CLARIFICATION OF CANE JUICE FOR FERMENTATION

By

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Abstract

Fermentation feedstocks in the sugar industry are based on cane juice, B molasses or final molasses. Brazil has been producing ethanol by directing sugarcane juice to fermentation directly or using lower quality juice as a diluent with B molasses to prepare the fermentation broth. One issue that has received only limited interest particularly from outside Brazil is the most appropriate conditions for clarification of the juice going to fermentation. Irrespective of whether the juice supply is the total flow from the milling tandem or a diffuser station or a part of the total flow, removal of the insoluble solids is essential. However, the standard defecation process used by sugar factories around the world to clarify juice can introduce unwanted calcium ions and remove other nutrients such as phosphorus and nitrogen that are considered essential for the fermentation process. An investigation was undertaken by SRI to assess the effects on the constituents of cane juice when subjected to the typical clarification process in an Australian factory and what conditions would be needed to provide a clarified juice suitable for fermentation. Typical juices from one factory were clarified in laboratory trials under a range of pH conditions and the resulting clarified juices analysed. The results indicated that pH had a major effect on the residual concentrations of key constituents in the clarified juice and that the selected clarification conditions are determined by the nominated quality criteria of clarified juice feedstock for fermentation. Further trials were conducted in overseas factories to confirm the results obtained in Australia. It became apparent that the preferred specifications for clarified juice going to fermentation varied from country to country. Each supplier of fermentation technology had criteria applying to clarified juice feedstock that would have a major impact on the standard of clarification required to achieve compliance with the criteria.

Introduction

Although the sugar industry in Brazil has been producing ethanol by directing sugar cane juice to fermentation for many years and has developed significant process technology and experience in this field, the sugar industries in other countries are just embarking on this option. The impetus for the change from crystal sugar to a combination of crystal sugar and ethanol includes the continuing low prices for raw sugar, the rising operating costs and the increasing worldwide demand for biofuels to replace fossil fuels.

One issue that has to be addressed is the most appropriate conditions for clarification of the juice going directly to fermentation. Irrespective of whether the juice supply is the total flow from the milling or diffuser station or a part of the total flow, removal of the insoluble solids is essential.

However, the defecation clarification process used by sugar factories around the world introduces unwanted calcium ions and removes other nutrients such as phosphorus and nitrogen that are considered essential for the yeast.

A review was undertaken by SRI to assess the effects on the constituents of cane juice when subjected to the typical clarification process in an Australian factory.

Optimum juice conditions

There is not a lot of information available in the public domain that defines the optimum conditions for clarified juice going to fermentation. However, it is generally agreed that:

- The juice should be free of all insoluble solids;
- The maximum concentration of nutrients should be retained within the juice;
- The turbidity of the clarified juice may not be relevant although one fermentation technology provider has specified a maximum turbidity of about 15 (based on ICUMSA method GS7-21);
- Inversion of sucrose in acidic conditions to fermentable monosaccharides is beneficial; and
- Juice should not be contaminated with bacteria.

As an example, the clarified juice shown in Figure 1 (left) would be unsuitable for a sugar manufacturer but is considered to be suitable for fermentation by some fermentation technology and equipment suppliers.

Fig. 1—Samples of turbid clarified juice suitable for fermentation (left) and clear clarified juice suitable for sugar production (right).

Fermentec Ltda of Brazil (A. Godoy, 2008; *pers comm.*) provided the data shown in Table 1 on optimum conditions for clarified juice going to fermentation.

Table 1—Optimum conditions for fermentation juice as specified by Fermentec,

Parameter	Nominated value
pH of limed juice	5.8–6.0
pH of clarified juice	5.0–5.5
Insoluble solids	<0.2%
Calcium	As low as possible (<800 mg/kg)
Manganese	2.0 mg/kg
Zinc	2.0 mg/kg
Magnesium	200 mg/kg
Phosphorus	50–65 mg/kg
Nitrogen (as NH_3OH)	60–70 mg/kg
Turbidity	Not important

Another fermentation company in South America provided the information shown in Table 2 for the required quality of clarified juice going to fermentation.

Table 2—Another specification for fermentation juice in South America.

