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Monitoring Saccharification Process in Brewery Industry Using Quality Control Charts

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Abstract

The aim of this study was to establish a control system for saccharification process using quality control charts. To achieve this goal, temperature, pH and brix were measured at 12 minutes intervals for 15 consecutive batches which took 2 hours each. The time variations for three process parameters were assessed to establish a good understanding of the saccharification process. The temperature varied between 58˚C and 62˚C while the pH decreased slowly due to oxidation, values of which varied between 5.7 and 5.0. Brix values increased linearly with time. The initial and final values of the three parameters varied from one batch to another. Of the three parameters, brix was not well represented on the quality control charts due to wide difference between initial and final values during saccharification. The final brix values varied between batches, from 10.6% to 11.6%. The control charts used in this study were X-bar and Range charts. The rules for interpreting control charts were implemented for both X-bar and R charts, results of which showed that the process was out of control, although some rules were not violated due to little number of batches studied. The values of $\bar{R}$ for temperature and pH data (2.27˚C and 0.35, respectively) were lower compared to brix data (11.2%). The corresponding values of span between control limits, SP_x and SP_R for temperature and pH were also comparatively lower than those established from brix data. Due to larger values of $\bar{R}$ for brix measurements, the corresponding control charts for brix were insensitive in identifying out-of-control points during saccharification process.

Keywords

Saccharification, Batch Processing, Statistical Process Control, Control Charts, Control Limits, Number of Subgroups

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1. Introduction

Quality control is defined as a group of activities designed to assure standard of excellence in manufacturing, production, management and other engineering aspects. Controllable factors that either positively or negatively influence the finished product are called quality control. Dar Brew, located in Dar es Salaam, Ubungo area, is a company that produces beer through steeping a starch source (commonly cereal grains) in water and then fermenting with yeast Several factors affect the product from each batch, such as pH, temperature and brix level. This study was conducted to monitor the saccharification process while implementing quality control charts as the process control tools for simultaneously measured pH, temperature and brix data.

The hydrolysis of polysaccharides to soluble sugars is called saccharification. Malt made from barley is used as a source of β-amylase to break down starch into the disaccharide maltose, which can be used by yeast to produce beer. Prior to fermentation, the cereals used by Dar Brew require saccharification or hydrolysis of carbohydrates such as cellulose and starch into sugars. Saccharification is a process of breaking a complex carbohydrate (starch or cellulose) into its monosaccharide components, which converts glucose into simple soluble fermentable sugar by hydrolyzing a sugar derivative or complex carbohydrate. Hydrolysis is a chemical reaction during which one or more water molecules are split into hydrogen ion (a cause of acidity) and hydroxide ions in the process of chemical mechanism, and the OH group is then used to break the complex polymers. Enzymes operating at suitable conditions are required to achieve this process [1] [2]. Carbohydrates are starch fragments left behind from incomplete enzyme degradation, and must be broken down by the commercial enzymes during fermentation [3].

A control chart is a graphical statistical tool used to distinguish between variation in a process resulting from common causes and variation resulting from special causes for the purpose of quality control. It presents a graphical display of process stability or instability over time. The first control chart was developed by Walter A. Shewart [4]. Since then, these charts have been widely applied in industry and also received intensive attention from researchers [5]-[7], forming the so called Statistical Process Control (SPC).

Thus, control charts are running records of the performance of the process and, as such, they contain a vast store of information on potential improvements being the main reason SPC is used to accelerate the learning process and to eventually produce an improvement in the processes. These charts are also useful in communicating the results to leaders, suppliers, customers, and others interested in quality improvement.

2. Literature Review

More sophisticated sensors to measure product attributes such as gas chromatographs, mass spectrometers, and spectrophotometers are available for on-line use in food industries. However, these devices require an increase in sophistication of users and increased maintenance. SPC is the application of statistical methods to the monitoring and control of a process to ensure that it operates at its full potential to produce conforming product with the least possible waste. While SPC has been applied most frequently to controlling manufacturing lines, it applies equally well to any process with a measurable output. The key tools in SPC are control charts, a focus on continuous assessment and improvements, which makes SPC more effective in process monitoring

Much of the power of SPC lies in the ability to examine a process and the sources of variation in that process using tools that give weight to objective analysis over subjective opinions and that allow the strength of each source to be determined numerically. Variations in the process that may affect the quality of the end product or service can be detected and corrected. With its emphasis on early detection and prevention of problems, SPC has a distinct advantage over other quality control methods, such as inspection, that apply resources to detecting and correcting problems after they have occurred. Good performance may also result from using SPC data to identify bottlenecks, waiting times, turnaround time and other sources of delays within the process. Process cycle time reductions coupled with improvements in yield have made SPC a valuable tool from both a cost reduction and a customer satisfaction standpoint. Much of the power of the control charts and SPC in general, lies in the ability to monitor both process center and its variation of the mean about that center. By collecting data from samples at various times within the process, variations in the process that may affect the quality of the end product or service can be detected and corrected, thus reducing waste as well as the likelihood that problems will be passed on to the customer. Control charts have an emphasis on early detection and prevention of problems.

The x-control charts are the most used statistical control charts when continuous variables are used to measure quality characteristics. With the widespread usage of automatic data acquisition system for computer charting and analysis of manufacturing process data, there is a need to automate the analysis of process data with little or no human intervention [4]. Many researchers tried to automate the analysis of control chart patterns by developing Expert Systems to limit the human intervention in the analysis process of the control chart [4] [5].

Control charts patterns are categorized as natural and unnatural patterns. The presence of an unnatural pattern such as runs, shifts in process mean, or trends means that a process is out of control [7]. The accurate identification of these unnatural patterns will help the quality practitioners to determine the assignable causes for process variation; because each unnatural pattern has its related assignable causes [6].

Traditional control charts use only recent sample data point to determine the status of the process based on the control limits only. They do not provide any pattern related information. To increase a control chart sensitivity many supplementary rules like zone tests or run rules have been suggested by different researchers [8]-[10] to assist quality practitioners in detecting unnatural patterns. Other researchers are using control charts in software process management [11]. The primary problems with applying run rules are that the application of all the available rules simultaneously can yield an excess of false alarms due to the natural variability in the process.

A run is a sequence of observations of increasing (or decreasing) points or a sequence of observations above or below the process mean [6] [12]. It is assumed that a process starts in control (has natural pattern) and then may undergo only one out of control pattern at a time. For simplicity, only cases of upward and downward shift and trend patterns have been investigated in this study and also because of the short time series (15 batches only). For complex systems with automated data collection, algorithm exist aimed at providing researchers with reliable and automated identification tool for (the so called artificial networks) which are designed to maximize the probability of success in identifying basic patterns accurately [6] [13]-[17].

A control chart comprises of a centerline, an upper control limit (UCL), and a lower control limit (LCL) on which fraction defective (attributes) statistics or sample average and variation (variables) statistics are plotted over time. The statistics are usually determined for selected variables which represent the key performance indicators of the process. The time scale is represented by number of batch processed and assessed, if one batch is processed per day. Batch number indicates number of days elapsed or a measure of time. Control limits represent the limits of variation that should be expected from a process in a state of statistical control. Control limits should not be confused with specification limits, which represent the desired process performance. Because the control chart is based on actual process statistics, most points (99.73 out of 100) plotted on the control chart are expected to fall somewhere between the upper and lower control limits. In fact, most points will lie on or around the centerline with few points much larger or smaller than the average. Points falling outside the control limits indicate lack of statistical control.

