

PILOT PLANT TRIALS WITH GRANULAR ACTIVATED CARBON AT THE MALELANE REFINERY

M MOODLEY¹, SB DAVIS², ED HARDWICK³ AND TC KERMEEN⁴

¹*Transvaal Sugar Limited, PO Box 47, Malelane, 1320*

²*Sugar Milling Research Institute, University of Natal, Durban, 4041*

³*Ionchem, PO Box 446, Isando, 1600*

⁴*Chemviron Carbon, Number One Southlink, Oldham, UK*

Abstract

The refinery at Malelane has a conventional carbonation/sulphitation decolourisation process and the maximum colour removal achieved is around 50%. To be competitive on both the local and export markets, it is necessary for Malelane to improve the decolourisation in the refinery. To achieve this objective a programme was implemented whereby other refining technologies are being evaluated. This paper describes tests done with activated carbon on a pilot plant scale at the Malelane Refinery.

Activated carbon decolourisation trials were performed on fine liquor at the Malelane refinery with a four column pilot plant provided by Chemviron Carbon Ltd. Two trials were run with an average contact time of two hours for a total period of 33 days, the first with fresh carbon and the second with partly spent carbon in two columns and fresh carbon in the polishing columns. Daily composite samples were taken of the feed, intermediate streams and the product, and analysed for colour at pH 4, 7 and 9. Samples of the feed and product streams were sent to the Sugar Milling Research Institute (SMRI), where the liquor was boiled to produce sugar. Output colours increased slowly over the duration of the trials, with an average of 60% colour removal. No sharp colour breakthrough was observed, but some peak colour shaving took place. The magnesite buffered carbon that was used prevented pH drops in the columns and hence inversion losses. Sufficient data were gathered from the trials to enable an initial estimate to be made of the configuration of a full scale plant, and a further trial with a longer contact time may be required.

Introduction

The first phase of the optimisation of the Malelane refinery with regard to refined sugar quality has been completed and is described in detail by Moodley and Schorn (1997) and Moodley *et al.* (1997). The next phase was to evaluate other refining decolourisation processes such as ozone and activated carbon. Full scale tests have been done with ozone and are described by Davis *et al.* (1998).

High (83%) levels of liquor decolourisation have been reported by a refinery using granular activated carbon (Mabillot, 1996). Pilot plant tests were therefore done with granular activated carbon at the Malelane refinery. Carbon treatment is primarily based on a naturally occurring pheno-

menon called adsorption, in which the solute molecules in a liquid or gas are attached to the surface of the solid. Activated carbon has a very high surface area and is thus an ideal material for adsorption (Anon, 1995).

The carbon chosen for the tests offered the following benefits (Anon, 1996):

- A high adsorption capacity for colour bodies ensuring a high decolourisation.
- Magnesite bonded into the carbon structure which provides a consistent buffering capacity. The other major benefit of carbon is that it can be reactivated several times. During the reactivation process, which is carried out at 850°C, the adsorbed impurities are destroyed.

It was decided to evaluate activated carbon as a polishing process at the Malelane refinery, and pilot plant tests were done on fine liquor leaving the sulphitation station. A schematic diagram of the Malelane refinery showing the point at which the fine liquor was taken off the main stream and pumped to the pilot plant is given in Figure 1.

Experimental

Description of the pilot plant

The pilot plant was specially designed by Chemviron for the evaluation of liquor decolourisation in a refinery. The plant is made up of four jacketed stainless steel columns which are mounted vertically within a container framework, as shown in Figure 2. The plant is designed to operate with four columns in series (i.e. one inlet and one outlet) in downflow operation, but it can also be configured for parallel operation. Each column has an internal diameter of 0,2 m and a volume of 110 litres and is equipped with pressure and temperature indicators, a rupture disk and sample points. The pilot plant is skid mounted for rapid and easy installation and the unit is equipped with a pump, heat exchanger and flow meter. The four columns are piped up to be operated in a merry-go-round system. The pilot plant specifications are given in Table 1, and the proposed operating conditions are given in Table 2.