Parameter	Nominated value
Temperature	70°C
pH	5.8–6.2
Reducing sugars	13–14 g/100 mL juice
Sulphur	0
Bacterial infection	<1.00 E+06
Bagacillo	<0.40%
Insoluble solids	0
Turbidity	1000–1200 IU
Dextran	<250 ppm
Volatile acids	500–1000 ppm

However, there is considerable variation in the target values of quality parameters at factories that are already sending juice directly to fermentation.A survey of some factories in Central and South America (see Table 3) indicates a wide range in pH of the feed juice.

Table 3—Range of pH values of clarified juice going to fermentation.

Factory	pH of clarified juice	Factory	pH of clarified juice
#1	6.8 – 6.9	#6	6.2 – 6.4
#2	6.0 – 6.2	#7	5.4 – 5.6
#3	6.0 – 6.2	#8	6.6 – 6.8
#4	6.0	#9	<6.8
#5	5.3 – 5.5	#10	6.3

In South America, one new factory with a cane diffuser has eliminated the clarification step. The justification relies on the assumption that the fermentation process and the viability of the yeast can tolerate an insoluble solids level of up to 0.2%. The installed process removes gross solids by filtration before sending the juice to fermentation.

The quality criteria are set to maximise the efficiency of the fermentation process and the viability of the yeast cells but appear not to consider the scaling potential in the evaporator or distillation columns.

Experimental

Settling rate tests were conducted on samples of mixed juice that were collected from an Australian factory to examine the effect of different pH levels on the settling rate, turbidity and residual impurities of the resulting clarified juice. The tests involved the following procedure:

- Heat the juice to either 75°C or 90°C;
- Adjust the pH of each one litre sample using lime saccharate to a selected value between 5.8 and 7.6 (the raw juice pH was 5.3);
- Heat the limed juice to boiling;
- Transfer the juice to a settling tube and measure the settling rate and settled mud volume; and
- Collect subsamples for analyses.

Mitsui Superfloc flocculants were tested at 3 mg/kg of juice. These flocculants had molecular weights of 24 × 10^6 and hydrolysis levels ranging from 24% to 38% and covered the full range of hydrolysis levels normally used in raw sugar manufacture.

The samples of clarified juice were analysed for turbidity (ICUMSA GS7-21), total phosphorus (ICUMSA GS7-15), calcium by inductively coupled plasma mass spectrometry and total organic nitrogen (Kjeldahl).

Results

Settling tests

The settling rates were very similar for each test. There appeared to be no effect of pH on the settling rates. Most settling rate results were in the range of 47–52 cm/min. The flocculants used were:

Sample 1	A2120, A2130
Sample 2	A2120
Sample 3	A2120
Sample 4	A2120, A2115 and A2125

As expected, the mud volume increased as the pH of clarification increased. The results are shown in Figure 2. Juice sample 3 contained less mud solids than the other juice samples. This is reflected in the lower mud volumes shown in Figure 2. Sample 3 also had higher settling rates of 61–64 cm/min because of the reduced effects of hindered settling.