If the process is stable, then the distribution of subgroup averages will be approximately normal. The limits are determined by estimating the "short-term" variation in the process, and defining process stability (or process control) as when the short-term variation provides a good model (or estimate, or prediction) of the longer-term variation. This is perhaps the most critical component towards the effective use of these control charts, which requires attention. One goal of using control charts is to achieve and maintain process stability, defined as a state in which a process has displayed a certain degree of consistency in the past and is expected to continue to do so in the future. This consistency is characterized by a stream of data falling within control limits based on $\pm 3\sigma$ of the centerline. The process can only be improved by removing common causes of variation [18]. Such efforts include rebuilding or replacing worn out equipment (like pumps, heat exchangers and steam generators), training employees, and employing preventive maintenance scheme. Improving the process can be expensive but worth the benefits received. However, some variation may be the result of causes which are not normally present in the process (special cause variation). Experience has shown that limits based on less than $\pm 3\sigma$ may lead to false assumptions about special causes operating in a process. In other words, limits which are less than 3σ from the centerline may trigger a hunt for special causes when the process is already stable. The three standard deviations are sometimes identified by zones. Each zone's dividing line is exactly one-third the distance from the centerline to either the upper control limit or the lower control limit. To create the zones, the span between the upper and lower control limits is divided into six equal parts. Calculation of the upper control limit (UCL) and lower control limit (LCL) for the averages of the subgroups give the uniqueness of the control chart to the problem at hand. Control limits define the parameters for determining whether a process is in statistical control or not.

The process is out of control if: a single point falls outside the 3σ limit, *i.e.*, beyond Zone A; two out of three successive points fall in Zone A or beyond; four out of five successive points fall in Zone B or beyond; and if eight successive points fall in Zone C or beyond[1]. These four criteria are also called unnatural patterns in the data. **Figure 1** shows the probability density function for the temperature data during saccharification process presented to show the control limits and zones of the control chart. The plot indicates zones for temperature control chart during saccharification process (data collected for 15 batches, leading to N = 165 data points) also the control limits at 1σ, 2σ and 3σ (upper control limits only).

Process performance is monitored with control charts by taking periodic subgroup samples from a process and plotting the sample points on a control chart. Sample points are compared against the control limits and evaluated for trends. The process is deemed to be in statistical control when all points fall within the control limits and there are no unnatural patterns (*i.e.*, trends) in the plotted data.

3. Methodology

3.1. Data Collection Methods

The study was conducted at Dar Brew in the quality assurance laboratory. This is one of the food industries in Tanzania in which sorghum beer is produced. In the quality assurance laboratory, the quality of raw materials, beer in process and final beer product is checked. Measurements of the parameters that have high significance to the quality of the beer produced were conducted (that is pH, Brix and temperature) in order to establish controllability of the process using control charts. Beer production at Dar Brew takes two main processes: saccharification (for about 2 hours) followed by fermentation (72 hours). This paper focuses on saccharification process.

The pH values were measured by using a pH meter, while temperatures were measured using a digital thermometer with a range of 0˚C - 100˚C. The sugar content of a fermentation broth (brix) was measured by using a refractometer. The amount of sugar in the broth will determine what the alcohol yield should be and when fermentation has reached completion. The refractometer was calibrated by using distilled water drops without sugar. The scale was adjusted by using a screw until the scale reads 0 when it is measuring the water sample. Then the sample was tested by the refractometer was checked for calibration before every sample was measured until when it was stable. Measurements separated by long lapse of time necessitated calibration. Readings were taken to the nearest 0.1 percent. The refractometer was dried with tissue paper and rinsed with water after each reading. Whenever several samples were collected simultaneously, drying of the refractometer was done after 10 to 15 measurements.

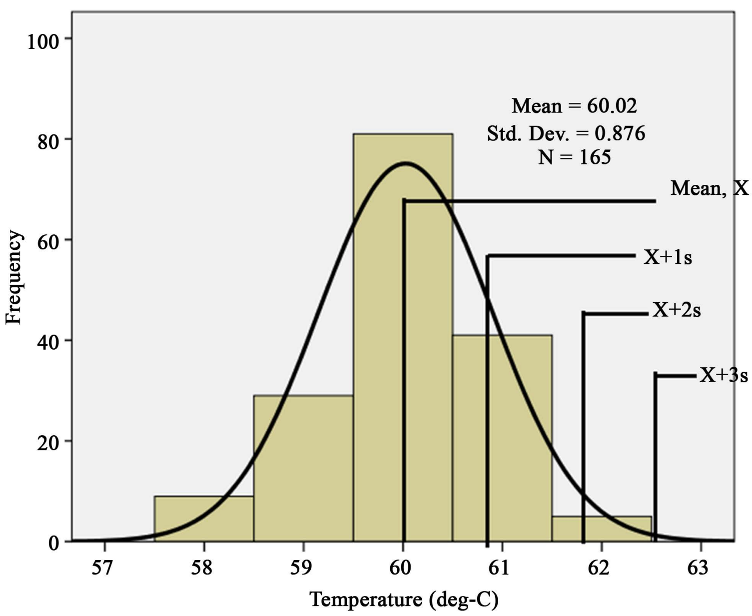


Figure 1. Control chart zones for temperature data during saccharification superimposed on a histogram (15 batches, N = 165).

[1]http://www.sixsigmaspc.com/spc/x-bar_and_range_charts.html.

3.2. Steps for Calculating and Plotting Control Charts

The steps for collecting, organizing, calculating and plotting data for variables X-Bar and R charts begins with determining the data to be collected (*i.e.*, temperature, pH and Brix), collecting and entering the data by subgroups. The individual subgroup measurements in time sequence, X_i, were entered in the portion of data collection section of the control [19], followed by calculating the averages and ranges of each subgroup. If there are k subgroups, then the average for each subgroup is given by Equation (1):

$$\bar{X}_j = \frac{\sum_{i=1}^{k} X_i}{k} \tag{1}$$

and the range for each subgroup is given by Equation (2):

$$R_i = X_{\max} - X_{\min} \tag{2}$$

If j is the number of subgroups, which is the same as number of batches studied, then the grand mean (or $\bar{\bar{X}}$) is calculated from Equation (3):

$$\bar{\bar{X}} = \frac{\sum_{i=1}^{j} \bar{X}}{j} \tag{3}$$

while the average of subgroup ranges is given by Equation (4):

$$\bar{R} = \frac{\sum_{i=1}^{j} R}{j} \tag{4}$$

The lower and upper control limits of subgroup averages are calculated from Equations (5) and (6):

$$LCL_{\bar{X}} = \bar{\bar{X}} - A_2\bar{R} \tag{5}$$

and

$$UCL_{\bar{X}} = \bar{\bar{X}} + A_2\bar{R} \tag{6}$$

The lower and upper control limits for ranges are calculated using Equations (7) and (8):

$$LCL_R = D_3\bar{R} \tag{7}$$

and

$$UCL_R = D_4\bar{R} \tag{8}$$

where the values A_2, D_3, D_4 depend on the subgroup size ($k = 11$), used in determining control limits for variables charts, were 0.373, 0.136 and 1.864, respectively[2].

The span between control limits for X-bar chart, SP_x is defined as per Equation (9):

$$SP_x = UCL_{\bar{X}} - LCL_{\bar{X}} \tag{9}$$

While the span between the R-chart control limits is given by Equation (10):

$$SP_R = UCL_R - LCL_R \tag{10}$$

3.3. Rules for Interpreting X-Bar and Range Control Charts

Rules for interpreting an X-Bar and R control charts exist as basic set of criteria for interpretation. The focus in this study is X-bar and R control charts, as summarized in **Table 4**. Whenever a single point falls outside the 3σ control limits, in a row in Zone A or beyond, a lack of control is indicated. Also, whenever at least 2 out of 3 successive values fall on the same side of the centerline and more than 2σ away from the centerline (in zone A or beyond), a lack of control is indicated. Note that the third point can be on either both sides. Whenever at least 4 out of 5 successive values fall on the same side of the centerline and more than one sigma unit away from the centerline (in Zones A or B or beyond), a lack of control is indicated (the fifth point can be on either side of the

[2]http://www.scribd.com/doc/686669/Spc-1.

centerline). Observations which suggest out of control situations can be listed as: 9 points in Zone C or beyond (on one side of central line), 6 points in a row steadily increasing or decreasing [9], and 14 points in a row alternating up and down, which can be used depending on the available number of subgroups of data. The test which provides an "early warning" of a process shift is a "2 out of 3 points in a row in Zone A or beyond", which can be easily tested and implemented with fewer number of subgroups available. The last rule is based on 15 points in a row lying in Zone C (above and below the center line).

