

## MINIMISING OF DECOLOURISATION COST FOR INVERT CANE SYRUP PRODUCTION USING LOW COLOUR SUGARCANE VARIETIES

By

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### Abstract

PRODUCTION of invert cane syrup is an alternative diversification product from sugarcane. The main problem with this product is the high cost of the associated decolourisation process for producing syrup with attractive colour for the consumer. This research project was conducted to study the effect of raw cane juice colour from different cane varieties on the colour of clear juice after purification and decolourisation steps as part of the process for the production of invert cane syrup. Purification of the raw cane juice was conducted using carbonatation-phosphatation followed by decolourisation of the clear juice using powder activated carbon (PAC). Sugarcane varieties were grouped into high, medium and low colour varieties having raw cane juice colour >20 000 IU, 10 000–20 000 IU and <10 000 IU, respectively. The correlation between raw cane juice colour and clear juice colour after the purification and decolourisation processes was very high. The colour of clear juice after purification followed by decolourisation using PAC at 1.6% on brix using high, medium and low colour cane varieties were 4500 IU, 2100 IU and 680 IU, respectively. In order to get the same colour as clear juice from low colour cane varieties, additional decolourisation processing was needed for clear juice from the high colour cane varieties. This investigation was able to show that using selective low colour sugarcane varieties in the production of invert cane syrup minimised the cost of decolourisation.

### Introduction

Invert syrups are widely utilised by different segments of the food industry such as soft drink and baking industries due to their sweetness and specific physical-chemical properties. Recently, several efforts were conducted to develop a continuous process for making invert syrups from raw cane juice without crystallisation that was economical on a large scale (Serna-Saldivar and Rito-Palomares, 2005; Ginslov and Peter, 2000). Invert cane syrup produced using processes developed by Indonesian Sugar Research Institute (ISRI) still contains minerals and other non sugars such as amino acids and vitamins originating from the cane juice. The challenge of technologies for invert cane syrup production concerns juice purification and decolourisation processes to produce attractive colour in the clear cane syrup.

Several studies have been published on the changes of raw juice colour to the colour of clear juice after purification processes (Sharma *et al.*, 1980; Sharma and Johary, 1984) and the changes of colour over each step of plantation white sugar production (Keskar and Nimbalkar, 1999). Johary and Singh (2001) reported on the effects of sugarcane varieties on juice colour. Of the varieties investigated, sugarcane variety CoS 91269 produced juice with the highest colour (12 985 units) compared with 5730 units for CoS 88230 variety, which produced the lowest coloured juice. Results of the study showed that, when the percentage of CoS 91269 variety in the total crop reduced from 47.55% to 37.33%, the colour of the sugar also decreased from 206.5 IU to 126.5 IU. These results show that the colour of raw juice depends on the variety and has a direct effect on the colour of sugar products.

This research project examined the possibility of minimising decolourisation costs of invert cane syrup production by using low colour cane varieties. The project investigated the effect of raw juice colour from different cane varieties on the colour of clear juice after purification and decolourisation steps as part of the process for the production of invert cane syrup.

## Materials and methods

Laboratory experiments were conducted on the juice from various sugarcane varieties. The juices were purified with three different methods. Clear juices from the best purification method were decolorised using powder activated carbon (PAC). A pilot scale experiment was conducted only on the juice from the lowest colour cane variety using the best purification technique, followed by decolourisation using the optimum dose of PAC. The purpose of the pilot scale experiment was to check the consistency of the best result from the laboratory experiment using the larger scale process.

### Laboratory experiments

Eight sugarcane varieties with various levels of juice colour were used in the laboratory stage experiments. The cane samples used in this experiment consisted of clean stalk without tops and trash. The cane was pressed twice using a small mill and the raw juice was collected for purification. Cane juice purification was conducted using three different methods namely simple phosphatation process (P); double carbonatation process (DC); and purification using a modified carbonatation-phosphatation process (DCP).

