OPTIMIZING PRE LIMING PH FOR EFFICIENT JUICE CLARIFICATION PROCESS IN SRI LANKAN SUGAR FACTORIES

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Abstract - **This study was conducted treating with Milk of Lime to reach different pH levels (T1- with Initial pH, T2, T3 and T4 with 6.5, 7.5 and 8.5 of pH respectively) to determine the optimum pre-liming pH which could result in best cane juice clarification in Sri Lankan sugar industries. The experiment design used was RCBD with five replicates. ANOVA followed by Duncan's Multiple Range Test (DNMRT) were used to identify significant mean differences. Regression analyses were carried out to model the variation of turbidity, mud volume and CaO with change of juice pH. Quadratic model (R2 = 99.2 %, p <0.001) best fitted to explain the effect of pH on turbidity of juice. Effect of pH on deposited mud volume and CaO were explained by cubic models with R2 = 99.4 % (p <0.001) and R2 = 93.9 %, (p <0.001) respectively.**
Among tested treatments, pH 7.5 is selected as the best for turbidity improvement of the clarified juice while pH 8.5 is the second best. However pH 8.5 (370 ml) was able to deposited significantly high mud volume than pH 7.5 (270 ml). Further, the amount of residual Ca2+ ions in the clarified juice at pH 7.5 (2715 ppm) is clearly lower than the amount of Ca2+ ions remaining in the clarified juice at pH 8.5 (2945 ppm). It is expected to obtain high turbidity and higher mud volume with low sugar inversion at optimum pH. Therefore the results suggest optimum pH range lie around pH 7.5 to 8.5. Conducting similar experiment by using mixed juice extracted from sugar factory mills with pH range around 7.0 to 8.4 at 0.2 increments is suggested to validate the optimum pH.

Keywords - **Defecation, Degradation, inversion, Sugar cane, pH, reducing sugar, Turbidity**

I. INTRODUCTION

Clarification is the one of main important process in the sugar manufacturing process. Because clarification affects the juice filterability, sucrose crystallization and the quality and yield of raw sugar produced. The main purpose of sugar cane juice clarification is to produce clarified juice (CJ) with the lowest concentration of insoluble and soluble impurities. Screening of juice eradicates only the coarse particles since flocculation is necessary to remove the fine and colloidal particles. Therefore flocculation technique is used in clarification process to provide clarified Juice [1].

The conventional method for juice clarification in Sri Lankan sugar industry is that defecation. During the defecation process, Mixed Juice is heated from ~35-55°C to ~76 0C and treated with Lime as milk of lime or lime saccharate to raise the pH from ~5.2 to 7.5–7.8 [2]. Lime is added to react with inorganic phosphate present in the cane juice to form calcium phosphate floc. These macro-flocs have a higher density relative to juice and settle by gravity. The settled flocculated mud impurities are extracted from the clarifier to recover trapped sucrose. In order to recover the trapped sucrose, rotating vacuum filters are used. The filtrate is recirculated and combined with Mixed Juice [3].

In sugarcane, the natural phosphates are occurring in two forms; inorganic (soluble) and organic (insoluble) phosphates. Only the soluble phosphate will react with the lime to form a Calcium Phosphate precipitate. Since presence of phosphates in cane juice is essential for good clarification process, phosphate should be added externally before liming if natural P_2O_5 content (about 200 mg/l) low in mixed juice [4].

Apart from that during the defecation process, a wide range of chemical and physical reactions takes place in the juice. The main chemical reactions include: Precipitation of amorphous calcium phosphate, proteins denaturation (and other organics, such as pectins, gums and waxes), inversion of sucrose due to the combined action of pH and temperature, degradation of reducing sugars to organic acids due to high pH and temperature, precipitation of organic and inorganic acid salts, hydrolysis of starch by the natural amylase in the juice and formation of colour bodies due to the polymerization

(either enzymatically or thermally) of flavonoids and phenolic compounds [5].