Commissioning of the pilot plant

Initially the columns were sterilised with a bactericide and then thoroughly rinsed with water. The required quantity of carbon was then added to the columns, after which the columns were backwashed with cold water to remove the carbon

finer. Hot water was then circulated through the columns until the temperature of 70°C was reached. Fine liquor was then fed to the pilot plant.

Table 1. Pilot plant specifications.

Flow (L/h)	63 - 314
Operating pressure (kPa)	500
Temperature (°C)	100 maximum
Carbon quantity per column (kg)	62,5 maximum
Operating mass of plant (ton)	4,2
Material of construction	SS 316
Width (m)	2,3
Depth (m)	2,6
Height (m)	6,1

Table 2. Proposed pilot plant operating conditions.

Fine liquor flow rate (L/h)	94
Carbon charge (kg)	21,15
Contact time (h)	2
Liquor density (kg/L)	1,31
Brix of liquor	65
Temperature of liquor (°C)	60 - 70
Liquor pH	6,8 - 7,2
Fine liquor colour	400 - 600

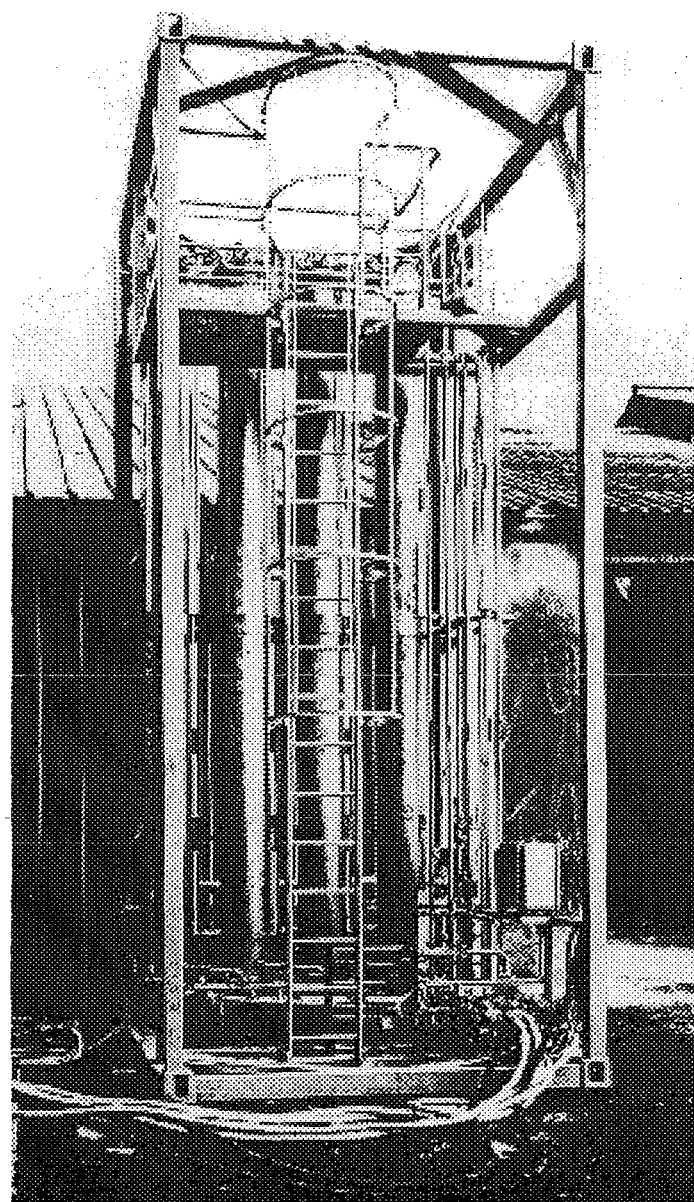


Figure 2. The Chemviron pilot plant.