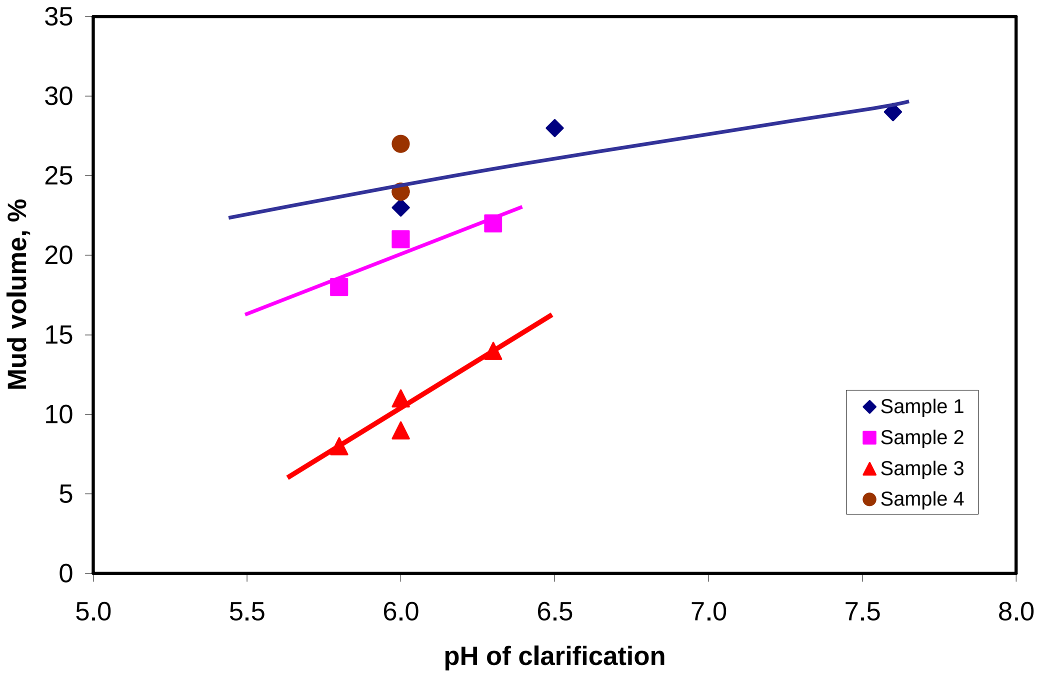

Fig. 2—Results showing the increase in mud volume as more lime saccharate is added to raise the pH of the juice.

Figure 3 shows that the turbidity decreased as the pH of clarification is increased. Under normal clarification conditions when the pH of the feed juice is 7.6 to 7.8, the turbidity would be expected to be less than 10. The results suggest that there was little or no reduction in turbidity between 6.5 and 7.6 pH. Usually there is a much larger reduction in turbidity over this pH range. These results are more a reflection of the properties of the particular juice rather than a typical response.

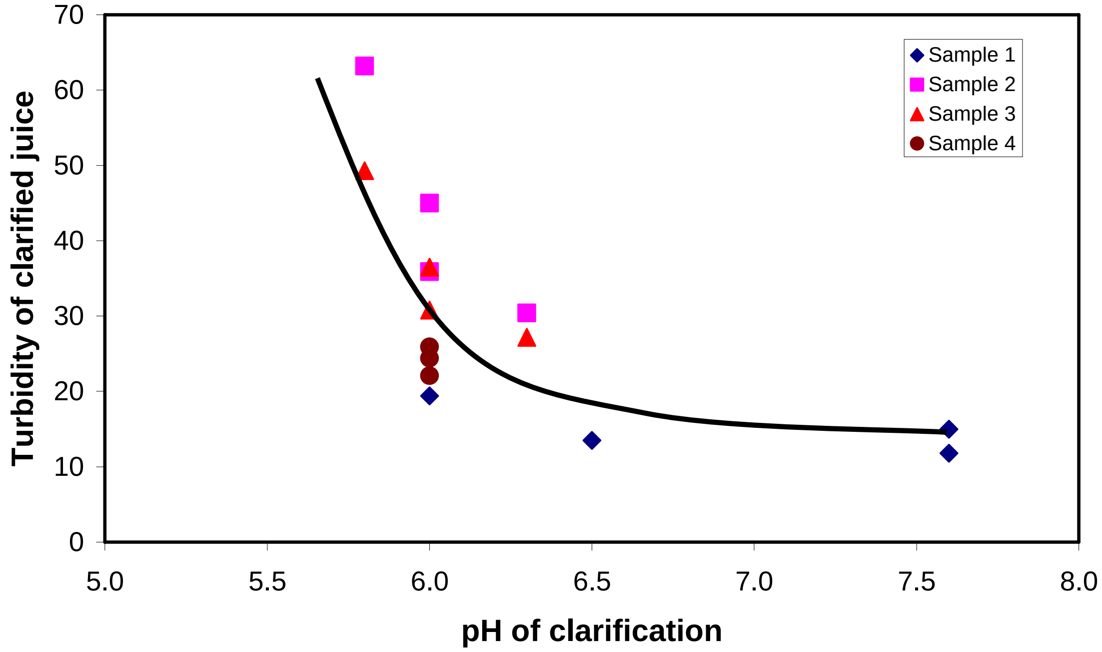

Fig. 3—Results showing the turbidity reduction as the pH of clarification increases.

Analyses of the clarified juice

Analyses of the clarified juice for calcium, phosphorus and nitrogen showed significant effects of pH as illustrated in Figures 4, 5 and 6.