The poor quality of clarified juice contributes to scaling of the evaporators and pans, and also increases the probability of sucrose loss to molasses. Clarification also have an impact on crystal morphology, color, crystal content, and polysaccharide and ash contents of raw sugar. And also Juice clarification has a great impact on factory evaporators' heat transfer coefficients particularly if scaling occurs from the excessive addition of lime [6]. Therefore it is important to optimize main operating parameters such as pH, temperature, type & dosage of flocculent, etc. to minimize impact to the subsequent process and overcome existing problems associated with the juice clarification process in local sugar factories. Among them pH is the one of important parameter in clarification, since pH of about 7 is necessary to neutralize the charge on the fine suspended particle in the juice to facilitate coagulation and settling. In addition, pH is important to the rate at which certain reaction occurs especially the precipitation of calcium phosphate. The juice pH was shown to have suggestions on the inversion losses, loss of sugar, color formation, sugar quality, and scaling in subsequent processes. Therefore this research is carried out some recent laboratory work to quantify the effects of different pH levels to clarified juice quality and floc settling behavior reflected by deposited mud volume.

II. MATERIALS AND METHODS

A. Preparation of Milk of Lime –

Fig. 1.For juice clarification in sugar factories milk of lime is prepared at a concentration of 6 to 10 °Be (degree Baume). Therefore, milk of Lime solution was prepared with 10 °Be during this study. For MOL, powdered, hydrate lime (Ca(OH)2) (37.6 g) was added to preheated (60°C) deionized water (400 ml) and mixed well.

B. Treatment structure –

The variety SL 96 128, which is the major commercial variety grown in Sri Lanka was used to obtain the juice for the preceding analysis. 5L of juice was extracted from the sugar mill (Mixed juice, MJ) and initial readings of Brix, Pol, Purity, reducing sugar, turbidity, TSS and TDS of mixed juice were taken. After that four samples were prepared by measuring 1 L of mixed juice in to conical flask separately. Then each of samples were treated with prepared Milk of Lime to reach different pH levels according to following table and required quantity of MOL volume was recorded for each sample.

Table - 1 Juice samples treating with different pH liming for clarification

Treatment No. (Sample)	Limed pH
T1 (Sample 1)	Initial pH ~ 5.44
T2 (Sample 2)	6.5 pH
T3 (Sample 3)	7.5 pH
T4 (Sample 4)	8.5 pH

Then each sample should be heated up to 101^0C (little above the boiling point). After that juice samples were taken and placed in to separate graduated cylinders of 1000 ml capacity for settling at least 2 and half hours. From the graduated cylinders, clarified juice (CJ) samples was taken and analysed for Brix, Pol, Purity, reducing sugar, calcium oxide, TSS, TDS, mud volume and turbidity. **The experiment was conducted using randomized complete block design with five replicates** (One block is defined as treatments done in a day).

C. Sample analysis methods –

Percentage of Brix was measured from the separated juice using digital brix meter. It is represented percentage of total soluble solids in given juice sample. Pol % juice (apparent sucrose) was measured from the same juice by using Polarimeter and Purity was calculated as Pol/Brix×100. Reducing sugars were measured from cane juice using Lane & Eynon (original) method [7]. The turbidity of the clear juice was determined according to the method of GS7-21 (2007). The concentration of calcium in clear juice were determined using EDTA method [7]. Brix%, Pol%, reducing sugar%, Purity%, Turbidity, TSS and TDS were calculated before and after the clarification.

D. Statistical analysis –

Data were analyzed using analysis of variance (ANOVA). The Duncan's Mean separation procedure was used to compare the four treatments and untreated mixed juice (UMJ) sample.
Regression analysis was carried out to model the relationship between turbidity vs pH, mud volume vs pH and CaO vs pH of the juice. Scatter plots were generated to identify the pattern of the data. The linear, quadratic and cubic forms were fitted using OLS method, and the best fit form were selected based on the magnitude of adjusted R^2 and the significance of the model components at 5 % probability level.
SAS (Ver 9.1) and Minitab (Ver 17) software were used for the data analysis.

III. RESULTS AND DISCUSSION

Turbidity, reducing sugar, Brix, Pol, Purity, Mud Volume, calcium oxide and color were measured to study the individual performances of the each treatments (T1(initial pH), T2(pH=6.5), T3(pH=7.5), T4(pH=8.5)). Lime was added to increase the pH to 6.5, 7.5, and 8.5 during clarification. However, the pH values were slightly dropped after heating to 6.25, 7.04, and 8.30, respectively.