For the simple phosphatation process, raw cane juice was heated to 60°C and 400 ppm P<sub>2</sub>O<sub>5</sub> (phosphoric acid) was added. Milk of lime at 5°Baumé was then added to raise the pH to 7 before the juice was heated to 100°C and then settled without flocculant addition. For the double carbonatation process, pH and temperature of the first carbonatation process were 10.5 and 55°C respectively. Milk of lime (8°Baumé) was used for the carbonatation process. The amount of lime milk added on raw juice was 10%(v/v). Raw juice was heated to 55°C and milk of lime was added until pH 10.5 was reached. The rest of the milk of lime was added to the juice during the addition of carbon dioxide to maintain the pH at 10.5. The temperature was kept constant at 55°C. After the reaction, the juice was filtered. Carbon dioxide was added to the filtered first carbonated juice to decrease the pH to 7.0, before the juice was further heated to 75°C and filtered. The purification process using the modified carbonatation-phosphatation process basically followed the double carbonatation process with phosphoric acid (27% v/v) added to the filtered first carbonated juice instead of carbon dioxide to reduce the pH to 7.0, before the juice was heated to 70°C and filtered. All filtration was conducted using filter paper (Whatman No.1). On the DCP process, kieselguhr was used as a precoat on the filter paper during the second filtration after the addition of phosphoric acid.

The clear juice from the purification processes were further decolourised using powder activated carbon (PAC) (analytical grade from Merck with an iodine adsorption value of 1220 mg/g) dosed at various concentrations. The mixture of clear juice and PAC was maintained at 70°C for one minute before it was filtered using Whatman No. 42 paper coated with kieselguhr.

Raw and clear juice samples were analysed for colour, brix (using refractometer), sucrose (measured by double polarisation), reducing sugar (Lane & Eynon method), CaO and MgO content (complexo-metric method), ash (conductivity) and turbidity.

Colour analysis of raw juice, clarified juice, and low colour juice after decolourisation using PAC was done by diluting the samples with distilled water to about 5, 10 or 15 brix or without dilution followed by method of ICUMSA GS1/3-7. The degree of sample dilution depended on the colour of the sample. The absorbance of the sample at 420 nm should be between 0.2 and 0.8. In the case of very low colour juice after decolourisation using PAC, there was no dilution needed for the sample. The turbidity on each juice was measured with a spectrophotometer at 900 nm using 50 mg

SiO<sub>2</sub> per litre of solution as a standard (Ananta and Martoyo, 1989). Raw juice was also analysed for phenolic content using the method described by Clarke *et al.* (1986) and the correlation between phenolic content and colour in raw juice was determined. The concentrations of CaO and MgO and phenolic content were calculated at 15 brix.

### Pilot scale experiments

In the pilot plant experiments, cane juice from the lowest colour cane variety was purified using the combined carbonatation-phosphatation process (DCP) detailed above in a batch system. For one trial, 200 L of cane juice from around 400 kg of sugarcane (PS 862) was processed over four batches. Clear juice was decolorised using PAC at a dosage of 1.6% PAC on clear juice brix. The PAC used in the pilot scale experiments was industrial grade with an iodine adsorption value of 1100 mg/g. The clear juice after the PAC treatment was adjusted to pH 5 and evaporated at 70°C to syrup for analysis.

## Results and discussion

### Laboratory experiments

Table 1 shows the cane varieties used in this research project and grouped into high, medium and low colour. The varieties with the highest and the lowest colour were also included in Table 1 separately for comparison. The cane was harvested almost at the same maturation i.e. more than 20 brix with an average reducing sugar content of 3.56%.

Seven cane varieties used in this experiment were collected from the same area (Pasuruan Experiment Station). These varieties were grown in similar soils, under similar climatic conditions, and were also cultivated with the same technique. Hence, the various colours in the raw juice reflect the inherent properties of the different varieties. One variety (PS 862) was collected from the Jengkol area. The Jengkol area had land with more porous and better drainage characteristics for good growth of the PS 862 cane and differed from the land characteristics in the Pasuruan area (compact soils and poor drainage).