Experimental programme

Catch samples of all streams were taken every four hours. From these catch samples a daily composite was made and analysed for colour, pH and brix. The temperature and pressure of all the liquor streams were recorded every four hours. The inlet liquor flow rate was also recorded every four hours. Once weekly, 25 kg each of the inlet and product streams were sent to the SMRI for crystallisation tests.

Analyses

Colour was measured at 420 nm and pH 4, 7 and 9, using 0,45 micron membranes for filtration. Crystal colour determinations were conducted on sugar samples affinated according to the ICUMSA method, and the sugar colours are reported on affinated sugars. The conductivity ash measurements were done according to the method described in the Laboratory Manual for South African Sugar Factories (Anon, 1985).

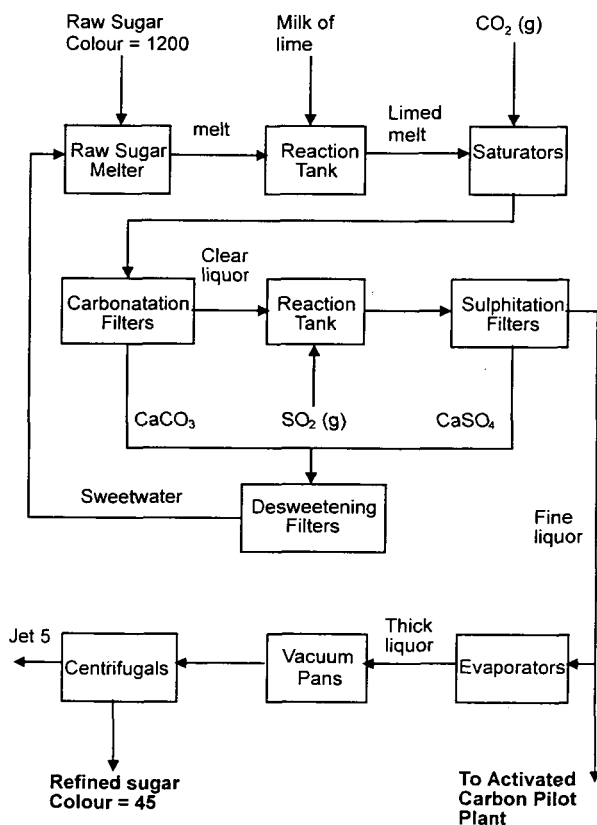


Figure 1. Simplified flow diagram of the Malelane refinery.

Results and discussion

Two trials were run with an average contact time of two hours for a total period of 33 days, the first trial with fresh carbon and the second with partly spent carbon in two columns and fresh carbon in the polishing columns. The configuration of the columns of the pilot plant during the two trials is shown in Figure 3. The average pilot plant operating conditions for the trial period are given in Table 3.

Table 3. Actual pilot plant operating conditions.

Flow rate (L/h)	148,4
Carbon charge (kg)	37
Contact time (h)	2,2
Brix	61 - 66
Temperature of inlet liquor (°C)	55 - 75
pH of inlet liquor	7,1 - 8,4
Colour of inlet liquor	452 - 705
Colour of product liquor	80 - 391
Average product colour	235

Temperature variation of inlet liquor

During the initial 24 hours of the running of the pilot plant, it was observed that the inlet liquor temperature was very low (40°C) and this caused the flow rate to drop to 15 L/h because of the higher viscosity and pressure drop across the column. This was due to the liquor in the 1 000 kg supply vessel cooling. To overcome this problem, a heating coil was inserted in the tank and hot water at 80°C was circulated through the coil. The inlet liquor temperature then stabilised at 65 to 70°C. However, there were periods when the liquor temperature was lower than normal, due to the liquor cooling in the supply tank, particularly at night.

pH of liquors

It was expected that the pH value of the treated liquor would drop by about 0,5 unit during the trial. However, the results of the trial showed no significant change in liquor pH as a consequence of the buffering capacity of the magnesite and possibly because of the lower than anticipated operating temperature.

Conductivity ash results

The average results (over 33 days) indicated no change in the ash content between the feed liquor and the outlet liquor from each column. This was expected as the grade of carbon used was not designed for ash removal.

