Decolorization of filtered liquor using bagasse fly ash at the pilot scale

C Ngasan¹, P Thiravetyan², P Suksabye³, P Leechart², A Simaratanamongkol² and U Pliansinchan¹

¹Mitr Phol Sugarcane Research Center Co. Ltd., Ltd. 399 Moo 1 Chumpae-Phukieo Road, Khoks-ata, Phukieo, Chaiyaphum 36110, Thailand; Chaiwtn@mitrphol.com
²School of Bioresources and Technology, King Mongkut’s University of Technology Thonburi, Bangkok, 10150 Thailand

Abstract In Thailand, decolorization in the sugar refinery process involves three steps: carbonation, filtration and ion-exchange resin. The main colorants in filtered liquor are removed by the resin. The regeneration of the resin with either NaCl or NaOH has problems in the recovery of Na⁺ and Cl⁻ ions. Activated carbon is an alternative decolorant used in many refineries around the world. This study examined at the laboratory- and pilot-scale level the usefulness of bagasse fly ash (BFA) for its decolorization ability on filtered liquor. BFA contains carbon and has a total surface area of 337 m²/g, an average pore diameter of 2.794 nm, and a specific volume of 0.2355 cm³/g. The ratio of medium pore-size volume to total volume is 65%, which makes BFA suitable for the removal of large molecules in solution, including color in liquor. The volume ratio of BFA to filtrate liquor used in the study was 1:14.4. The results showed that the efficiencies of BFA in color, turbidity and starch reduction in syrup were 72-94%, 97% and 86-91%, respectively. However, the removal efficiency for dextran was lower at 9-44%. The use of BFA did not change the concentration of sucrose, glucose, fructose, pol or Brix. However, BFA did increase the conductivity of the treated samples. During testing, the liquor samples were found to contain higher levels of calcium and magnesium when compared to the original syrup. This may need to be studied further to investigate the effects of these metals; whether or not they are acceptable, and if they exceed specific food product standards.

Key words Bagasse fly ash, decolorization, filtered liquor

INTRODUCTION

Bagasse fly ash (BFA) is a byproduct from the bagasse biomass power plant that uses bagasse as fuel to produce electricity. After the process of producing electricity there is a large quantity of BFA that requires disposal. During the 2014-2015 season 105.95 Mt of sugarcane was milled in Thailand. By using the method of Cordeiro et al. (2004) the calculated amount of BFA generated per year is approximately 661,587 t. Bagasse ash has potential as a soil additive, but the application rate of bagasse ash used for this is very low, so large areas of land are required to dispose of the remaining ash, dust and trash.

The properties of carbon has been studied in countries such as India, South Africa and Thailand. For example, a study in India by Gupta and Ali (2004) reported the properties of the BFA with a density of 1,010 kg/m³, porosity of up to 0.36% and with a surface area of 450 m²/g (particle size 200-250 μm). In Thailand. They used bagasse fly ash from a local sugar mill as an adsorbent for decolorization of sugar syrup. Approximate analyses of BFA were 16% volatile matter, 65% fixed carbon and 18% ash. Total surface area and average pore diameter of BFA were 256 m²/g and 3.243 nm, respectively (Thongpradistha 2007). BFA has also been used to eliminate toxins in the water. For sample, Srivastava et al. (2005) used BFA to eliminate phenol, Gupta et al. (2003) used BFA to eliminate cadmium and nickel, Gupta et al. (2002) used BFA to eliminate lindane and malathion, and Gupta and Ali (2004) used BFA to eliminate lead and chromium.

Mitr Phol staff believed that single-use BFA could be an effective color sorbent in cane syrup during the decolorization process. A laboratory-scale study showed that BFA had a decolorization efficiency in sugar syrup of 73%, similar to resin with a decolorization efficiency of 78% (Thongpradistha 2007). It is possible that BFA could be used to replace the current procedure that reduces syrup color using ion-exchange resin, and as a result, reduce the overall cost of production.
MATERIALS AND METHODS

Bagasse fly ash preparation

The BFA was collected from the waste pond of the Mitr Phol Bio Power Plant in Dan Chang district, Supanburi Province, washed to remove sand and dirt, and filtered through a 20 mesh screen (nominal sieve opening 841 μm). The particles were treated with water in a 1:10 weight ratio and kept for 1 hour. The bagasse fly ash was washed with distilled water until a pH of 7.5 was achieved. It was then dried in sunlight and stored in sealed plastic bags.

