig. 5: Variation of the effluent invert sugar percentage with supersaturation at different temperatures

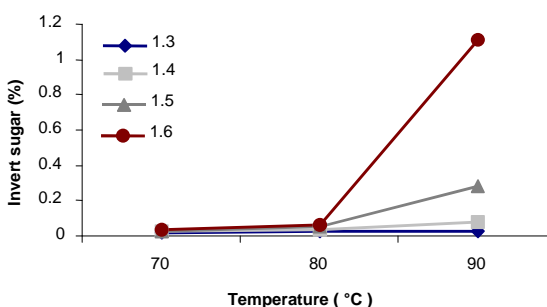


Fig. 8: Variation of the invert sugar percentage of rock candy with temperature at different supersaturations

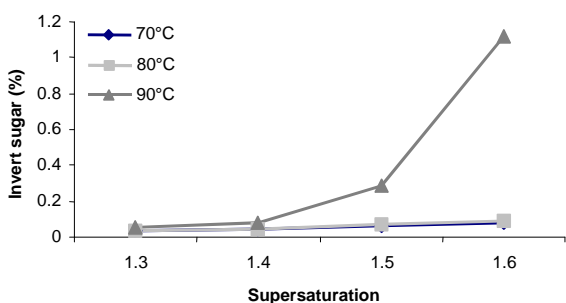


Fig. 6: Variation of the effluent invert sugar percentage with temperature at different supersaturations

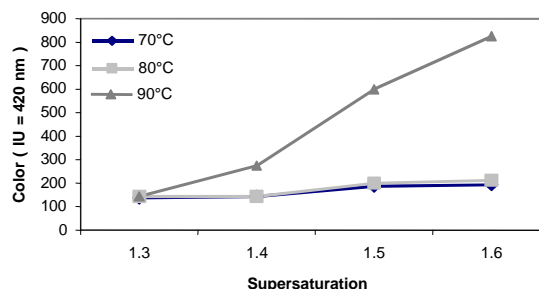


Fig. 9: Variation of the effluent color with supersaturation at different temperatures

the more hydrolysis of sucrose, the more inverts sugar in the effluent and rock candy. At the most supersaturation (1.6) the effluent viscosity is so much that the rock candy is not easily separated from it, because of high inversion. The amount of invert sugar of rock candy at low temperatures and supersaturations is rarely, its reason is small sucrose hydrolysis. The increase in the mentioned parameters is caused sucrose inversion (Figs. 7 and 8). The invert sugar percentage in traditional rock candy more than that in experimental rock candy because of critical temperatures, time and the use of wastage rock candy. At the same brix, these amounts are 0.551 and 0.055, respectively.

**Color:** Because the effluent stays at high temperature for a long time, therefore, its invert sugar and color are increased. Figs. 9 and 10 show the effect of temperature and supersaturation on effluent color. The color of effluent which stays at 90°C has significant difference when compared whit two other temperatures (70 and 80°C) especially at high supersaturations (1.5 and 1.6) because its first syrup has been prepared at higher temperature in comparison with the first syrup of the mentioned temperatures and thus has most amount of invert sugar and color. Of course, this amount of color is less than that in produced effluent of traditional way. In traditional way

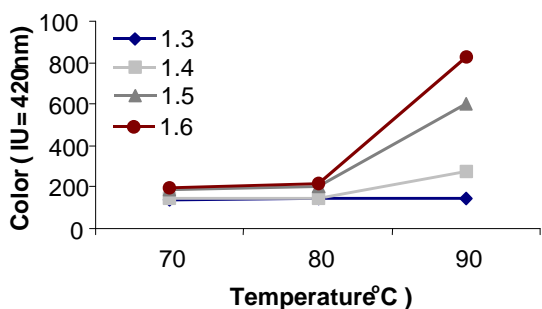


Fig. 10: Variation of the effluent color with temperature at different supersaturations

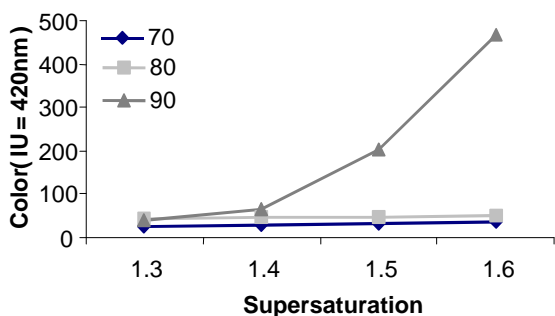


Fig. 11: Variation of the color of rock candy with supersaturation at different temperatures

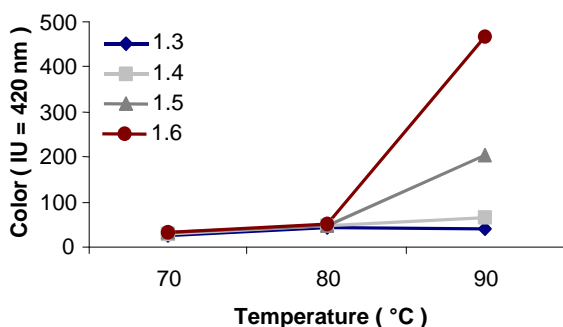


Fig. 12: Variation of the color of rock candy with temperature at different supersaturations

the color of the produced effluent of the first syrup (the brix of first syrup is 85.17) is 1200 ICUMSA units whereas at the same conditions in experimental way this amount is 203 ICUMSA units that its reason is high temperature and time, primary invert sugar and low quality of cosume first syrup. The achieved results showed that the increase in the temperature and supersaturation will led to the significant difference of means.