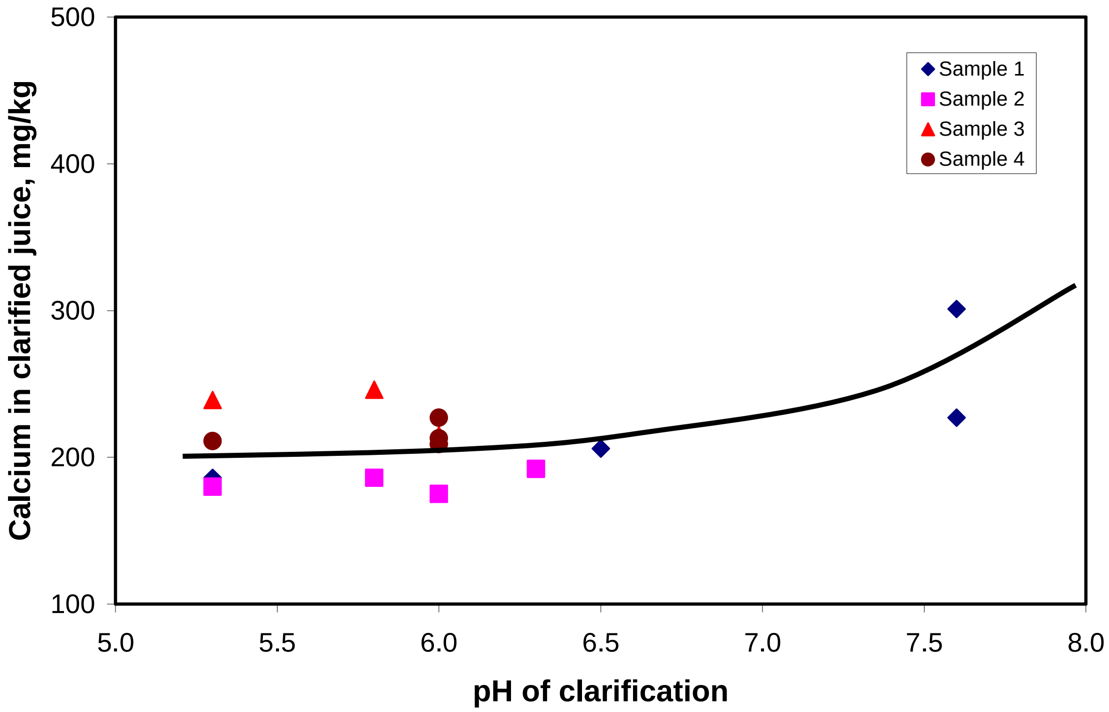

Fig. 4—Changes in the residual calcium of clarified juice due to changes in the pH of clarification.