3.4. Description of the Measurements during Saccharification

The three important parameters (brix, pH and temperature) were measured at the same time, during saccharification process. Saccharification takes two hours before fermentation starts, during which measurements were taken after every 12 minutes giving 11 data points during the process (defined as subgroups). The study was conducted for fifteen (15) batches, as summarized in **Table 1**.

3.5. Control Chart Data Collection Plan

The assumptions made on choice of subgroups include: the observations within a subgroup are from a single, stable process; the subgroups are formed from observations taken in a time-ordered sequence; the observations within the subgroups are independent, implying that no observation influences, or results from, another. It is also assumed that the numbers exist on a continuum, *i.e.*, there will be many different values in the data set. In the real world, the data are never completely continuous. It is thus assumed that the fluctuation of the points between the control limits is due to the variation that is intrinsic (built in) to the process. The last assumption is that a process starts in control (has natural pattern) and then may undergo only one out of control pattern at a time [6]. However, when there are only a few numbers that appear over-and-over it can cause problems with the analysis. A statistical analysis of the data (mode, standard deviation, skewness, kurtosis, and probability functions) will be conducted to ascertain the data spread. A common problem is that when few numbers repeat more often in the data set, the R chart will underestimate the average range, causing the control limits on both the average and range charts to be too close together.

In this study, subgroups were made by following the time period to process a batch, that is, within 120 minutes. With samples taken at intervals of 12 minutes, the number of subgroups was, $k = 11$, which determines the values of UCL and LCL for both X-bar and Range charts, as shown in **Table 2**. The larger the subgroup, the more sensitive the chart will be to shifts, provided a rational subgroup can be formed. A rational subgroup is simply a sample in which all of the items are produced under conditions in which only random effects are responsible for the observed variation.

4. Results and Discussion

4.1. Variation of Brix, Temperature and pH during Saccharification

Sugar content is a parameter which determines the amount of alcohol that can be made during fermentation process, while saccharification is the sugar making process. **Figure 2** shows the variation of brix (%) with time measured during saccharification process for 15 batches.

Table 1. Summary of data from saccharification process.

	Parameters	Values
1)	Duration for each batch (minutes)	120
2)	Time interval between measurements	12 minutes
3)	Number of subgroups, k	11
4)	pH	Decreases from 5.5 to 5.1
5)	Temperature	60˚C
6)	Brix	Increase from 0% to 12%
7)	Number of batches	15

Table 2. Sample data collection and calculations template for temperature during saccharification.

Time (min)	K	BATCH 1	BATCH 2	BATCH 3	…	BATCH 14	BATCH 15
0	1	59	59	61	…	60	59
12	2	60	60	62	…	60	59
24	3	60	59	60	…	60	60
36	4	61	59	59	…	60	60
48	5	60	59	59	…	61	59
60	6	59	60	59	…	61	60
72	7	60	60	58	…	62	59
84	8	60	60	58	…	61	58
96	9	60	60	58	…	61	58
108	10	60	60	58	…	60	59
120	11	60	59	59	…	60	60
MAX		61	60	62	…	62	60
MIN		59	59	58	…	60	58
$\bar{X}$		59.91	59.55	59.18	…	60.55	59.18
R		2	1	4	…	2	2
$\bar{\bar{X}}$		59.67	59.61	59.64	…	59.86	59.18
$\bar{R}$		2.20	2.27	2.27	…	2.27	2.27
UCL_{-x}		60.67	60.67	60.67	…	60.67	60.67
LCL_{-x}		59.38	59.38	59.38	…	59.38	59.38
UCL_{-R}		3.84	3.95	3.95	…	3.95	3.95
LCL_{-R}		0.56	0.58	0.58	…	0.58	0.58
$\bar{\bar{X}}+3\sigma$		60.67	60.67	60.67	…	60.67	60.67
$\bar{\bar{X}}+2\sigma$		60.45	60.45	60.45	…	60.45	60.45
$\bar{\bar{X}}+1\sigma$		60.24	60.24	60.24	…	60.24	60.24
$\bar{\bar{X}}$		59.67	60.02	60.02	…	60.02	60.02
$\bar{\bar{X}}-1\sigma$		59.81	59.81	59.81	…	59.81	59.81
$\bar{\bar{X}}-2\sigma$		59.59	59.59	59.59	…	59.59	59.59
$\bar{\bar{X}}-3\sigma$		59.38	59.38	59.38	…	59.38	59.38

Result show that brix increases linearly with time during saccharification, a relationship represented by a linear Equation (11):

$$B_s(t) = 0.0938t + 0.0807 \tag{11}$$

(with $R^2 = 0.9917$). On average, the final brix for all batches was 11%, although final values ranged from 10% to 12%. **Figure 2** shows that brix increases with time during saccharification attributable to the formation of sugars, as starch (the main storing form of sugar and energy in seeds), is converted into fermentable sugar. Although all batches start with the same Brix level (of about 0%), they end up with slightly different Brix values after 2 hours depending on process conditions (temperature and pH). It can also be stated that, at a given saccharification

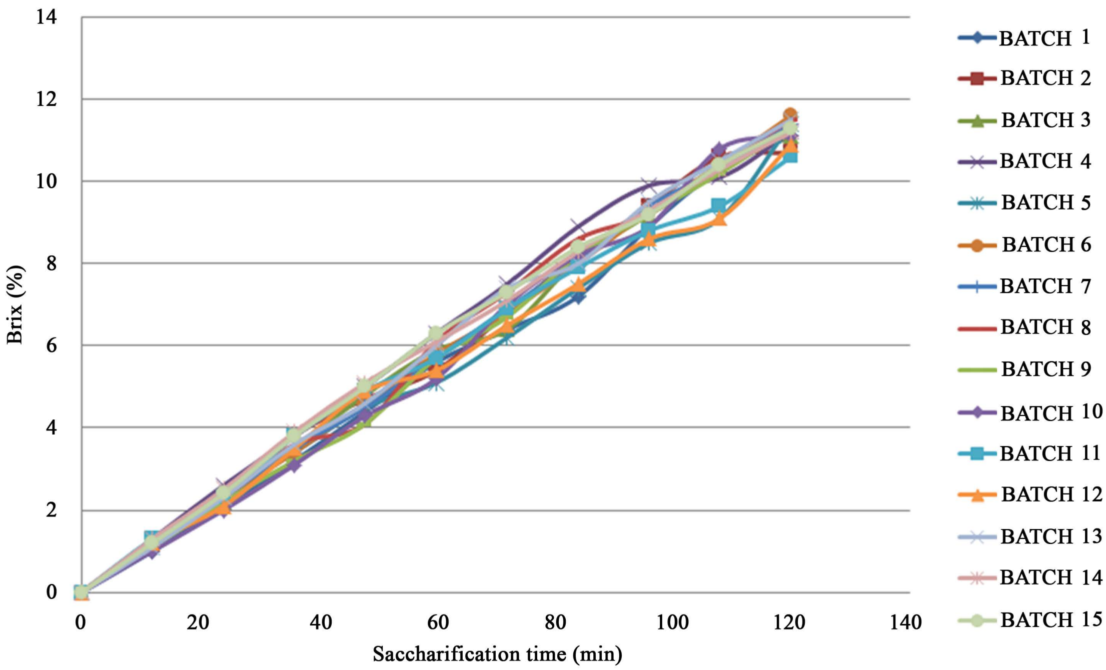


Figure 2. Variation of brix (%) with time during saccharification.

time, the brix values spread from the mean values between different batches towards the end of the process, results of which require assessment.

