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# THE INVESTIGATION INTO THE DECOLOURISATION OF JOHNSON FILTER SWEETWATER USING ULTRAFILTRATION

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## Abstract

The decolourisation of Johnson filter sweetwater using various inorganic ultrafiltration membranes was investigated and the effectiveness of the colour removal was examined by comparing the quality of the crystals which could be produced from the feed and permeate solutions. Point ICUMSA colour retentions in Johnson filter sweetwater ranged from 28 to 50% for the various membranes used. Colour retention in solutions of refined sugar and affinated refined sugar ranged from 34 to 52% respectively demonstrating that ultrafiltration has the ability to retain those colour molecules which were included in the refined sugar crystals. Solutions of Johnson filter sweetwater feed and ultrafiltration permeate were crystallised and the quality of the crystals was compared. Crystals from the ultrafiltered sweetwater had up to 27% less colour than those produced from the feed Johnson sweetwater.

## Introduction

The raw sugar melter is the one vessel in the sugar refinery where impurities, or non-sugars, are returned to the sugar solution. This is due to the low purity of the refinery sweetwater used for dissolving the VHP sugar. The vast majority of recycled sweetwater arises from the filtrate and wash product of the Johnson filter presses and is termed Johnson filter sweetwater.

In the course of this investigation it was estimated that the increase in the ICUMSA colour load to the melter at Hulett Refineries due to recycled Johnson filter sweetwater can be up to 31% based on weekly average quality control data from April 1991 to October 1993.

Johnson filter sweetwater poses a problem in that, although it is not a product stream *per se*, it still has some intrinsic value due to its sucrose content and cannot simply be discarded. Johnson filter sweetwater has an average brix value of 9,7. From an economic point of view, the practise of melting raw sugar in high quality sweetwater is correct and is general practice in sugar refining. However, little attention has been paid to the merits of improving the quality of this sweetwater prior to the melter and it has not been considered economically feasible to devote a colour removal plant, based on existing sugar decolourisation processes, specifically to the decolourisation of Johnson filter sweetwater.

The process of ultrafiltration, a class of the science of membrane technology, offers an alternative colour removal process to those currently being employed by the sugar refinery.

## The nature of ultrafiltration

Ultrafiltration is a relatively new separation technique employing a selective membrane to fractionate a mixture of solutes. The following definition of a membrane was compiled from several definitions listed by Lonsdale (1989).

*A membrane is a thin permeable barrier, interposed between two phases, which offers varying degrees of selectivity to the passage of different constituents, as a function of their specific transport properties, under the influence of a driving force.*

Hence the ultrafiltration membrane acts as a selective barrier, retaining certain solutes while allowing the passage of others, resulting in the enrichment of either the retentate or the permeate in one or more components of the process fluid. Ultrafiltration, by definition, retains molecules or particles in the range 0,002 to 0,02 μm. From a molecular mass point of view, ultrafiltration retains molecules larger than 500 daltons (Gekas (1988)).

As with all membrane processes, ultrafiltration membranes can be classified according to:

- the membrane material which may be organic, inorganic or liquid.
- the criteria for retention based on, pore size, pore shape, chemical composition of the membrane or physical state of the membrane.

Ultrafiltration processes operate under cross-flow conditions as opposed to dead-end conditions. Cross-flow utilises the shearing effect, caused by the flow of the feed solution parallel to the retention surface, to prevent the formation of filter cake and limit blockage of the membrane pores by the solute molecules.

Ultrafiltration has previously been used to decolourise various sugar streams. Patel (1992) reported that a colour retention of 60% was possible using a Carbosep 10 000 dalton molecular mass cut-off membrane for the ultrafiltration of refinery sweetwater. It is therefore evident that a certain degree of colour retention, based on ICUMSA colour, is achievable using ultrafiltration. However, what is not clear is what degree of colour retention is required to have a positive influence on the sugar refinery. Simply improving the colour of the Johnson filter sweetwater may not be beneficial if the membrane only retains those colour molecules which are easily removed by the ion exchange and crystallisation decolourisation processes. However, if the membrane retains those molecules which are not removed by ion exchange, which foul the ion exchange resin or which are preferentially included during crystallisation, the ultrafiltration of Johnson filter sweetwater will be beneficial irrespective of the degree of colour retention based on ICUMSA colour.

A series of experiments was designed to assess the effectiveness of ultrafiltration for the decolourisation of Johnson filter sweetwater.

## Experimental

### *The selected membranes*

Following an extensive literature survey into the available ultrafiltration membranes, it was decided only to investigate inorganic membrane modules due to their ability to tolerate the high temperature process conditions of the Johnson filter sweetwater stream. The average temperature of the sweetwater is about 83°C.

Of the available inorganic membranes it was decided to investigate the CeraMem module LMDA-20-P1, the M5 Micro Carbosep 60 module borrowed from the SMRI and the Membralox 1P19-40 module borrowed from the Atomic Energy Corporation. The CeraMem and Carbosep membranes were selected due to their low specified molecular mass cutoff ranges, while the Membralox membrane was selected in order to investigate the potential of secondary membrane formation.

Membrane specifications are summarised in Table 1 while cross-sections of the membrane modules are presented in Figure 1.

The Membralox module was modified by the formation of a dynamic, dual layer $ZrO_2$/polyacrylic-acid (PAA) membrane on the support surface. The procedure followed was that described by Cawdron and Neytzel-de Wilde (1986).

### *Pilot plant equipment*

Two pilot ultrafiltration rigs were used for the experiments.