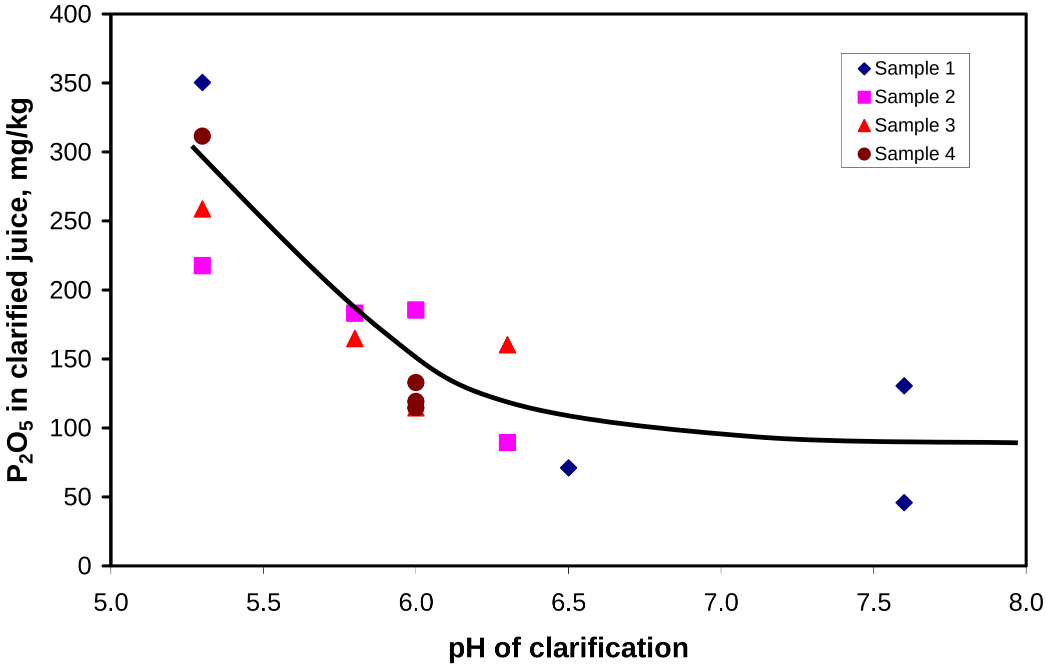

Fig. 5—Changes in the phosphorus content of clarified juice due to changes in the pH of clarification.

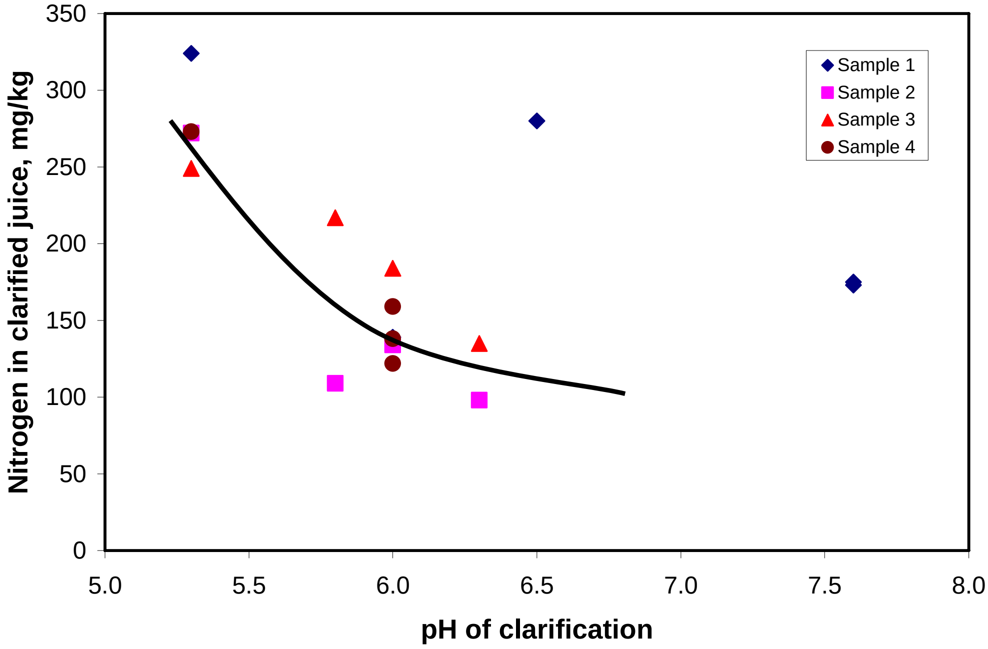

Fig. 6—Changes in the total nitrogen content of the clarified juice due to changes in the pH of clarification

The reason for the different levels of nitrogen in sample 1 is unknown. The clarified juice samples were not analysed for insoluble solids as the levels were estimated to be significantly below the 0.2% limit suggested by Fermentec.

Visually, the clarified juice samples appeared to contain no insoluble solids. This observation was supported by the relatively low turbidity values of 30 and lower for pH levels of 6.0 and above.

Another series of tests was conducted in an overseas factory using the juice from a bagasse diffuser. Characteristics of this juice include:

- Very low mud solids loadings;
- Low brix;
- Higher levels of impurities; and
- Low phosphorus content.

The implication is that clarifying this juice separately becomes very difficult.

The same test procedure was used but only settling rates, turbidity and mud volume data were collected.

Settling rates were difficult to measure because the mud flocs were relatively light and tended to be affected by any flow currents inside the cylinder. The low number of flocs made it difficult to identify any interface between the juice and the settling mud flocs. The best estimate of settling rate was that it varied between about 15 and 40 cm/min. The settled mud was of lower density than that normally expected from juice flowing from a milling tandem or even from a cane diffuser. The results are shown in Figures 7 and 8.

