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Study of Sugarcane Juice Clarification with Additives by Continuous Centrifugation

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The quality and yield of sugar influenced by the raw material and the operational stages. The main objective of treatment of the broth is to eliminate unwanted particles in sugarcane that increase equipment wear and incrustations, reducing productivity. The procedure used in the clarification defines the type of sugar produced. Initially chemical additives are used with subsequent heating which favors decantation, necessitating effective control so that the impurities are effectively removed and do not influence the subsequent steps. This work evaluated the clarification of the sugarcane juice by caleation, addition of phosphoric acid in different concentrations and continuous centrifugation, through the physic-chemical parameters. Analyzes were pH, °Brix, turbidity, conductivity and ICUMSA color. It adopted for the experimental results a factorial planning 2^2 (STATISTICA 7.0) to evaluate the statistical representation and interaction between independent and dependent variables. They compared to the means by Tukey's test ($p < 0.05$) and to evaluate the level of association between the dependent variables studied was adopted Pearson's correlation coefficient (R), through analysis of variance (ANOVA) and by the F test. The results of the physic-chemical analyzes that indicated the best operating conditions that caused greater reduction of color and turbidity was point 5 (90°C and 150 ppm of phosphoric acid). In the statistical planning, the color reduction did not present statistical significance and the turbidity was statistically significant, making it possible to present the analysis of variance (ANOVA) and the correlation coefficient. The model fits well with the experimental data regarding turbidity reduction, that is, the model is statistically significant at the 95% confidence level for the studied range.
Keywords: Sugarcane, centrifugation, clarification.

1. Introduction

The sugar manufacturing process aims at the proper preparation of the raw material which consists of efficient cleaning, extraction of the sugarcane juice, adequate treatment, concentration, formation of the crystals (cooking), crystallization, centrifugation and drying (Payne, 1989). The Brazilian production of sugar is made from the extraction of sugarcane juice (Sacharum officinarum), where it involves an industrial process. This based on grinding, treating, filtering and boiling the broth so that then the centrifugation of the sugar produced by separating it from the molasses and defining its type according to the chemical inputs used (Costa et al, 2018). The quality of the sugar produced is directly associated with the process of clarification of the broth, once the treatment is ineffective, it produces a lower quality sugar, with presence of more intense colors, impurities and black dots (Albuquerque, 2011). In most Brazilian plants, clarification carried out by burning sulfur, producing sulfur dioxide in the sulfation process, which questioned by safety standards and

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environmental issues, since it can form sulfuric gas and later sulfuric acid, which is mainly responsible for acid rain (Hamerski, 2009). Therefore, the tendency is for this process replaced by others, such as ozonization, carbonation or bicarbonation. In the food industry, it is common to use centrifugation to separate mixtures, which not easily separated by decantation of one or more components being liquid / liquid or solid / liquid. Centrifugation is a mechanical process, which has the function of separating or clarifying a mixture, from different densities of its components. This process used in the development of this study, aiming to clarify the sugarcane juice, reducing the amount of inputs used and optimizing the process steps.

2. Materials and Methods

2.1 Materials

Phosphoric acid, hydrochloric acid, sodium hydroxide manufactured by Merck Ltd. United States. The calcium oxide used was provided by Cal Norte S.A., Rio Grande do Norte, Brazil.

2.2 Sugarcane Juice

The analysed sugarcane juices extracted at the Monte Alegre Plant, Mamanguape - Paraíba, Brazil, from the first milling stage. 1.0 L broth used for each experiment. The broth filtered on a polyethylene filter, added with phosphoric acid at different concentrations according to the experimental planning. Then calcium oxide solution (10%) added, the pH adjusted to 7.0 to 7.2.

2.3 Experimental planning 2^2

From the experimental planning 2^2 the results go through an analysis of variance (ANOVA), by the test F. The means were compared by the *Tukey test* at a 5% probability level ($p < 0.05$), using statistical software (STATISTICA 7.0). The Pearson correlation coefficient (r) used to assess the level of association between the dependent variables studied (Barros Neto et al, 2007).

