COLOUR REMOVAL WITH THE SPARAC PROCESS: PRELIMINARY RESULTS

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Abstract

Following previous tests done on chemically regenerable carbon, Norit has developed a new grade of carbon that has faster adsorption kinetics. The effect of repeated cycles and chemical regenerations on the colour removal performance of this carbon has been investigated. Decolourisation trials were performed on brown liquor with a pilot plant supplied by Norit at the SMRI. Tests were carried out at 1 bed volume per hour (BV/h) at 80°C. Preliminary results show that calcium salt fouling can reduce the effectiveness of colour removal. The use of a pre-filter of spent carbon and different regeneration procedures is discussed. Overall the colour removal performance of SPARAC is lower than expected. Reasons for the poor performance are discussed.

Keywords: granular activated carbon; chemical regeneration.

Introduction

Granular activated carbon (GAC) is a very effective method of removing colour during sugar refining. However conventional GAC needs to be thermally regenerated requiring special kilns for this purpose. Norit has recently developed a new type of GAC (SPARAC) which can be regenerated with sodium hydroxide offering capital and carbon savings, and ease of application. After extensive treatment with this GAC at the Malelane factory by Moodley *et al*. (2000), Norit developed an improved SPARAC that has faster adsorption kinetics. This new type of carbon needed to be evaluated in terms of its lifetime and colour removal performance at higher throughputs. A pilot plant was made available by Norit for evaluation work on the new carbon.

Experimental

Pilot plant

The pilot plant consists of four 2800ml glass columns heated by hot water circulation and a peristaltic pump and a magnetic flow meter to provide a controlled and consistent liquor flow. The pilot plant is described in detail by Davis and Barker (2001). Table 1 describes the operating conditions.

Table 1. Operating conditions for the Norit pilot plant.

Carbon size	0,7mm - 1,2mm	**Feed**	Brown liquor
No of columns	2	**Feed colour**	650-750 ICUMSA
Vol carbon /column	1920 ml	**Feed pH**	7,8 -8,8
Flow rate	1BV/h*(64 ml/min)	**Feed Bx**	65°
Contact time	1 hour	**Temperature**	80°C

*BV = carbon bed volume per hour

Two columns were used instead of four as the logistics of fetching and returning the liquor from the refinery restricted the quantity of liquor that could be processed daily. Samples of feed and product were taken once or twice a day and at the same time temperatures and pressures, feed flow rate and feed volume were recorded.

Run cycles
A cycle consisted of sweetening on and running liquor through the columns until the decolourisation dropped to approximately 20%. At this point the columns were sweetened off and the carbon was regenerated. The average duration of a cycle was about 6 days. Shorter run cycles (2 to3 days) were also tried to investigate if this would increase the carbon performance.

Regeneration
The proposed procedure for carbon regeneration was as follows:

- Sweeten off at 2 bed volumes per hour (BV/h) until the outlet Brix is 1.
- Regenerate with 2% NaOH for 3 hours at 1,33 BV/h in down flow.
- Rinse with water for 1 hour at 2 BV/hr
- Neutralize with 0,6% citric acid at 2 BV/hr for 1 hour in down flow.
- Rinse with water at 2 BV /hr.
- Sweeten on at 1 BV/hr.

However since colour removal performance was lower than expected several modifications were done to try to improve performance. The use of HCl by Moodley appeared to remove excessive calcium from the carbon and this approach was tried in the early cycles. However during cycle 5 there was severe fouling of the carbon with calcium salts. After discussing the matter with Norit it was decided to add a filter to remove any precipitate, thereby eliminating the need for the acid step. The pre-filter consisted of spent carbon from the first column which was replaced with carbon from column 2 and column 2 was topped up with virgin carbon.

Regeneration with sodium hydroxide was increased from 3 to 6 hours to try to increase performance.

It was also noticed that after neutralizing with citric acid, the feed liquor pH dropped about 4 units whilst sweetening on leading to unacceptable levels of inversion and sucrose losses. Phosphoric acid and acetic acid were also used but with the same results. The purpose of neutralization is to prevent extra colour formation at higher pH values when sweetening on. However it is better to form colour rather than lose sucrose and it was decided to remove the neutralisation step from the regeneration procedure. Instead the rinse water cycle was increased until the pH of the effluent was approximately 9. Removal of this step appeared to have no effect on colour removal and there seemed to be no problem with pH whilst sweetening on.

Results

Carbon performance can be best visualized by plotting the colour loading against bed volumes. Colour loading is the cumulative units of colour removed multiplied by the mass of Brix passed divided by the mass of carbon. Comparison of curves from different cycles gives information about the effectiveness of colour removal and this can be linked to operating and regenerating procedures.

Prefiltration
Figure 1 shows the performance of virgin carbon (cycle 1), cycles without the use of a prefilter (cycles 2 to 5) and cycles with the use of a pre-filter (cycles 6 to 9). The two straight lines shown in the Figure 1 represent the effect of filtration and were obtained from linear regression analysis.