The response of each treatment conditions to select main quality parameters are described separately as below.

Table - 2 Average Physical and Chemical characteristics data obtained from clarification test (n=5)

Treatment No.	pH	Turbidity (IU)	Pol %	Brix %	Purity %	TSS (mg/L)	TDS (mg/L)	RS %	CaO (ppm)	Mud Volume (ml)
UMJ (Untreated mixed juice)		218.76a	15.07b	17.56b	85.78ab	11750a	158360b	0.399c		
T1	5.44 (Initial pH)	153.66b	17.13a	20.14a	84.98b	12140a	192700a	0.584a	0 c	0 d
T2	6.5	64.42c	17.08a	19.44a	87.9a	10430a	186710a	0.514b	2445b	60c
T3	7.5	3.64d	17.27a	19.66a	87.83a	10470a	188940a	0.559a	2715ab	270b
T4	8.5	6.12d	17.34a	19.66a	88.19a	10590a	190970a	0.540ab	2945a	364a

TSS = Total Suspended Solids, TDS = Total Dissolved Solids, RS = Reducing Sugar

*For each clarification parameter and CJ composition, figures with the same letters are not significantly (P > 0.05) different.

Average physical and chemical characteristics data obtained from clarification test are shown in table 2.

Turbidity

Turbidity value of juice samples were decreased as the increase of pH values from initial pH 5.44 (T1) to pH 7.5 (T3), but turbidity value was begun to increase again when the pH increases to 8.5. Therefore pH 7.5 shows the lowest turbidity value.

Juice turbidity mainly caused by suspended impurities. Thus, turbidity removal is considered as primary objective and therefore turbidity measurement can be used as a high degree of confidence measurement to measure the efficiency of clarification [8]. Lime was added to neutralize the juice and form insoluble lime salts such as Calcium Phosphates. Colloidal matters such as pectins, hemicelluloses, proteins and coloured compounds are absorbed by the precipitated ions and some colloids are flocculated by heat. Therefore turbidity of treated juice samples decreased compare to UMJ, due to removal of impurities. And also T1 and T2 were shown significantly high values of turbidity compared to T3 and T4. Since low liming pH in the process leads to limitation of reaction with existing phosphate content and reduction in catching impurities. But excess lime to 8.5 pH gave adverse effect on turbidity. Therefore, liming to a certain pH is necessary to achieve lower turbidity. Among the tested treatments liming to pH 7.5 (T3) with heating is suggested in

cold liming process to achieve lower turbidity in clarified juice. Further, regression analysis revealed that the quadratic model ($R2 = 99.2$ %, p <0.001) best fitted to explain the effect of pH on turbidity of juice (Equation 01). The fitted line plot and the 95% confidence Interval (95% CI) for turbidity and pH is depicted in Fig. 1 and it revealed that the data are randomly spread about the regression line and majority of data points are within the confidence limit.

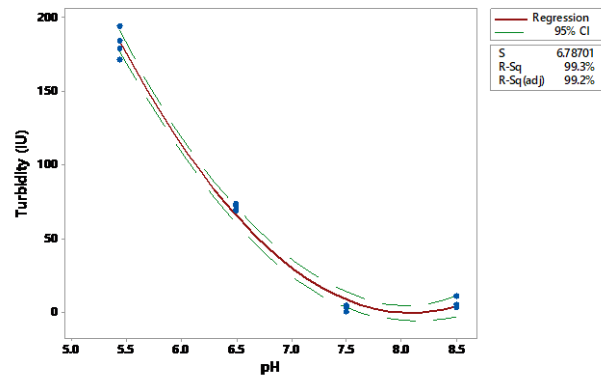$$Turbidity = 1721 - 425.6 \, pH + 26.32 \, pH^2 \quad\quad (1)$$

Fig. 1 Effect of pH on the turbidity of clarified juice.