In the treatment of sugarcane juice by centrifugation, the temperature and the concentration of phosphoric acid (independent variables) with respect to colour and turbidity (dependent variables) were evaluated, as well as to verify the interaction between these parameters.

Table 1 show the matrix and the variables used in the treatment of the juice by centrifugation.

Table 1 - Matrix 2² of experimental planning used

Experiment	Temperature (◦C)	Phosphoric acid (ppm)	Variables	
1	75	0	(-1)	(-1)
2	75	300	(-1)	(+1)
3	105	0	(+1)	(-1)
4	105	300	(+1)	(+1)
5	90	150	0	0
6	90	150	0	0
7	90	150	0	0

2.4 Centrifugation

Centrifuges apply relative centrifugal force and acceleration perpendicular to the fixed axis to separate the suspended particles from a solution (Cremasco, 2011). In this study the cane juice was fed by the top of the centrifuge (Brand: West), capacity of 50L/ h, gravity 9 g, 1.375 rpm, being removed on the upper side.

The relation between the units of quantification of the centrifugation determined by Eq. (1):

$$\mathrm{G\ force} = 1{,}12.\,\mathrm{r}.\left(\frac{\mathrm{RPM}}{1000}\right)^2 \tag{1}$$

Where: r is the radius of the rotor in millimetres (42.5 mm); G force = 9

The gravitational force on a particle quantified by applying Eq. (2).

$$\mathrm{F_g} = \mathrm{m.g} \tag{2}$$

Where: m: particle weight (g); g: acceleration of gravity (m/s^2)

The centrifugal force is quantified from Eq.(3).

$$\mathrm{F_C} = \mathrm{m.r.w^2} \tag{3}$$

Where: m: particle weight (g); r: particle radius (m); w: angular velocity (rad/s^2)

Relating the centrifugal and gravitational forces and converting the angular velocity to revolutions per minute (rpm), we have Eq. (4) which simplified in Eq. (5):

$$\frac{F_c}{F_g} = \frac{m.r.w^2}{m.g} = \frac{rw^2}{g} = \frac{r}{g}.\left[\frac{2.\pi.N}{60}\right]^2 \quad (4)$$

$$\frac{F_c}{F_g} = 0.00118.r.N^2 \quad (5)$$

Where: N revolutions per minute (rpm)

2.5. Physical-chemical analysis

To examine the influence of the treatment on the sugarcane juice by: adding phosphoric acid, caleation, heating and centrifugation physical-chemical analyses were performed: ºBrix, pH, and turbidity, conductivity and ICUMSA colour before and after the centrifugation experiments using specific methodologies of the sugar-alcohol area (ICUMSA, 2009). The ambient temperature (22 °C) maintained so as not to cause changes in the values of the analyses.

2.5.1 °Brix

To determine the percentage of solids dissolved in solution, expressed in ° Brix degrees, by refractometer, 50.0 mL of the filtered sample the filtered sample, if the refractometer reading.

2.5.2 pH

The determination of the concentration of H^+ ions performed by potentiometric method. The equipment calibrated at room temperature and pH measured.

2.5.3 Turbidity

The solution was to stand for 5 minutes to eliminate the bubbles, and then reading held in turbidímetro.

2.5.4 ICUMSA Colour

To determine the colour of the broth by the ICUMSA method, a broth solution was prepared with 100.0 mL at 5 ° Brix, filtered using 0.45 µm aperture membrane. The pH adjusted to 7.0 with solution of hydrochloric acid or sodium hydroxide (0.1N), then the reading was carried out with the spectrophotometer at 420 nm and the colour was quantified using Eq.(6), (ICUMSA, 2009).

$$\text{ICUMSA Color} = \frac{(1000xA)}{(BxC)} \quad (6)$$

Where: A = absorbance of the measured solution at 420 nm; B = thickness of the cell in cm (1,0 cm); C = concentration of the filtrate in g/mL in function of 5 °Brix to 100.0 mL of the solution.