**Table 1**—Sugarcane varieties used in experiments and their analyses.

Group	Sugarcane varieties	Brix	Sucrose purity (%)	Reducing sugar (% on Brix)	Colour of raw cane juice (IU)
Highest colour	PS 921	21.9	91.12	1.28	42 384
High colour	PS 891, PS 851, PS 921, PSJT 9344	20.72 ± 1.2	87.42 ± 4.11	3.73 ± 2.45	31 380 ± 5500
Medium colour	PS 864, PS 97226	21.14 ± 0.7	86.74 ± 1.4	3.47 ± 0.45	16 756 ± 2105
Low colour	PS 951, PS 862	21.50 ± 0.8	89.81 ± 0.18	3.48 ± 0.28	8357 ± 1153
Lowest colour	PS 862	21.54	89.63	3.76	7289

Raw cane juice from each cane variety was purified using the three different processes (P), (DC) and (DCP). These three purification techniques were chosen with consideration that the clear juice resulting from these processes will be safe for direct consumption. Other purification processes such as sulfitation were not selected because the residual SO<sub>2</sub> in the clear juice was considered harmful to human health.

Table 2 shows the performance of the three clarification processes averaged for all cane varieties. The DCP purification process produced the best results in terms of high purities, low residual calcium and magnesium ions (CaO+MgO) and low turbidity, followed by the DC and P processes. The improvement in sucrose purity for purification using DCP, DC and P were 3.21, 2.98 and 1.05 units respectively and the reduction of CaO+MgO content were 27.6, 22.2 and 39.9% for

the DCP, DC and P processes respectively. Clear juice with low turbidity values resulted from the DC and DCP purification processes.

**Table 2**—Effect of purification process using phosphatation (P), double carbonatation (DC) and combination of double carbonatation+phosphatation process (DCP) on sucrose purity, CaO+MgO and turbidity.

Purification process	Sucrose purity (%)	CaO+MgO (ppm in juice at 15 brix)	Turbidity (ppm SiO <sub>2</sub> in juice at 15 brix)
Before purification	87.85 ± 3.67	742 ± 124	1156 ± 298
Phosphatation	88.90 ± 3.75	537 ± 176	16 ± 10
Double carbonatation	90.83 ± 2.90	577 ± 120	10 ± 7
Double carbonatation+phosphatation	91.06 ± 2.16	446 ± 144	7 ± 10

The changes in colour during each purification process are shown in detail for each group of cane varieties in Figure 1. The results show that the average decrease in colour of the juice from all groups of sugarcane varieties after purification using P, DC and DCP were 14.4, 56.6 and 71.7% respectively. The best colour removal resulted from purification using DCP. A review by Sharma and Johary (1984) indicated that removal of colorants during purification of cane juice took place either by way of precipitation / coagulation or through adsorption on the surface of various precipitates produced during the clarification process. Previous experiments conducted by Sharma *et al.* (1980) and Sharma and Johary (1984) support the results of this research in that the mud produced during the carbonatation process could adsorb larger magnitudes of colorants as compared to sulfitation and defecation processes. The juice purification process trialled in this research based on simple phosphatation (P) was basically similar to the defecation process with the addition of phosphoric acid (400 ppm P<sub>2</sub>O<sub>5</sub>).