Physical and chemical characteristics of BFA

Determination of surface area and the pore diameter

The particle size distribution, average size, surface area and the pore diameter of BFA were measured by nitrogen adsorption isotherm using BEL-sorp instrument and by the Brunauer-Emmett-Teller (BET) method, using Belsorp Adsorption/desorption analysis software.

Inductive Coupled Plasma Spectroscopy (ICP) analysis

Determinations of heavy metals in syrup before and after the adsorption process were analyzed by ICP to determine the concentrations of iron, calcium, magnesium, silicon and aluminum.

Decolorization efficiency of BFA on filtered liquor

Laboratory-scale experiment

The decolorization efficiency on filtered liquor was studied in a glass column (diameter 5 cm and length 45 cm), containing 353 g of packed BFA and filled with about 3.5 L of filtered liquor. The liquor passed through the BFA under gravity and liquor samples were collected to analyze the quality.

Pilot-scale experiment

The equipment was set up shown in Figure 1. Sand was placed into the base of the column (diameter 20 cm, 152 cm height), then 3.46 kg of BFA was packed downwards. The 56°Bx filtered liquor was continuously pumped downwards through the packed column for about 1 hour. The pressure was maintained at 100 kPa and the temperature of the liquor was controlled at 80°C. Liquor samples were collected before and after treatment and analyzed for decolorization efficiency, Brix, pol, conductivity ash, turbidity, starch, dextran and heavy metals content.

Sample analysis

Color in liquor samples was analyzed using the ICUMSA method GS1/3-7. Turbidity was analyzed using ICUMSA Method GS 2/3-18. The conductivity ash in all the samples collected was measured using ICUMSA Method GS1/3/4/7/8-13. Starch was analyzed in accordance with the SASTA manual (Anon. 1985). Dextran concentrations in samples were determined by the haze method following the modified alcohol method by ICUMSA Method GS1-15.
**RESULTS AND DISCUSSION**

**Physical properties of bagasse fly ash**

The BFA was analyzed for total surface area, BET surface area, total pore volume, and pore size (Table 1). The size of the BFA was smaller than what Gupta and Ali (2004) reported, but larger than that used in the Thongpradistha (2007) study. The ratio of medium pore size volume to total volume shows that the holes are medium sized; therefore, it is appropriate to remove color which exists as large molecules in the liquor.

**Table 1. Physical properties of the BFA.**

<table>
<thead>
<tr>
<th>Total surface area (m²/g)</th>
<th>Total pore volume (cm³/g)</th>
<th>Mesopore volume (cm³/g)</th>
<th>Micropore volume (cm³/g)</th>
<th>Average pore diameter (nm)</th>
<th>Proportion mesopore (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>337.15</td>
<td>0.2355</td>
<td>0.15265</td>
<td>0.08285</td>
<td>2.794</td>
<td>64.82</td>
</tr>
</tbody>
</table>

**Decolorization efficiency of filtered liquor at the laboratory scale**

The laboratory tests found that the BFA particles can reduce the amounts of starch, dextran, color and turbidity (Table 2). The color reduction was much higher than the 73% achieved by Thongpradistha (2007). In addition, decolorization efficiency of ion-exchange resin was 78%. When BFA was activated, the highest decolorization efficiency was 93-96%.

**Table 2. Effects of using BFA as adsorbent in syrup filtrate liquor at the laboratory scale.**

<table>
<thead>
<tr>
<th>Stage</th>
<th>Color (IU)</th>
<th>Turbidity</th>
<th>Conductivity ash (%)</th>
<th>Starch (ppm)</th>
<th>Dextran (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filtered liquor (Before treatment)</td>
<td>668</td>
<td>177</td>
<td>0.07</td>
<td>125</td>
<td>901</td>
</tr>
<tr>
<td>Filtered liquor (After treatment)</td>
<td>33</td>
<td>106</td>
<td>0.07</td>
<td>19.26</td>
<td>530</td>
</tr>
<tr>
<td>Removal, %</td>
<td>95.1</td>
<td>40.1</td>
<td>0.0</td>
<td>84.6</td>
<td>41.2</td>
</tr>
</tbody>
</table>
Decolorization efficiency of filtered liquor at the pilot scale

When the experiment was initially conducted, there was a steady increase in the internal pressure in the PVC column. This led to leakages of the joints in the column. Clogging within the bagasse fly ash bed was leading to an increase in pressure in the column. As a result, the PVC column was replaced with a steel column.