Reducing sugar

The reducing sugar percentage was also significantly affected by the pre liming pH value. The pH adjustment of sugar cane juice via the addition of MOL is a critical step during the clarification process to avoid sucrose inversion by hydrolysis in acidic conditions ($\leq$ pH 4) and alkaline degradation ($\geq$ pH 8) [13]. Sucrose mostly hydrolyses to make reducing sugars, glucose and fructose, which are available in the form of fructosyl oxocarbenium cation and D–glucose at the extreme acidic and alkaline juice conditions.

According to Table 2, highest value of reducing sugar was recorded in the T1 (pH = 5.44) due to hydrolysis of available sucrose in to reducing sugars in acidic conditions. And also reducing sugar value was begun to reduce again when the pH rises to 8.5 (T4) due to alkaline degradation. Therefore, liming to a certain pH is necessary to maintain optimum level of reducing sugar in the given sugarcane juice sample.

Pol/Brix/Purity

Sucrose is most stable ~ pH 8.3, whereas glucose and fructose (invert sugars) are most stable at severe acid conditions such as pH 3-4, thus balancing both sucrose and invert sugars at optimum level is a real challenges to sugar industries [9]. In the above paragraph, the variation in Glucose and Fructose (Reducing Sugar) was analysed at different pH levels, whereas

the variation in Sucrose (Pol %) at different pH levels is analysed in this paragraph.

Since some of the dissolved non sugars removed from the mixed juice (MJ), the pol of the Clarified Juice (CJ) samples (T1, T2, T3 and T4) significantly exceeds that of the UMJ sample. And also brix value of Clear Juice (CJ) samples (T1, T2, T3 and T4) significantly exceeds that of the UMJ due to increase of dissolved ions with introducing liming. However, the ratio of pol to Brix (Purity %) of the treatments T1 to T4 is not significantly different from that of the UMJ.

However there are no significant difference between treatments (T1 to T4), for pol % and Brix %, while purity of T1 significantly lower compared to other treatments.

Mud Volume
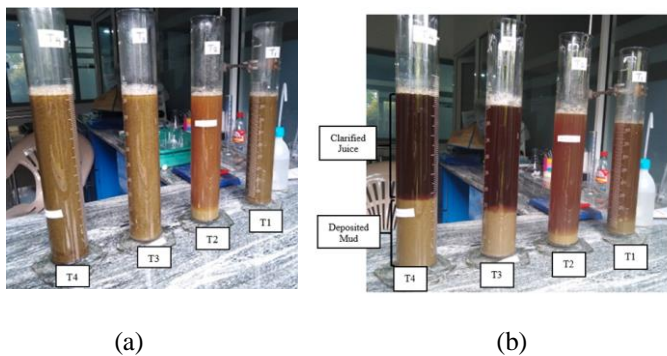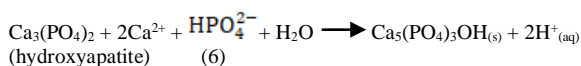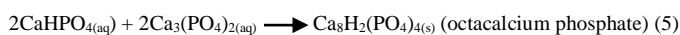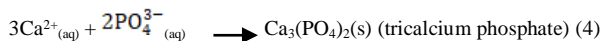

(a)   (b)

Fig. 2. (a) Before clarification (b) After Clarification

After treating with different pH values, Precipitation of various calcium phosphates forms are occurred in sugarcane juice samples. Equation 2 is shown the dicalcium phosphate form of precipitation. Secondary reaction takes place to form intermediate calcium phosphate phases due to the creation of unstable and insoluble dicalcium phosphate in water. Therefore the most stable compound of the calcium phosphate phases are shown in Equations 3, 4, 5 and 6 [3],[10].

$$Ca^{2+}_{(aq)} + HPO_4^{2-}_{(aq)} \longrightarrow CaHPO_{4(s)} \text{ (dicalcium phosphate)} \quad (2)$$

$$Ca^{2+}_{(aq)} + 2H_2PO_4^-_{(aq)} \longrightarrow Ca(H_2PO_4)_{2(s)} \text{ (monocalcium phosphate) (3)}$$

$$3Ca^{2+}_{(aq)} + 2PO_4^{3-}_{(aq)} \longrightarrow Ca_3(PO_4)_2(s) \text{ (tricalcium phosphate) (4)}$$

$$2CaHPO_{4(aq)} + 2Ca_3(PO_4)_{2(aq)} \longrightarrow Ca_8H_2(PO_4)_{4(s)} \text{ (octacalcium phosphate) (5)}$$

$$Ca_3(PO_4)_2 + 2Ca^{2+} + HPO_4^{2-} + H_2O \longrightarrow Ca_5(PO_4)_3OH_{(s)} + 2H^+_{(aq)}$$
(hydroxyapatite)   (6)