Figure 3 shows the final values of brix levels reached during saccharification for different batches. The final values vary between 10.6% and 11.6%, with standard deviation of 0.289%. Such a variation show that the amounts of sugars in the fermentation broths were different, which leads to different product quality between batches.

The pH is an important parameter in the saccharification process control since enzymes work best under a certain acidity or basic condition, unless otherwise, the desired product will not be produced. **Figure 4** shows the values of pH measured during saccharification process for 15 batches. The pH data signifies a measure of acidity of the syrup produced. Results show a slow decrease in pH during the 2 hours of saccharification because some oxidation occurs as aldehyde and ketones are formed and changed into weak acids. Thus acidity increases slowly with time during saccharification since the reaction is enzyme catalyzed, as shown in **Figure 4**.

The conversion of ketones to weak acid during saccharification is very limited due to lack of the critical hydrogen for the elimination to occur (a major difference between aldehydes and ketones). Under acidic conditions, the aldehyde is oxidized to carboxylic acid. Such metabolic processes are typically highly susceptible to even slight changes in pH and therefore, proper control of this parameter is critical. Precise manipulation of pH can determine the relative yield of the desired species over competing by-products. Moreover, enzymes works best in a narrow range of pH and deviations of as little as 0.2 to 0.3 may adversely affect a batch quality in some cases.

Figure 5 shows the values of initial and final pH of the saccharification broth for different batches. The range of pH during saccharification process has to be from 5 to 5.5, as observed in this study. A slight difference was observed in the initial pH range for which most of batches started with pH higher than the limit of 5.5, although the final pH was within the range. Batches starting with higher pH (e.g., batches #8 to 11) also ended with higher final pH values. There is a clear variation in terms of controllability of the process, such that the initial pH values are not the same and hence the final pH values are also different. As a result, product quality variations are inevitable. The final pH during saccharification shown in **Figure 4** and **Figure 5** become the initial pH during fermentation, unless adjustment is performed. For all batches studied, the final saccharification pH values were below the maximum limit of 5.5.

Temperature is an important parameter in process monitoring and control during saccharification. For the enzyme to work best, they require a certain constant temperature about 60˚C. **Figure 6** shows the variation of

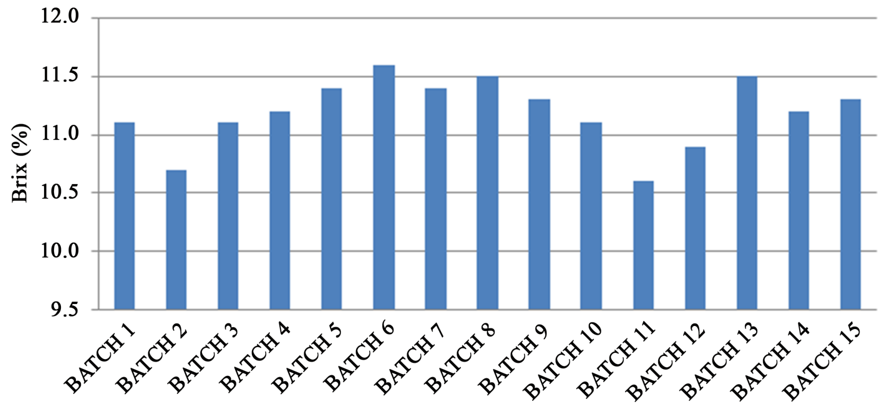


Figure 3. Final brix levels for different batches during saccharification.

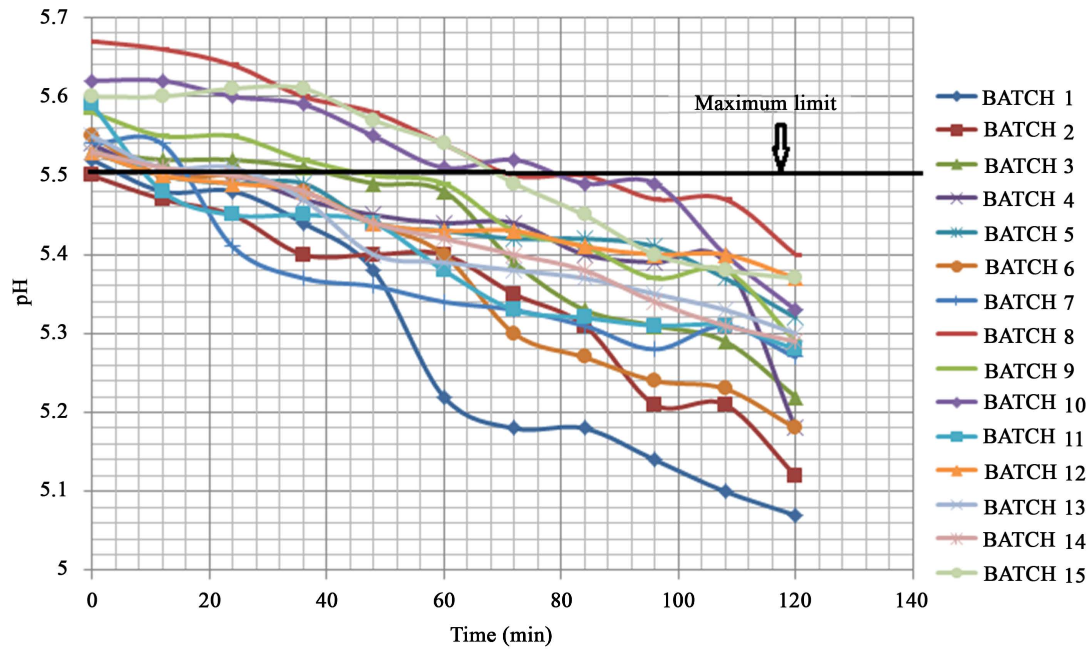


Figure 4. The variation of pH with time (minutes) during saccharification.

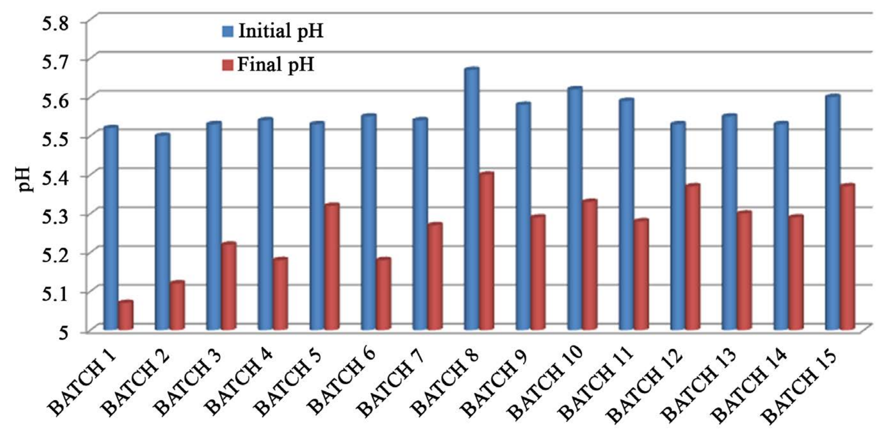


Figure 5. Values of initial and final pH of the saccharification broth for different batches.