Base on the mentioned reasons, the more color of effluent causes the more color of rock candy. The color of sugar and rock candy at low temperatures and supersaturations are close to each other because of the small sucrose hydrolysis (Figs 11 and 12). The produced

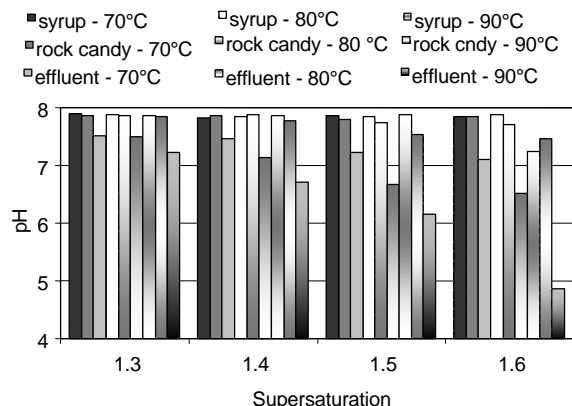


Fig. 13: Variation of pH in the first syrup and produced effluent and rock candy at different temperatures and supersaturations

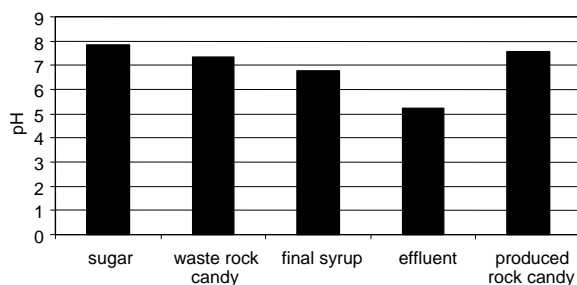


Fig. 14: Variation of pH in primary material, final syrup and the produced effluent and rock candy in traditional way

rock candy in such conditions is white perfectly without using blankit as a bleacher. The color of traditional rock candy more than that experimental rock candy because of mentioned reasons before. At the same brix of first syrup, the color of traditional and experimental rock candy is 253.18 and 48.35 ICUMSA unit, respectively.

The effect of final syrup quality on the various parameters of the effluent and rock candy.

Because the low efficiency of the rock candy production thus in order to the production process is justifiable economically, the effluent and wastage rock candy should be used on successive occasions. Since the quality of the produced effluent and rock candy is directly affected by the first syrup quality, hence its preparation conditions are very important. The time and temperature are vital factors in this area that the syrup will be produced in the best conditions by their exactly control.

**pH:** According to Fig. 13, the lower brix in syrup, the higher pH in its produced effluent and rock candy. The achieved effluent of critical conditions (SS=1.6 and 90°C)

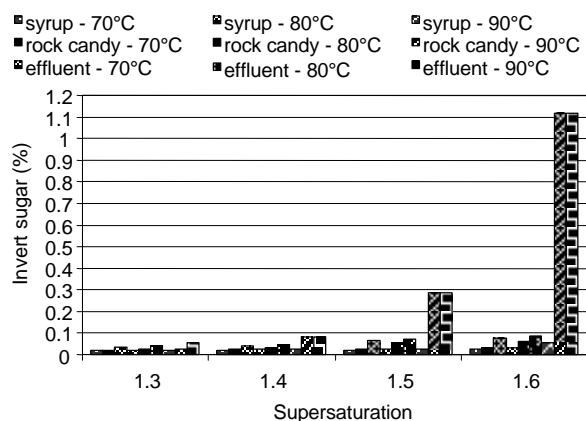


Fig. 15: Variation of invert sugar percentage in the first syrup and produced effluent and rock candy at different temperatures and Supersaturations.

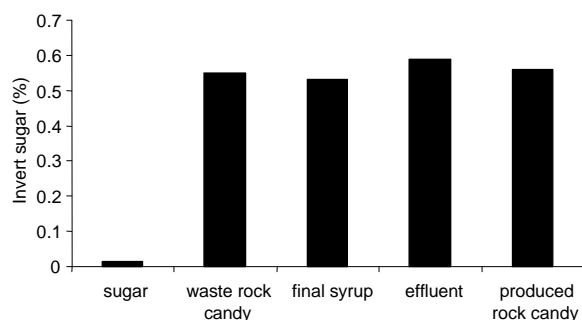


Fig. 16: Variation of invert sugar percentage in primary material, final syrup and the produced effluent and rock candy in traditional way

should not be reused because it decreases the pH of the final syrup intensely when it is used in subsequent productions.