Type of calcium phosphate phase which is formed during clarification process is depend on the concentration of calcium

and phosphate, pH and the nature of the particle interface [3]. Therefore settling rate and final mud volume are differed with pH values of the treatments. Final mud volume was increased with the increase of pH liming and it was highest in treatment 4 (pH=8.5). That leads to highest juice clarity.

Regression analysis revealed that the cubic model (R^2 = 99.4 %, p <0.001) best fitted to explain the effect of pH on deposited mud volume (ml) (Equation 7). The fitted line plot and the 95% confidence Interval (95% CI) for mud volume and pH is depicted in Fig. 3 and it revealed that the data are randomly spread about the regression line and majority of data points are within the confidence limit.

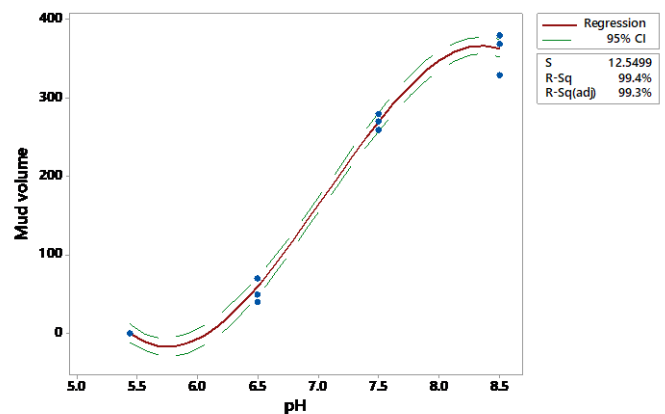$$\text{Mud volume} = 13805 - 6240 \text{ pH} + 916.0 \text{ pH}^2 - 43.29 \text{ pH}^3 \quad (7)$$

Fig. 3. Effect of pH on the deposited Mud volume after Clarification

CaO

When the pH liming increases, the mud volume increases but it was observed that the residual concentration of calcium also increased with the pH. Introduced Ca^{2+} ions in to juice samples react with P_2O_5 to form a calcium phosphate precipitation, but some amount of Ca^{2+} ions are remained in the clarified juice due to limited amount of natural occurring P_2O_5 in the sugarcane juice. Therefore calcium oxide content of clear juice increased with increased levels of liming pH according to the above Table 2. Therefore higher calcium level in the clarified juice obtained with higher pH (pH ~ 8.5) result in an increase in scale formation in the evaporators.
The regression analysis revealed that the cubic model (R^2 = 93.9 %, p <0.001) best fitted to explain the effect of pH on CaO (ppm) of clarified juice (Equation 8). The fitted line plot and the 95% confidence Interval (95% CI) for CaO and pH is depicted in Fig. 4.

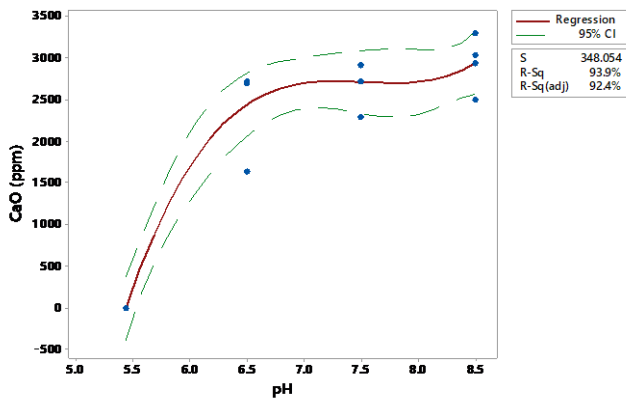$$\text{CaO} = -131455 + 53651 \text{ pH} - 7142 \text{ pH}^2 + 316.5 \text{ pH}^3 \quad (8)$$

Fig. 4. Effect of pH on CaO content in clarified juice

Color

The formation of colourants produced during factory processing is mainly due to sugar degradation reactions. Reducing sugars, such as glucose and fructose, formed by the inversion of sucrose, play an important role in the formation of colour. These sugars degrade due to changes in operating conditions such as pH and temperature to form highly reactive intermediates, which undergo condensation and polymerisation reactions to form highly coloured polymers [11]. According to the Fig. 2, clarified juice color was darkened with the increase of pH liming from T1 to T4.