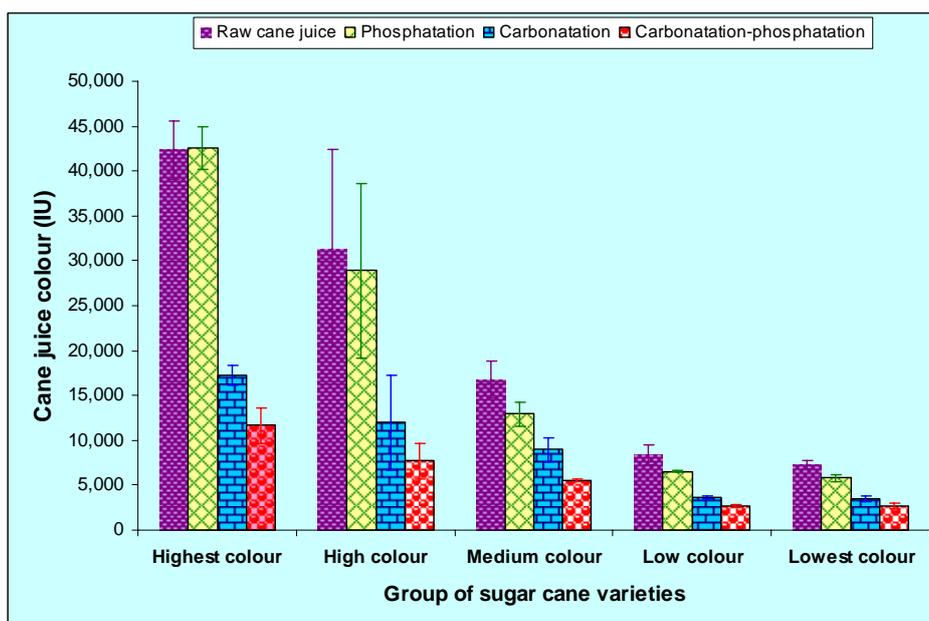


Fig. 1—Changes in juice colour after purification using phosphatation (P), double carbonatation (DC) and a combination of double carbonatation+phosphatation process (DCP) for each group of cane varieties.

The laboratory trials clearly showed that the clear juice colour was significantly influenced by the colour of the raw juice. The trials showed that higher raw juice colour resulted in the highest clear juice colour after the purification processes. The clear juice colour from simple purification using P process of the low colour group of cane varieties was similar to the clear juice colour resulting from the high colour group of cane varieties using more complex purification techniques like DCP.

One of the major colorant groups in the cane plant is the phenolic compounds (Clarke *et al.*, 1986). Figure 2 shows the correlation between the colour of raw juice and the phenolic content calculated for each juice at 15 brix. Higher raw juice colours correlated with a higher content of phenolic compounds. The correlation  $R^2$  value was 0.77.

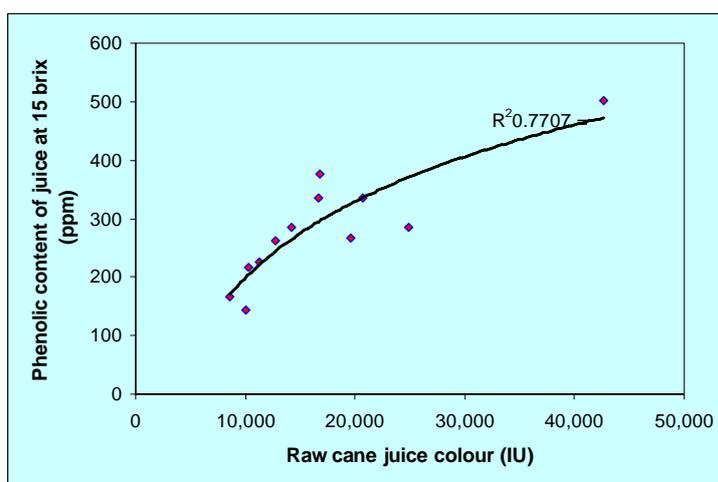


Fig. 2—Correlation between colour of raw cane juice and the phenolic content.

The clear juice resulting from the DCP purification process was further decolorised using various concentrations of PAC including 0; 0.1; 0.2; 0.8; 1.6; 3.2 and 6.4% on clear juice brix (see Figure 3). The results showed that the colour of clear juice after decolourisation using PAC was influenced by the colour of the clear juice before PAC treatment. The colour of the clear juice after decolourisation using PAC was influenced by the colour of raw juice before the purification process. The magnitude of the colour reduction declined as the concentration of PAC was increased. The lowest colour juice (from cane variety PS 862) reached a minimum value when the PAC reached 1.6% on juice brix. These results showed that not all of the colour could be removed using PAC.