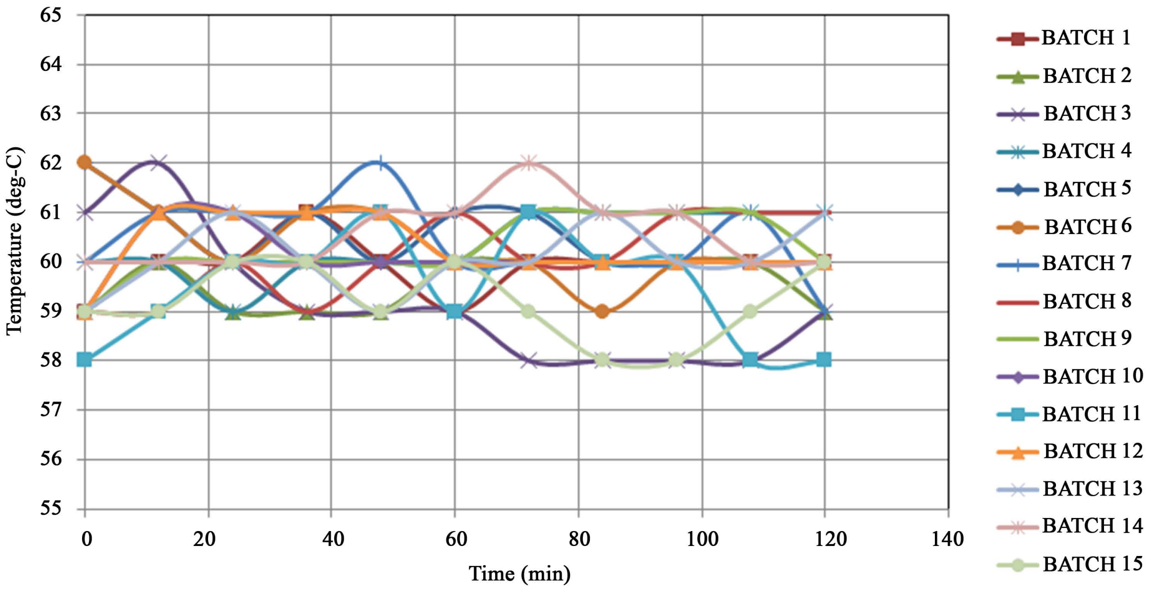


Figure 6. Variation of temperature with time during saccharification.

temperature with time during saccharification for different batches. During saccharification, the temperature has to remain constant because the enzymes act best at a temperature of 60˚C. However, due to process fluctuation, it ranged from 58˚C to 62˚C, which is a narrow range. Based on measured values (**Figure 6**), the temperature was varying from batch to batch, which affects the product quality. Temperature control is one of the most important processes in a brewery industry, for promoting growth of microorganisms and controlling the yield, in order to achieve the desired flavor profile in the finished product.

The statistical analysis of all temperature data collected during saccharification of 15 batches was presented in **Figure 1** (N = 165), which follows a normal distribution, with a mean of 60.02˚C. As discussed earlier, the initial and final temperatures during saccharification were different for different batches. **Figure 7** shows the values of minimum, average and maximum temperatures observed during saccharification for the batches studied, from which a variation is clearly depicted between different batches. One of the control measures can include having a constant initial temperature before the saccharification begins, while allowing the final temperature to vary within the acceptable range.

While most of the process stability studies utilizing control charts focused on the variability within subgroup averages, in this study the variations of the data across the batches at constant values of k or time was also assessed. The probability density functions of the data at different values of k are shown in **Figure 8** for pH and temperature data. While the temperature data shows a high uniformity across the batches (overlapping probability density functions at the same mean value and with similar bell shapes, low skewness), the pH data shows probability density functions with different mean values and shapes. The probability density functions for pH data have very long tails on either side (highly skewed), showing that the incoming batches are operated at different conditions. The results show that, if the product quality depends only on temperature, a uniform product quality will be expected, however, since the quality depends on several other factors (including pH and brix level), acting at the same time, the products will not attain the same quality.

There is a wide variation of the operating conditions between batches as depicted in **Figure 8**. It is expected for the pH, brix and temperature values to be closer at any selected time as the different batches are processed. But due to process variations, the values are different, leading differences in standard deviation as the saccharification time is considered. If the process is stable, then the distribution of subgroup averages will be approximately normal.

4.2. Application of Quality Control Charts to Saccharification Process

When points exceed the control limits, we assert that the process must have shifted, since the chance of this happening is so small. One advantage of the batch means X-bar chart is that it controls both the within-batch

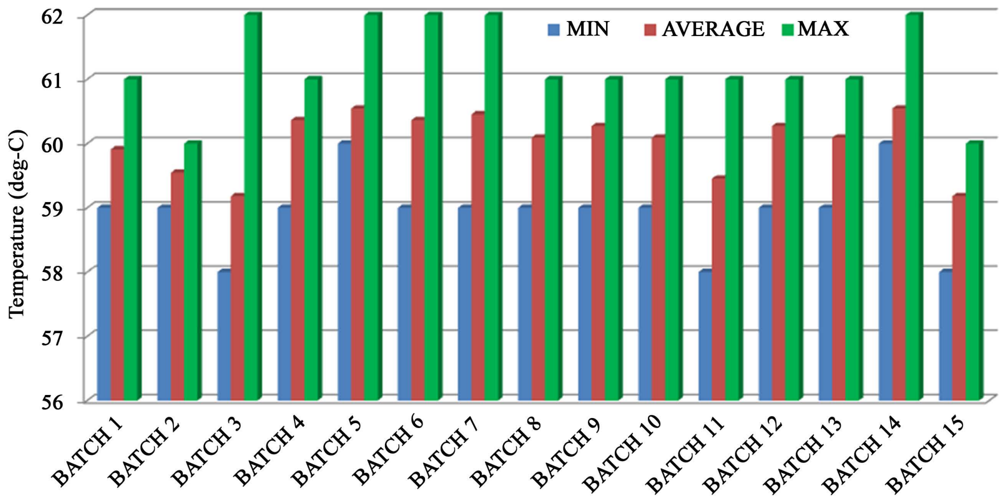


Figure 7. Variation of minimum, maximum and average temperatures among the bathes during saccharification.

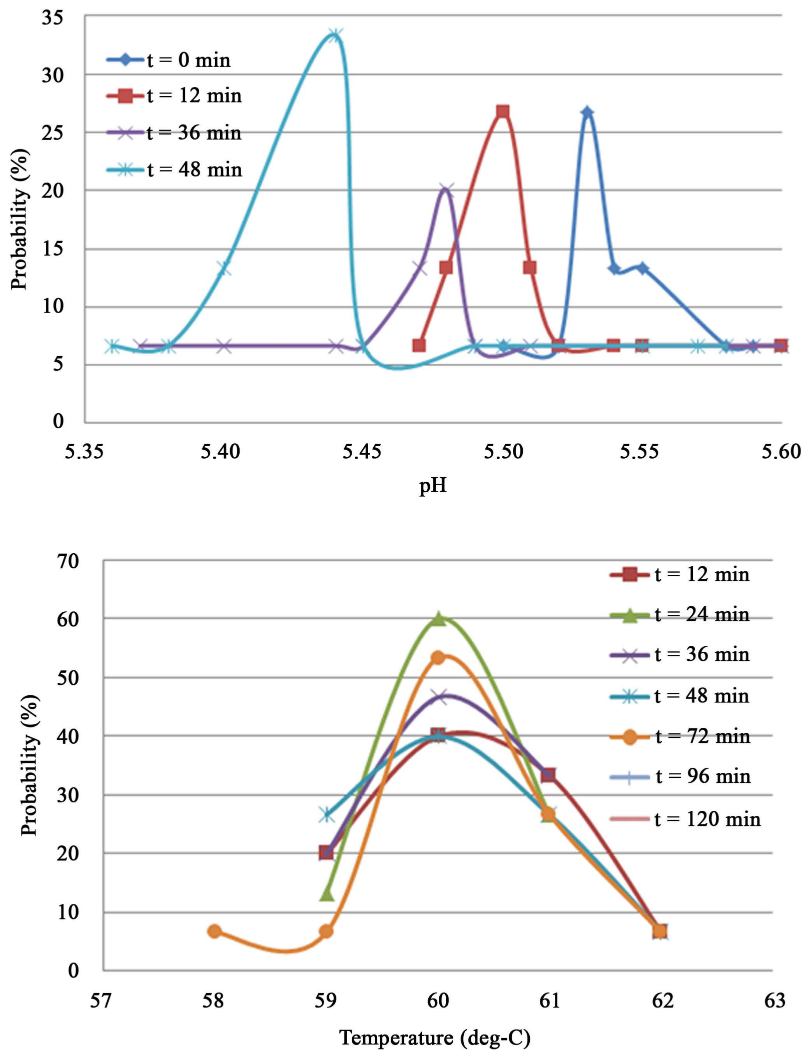


Figure 8. Comparison of variations between batches for pH and temperature data.