2.5.5 Conductivity

For conductivity determination, the instrument initially gauged, the electrode washed with distilled water, wiped it and immersed in the sample, waiting until the numbers on the display stabilized.

The determination of the conductimetric ashes carried out from Eq. (7):

$$cz(\%\, ^m/_V) = (16{,}2 + 0{,}36.D).10^4.C.f.K \quad (7)$$

Where: D: Sample mass used in grams per 100 mL

C: C1-C2

C1: Conductivity in µS/ cm at 20^{o}C of the solution; C2: Conductivity in µS/ cm at 20 ^{o}C of distilled water; f: Solution Dilution Factor = 5/ S; S: Sample weight in grams; K: Conductivity cell constant (cm^{-1});

The values obtained raised to 10^6 to give the representation in ppm.

3. Results and discussion

3.1. Centrifugation Quantification

Applying Equation (1), the rotations per minute that the centrifuge operated quantified, resulting in 434.8 rpm. Then, Equation (4) applied determining the separation ratio of the particles of the sugarcane juice treated with the mass, the radius and number of revolutions (N - rpm) being constant. The value obtained for the relation between the centrifugal and gravitational forces acting on the separation using Equation (5) was 8,983.93.

3.2. Physical-chemical analyses of sugarcane juice

Table 2 presents the results of the physical-chemical analyses of the raw sugarcane juice obtained from the experimental design 2^2 elaborated from dependent and independent variables defined experimentally and through the literature (Hamerski, 2009).

Table 2 - Physical-chemical analyses of raw sugarcane juice

Experiment	Before Treatment				
	pH	°Brix	Turbidity (NTU)	ICUMSA Colour	Conductivity (mS)
1	4.30	14.0	568	23079.40	3.31
2	4.30	14.0	568	23079.40	3.31
3	4.28	13.9	568	23647.50	3.63
4	4.28	13.9	568	23647.50	3.63
5	4.65	13.0	568	23957.50	3.50
6	4.65	13.0	568	23957.50	3.50
7	4.65	13.0	568	23957.50	3.50

The Table 3 presents the results of the physical-chemical analysis of the sugarcane juice after caleation (addition of 10% CaO solution) to pH close to 7.0, addition of phosphoric acid in the proportions of experimental design and centrifugation.

Table 3 - Physical-chemical analysis of the broth with corrected pH and additions, heated and centrifuged

Experiment	After Treatment							
	T (°C)	H_2PO_4 (ppm)	pH	°Brix	Turbidity (NTU)	ICUMSA Colour	Conductivity (mS)	Reduction Colour (%)	Reduction Turbidity (%)
1	75	0	6.98	13.9	474	22469.70	3.92	2.64	16.55
2	75	300	7.02	13.8	227	19538.90	4.01	15.34	60.04
3	105	0	6.80	13.5	535	22509.47	3.94	4.81	5.81
4	105	300	7.00	13.9	529	22175.45	2.91	6.22	6.87
5	90	150	6.90	13.8	462	18823.70	3.35	21.43	18.66
6	90	150	7.00	13.6	478	19767.80	3.35	17.49	15.85
7	90	150	6.90	13.6	466	19079.40	3.35	20.36	17.96

The results of the physical-chemical analysis of the treated and centrifuged sugarcane juice indicated that the best operating conditions that caused the greatest colour reduction and turbidity were point 5.

3.3 Colour reduction (%) of sugarcane juice – STATISTICA 7.0

The physical-chemical results obtained experimentally were analysed in the STATITICA 7.0 program and the estimates of the effects for the reduction of colour (%) in the cane juice submitted to the treatment of: caleation, phosphoric acid and centrifugation are presented in Table 4.