Colourants such as caramels and melanoidins are pH insensitive; therefore their colour does not change across pH 5.44–8.5. But flavonoids and phenolic compounds (i.e., colour precursors) are highly pH sensitive. Therefore, these types of compounds are lightly coloured at pH 5.44 (lower liming pH) and darken greatly at pH 8.5 (highly alkaline conditions) [11]. This is because at pH 8.5, the ionization of these compounds is almost complete. Hence, these compounds are more highly coloured in their anionic form than in their neutral form. That's the reason behind the color variation of clarified juice which is mentioned in Fig. 2.

Main juice quality parameters and mud volume were separately analyzed to identify the individual performances of the each treatments (T1 (5.44), T2 (pH=6.5), T3 (pH=7.5), T4 (pH=8.5))., However, turbidity measurement can be used as a high degree of confidence measurement to measure the efficiency of clarification since turbidity removal is the primary objective of the juice clarification process. Therefore, the variation of other key parameters with turbidity is further elaborated using the contour graphs.

Effect of pH on turbidity and pol in juice

In order to achieve high clarification efficiency, the turbidity value should be low. Thus according to table 2, the lower turbidity values are recorded at 7.5 pH (3.64 IU) and 8.5 pH (6.12 IU). When analyzing this lower turbidity with pol in juice parameter, pol in juice value should be a higher one. The darkest green color represents the highest pol in juice according to the contour graph mentioned in Fig. 5. By considering these two phenomena's, the most suitable region with best pH range is between 8.0 to 8.5 (pol % > 17.3%). But the pH range from 7 to 8.0 can also be taken in to consideration since it shows less variation in pol in juice > 17.25 %.
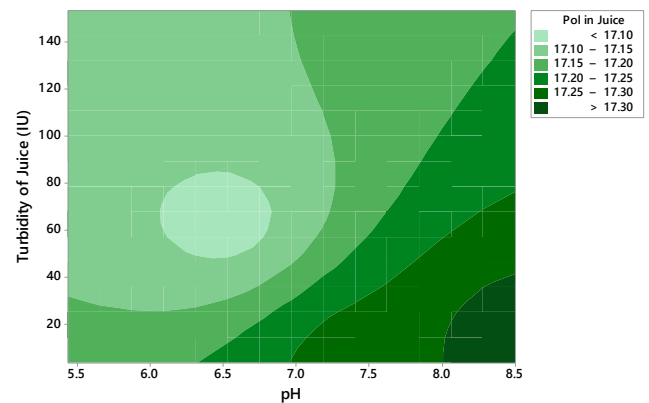

Fig. 5. Contour Plot for variation of Pol % and Turbidity with pH of juice

Effect of pH on turbidity and reducing sugar in juice

In order to achieve high clarification efficiency, the turbidity value should be low. According to table 2, the lower turbidity values are recorded at 7.5 pH (3.64 IU) and 8.5 pH (6.12 IU). When analyzing this lower turbidity with reducing sugar parameter, reducing sugar value should be low. The darkest blue color represents the lowest reducing sugar according to the contour graph mentioned in figure 6. By considering these two phenomena's, the most suitable region with best pH range is between 7.0 to 8.5. Even though the pH 8.0 to 8.5 shows much better reducing sugar value than the value showed in pH range of 7.0 to 8.0, this cannot be considered since the reducing sugar started to invert at high pH. As a conclusion, the best pH range is 7.0 to 8.0 by considering both Fig. 5 & Fig. 6.
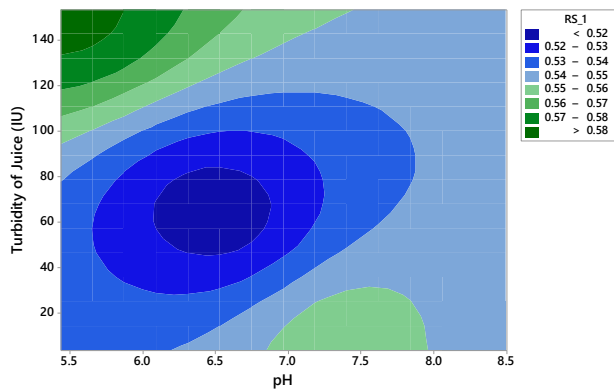
Fig. 6. Contour Plot for variation of reducing sugar % and Turbidity with pH of juice