variation (on the range chart) and the between-batches variation (on the X-bar chart). **Figure 9** shows X-bar charts for brix, temperature and pH measurements. For the case of brix data, all values are within the control limit, and very close to the center line.

The X-bar chart for brix shows no signs of variation as all the points fall within region C (very close to the centerline). This shows that there is a limit on using quality control charts when it comes to wide range variation data. This is mainly due to wide variations of brix data, leading to higher range value and hence higher $\bar{R}$. Since the limits of the X-bar chart depends on $\bar{R}$, the span between the limits were very high (8.35 units for brix compared to 0.26 and 1.69 units for pH and temperature, respectively.

The temperature control chart, on the other hand, shows runs with several points above the centerline from batch #4 and #10 and also from batch #12 to #14. The first run comprise of seven (7) points in a row indicating

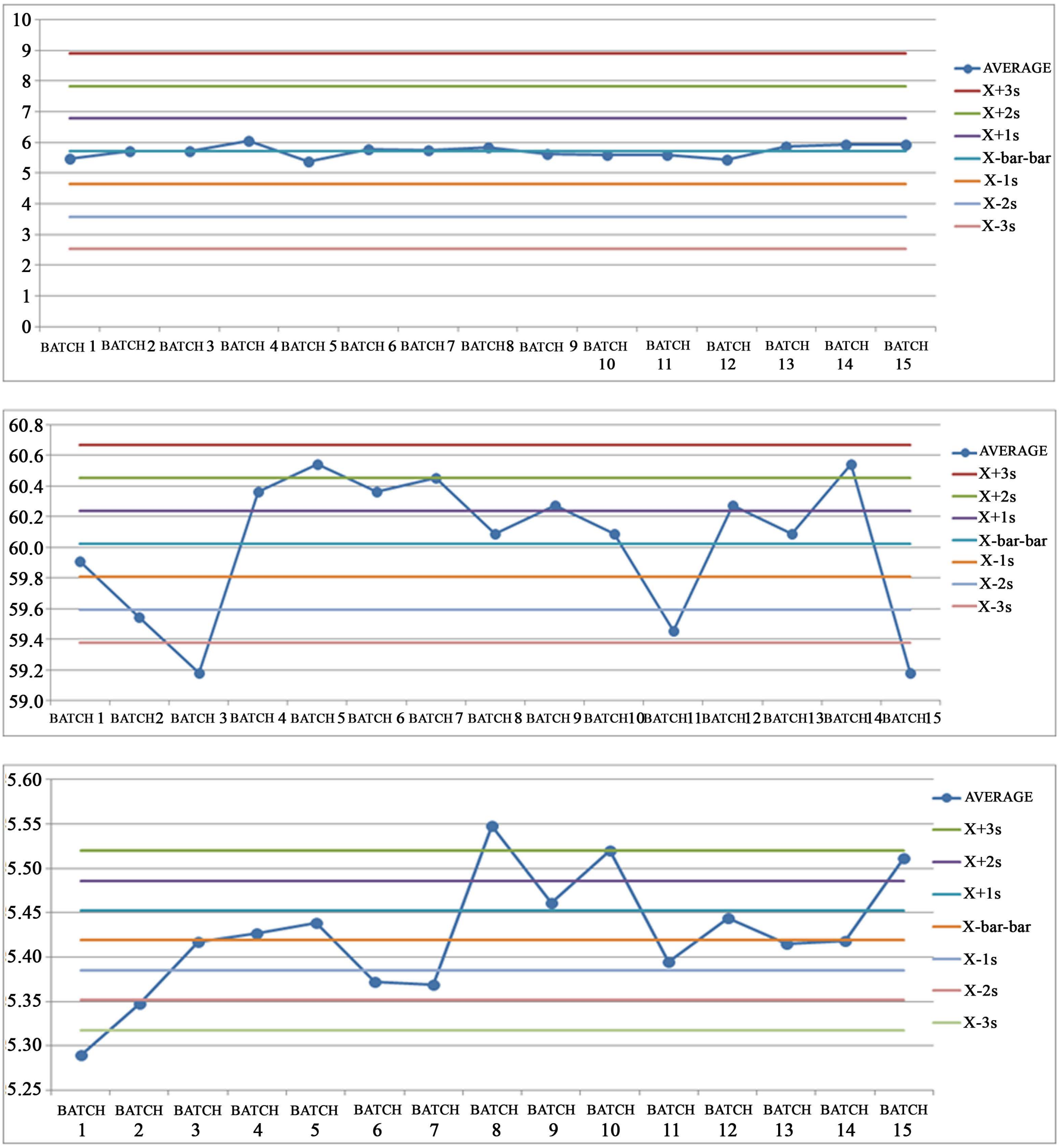


Figure 9. X-bar chart for brix, temperature and pH data during saccharification process.

that there is an abnormality in the process which requires an adjustment. Another run on temperature data was from batch #1 to #3, during which the average temperature was decreasing continuously. The pH data, on the other hand, shows runs of 3 points below the centerline (#1 to #3) and #8 to #10, which are not significant.

Trends were also observed in the pH and temperature data. In the temperature data, decreasing trends were observed from batch #1 to #3, while an increasing trend was observed from batch #9 to #11, both comprising of three points only. In the pH control chart, on the other hand, an increasing trend of batches #1 to #5 and a decreasing trend from batches #5 to #7 were also observed. Such trends indicate that machine adjustments are required.

The X-bar charts plotted in **Figure 9** allow process observation through quality characteristic measurements within each subgroup. The relationships between process observations are based on the assumption that the observations are independent variables. The size of shift to be detected was up to 3σ. Critics of this approach argue that control charts should not be used when their underlying assumptions are violated, such as when process data is neither normally distributed nor binomially (or Poisson) distributed. Such processes are not in control and should be improved before the application of control charts. Additionally, application of the charts in the presence of such deviations increases the type I and type II error rates of the control charts, and may make the chart of little practical use. According to the rules, the saccharification temperature has violated rule number 1(two values are outside the LCL_x, batch #3 and #15), from which, lack of control is indicated. The chart indicates that the process is in need of adjustments.

Figure 9 shows also X-bar control chart for pH measurements during saccharification process. Some values are within the control limit and some are out of the control limits, for instance Batches #1 and #8. This means that the Rule #1 has been violated. The pH as a process parameter shows that the process is not under control. Special cause variations result from either process stability or a shift in process average implying that the process performance is unpredictable and unmanageable until special causes of variations are removed from the process. While special causes occur relatively infrequently, they represent excessive process variation. Further analysis based on the 8 rules for interpreting control charts is presented in **Table 3**. While controlled variation is characterized by a stable and consistent pattern of variation over time (associated with common causes with an

Table 3. Detailed analysis of X-bar control charts by test type and indicators on the process.