Table 4 - Estimates of the effects on colour reduction (%) in the sugarcane juice subjected to the treatment

	Effect	Standard Deviation	p	Confidence interval (-95%)	Confidence interval (95%)
Mean	12.61	3.63	0.040137	1.07	24.16
X1	7.06	9.60	0.515518	-23.49	37.60
X2	-3.47	9.60	0.741491	-34.02	27.07
X1. X2	-5.64	9.60	0.597890	-36.19	24.90

Where: X1: concentration of phosphoric acid, X2: temperature, p: probability of significance ($p< 0.05$).

Evaluating the data in Table 4, it was possible to observe that none of the factors, presented statistical significance ($p <0.05$ - statistically significant at 95% confidence) for the studied range.

3.4 Reduction of Turbidity (%) of the sugarcane juice – STATISTICA 7.0

The physical-chemical results obtained experimentally were also analysed in the software STATITICA 7.0 for the reduction of turbidity and the estimations of the effects presented in Table 5.

Table 5 - Estimation of the effects for the reduction of turbidity (%) in the sugarcane juice

	Effect	Standard Deviation	p	Confidence interval (- 95%)	Confidence Interval (95%)
Mean	20.25	1.45	0.000798	15.63	24.87
X1	22.27	3.84	0.010201	10.05	34.49
X2	-31.95	3.84	0.003637	-44.18	-19.73
X1. X2	-21.21	3.84	0.011684	-33.44	-8.99

Where: X1: concentration of phosphoric acid, X2: temperature, p: probability of significance ($p < 0.05$)
By evaluating the data in Table 5, it can be seen that the factor X1 (phosphoric acid concentration) presented statistical significance for the studied range ($p < 0.05$ to 95% confidence). Therefore, the treatment of caleation with addition of acid phosphoric acid and centrifugation have been able to reduce turbidity statistically and operationally, since this reduction benefits the following stages of sugar production.
Statistically the concentration of phosphoric acid, temperature and the interaction between these effects are significant, that is, they interfere positively in the reduction of turbidity of the sugarcane juice with the methodology applied.

The coded model (Equation 8) representing the turbidity reduction of sugarcane juice generated from of the regression coefficients.

$$\mathrm{Turbidity}(\%) = 20.25 + 11.14\mathrm{X}_1 - 15.98\mathrm{X}_2 - 10.61\mathrm{X}_1\mathrm{X}_2 \quad (8)$$

One of the ways to assess the quality of fit of the model is through the coefficient of determination or regression (R^2). This coefficient indicates how much the model was able to explain the data collected, so the closer to 100% the better the adjustment. Table 6 shows the analysis of variance (ANOVA) for turbidity reduction. As the correlation coefficient was high (R^2 = 97.80%) and the value of F calculated for the regression presented a value higher than the table ($F_{0.05;3;3} = 9.28$) it is possible to conclude that the model fits well to the experimental data, that is, the model is statistically significant at the 95% confidence level for the studied range.

Table 6 - ANOVA evaluation for turbidity reduction response (%)

	Quadratic sum	Freedom Degrees	Quadratic Mean	F Test	R^2 (%)
Regression	1967.14	3	655.71	44.47	97.80
Residue	44.24	3	14.75		
Lack of fit	39.94	1			
Pure error	4.30	2			
Total	2011.38	6			

$F_{0.05; 3; 3} = 9.28$
Explaining Table 6, we have the regression that represents the fit to the model presented in Equation (8), the residual is equivalent to the sum of the lack of fit to the model and to the pure error and the sum of the quadratic sum is equivalent to regression plus the residue.
The ANOVA results indicate that the linear models (R^2) can represent the turbidity removal data. When comparing the percentages of turbidity removal in Table 5 with the ANOVA results, Table 6 shows agreement between the results. For the highest percentages of turbidity removal obtained in treatments with 90 °C, both the ANOVA and the estimation of the effects, presented as a significant factor, on the ability to remove the turbidity. The results obtained regarding the reduction of the turbidity of the sugarcane juice, in relation to the temperature, are close to those used by Hamerski, (2009).
Figure 1 presents the estimation of the effects on turbidity reduction in the treated broth in the form of the Pareto Diagram.