Thongpradistha (2007) studied the effect of contact time on the decolorization efficiency of sugar syrup. He used 10% BFA, 10% resin and 10% activated carbon in a packed bed column system. The results showed that BFA had a stable decolorization efficiency of sugar syrup with a contact time of 1 hour.

At the pilot scale, BFA was very effective in reducing color, turbidity and starch; the results being 72-94%, 97% and 86-91% respectively (Table 3). For each 50 L of syrup, 3.5 kg of BFA were absorbed at a ratio BFA:filtered liquor of 14.9:1. According to this ratio, the usage of BFA is estimated to be 69.4 kg for 1 t of melt liquor in 1 hour, while 0.08-0.15 L of resin is used to decolorize 1 t of melt liquor (Rein 2007). Total useful capacity of the fly ash column will be investigated in the future.

Table 3. Effect of using BFA as the adsorbent for filtered liquor in the pilot-scale experiments.

<table>
<thead>
<tr>
<th>Stage</th>
<th>Color (IU)</th>
<th>Turbidity</th>
<th>Conductivity ash (%)</th>
<th>Starch (ppm)</th>
<th>Dextran (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filtered liquor (Before treatment)</td>
<td>344</td>
<td>528.5</td>
<td>0.1592</td>
<td>95.94</td>
<td>1297</td>
</tr>
<tr>
<td>Filtered liquor (After treatment for 1 h.)</td>
<td>95</td>
<td>24</td>
<td>0.138</td>
<td>12.69</td>
<td>1065</td>
</tr>
<tr>
<td>Removal, %</td>
<td>72.4</td>
<td>95.5</td>
<td>13.3</td>
<td>86.6%</td>
<td>17.9</td>
</tr>
</tbody>
</table>

BFA adsorption of dextran was low to moderately effective (9-44%) at the levels tested at the pilot scale and tests at the laboratory scale. Both the column and laboratory tests showed that the BFA does not absorb sucrose. The conductivity ash of the liquor after BFA treatment showed a slight decline. This may be because the bagasse ash may absorb some of ash components. Table 4 indicates that aluminum ions increase and iron ions decrease significantly during the carbonatation process, but the aluminum ions reduce significantly during the treatment with BFA. Calcium, iron and magnesium ions are transferred from the BFA to the liquor during the decolorization process.

Table 4. Analyses of liquor for metal ions before and after treatment.

<table>
<thead>
<tr>
<th>Stage</th>
<th>Si</th>
<th>Al</th>
<th>Fe</th>
<th>Ca</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>Before carbonation</td>
<td>5.66 ± 0.16</td>
<td>1.083 ± 0.033</td>
<td>1.45 ± 0.017</td>
<td>210.44 ± 0</td>
<td>72.2 ± 3.7</td>
</tr>
<tr>
<td>Filtered liquor (before)</td>
<td>7.15 ± 0.07</td>
<td>2.713 ± 0.041</td>
<td>0.183 ± 0.005</td>
<td>153.05 ± 0</td>
<td>66.2 ± 0.9</td>
</tr>
<tr>
<td>Filtered liquor after 1 h.</td>
<td>5.45 ± 0.10</td>
<td>0.610 ± 0.014</td>
<td>0.319 ± 0.006</td>
<td>210.44 ± 0</td>
<td>241.3 ± 2.1</td>
</tr>
</tbody>
</table>

This ion transfer may need to be studied further particularly to determine if the new concentrations exceed the standards specified for these food grade products.

CONCLUSIONS

Bagasse fly ash has been shown to be effective at reducing the color, turbidity and starch in filtered liquor at a good level, but there are limited reductions in ash and dextran. This suggests it is possible to develop a bagasse fly ash fixed base system as a replacement for the ion-exchange resin system.