Rule	Violation and indicators on the process
RULE #1: Whenever a single point falls outside the 3σ control limits, in a row in Zone A or beyond, a lack of control is indicated.	a) Temperature control chart Batch #3 and #15 are outside 3σ control limits. b) pH control chart Batch #1 and #8 are outside the 3σ control limits. This test provides an “early warning” of a process shift. Since the probability of this to happen is rather small, it is very likely not due to chance.
RULE #2: Conducting 2 out 3 beyond 2σ	a) Temperature control chart Batch #2 and #3 are 2σ away from centerline. b) pH control chart Batch #1 and #2 are 2σ away consecutively below the centerline. c) pH control chart Batch #6 and #7 are consecutively 2σ away below centerline.
RULE #3: 3 out of 5 connectively fall on same side of centerline and more than 1σ away from centerline	Temperature control chart Batches #4, #5, #6 and #7 are 1σaway from the centerline, consecutively. Like the previous test, this test may be considered to be an “early warning indicator” of a potential process shift. The false-positive error rate for this test is also about 2%.
RULE #4: Whenever at least 8 points in a row in Zone B, A, or beyond, on either side of the center line (without points in Zone C) a lack of control is indicated.	Not violated
RULE #5: 6 points in a row steadily increasing or decreasing	Not violated
RULE #6: 9 points on one side of centerline	Not violated
RULE #7: 14 points in a raw alternating up and down	Not violated
RULE #8: 15 points in **Zon**e C	Brix control chart, all points in Zone C. This test indicates a smaller variability than is expected (based on the current control limits).

outcome that is predictable within the bounds of the control limits), the process under review indicated uncontrolled variations characterized by variation that changes over time and is associated with special causes. The outcomes of uncontrolled process are unpredictable. A customer may be satisfied or unsatisfied with the product given this unpredictability.

4.3. Interpretation of X-Bar Control Charts

Control charts provide the operational definition of the term special cause. A special cause is simply anything which leads to an observation beyond a control limit. However, this simplistic use of control charts does not do justice to their power. Control charts are running records of the performance of the process and, as such, they contain a vast store of information on potential improvements. **Table 3** shows the detailed analysis of X-bar control charts by test type and indicators on the process. While some guidelines are presented here, control chart interpretation is an art that can only be developed by looking at many control charts and probing the patterns to identify the underlying system of causes at work.

4.4. Range Control Charts for Saccharification Process

The R chart shows changes in the dispersion of the process, being particularly useful as it shows changes in mean value and dispersion of the process at the same time. This makes it a very effective method for checking abnormalities within the process. Since the R chart is simple to calculate, it can also be plotted while the process is in progress so that it points out a problem in the production flow in real time mode [20]. The popularity of the R chart is only due to its ease of calculation, dating to its use before the advent of computers. The range statistic is, however, a poor estimator of process sigma for large subgroups, which necessitates use of X-bar chart, simultaneously.

In order to assess the indications on the process, tests were conducted on the range control chart using the rules, with reference to the data presented in **Figure 10**. Rule #1which states that whenever a single point falls outside the 3σ control limits, in a row in Zone A or beyond, a lack of control is indicated. This was violated by only Batch #3 on temperature range control chart. Rule #8, which states that 15 points in a row in Zone C (above and below the center line), was violated only by in brix range chart for which all range values for 15 batches fell in Zone C. All other rules were not violated, indicating that R charts were not efficient in detecting process variations.

4.5. Analysis of the Control Chart Parameters for Saccharification Process

The control chart for an increasing function like the brix data during saccharification leads to high range and hence too far apart control limits (Equations (5) to (8)), making the chart unable to capture variations outside the limits. This was observed for the case of brix data, which was increasing from 0 to around 11%. With $\bar{R}$ = 11.193%, the corresponding values of UCL-x and LCL-x were far apart (8.900 and 2.520), respectively, compared to pH and temperature for which the control limits were closer.

Comparing the control charts for brix (with $\bar{R}$ = 11.193%) with those of temperature and pH, it can be seen that all the points are in region C for brix, while the data points are spread in all regions for temperature and pH (**Figure 9**). This shows that the brix control chart was not capable of identifying out-of-control points during saccharification process, different from temperature and pH control charts in which out-of-control points were detected. **Table 4** compares the control chart parameters for the three parameters: brix, temperature and pH.

The value of $\bar{\bar{X}}$ presented in **Table 4** resembles the mean value reported in **Figure 1**, when all temperature data were analyzed, because the former is the mean of all averages from the subgroups. The control chart parameters for the three variables studied (pH, brix and temperature) were compared as shown in **Figure 11**. The variables were measured simultaneously during the saccharification process, which, however, gives different signals regarding the controllability of the process.

Figure 11 shows that the UCL_X for brix was very high due to higher values of $\bar{R}$ compared to pH and temperature data. It should be noted that, the temperature data is only presented in the Table but not on the plot due to higher values of $\bar{\bar{X}}$, UCL_X and LCL_X. Based on the equations used for determination of upper and lower control limits for both X-bar and R-chart, Equations (5) to (8), it is evident that the effect of $\bar{R}$ is critical in the

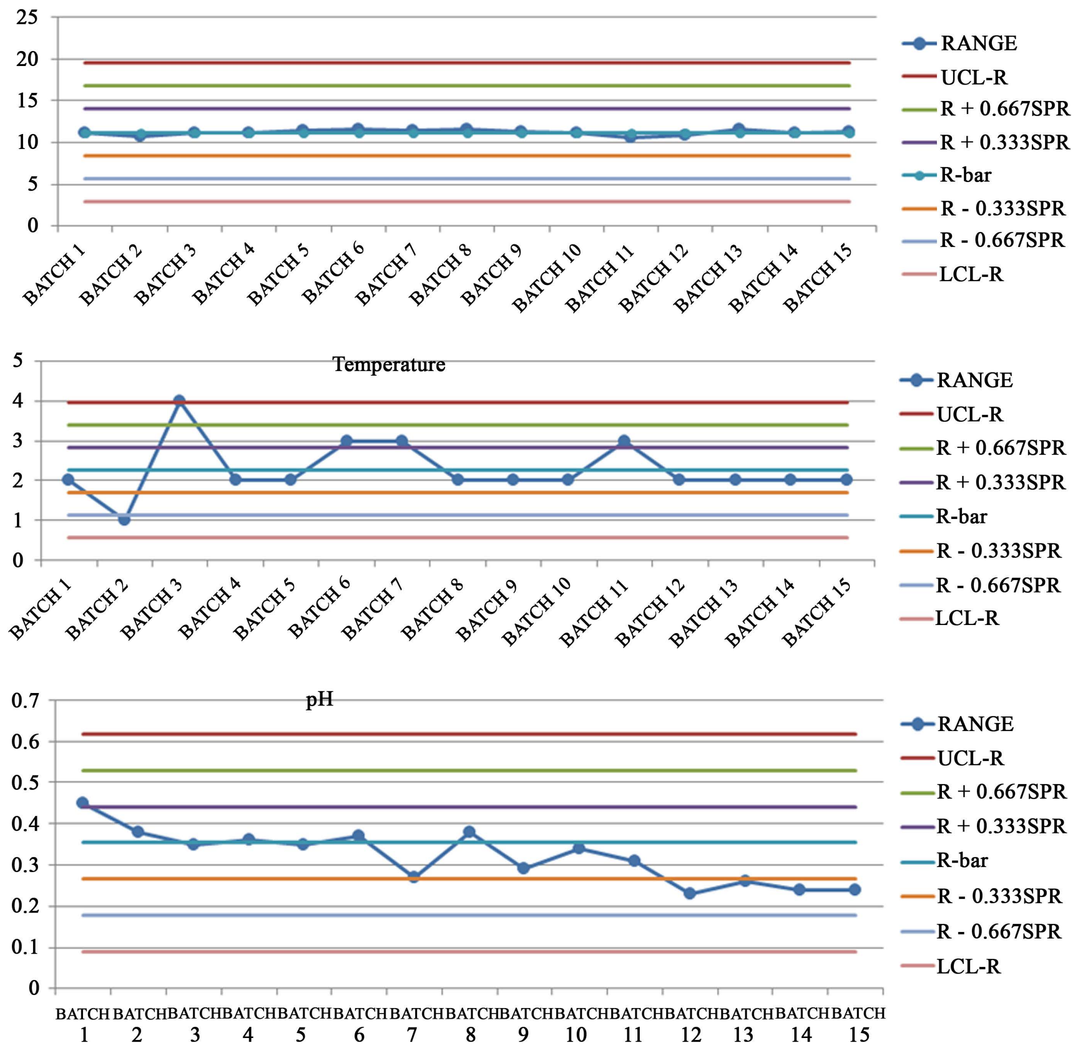
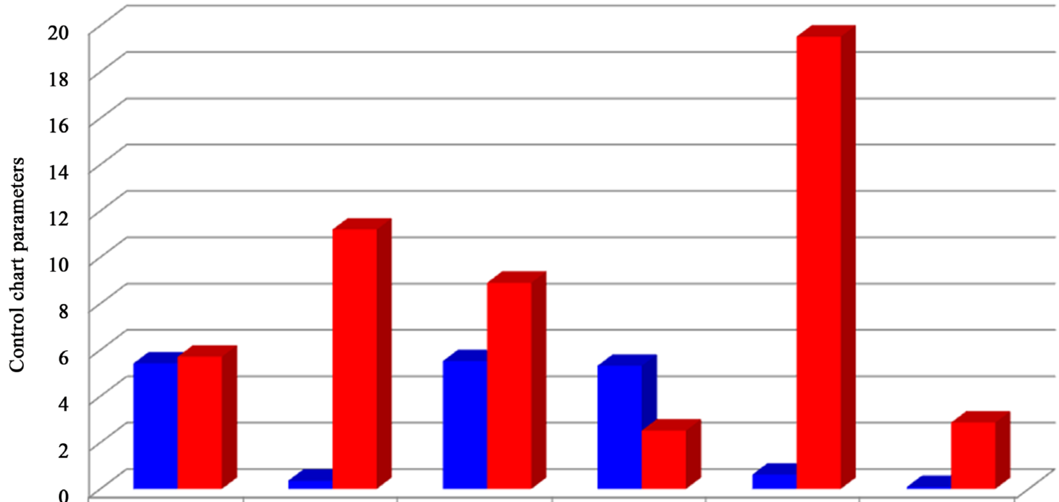