The quality of produced effluent and rock candy in traditional way is less than that in experimental way. In the former, the pH of the effluent and rock candy is 5.21 and 7.56, respectively (Fig. 14). While in respect of the latter, the pH of the effluent and rock candy is higher because of decreasing in the temperature and time of cooking operation and control of sugar and water mixing ratio.

**Invert Sugar Percentage:** The increase in the sugar to water ratio of syrup is led to the increase of dissolving of time and temperature and this means the intensification of sucrose hydrolysis. The produced effluent and rock candy from critical syrup (SS=1.6 and 90°C) will have higher invert sugar amount (Fig. 15).

In the experimental production because the temperature and time factors usually is not critical hence, the invert sugar percentage is not so considerable. This

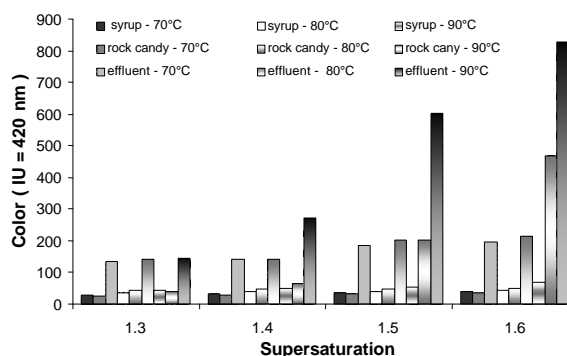


Fig. 17: Variation of color in the first syrup and produced effluent and rock candy at different temperatures and Supersaturations

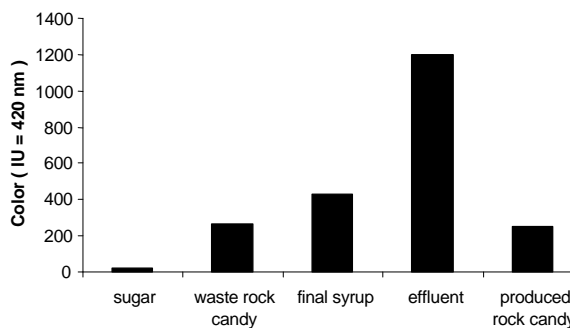


Fig. 18: Variation of color in primary material, final syrup and the produced effluent and rock candy in traditional way

state is observed at 70 and 80°C for all 4 supersaturation levels which in traditional way, the excessive water increases the required time of cooking. Thus, the syrup must stay at the temperature above 100°C for long time so that its surplus water is evaporated and this topic means the intensity of inversion in produced syrups in this way. Of course the great amount of invert sugar in the wastage rock candy which is used accompaniment by sugar in production process will lead to the more hydrolysis of sucrose. In the same conditions, the amount of invert sugar percentage in the first syrup and its produced effluent and rock candy in experimental way was 0.023, 0.055 and 0.072, respectively, while for traditional way the mentioned amounts above 0.5.

**Color:** The increase in the invert sugar percentage will lead to the increase in the color. The caramelization and hydrolysis of cyclic compounds as HMF which take place at high temperature, increases the amount of color. Caramelization reactions (which do not involve proteins)

are strongly dependent on temperature and have high activation energy values. The presence of coloring agents have various effects on sucrose solubility [6]. Because at high brix, the high temperature and time need to dissolve of sugar, thus color intensity will be more (Fig. 17).

The critical temperature and time and the amount of excessive water in traditional way justify the more color in the first syrup that it lead to the increase in the color of its produced effluent and rock candy. The amount of color in traditional way is more than that in experimental way (Fig. 18).

### CONCLUSIONS

According to achieved results, the range of supersaturations (1.3-1.6) at 90°C has undesirable effect on pH, color and invert sugar percentage parameters. Therefore, the use of the temperature at 90°C is not suitable any of supersaturations. Also, produced effluent at 1.6 is complete viscose. Since the amount of color and invert sugar percentage at 1.5 and 1.6 is more than that at 1.3 and 1.4 thus the production process should be concentrated on 1.3 and 1.4 at 70 and 80°C. Because the

amount of pH, color and invert sugar percentage at two aforesaid temperatures and supersaturations (1.3 and 1.4 at 70 and 80°C) almost is equal, in order to production of rock candy ss=1.4 and 80°C is suggested because of the supersaturation importance as a driving force and temperature as an effective factor in mass transfer.

### REFERENCES

1. <http://www.sugarindia.com/candy.htm>
2. Vauck/Müller Grundoperationen chemischer Verfahrenstechnik. 1994, 10. Auflage, Deutscher Verlag für Grundstoffindustrie, Leipzig. Stuttgart. pp: 625-646.
3. [http://www.googler.de/Candy + Sugar + patented](http://www.googler.de/Candy+Sugar+patented).
4. International commission for uniform methods of sugar analysis, Methods Book 1994.
5. Reinefeld D. and F. Schneider, 1978. Analytische Betriebskontrolle der Zuckerindustrie. Verlag Dr. Albert Bartens. Berlin.
6. Grimsey, I.M. and T.M. Herrington, 1996. The incorporation of colored compounds in sucrose crystals, Zuckerindustrie 121 Nr. 1, pp: 40-45.