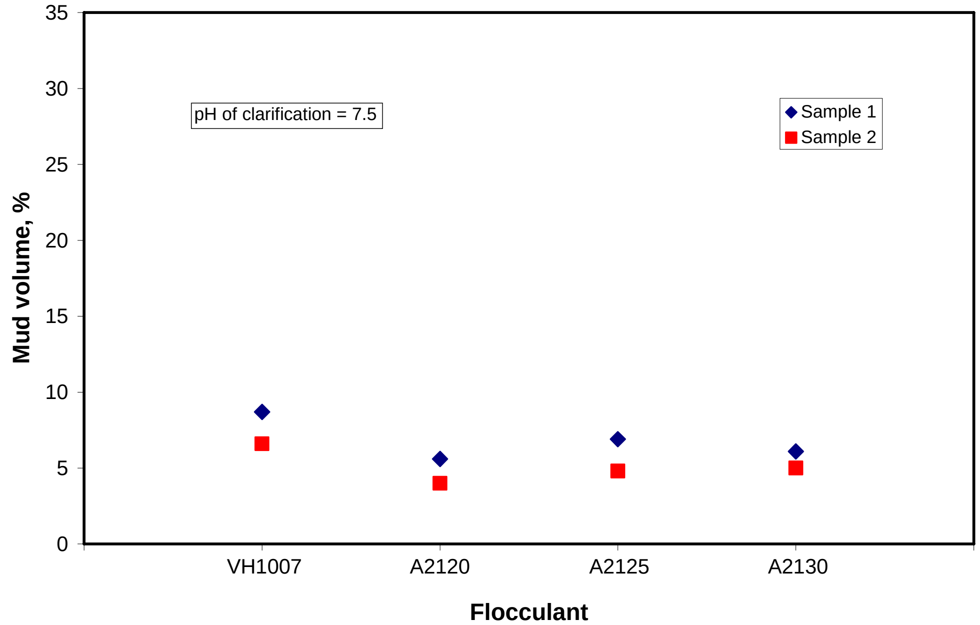

Fig. 7—Mud volumes from bagasse diffuser juice for different flocculant types.

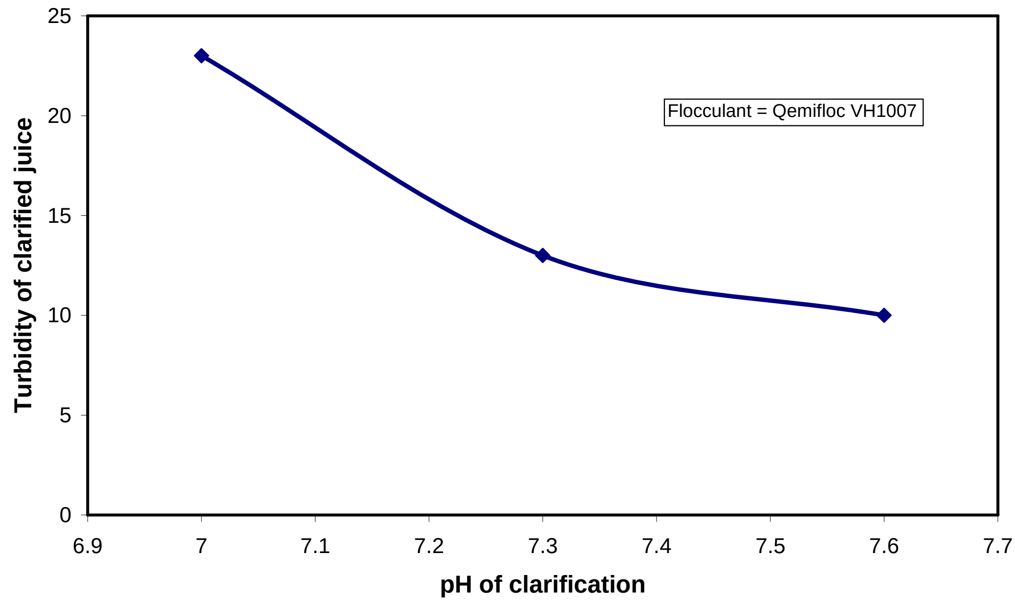

Fig. 8—Turbidity data from the bagasse diffuser experiments.

For the samples tested, increasing the pH just between 7.0 and 7.6 made a large difference in the turbidity of the clarified juice. The criteria for clarified juice at this distillery were for a pH of not more than 6.8 and a maximum turbidity of 15. The results suggest that it would be difficult to achieve compliance with the criteria.