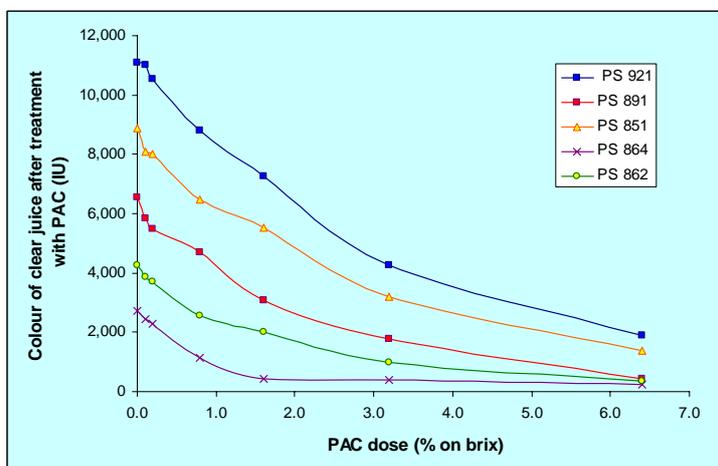


Fig. 3—Clear juice colour from purification using the double carbonatation+ phosphatation process followed by decolourisation using various concentrations of PAC.

Figure 4 shows the colour of clear juice from each group of cane varieties after purification using the DCP process followed by decolourisation using PAC at a dosage of 1.6% on clear juice brix. The colour of clear juice after purification by the DCP process followed by decolourisation using PAC at 1.6% on brix for the high, medium and low colour varieties were 4500 IU, 2100 IU and 680 IU, respectively. In order to get the same colour as clear juice from low colour varieties, additional decolourisation (or higher dosage of PAC as shown in Figure 3) would be required for the clear juice from high colour varieties. This result showed that the cost of decolourisation on invert cane syrup production was able to be minimised using low colour sugarcane varieties.

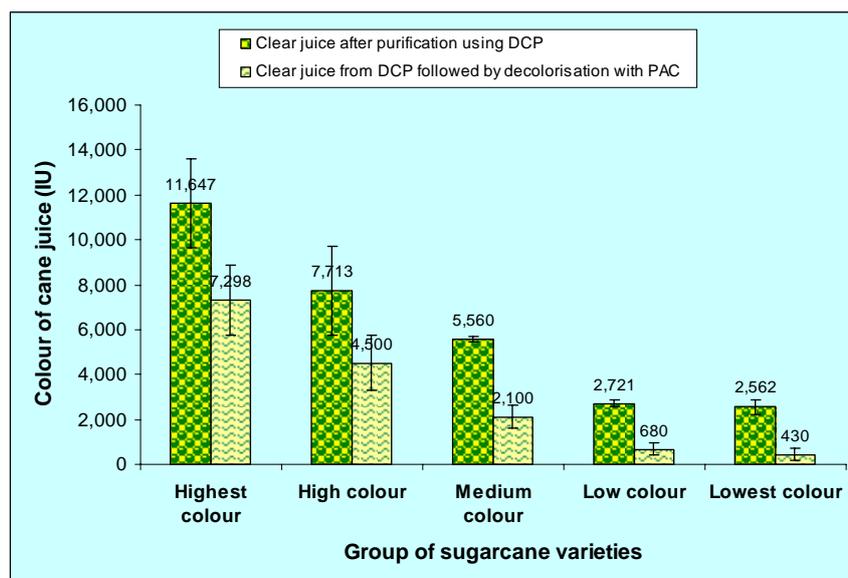


Fig. 4—Average colour of clear juice from each group of cane varieties after purification using the DCP process followed by decolourisation using PAC at 1.6% on clear juice brix.