Liquor colour results

The results of the liquor colours for the initial thirteen days are plotted in Figure 4. The results indicate a sharp increase in the outlet colours within the first two days, thereafter the colour breakthrough is relatively slow. There was a wide variation in the feed liquor colours, ranging from 452 to 705

with an average of 551. Despite this variation, the activated carbon performed fairly well and the overall liquor decolourisation ranged from 40 to 90% with an average of 60%. After 13 days, it was decided to replace the carbon in columns one and two. Columns three and four then became the leading columns and columns one and two the polish columns.

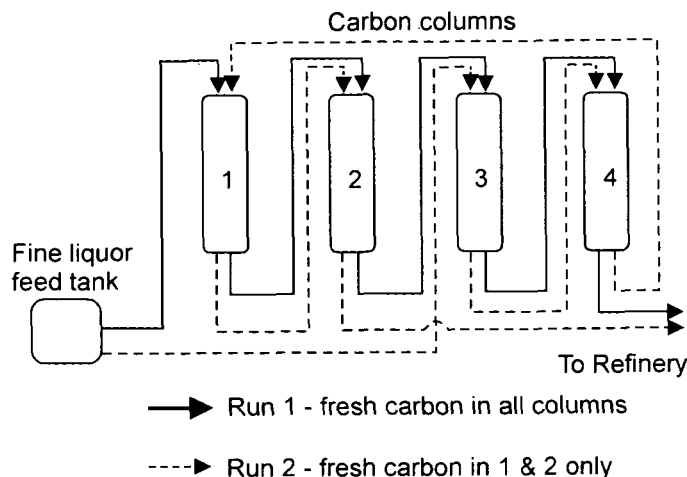


Figure 3. Configuration of the columns of the pilot plant during the two trials.

Liquor colour results for the balance of the trial (14 to 33 days) are given in Figure 5. Here again the outlet colours rose sharply initially, after which the colour breakthrough was slow. The dip in liquor colour at day 25 is due to recycling as a result of problems in the refinery. The average overall liquor decolourisation was similar to the first test. The results have shown that high levels of decolourisation have been achieved with the carbon. The overall decolourisation for both the tests (product/inlet) are plotted in Figure 6. The average liquor decolourisation for the trial was 60%, which is very encouraging. However, Lionnet (1990) found that liquor colour is not always a good indicator of crystal colour, and so a crystallisation step was included in the experimental work. Samples of the feed and product liquors were boiled in the SMRI pilot pan under identical conditions and the resulting sugars were affinated. The sugar crystal colours are plotted in Figure 7 against the pan feed liquor colour. The equation for the sulphited and carbon treated liquors is as follows:

$$\text{Crystal colour} = 0,76 + 0,168 * \text{feed liquor colour} \quad (1)$$

(n = 22, r = 0,93)

It is encouraging to note that the colour transfer of carbon-treated liquors is similar to those produced by the existing process at Malelane. Work done by the SMRI (Lionnet and Moodley, 1996) has shown that the colour transfer (crystal colour/feed liquor colour) of carbonatation/sulphitation refineries is lower than at refineries with ion exchange. The Sugar Milling Research Institute (SMRI) has done a number of boilings with liquors decolourised by ion exchange and these results, also plotted in Figure 7, confirm the previous findings. This is a consequence of the different mechanisms by which these decolourisation processes work, ion exchange

being not as effective as carbonatation/sulphitation or activated carbon in removing colour bodies that are preferentially transferred to the crystal.

In this regard it is interesting to look at the results of the colour measurements done at pH 4 and 9, which are shown in Figures 8 and 9. The colours measured at pH 4 indicate that the carbon has a relatively low capacity for pH-insensitive colour, which arises mainly from factory produced colour. This can be seen from the way in which the product colour follows the changes in inlet colour closely, although at a smaller magnitude. In particular, the large spikes on days 18 and 30 caused a rise in the product colour shortly afterwards, showing that there is little 'peak-shaving' of this type of colour; in other words, the short term spikes are not removed. The short delay in the peak in the product colour is probably due to the equilibrium concentration of the colour bodies on the carbon changing with feed colour. In other words, more colour will be adsorbed when there is a high feed colour, but when the feed colour drops, some of the adsorbed colour will be released back into the liquor. This is not a serious problem, as this type of colour has a low transfer rate to the crystal.