Discussion

It is clear from the results obtained that the pH of clarification should be maintained at values below 6.2 if the criteria set by Fermentec are to be maintained. This is primarily to satisfy the minimum phosphorus criterion as compliance with other criteria is maintained at pH levels up to about 6.5.

An advantage of maintaining a pH below 6.5 is that alkaline degradation of reducing sugars to organic acids is avoided. According to Fernando Perez (*pers comm., 2008*) concentrations of organic acids above about 5000 ppm are detrimental to the fermentation process.

Previous experiments by SRI determined that the residual calcium levels in clarified juice started to rise once the pH was increased above about 6.5. Those experiments also determined that other inorganic ions such as manganese, magnesium and zinc were not affected by the normal defecation process used for the clarification of cane juice. The present data support that conclusion with respect to the residual calcium level. The clarified juice samples collected during the investigation reported here were not analysed for magnesium, manganese or zinc. At pH levels below 6.5, all the calcium added as calcium saccharate reacted with phosphorus in the juice to form calcium phosphate precipitate. There was no increase in residual calcium above the base level provided the pH of clarification remained below about 6.5.

Precipitation of calcium phosphate by the addition of lime to react with the phosphorus that occurs naturally in the juice is an essential pre-requisite to the standard clarification process. The calcium phosphate forms a bridge between the anionic flocculant and the insoluble solids. The large flocs formed by this linkage ensure high settling rates and provide an effective mechanism for the removal of other solids from the juice. Without the calcium phosphate precipitate, more expensive processing options would be required to remove the insoluble solids from the juice.

The option of avoiding a clarification step for diffuser juice takes advantage of the low initial solids level in the juice, typically 0.1 to 0.25%. Potentially most of the remaining solids could be removed by self cleaning rotary filters with small aperture (<100 µm) screens. Heating and evaporation steps are still required and higher scaling rates in the evaporator and distillation columns are expected.

When these results are applied to a decision on the type of clarifier and the residence time built into that clarifier, the obvious conclusion is that the installation of a clarifier with a long residence time offers no advantage. This conclusion is based on the following:

- The settling rates are high, even at the low pH levels.
- There are no chemical reactions (except sucrose inversion) that occur in the clarifier that could benefit the performance of the clarifier or the subsequent fermentation process.

The SRI NG clarifier with its short residence time can be applied to the clarification of juice for fermentation. The latest design has been developed through the application of computational fluid dynamics to examine the trajectories of floc particles under a range of operating scenarios to provide optimum conditions that will ensure the effective separation of the insoluble solids from the juice. Other clarifier designs can also be used for this application provided they are operated efficiently.

The experimental data are applicable to the juices being processed at the various factories during the period when the samples were collected. The results may not be fully applicable to other factories although the trends should be similar irrespective of the source of the cane supply. The settling rates of other juices will vary depending on a number of properties including mud solids content, phosphorus concentration and the flocculant being added.

It is most likely that some juices will have lower settling rates, particularly when operating at the low pH levels or when the mud solids content is higher. Low settling rates can also result when the juice pre-treatment conditions deviate from set points. In these cases the risk of carryover of small mud particles will increase.

The settling rate of the mud is the most important parameter to be used when selecting the appropriate size of clarifier for a nominated juice flow rate. Realistic settling rate data must be used when selecting the diameter of the clarifier so that clarification standards are maintained during periods when low settling rates exist. Thus it is important for the design team to work with the client to determine the most appropriate size of clarifier to suit their particular application.

Maintaining a balance between the charge density on the mud particles and the flocculant is an important consideration irrespective of the destination for the clarified juice. When operating at low pH levels, it becomes more difficult to achieve a consistently high clarification standard because the charge density on the mud particles can be much lower.

Conclusions

Experiments conducted by SRI have shown that the optimum pH for the clarification of juice going to fermentation is below 6.2 if the criteria nominated by fermentation technology providers are to be maintained.

The results indicate that the short residence time clarifiers such as the SRI NG clarifier are also suitable for this application irrespective of the specification applied to the clarified juice. Where clarified juice of low turbidity is required, it is important to get the chemistry of the clarification process correct. If tight control of the chemical conditions is not maintained, it will be difficult to comply with the nominated standards.