Purification of the lowest colour cane varieties with the DCP process followed by decolourisation using PAC at 1.6% on clear juice brix was further tested in pilot scale trials as part of the process for invert cane syrup production.

#### Pilot scale experiments

The analysis of cane juice over each step of the DCP clarification process, decolourisation and evaporation to produce cane syrup is shown in Table 3. The data are the average of eight trials. Around 400 kg of cane was processed for each trial. The results showed that the average brix of raw juice was 21.1, purity 87.7%, reducing sugar 4.95%, CaO+MgO 372 ppm and colour 9865 IU. The quality of raw juice used in the pilot scale experiments was similar to that of the laboratory experiments. The purification process resulted in the juice purity increasing by 1.36 units, colour decreasing by 70.7%, while the reducing sugar and ash conductivity (% on brix) were almost constant.

After the purification process the clear juice was decolourised using PAC at 1.6% on clear juice brix. The decolourisation step decreased the clear juice colour by 74.6% (average 738 IU). The clear juice was then evaporated. There was a slight increase in colour after evaporation from 737 IU to 991 IU and the thick juice was still very clear with just a slight increase in turbidity.

The low colour thick juice will be used to produce invert cane syrup through sucrose inversion with a final decolourisation step. Sucrose inversion using immobilised invertase is being developed by ISRI. The final decolourisation step on invert cane syrup production uses weak anionic or strong anionic resin regenerated using disodium hydrogen phosphate (Triantarti and Toharisman, 2008).

**Table 3**—Results of cane juice analyses following clarification using the DCP process, decolourisation using PAC and evaporation.

Parameter	Result of analysis			
	Raw cane juice	Clear juice after purification with DCP	Clear juice after purification with DCP and Decolourisation using PAC	Thick juice
Brix	21.09 ± 0.67	14.98 ± 1.35	14.03 ± 1.09	74.01 ± 2.23
Sucrose (%)	18.50 ± 0.84	13.33 ± 1.14	12.55 ± 0.87	66.44 ± 2.64
Purity (%)	87.66 ± 2.12	89.02 ± 1.82	89.47 ± 1.95	89.75 ± 1.09
Reducing sugar (%)	4.95 ± 1.24	4.36 ± 0.93	4.55 ± 0.89	4.19 ± 0.99
Colour (IU)	9865 ± 2,109	2890 ± 585	738 ± 250	991 ± 539
CaO+MgO in juice at 15 brix (ppm)	372 ± 144	368 ± 140	369 ± 140	202 + 70
Ash conductivity (% on brix)	1.24 ± 0.13	1.31 ± 0.31	1.39 ± 0.33	1.02 ± 0.11
Turbidity at 15 brix (ppm SiO <sub>2</sub> )	547 ± 36	18 ± 4	4 ± 2	9 ± 6 *

Note: Turbidity was calculated for juice at 60 brix

The result of the pilot scale experiment using the lowest colour cane variety (PS 862) showed a consistent result on low colour of clear juice compared with the laboratory result using the same cane variety, purification process and decolourisation process. The results of both laboratory and pilot scale experiments showed that minimising decolourisation costs of invert cane syrup production by using low colour cane varieties was quite promising.

However, the colour of raw juice not only depends on the cane variety but also on many factors such as agro-climatic conditions prevailing during the growth period, land characteristics and cultivation techniques. Before commercial scale production of invert cane syrup can commence, several trials have to be done by planting low colour cane varieties in different areas that will be cultivated and processed to determine the quality of raw juice produced (especially colour). The colour of raw juice is also influenced by the sugarcane maturation during harvesting, time between harvesting and milling and the cleanliness of the cane based on included dirt, tops and trash (Sens and Decagny, 2001). Good management of cane cultivation, harvesting and post harvest practices are very important to get a consistent, good quality sugarcane that produces low colour raw juice.