In contrast, the results of colour measured at pH 9 show that the pH-sensitive colour, consisting mainly of plant derived pigments, is well adsorbed by the carbon, which has a large capacity for this type of colour. Here peak shaving is very clear for most of the duration of the trial, and only after day 26 does the product colour rise with the increasing feed colour. In particular, the spike at day 18 has been completely removed by the carbon, and no spike is visible in the product. This is a major benefit in the refinery, as it is this pH-sensitive colour which is most readily transferred to the crystal.

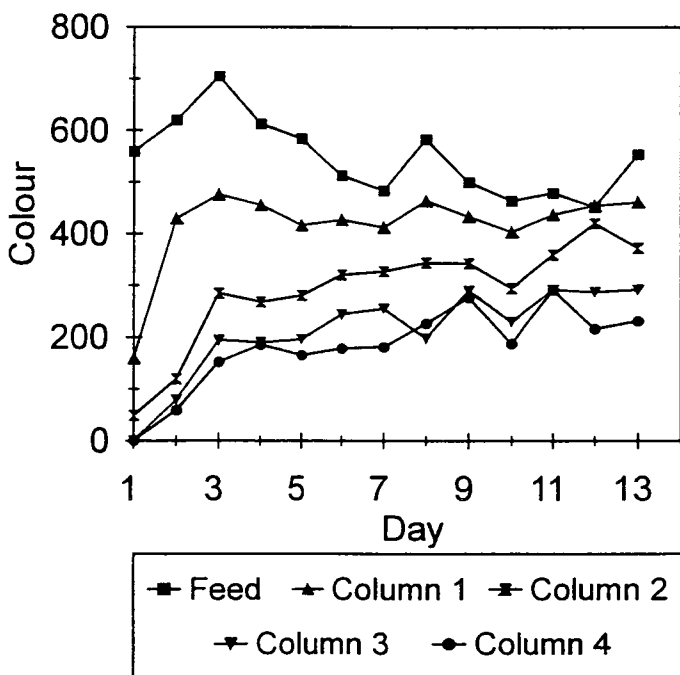


Figure 4. Liquor colour results for first 13 days.

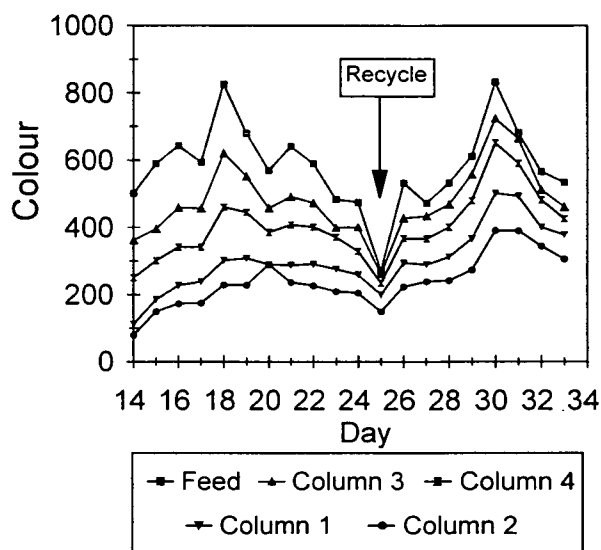


Figure 5. Liquor colour results for next 20 days.