Effect of pH on turbidity and purity in juice

By considering the purity of the juice, the best juice purity gives when the pH is between 8.0 to 8.5, according to figure 8. But when compare with the reducing sugar and pol in juice parameters, the optimum pH range is 7.0 to 8.0 with respect to Fig. 5, 6 & 7.
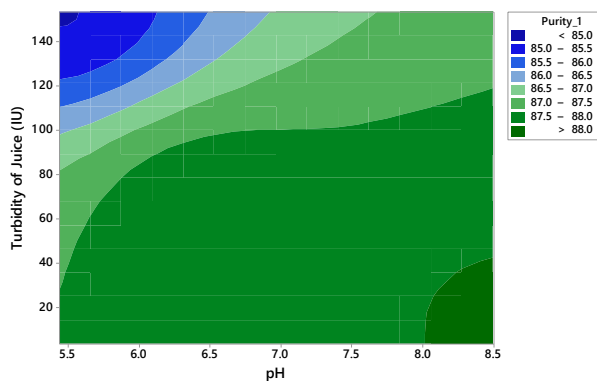

Fig. 7. Contour Plot for variation of purity % and Turbidity with pH of juice

Turbidity, reducing sugar, calcium oxide, Mud Volume, Brix, Pol, Purity and color were measured to study the individual performances of the each treatments (T1(initial pH), T2(pH=6.5), T3(pH=7.5), T4(pH=8.5)). However, turbidity, reducing sugar, calcium oxide and purity are more influential in indicating the juice clarification efficiency than the rest.

Over liming to pH ~ 8.5 can result in highly alkaline conditions and it was recorded separation of highest mud volume from the mixed juice. Although an alkaline environment can reduce sucrose losses due to inversion, but it would exacerbate scaling in the evaporators due to increase of residual Ca^{2+} ions in the clarified juice. As well as it promotes the formation of colourants (dark brown) and initiates to decrease reducing sugar due to the alkaline degradation of glucose and fructose. On the other hand deficit liming can cause the acidic conditions (pH = (5.44 - 6.5)) and it was recorded lowest level of residual Ca^{2+} in the clarified juice. So it is a good sign for the retardation of scale formation in the evaporators. But under deficit liming, no clear mud separation was observed with each treatment (T1 & T2). Furthermore, each treatments showed higher turbidity values and lower deposited mud volumes of the juice under the acidic conditions (pH = 5.44 & pH = 6.5).

IV. CONCLUSION

Since over liming and deficit liming are not good, neutral liming to pH~7.5 should be considered for sugar clarification process. Liming to pH 7.5 gave a lowest turbidity value. Although the precipitated mud volume at pH 7.5 (270 ml) is slightly lower than the precipitated mud volume at pH 8.5 (370 ml), but it is significantly higher than the precipitated mud volume at pH 6.5 (70 ml). In addition, the amount of residual Ca^{2+} ions in the clarified juice at pH 7.5 is clearly lower than the amount of Ca^{2+} ions remaining in the clarified juice at pH 8.5. Thus 7.5 pH is selected as the best performed pH out of tested pH values.

Among tested treatments, T3 (pH =7.5) is the best for turbidity improvement of the clarified juice while T4 (pH 8.5) is second best. In contrast T4 is deposited significantly high mud volume than T3. It is expected to obtain high turbidity and higher mud volume with low sugar inversion at optimum pH. Therefore the results suggest optimum pH range lie around pH 7.5 to 8.5. Conducting similar experiment by using pH range around 7.0 to 8.4 at 0.2 increments is suggested to validate the optimum pH.

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