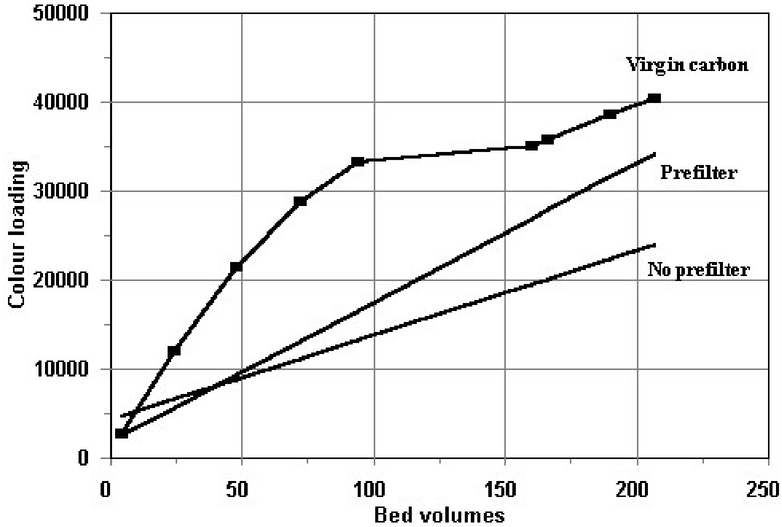

Figure 1. The effect of using a prefilter on carbon performance.

The results from Figure 1 can be summarised as follows:

- It is immediately apparent that there is a sharp decrease in performance of the carbon after the first cycle (virgin carbon).
- The use of a prefilter allows more colour to be loaded on to the carbon by preventing the calcium salts from blocking the carbon pores. This method is also more effective than using hydrochloric acid to remove the calcium.
- The performance of the carbon was quite constant after the first cycle and no deterioration in performance was noticed.

Short cycles

The effect of using shorter cycles on carbon performance is shown in Figure 2. Since this new type of carbon has faster absorption kinetics shorter cycles should increase performance. The results of the short cycles (cycles 10 to 17) were compared to virgin carbon and to the cycles with and without the use of a prefilter. All comparisons were made over 60 bed volumes, the approximate amount of feed liquor processed for each short cycle.

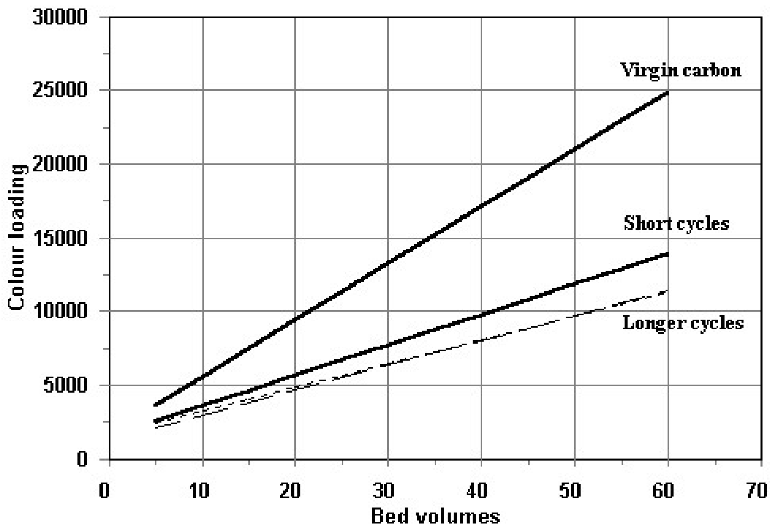

Figure 2. The effect of operating short cycles on carbon performance.

As can be seen from Figure 2 there is an increase in performance of the carbon when short cycles are used. This type of approach prevents excessive fouling of the pores and the carbon is able to adsorb colour faster. However chemical costs and the amount of effluent increase.

Discussion

Colour removal with this new type of activated carbon has been poor with the exception of the first cycle. The reason for the large drop in performance after the first cycle may be explained by considering that there are two types of colour in the brown liquor feed. Carbon has a high capacity for one type of colour (type 1) and a low capacity for the other type of colour (type 2). Type 1 colour is not effectively removed with alkali regeneration while type 2 is. Hence there is only a limited capacity for the type 1 colour in the carbon and this colour type seems to comprise the bulk of the feed liquor. Therefore the major decolourising effect is the removal of the type 2 colour in the liquor as the capacity for type one is quickly used up and is the reason why decolourisation rapidly decreases after sweetening on. Prefiltering the liquor would only affect the adsorption of the type 2 colour. Therefore it appears that neither increasing the quantity of caustic, nor performing a hydrochloric wash, nor filtering the feed was able to increase capacity for the type 1 colour.

Conclusions and Recommendations

The carbon has completed 17 cycles, processed about 7500 l of brown liquor and been operating for approximately 2000 hours (83 days). Excluding the cycle with virgin carbon, the carbon has shown consistent performance which can be improved by prefiltering the feed liquor or running shorter cycles.

Overall the carbon performance has been disappointing with decolourization between 20 and 40 % compared to Moodley who had values of approximately 50 %. However Moodley was using fine liquor from the Malelane factory as feed and was operating at half the feed rate (0,5 BV/h). Davis and Barker have shown that there are different proportions or types of colour in the Huletts liquor compared to the Malelane liquors by looking at colour transfers from liquor to crystal. This could explain the different behaviours of the liquors towards activated carbon.

The following tests are suggested to determine the reasons for the poor performance of the carbon.

- To determine whether the new carbon or the higher flow rate is the cause, cycles will be run with a lower flow rate used by Moodley.
- Run cycles with fine liquor from Malelane on a small scale to investigate if the feed liquor is the cause of the lower performance.
- In order to size and plan a larger scale pilot plant, of capacity 30 tons of Brix per hour, the Bed depth service time method (BDST) calculation method introduced by Hutchins (1973) and developed by Scholten and Staal (1998), will be used. This will require the use of four columns in series.

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