### Conclusions

Cane juice purification using a combination of double carbonatation and phosphatation processes provided the best quality juice (low colour, high purity) that can be used for invert cane syrup production. The colour of clear juice after purification and decolourisation using PAC is influenced by the colour of the raw juice. In the pilot scale experiments, the colour of thick juice produced using low colour cane varieties after purification using double carbonatation and phosphatation processes, followed by decolourisation using PAC at dosage 1.6% on brix of clear juice, reached 991 IU. However, further research is needed to determine cane cultivation conditions which affect the colour of cane juice especially for potential low colour cane varieties.

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## RÉDUCTION DU COÛT DE DÉCOLORATION DE SIROPS INVERTIS GRACE A DES VARIÉTÉS DE CANNE DE COULEUR FAIBLE

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**MOTS-CLEFS : Couleur, Canne a Sucre,  
Variété, Purification, Décoloration.**

### Résumé

LA PRODUCTION de sirop de canne à sucre inverti est un produit de diversification de la canne. Le principal problème avec ce produit est le coût élevé du processus de décoloration, pour la production d'un sirop avec une couleur attrayante pour le consommateur. Cette recherche a été réalisée pour étudier l'effet de la couleur du jus de canne provenant de différentes variétés sur la couleur du jus clair après les étapes de purification et de décoloration pendant la production de sirop de canne à sucre inverti. La purification du jus de canne brut a été menée à l'aide de la carbonatation-phosphatation suivie de décoloration du jus clair à l'aide de charbon actif en poudre (PAC). Les variétés ont été regroupées en couleur de jus haute, moyenne et faible, dans des fourchettes > 20 000 UI, 10 000 à 20 000 UI et 10 000 UI, respectivement. La corrélation entre la couleur de jus de canne brut et la couleur de jus clair après les processus de purification et de décoloration était très élevée. Les couleurs du jus clair après purification suivie de décoloration à l'aide du PAC (consommation 1.6% sur brix) pour les variétés de couleur haute, moyenne et basse ont été IU 4500, IU 2100 et 680 UI, respectivement. Afin d'obtenir la couleur de jus clair provenant de variétés à faible couleur, une décoloration supplémentaire était nécessaire pour les jus clair obtenus à partir des variétés de couleur haute. Ce travail a pu démontrer que l'utilisation de variétés à couleur faible pour la production de sirop de canne à sucre inverti réduit au minimum le coût de la décoloration.

## MINIMIZACIÓN DEL COSTO DE DECOLORACIÓN DE JARABE INVERTIDO USANDO VARIEDADES DE CAÑA DE BAJO COLOR

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**PALABRAS CLAVE:** Color, Caña, Variedad, Purificación, Decoloración.

### Resumen

LA PRODUCCIÓN de jarabe invertido de caña es una alternativa de diversificación de productos. El problema principal es el alto costo de los procesos de decoloración para obtener un producto atractivo al consumidor. Esta investigación fue orientada al estudio del efecto del color del jugo de diferentes variedades de caña en el color del jugo clarificado después de las etapas de purificación y decoloración como parte del proceso de obtención de jarabe invertido. La purificación se efectuó usando carbonatación- fosfatación seguida de decoloración del jugo claro usando carbón activado en polvo (PAC). Las variedades de caña se agruparon en bajo, medio y alto color con colores en jugo crudo de >20000 IU, 10000–20000 IU and <10000 IU, respectivamente. La correlación entre color de jugo crudo y color de jugo claro, después de los procesos de purificación y decoloración fue alta. El color del jugo claro después de purificación seguida de decoloración usando PAC a 1.6% en brix y usando variedades de alto medio y bajo color fue de 4500 IU, 2100 IU y 680 IU, respectivamente. Para obtener el mismo color del jugo claro obtenido a partir de variedades de bajo color, se requirió procesamiento adicional de decoloración con las variedades de alto color. La investigación mostró que el uso de variedades de bajo color en la producción de jarabe invertido, minimiza los costos de decoloración.