Figure 10. Range control charts for brix, temperature and pH values during saccharification.

Table 4. Control chart parameters for process data during saccharification.

Control chart parameter	Brix (%)	pH	Temperature (˚C)
$\bar{\bar{X}}$	5.71	5.42	60.03
$\bar{R}$	11.19	0.35	2.27
$\bar{\bar{X}}+1\sigma$	7.10	5.46	60.31
$\bar{\bar{X}}-1\sigma$	4.32	5.37	59.74
$\bar{\bar{X}}+2\sigma$	8.49	5.51	60.59
$\bar{\bar{X}}-2\sigma$	2.93	5.33	59.46
$UCL\text{-}x$	9.89	5.55	60.87
$LCL\text{-}x$	1.54	5.29	59.18
SP_x	8.35	0.26	1.69
$UCL\text{-}R$	20.86	0.66	4.23
$LCL\text{-}R$	1.52	0.05	0.31
SP_R	19.34	0.61	3.92



	X-bar-bar	R-bar	UCL-x	LCL-x	UCL-R	LCL-R
pH	5.419	0.354	5.52	5.318	0.617	0.091
Brix	5.7102	11.193	8.9	2.52	19.521	2.865
Temperature	60.032	2.2667	60.67	59.378	3.953	0.58

Figure 11. Control chart parameters for pH and brix during saccharification and fermentation processes (temperature values not plotted).

effectiveness of control charts in identifying out-of-control cases. The effectiveness of the control charts depends on the span between upper and lower control limits, as per Equations (9) and (10), values of which are presented in **Figure 12**. The fact that $\bar{R}$ was very high for brix data (approximately, 11.2%), the control limits, SP_X and SP_R, are excessively far apart, as shown in **Figure 12**, charts of which could not identify out-of-control points.

Thus, based on the data collected during saccharification, brix should not be used for process monitoring using control chart method based on its variations with time. Useful data for control charts can be, for example, final values reached during processing of batches or time to reach 50% of maximum value of required brix.

5. Conclusions

This paper presents the detailed analysis of process control in food processing industries using control charts. Data for the three parameters, brix, temperature and pH were organized presented using control charts. The time variations of the pH, temperature and brix during saccharification were studied thoroughly. There was an increase in brix as time increased to 2 hours due to formation of sugars, during which brix ranged from 0 to about 12% variations of which were defined as wide compared to temperature and pH. The final brix values during the processing of the batches also varied between batches, from 10.6% to 11.6%, indicating variations in product quality. Temperature during saccharification varies from 58˚C to 62˚C, which is very close to the optimal temperature for saccharification (that is, 60˚C), at which the enzymes work best.

The pH was observed to decrease with time due to oxidation of the aldehyde and ketone groups. Enzymes work best at a wide range of pH from 5.5 to 5.0. The initial and final pH during saccharification varied from one batch to another, with low final values and high initial pH values for some batches. In this study, X-bar and Range charts were plotted and studied.

Eight (8) rules for interpreting control charts were implemented for both X-bar and R charts, results of which showed that the process was out of control. By studying both X-bar and Range charts, it was observed that Rules #1, #2, #3 and #8 were violated, while the remaining rules were not violated due to few batches studied (as some rules require to study up to 15 points in a row on the chart).

This study reveals that the span between control limits, SP_x and SP_R depends strongly on $\bar{R}$. The values of $\bar{R}$ for pH and temperature were lower than the corresponding value for brix. Due to large values of SP_x and SP_R for brix, the control charts were not sensitive in identifying out of control points during saccharification.

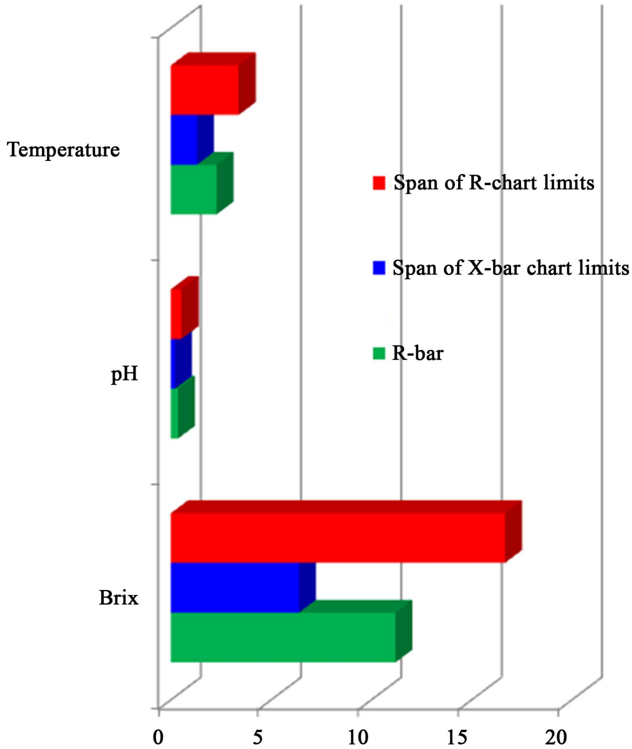


Figure 12. Comparison of $\overline{R}$ values and its effect on the control limits.

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