Depending on the specification of the clarified juice, treatment of diffuser juice may not require a clarification step. This presents an opportunity to investigate other processes that may satisfy the requirements for fermentation feedstock.

CLARIFICATION DU JUS DE CANNE POUR LA FERMENTATION

Par

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MOTS CLEFS: Jus, Clarification, Fermentation, Turbidité, Matières Insolubles.

Résumé

LES MATIERES premières pour la fermentation dans l'industrie du sucre sont les jus de canne à sucre, la mélasse B ou la mélasse finale. Le Brésil produit de l'éthanol directement a partir du jus de canne ou en utilisant des jus de qualités inférieures comme diluants avec la mélasse B pour préparer le bouillon de fermentation. Les conditions les plus appropriées pour la clarification du jus destine a la fermentation sont mal définies, excepte au Brésil. Indépendamment de la source du jus, par exemple le débit total du tandem ou de la station de diffusion ou une partie du flux total, il faut enlever les solides insolubles. Toutefois, le processus de défécation standard utilisé par les sucreries dans le monde entier afin de clarifier les jus peut introduire des ions calcium indésirables et supprimer les autres éléments nutritifs tels que le phosphore et azote qui sont considérés comme essentiels pour le processus de fermentation. Une étude a été entreprise par le SRI pour évaluer les effets sur les constituants du jus de canne lorsqu'il est soumis au processus de clarification typique dans une usine australienne; on a aussi étudie les conditions nécessaires pour fournir un jus clarifié adapté à la fermentation. Des jus typiques provenant d'une usine ont été clarifiés dans le laboratoire sous un éventail de conditions de pH et le jus clarifié résultant analysé. Les résultats ont révélé que le pH avait un effet majeur sur les concentrations des constituants principaux résiduels dans le jus clarifié et que les conditions de clarification sélectionnés sont déterminées par les critères de qualité nécessaires pour les jus destines a la fermentation. Des essais complémentaires ont été effectués dans des usines à l'étranger pour confirmer les résultats obtenus en Australie. On a trouve que les spécifications préférées pour les jus clarifiés destines a la fermentation varient d'un pays à l'autre. Chaque fournisseur de technologie de fermentation se sert de critères pour le jus clarifie qui ont un impact majeur sur le processus de clarification.

CLARIFIACION DE JUGO DE CAÑA PARA FERMENTACION

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PALABRAS CLAVES: Jugo, Clarificación, Fermentación, Turbiedad, Sólidos Insolubles.

Resumen

LA MATERIA prima para la fermentación en la industria azucarera está basada en jugo de caña, mieles B o miel final. Brasil ha estado produciendo etanol fermentando jugo caña o usando jugo de calidad para diluir la miel B y preparar el caldo de fermentación. Un tema que ha recibido poca atención particularmente en países diferentes a Brasil ha sido el de encontrar las condiciones más apropiadas para la clarificación del jugo que posteriormente se va a fermentar. Independiente si el flujo total del jugo es suministrado por un tándem de molinos ó por una estación de difusión, es esencial la remoción de los sólidos insolubles. Sin embargo, el proceso estándar de defecación usado por las industrias azucareras alrededor del mundo para clarificar el jugo puede introducir iones de calcio indeseados y remover otros nutrientes tales como fósforo y nitrógeno que son considerados esenciales en el proceso de fermentación. El SRI realizó una investigación para valorar los efectos en la composición del jugo de caña cuando es sometido al típico proceso de clarificación en un ingenio australiano y las condiciones que podrían ser requeridas para suministrar un jugo apto para la fermentación. Jugos de caña de un ingenio fueron clarificados en ensayos de laboratorio bajo un rango de pH. Los jugos clarificados fueron analizados. Los resultados indicaron que el pH tiene un efecto importante en las concentraciones residuales de los constituyentes claves del jugo clarificado y que las condiciones bajo las cuales se realice la clarificación debe se seleccionadas de acuerdo con los criterios de calidad requeridos para fermentar el jugo clarificado. Se pudo establecer que aparentemente las condiciones deseadas para un jugo clarificado que se va a fermentar varían de un país a otro. Las empresas que suministran las tecnologías de fermentación han establecido criterios para el jugo clarificado que podrían tener un mayor impacto en el nivel de clarificación requerido para lograr el cumplimento de estos criterios.