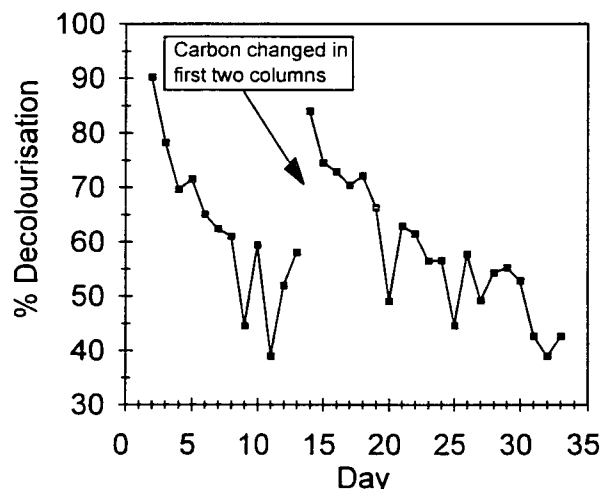


Figure 6. Overall liquor decolourisation results.

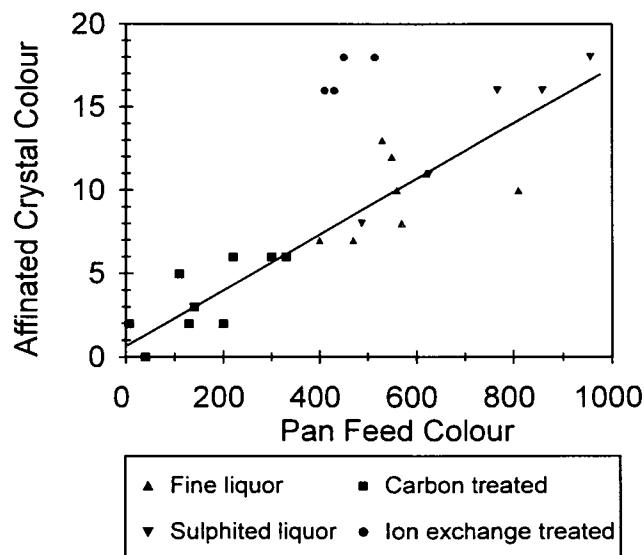


Figure 7. Liquor feed colour versus affinated crystal colour for the carbon trial and ion exchange results.

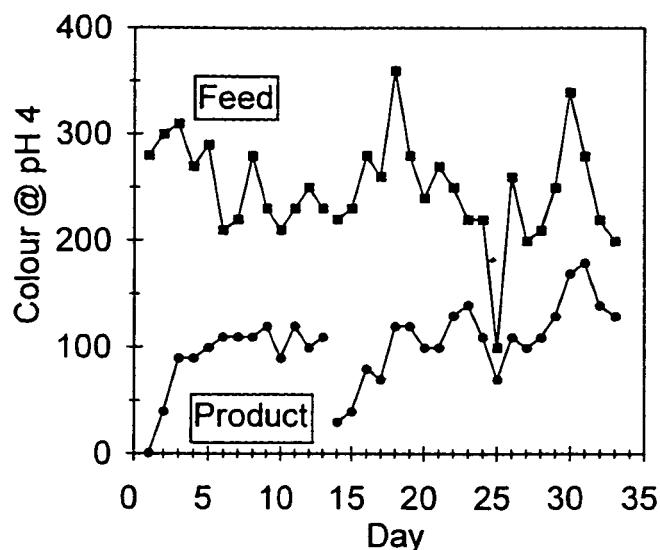


Figure 8. Liquor feed and product colours measured at pH 4.