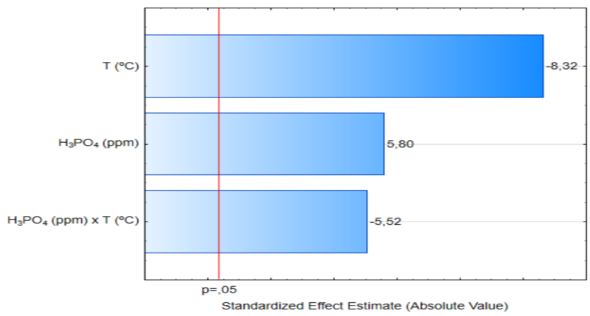

Figure 1. Pareto diagram for turbidity reduction of treated sugarcane juice

Evaluating Figure 1, we can see that effects whose rectangles are to the right of the dividing line (p = 0.05) are significant ($p > 0.05$), so in predictive models these effects must be included. The significant factors of temperature and phosphoric acid, obtained from the Pareto graph, coincide with those presented in the

analysis of variance. The factor that has the greatest significant effect on the reduction of turbidity is the temperature, acting reducing it. We can observe that the interaction between the evaluated factors was also representative. The temperature has a positive effect because it raises the concentration of the broth and, consequently, contributes to the separation of the soluble solids.
Figure 2 shows the response surface plot where the effects of temperature and addition of phosphoric acid can be visualized with respect to turbidity reduction.

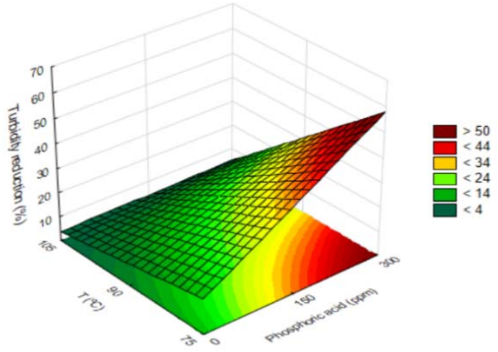

Figure 2. Response surface for turbidity reduction (%)

The response surface for turbidity removal (Figure 2) represents the effects of temperature and phosphoric acid, showing that the higher the concentration of phosphoric acid the greater the turbidity removal and the temperature did not influence the response considerably. The use of lower temperatures in broth treatment benefits the sugar production process because excess temperature degrades sucrose by increasing broth colour and reducing yield and quality. It`s found that in order to achieve the best turbidity removal temperatures near 90 ° C and high concentrations of phosphoric acid are required. Figure 2 presents graphically the independent variables (temperature and concentration of phosphoric acid) and the reduction of turbidity (variable response), obtained as a surface in three-dimensional space.

4. Conclusion

Experiments and literature (Hamerski, 2009) defined the dependent variables (turbidity and ICUMSA color) and independent variables (T (°C), phosphoric acid concentration);
The physic-chemical evaluation of sugarcane juice by experimental design indicated that the best treatment conditions for the reduction of colour (90 ° C, 150 ppm) and turbidity (75 ° C, 300 ppm);
The STATISTICA 7.0 indicated that the colour reduction was not statistically significant and the turbidity reduction presented statistical significance ($p > 0.05$);
It was possible to generate a coded model to represent the reduction of turbidity of the sugarcane juice from the regression coefficients. The correlation coefficient (R^2 = 97.80%) was high, (F cal. Regression = 44.47> $F_{0.05; 3; 3}$ = 9.28), allowing to conclude that the model fits well with the experimental data.

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