The main issue with using fly ash to remove color instead of resin may be sorting out the size of bagasse fly ash to reduce clogging and pressure problems occurring in the system. In addition, more research needs to be conducted about the
production of BFA pellets to solve such problems. Further research is also needed to address the issue of dissolved metals in liquor.

ACKNOWLEDGEMENTS

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REFERENCES


Décoloration à l’échelle pilote de la liqueur filtrée à l’aide de cendres volantes de bagasse

Résumé. La décoloration dans le processus de raffinage du sucre en Thaïlande comporte trois étapes: la carbonatation, la filtration et les résines échangeuses d’ions. Les colorants présents dans la liqueur filtrée sont éliminés par la résine. La régénération de la résine avec le NaCl ou le NaOH présente des problèmes pour la récupération des ions Na⁺ et Cl⁻. Le charbon actif est un autre décolorant utilisé dans de nombreuses raffineries. Cette étude a examiné à l’échelle laboratoire et pilote l’efficacité des cendres volantes de bagasse (BFA) pour décolorer la liqueur filtrée. BFA contient du carbone et a une superficie totale de 337 m²/g, un diamètre moyen des pores de 2,794 nm et un volume spécifique de 0,2355 cm³/g. Le rapport moyen volume de pores / volume total est de 65 %, ce qui permet à la BFA d’enlever les grosses molécules en solution, y compris la couleur dans la liqueur. Le rapport de volume de BFA à filtrer / liqueur utilisée dans l’étude a été 1:14. 4. Les résultats montrent que les gains d’efficience de la BFA en couleur, turbidité et réduction de l’amidon du sirop étaient respectivement de 72-94%, 97% et 86 à 91%, respectivement. Cependant, l’efficacité d’élimination du dextran était inférieure à 9-44%. L’utilisation de la BFA n’a pas modifié les concentrations de saccharose, glucose, fructose, pol ou Brix. Toutefois, la BFA a augmenté la conductivité des échantillons traités. Pendant l’essai, les échantillons de liqueur avaient des concentrations plus élevées de calcium et de magnésium par rapport au sirop original. Il faut étudier davantage les effets de ces métaux pour établir s’ils sont acceptable et si les concentrations dépassent les normes spécifiques de produits alimentaires.

Mots-clés: Cendres volantes de bagasse, décoloration, liqueur filtrée

Decoloración del licor filtrado empleando cenizas de bagazo, a escala piloto

Resumen. En Tailandia la decoloración en el proceso de la refinación de azúcar envuelve tres etapas: carbonatación, filtración y tratamiento con resinas de intercambio iónico. Los principales colorantes en el jugo filtrado se remueven por las resinas. La regeneración de las resinas tanto mediante NaOH ó NaCl, presentan problemas en la recuperación de los iones Na⁺ y Cl⁻. El carbón activado es una alternativa de decoloración, empleada en muchas refinerías en el mundo. El presente estudio examina a escala de laboratorio y planta piloto la utilidad de la ceniza de bagazo (BFA en inglés), por su habilidad decolorante de los licores filtrados. El BFA contiene carbón y...
tiene un área superficial total de 337 m²/g, diámetro promedio de poro de 2.794 nm y un volumen específico de 0.2355 cm³/g. La relación del volumen tamaño medio del poro al volumen total es de 65%, lo que hace al BFA adecuado para la remoción de grandes moléculas en solución, incluyendo el color en los licores. La razón de volumen de BFA al licor filtrado empleado en el estudio fue de 1:14.4. Los resultados muestran que la eficiencia del BFA en la remoción de color, turbidez y almidón en los siropes fue de 72-94%, 97%, 86-91% respectivamente; no obstante la eficiencia de remoción de dextrana fue mucho menor a 9-44%. El empleo del BFA no modificó la concentración de sacarosa, glucosa, fructosa, pol o Brix. Sin embargo, BFA incrementó la conductividad de las muestras tratadas. Durante las pruebas las muestras de licor mostraron contener altos niveles de calcio y magnesio, comparados con los del licor original. Esto puede requerir de estudios adicionales para investigar el efecto de estos metales, si estos niveles son o no aceptables y si exceden las normas específicas de los productos alimenticios,

**Palabras clave:** cenizas de bagazo, decoloración, licor filtrado