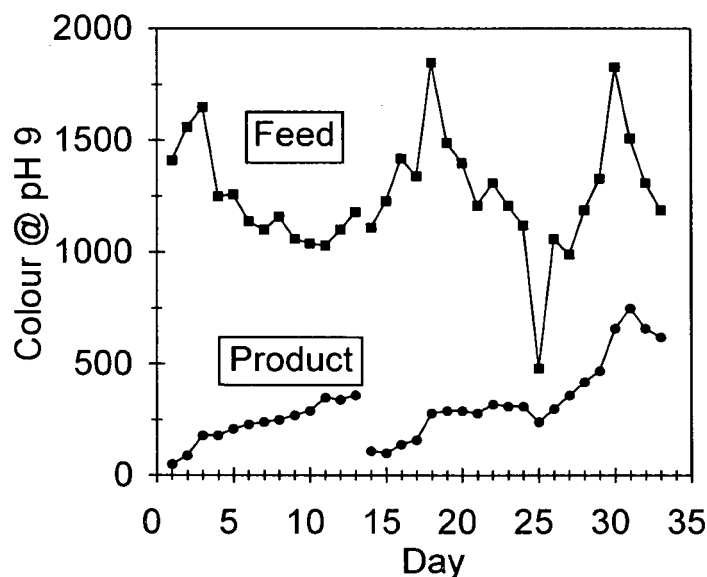


Figure 9. Liquor feed and product colours measured at pH 9.

Effect of feed liquor colour on refined sugar colour

Equation 1 has been used to calculate the expected refined sugar colour, from the feed colour. In the calculation it is assumed that the crystal consists of 80% of the total first sugar colour and also that the first sugar constitutes 30% of the total colour in refined sugar colour. The results are given in Table 4.

The average fine liquor colour for the season at Malelane was 567 and a refined sugar of 40 colour units was produced. If equation 1 is used to predict the refined sugar colour from the feed liquor of 567, then the expected refined sugar would be 43, and this value agrees reasonably well with the measured value. Although the feed colour to the carbon pilot plant varied from 452 to 705, the product colour did not exceed 400

colour units. The refined sugar produced from this fine liquor would be less than 31 colour units (Table 4) which is very encouraging.

Table 4. Feed and sugar colours.

Feed liquor	Crystal	First sugar	Refined sugar
100	2	3	10
200	4	5	17
300	6	7	24
400	7	9	31
500	9	11	38
600	11	14	45
700	13	16	52
800	14	18	59
900	16	20	66

Implications of trial results for a production unit

From the pilot plant data, the carbon usage can be calculated to be 2,12 kg of carbon per ton of dry sugar solids treated. This is for the second period of operation when partly exhausted carbon was used in two columns. This cannot be directly compared with results from other refineries using granulated activated carbon as the inlet colours and treatment objectives (the target output colour) vary, but this figure is considered to be extremely favourable.

For this trial, a treatment objective of 220 ICUMSA colour units was assumed, and it is clear from the data presented that it was rapidly exceeded by the output from the first three columns in the second stage of the trial, and was soon reached by the product stream. This indicates that the mass transfer zone where the colour bodies were being actively adsorbed occupied three of the four columns. For best carbon usage efficiency, this zone should be contained within a single bed. This would give a sharper breakthrough curve and a more definite indication when the carbon needed to be replaced. Under the conditions of the trial, greater efficiency could be achieved by decreasing the feed rate such that the contact time was increased from 2 hours 12 minutes to 3 hours 30 minutes.

Another method that is commonly used to shave the peaks of inlet colour is blending. The plant is set up with several columns in parallel, one of which is off-line for regeneration at any time, and the other three are at different stages of exhaustion. The outputs from the three columns that are on line vary in colour and are blended to make a product that meets the treatment objective. In this way, each column in turn can be allowed to produce a higher colour output before change over, and the carbon is used more efficiently. Using the results of the second stage of the trial, a three column blending system such as described would use an estimated 1,25 kg of carbon per ton of dry sugar solids, a great improvement over the series system.

Conclusions

This trial has proved the effectiveness of granular activated carbon in decolourising refinery liquors to produce low colour sugar. Naturally, a single set of operating conditions cannot be used to optimise a process, and further trials would be needed to obtain the most efficient configuration for a full scale production plant. Nevertheless, the trials have yielded useful data and some interesting results, which can be used to generate a preliminary design and costing.

Activated carbon has two main advantages over ion exchange, namely a lack of effluent that must be disposed of and the removal of a greater proportion of colour bodies that are readily transferred to the sugar crystal compared with ion exchange. For these reasons, granular activated carbon is likely to become an important process for any refinery aiming at reducing sugar colours.

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