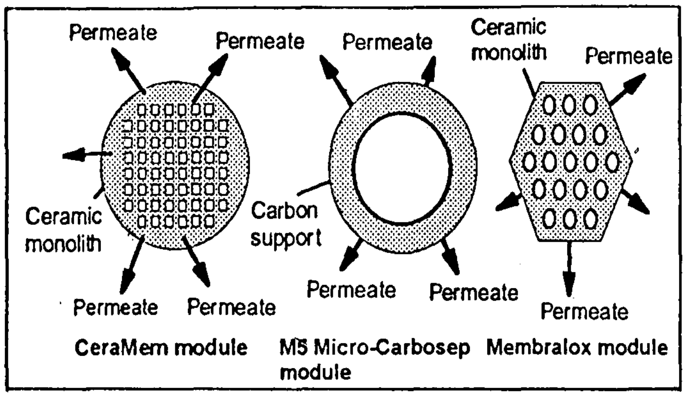

**FIGURE 1** Cross-Sections of the CeraMem, Carbosep and Membralox membrane supports.

The *Johnson sweetwater ultrafiltration rig* (Figure 2) was designed to run using feed directly from the Johnson filter sweetwater tank at Hulett Refineries. A 30 mm pipeline, connected to the Johnson filter sweetwater pump discharge line, supplies the ultrafiltration rig booster pump – an ETB2040-20 centrifugal pump. This delivers to a 12 mm line attached to the rig framework. Prior to connecting to the membrane housing, three separate lines are connected to the 12 mm feed line. The first line is the continuous feed sample port, while the second line is the feed purge line. The membrane isolation valve was closed when feed was being purged to the sump allowing the membrane module to be bypassed. The third line is the feed sample port. The retentate line following the membrane housing includes a

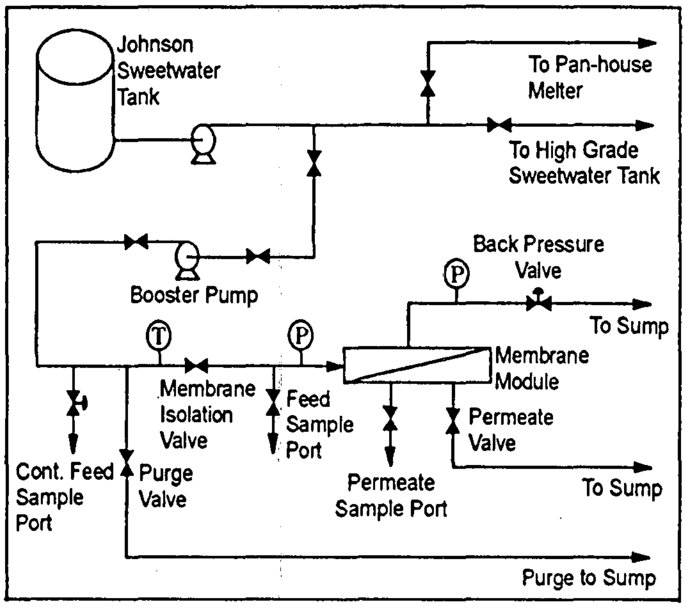

**FIGURE 2** Flow diagram of the Johnson sweetwater ultrafiltration rig.

**Table 1**
**Specifications of the inorganic ultrafiltration membranes**

| | CeraMem LMDA-20-P1 | M5-Micro Carbosep 60 | Modified Membralox 1P19-40 |
|---|---|---|---|
| Manufacturer | CeraMem | Rhone-Poulenc | Alcoa/SCT |
| Membrane material | $ZrO_2$ | $ZrO_2/TiO_2$ | Dynamic $ZrO_2$/polyacrylic-acid |
| Support material | $ZrO_2$ | Carbon | Alumina |
| Molecular weight cutoff (daltons) | 10 000 to 15 000 | 10 000 | |
| Geometry of support | Circular monolith – 60 square section channels | Tubular | Hexagonal monolith – 19 circular channels |
| Channel ID (mm) | 2 | 6 | 4 |
| Channel length (mm) | 33 | 600 | 869 |
| Membrane area (m²) | 0,1585 | 0,01131 | 0,2075 |
| Max T (°C) | 130 | 300 | 1 000 |
| Max P (kPa) | 700 | 1 500 | 10 000 |
| pH range | 5 to 12 | 1 to 14 | 1 to 14 |

control valve which allows the system back pressure, and hence the trans-membrane pressure, to be controlled. Pressure gauges are located at the inlet and outlet ends of the housing module. When not being sampled from the permeate sample port, permeate is discharged to the sump through the permeate valve. Retentate is discharged to the sump.

The *refined sugar ultrafiltration rig* (Figure 3) has a 40 ℓ feed tank which is connected to an M-range, 532 Mono pump. A heating coil comprising a 12 mm stainless steel tube is located in the feed tank and is connected to a separate hot water tank by means of rubber tubes. The temperature of the hot water tank is maintained by a 2 kW heater and PID controller. The temperature of the feed tank is controlled at the desired set point by a PID controller which switches on and off a small magnetic centrifugal pump (Serfilco Model A) in the hot water circuit. The purpose of the hot water coil is to provide gentle heating to the refined sugar solutions. The membrane housing module is attached to a stand immediately above the feed tank. Feed solution may be pumped either directly to the membrane module, may be returned to the feed tank via a bypass line, or may be pumped to drain. Retentate flows through a metric 24-G rotameter before returning to the feed tank. The retentate may also be diverted to drain. Trans-membrane pressure is controlled by adjusting the valves in the retentate return and bypass return lines. Pressure gauges are located at the inlet and outlet ends of the housing module. All flow lines, except the pump supply and discharge lines and the flexible stainless steel hoses connecting to the housing module, are 12 mm stainless steel tubes. The pump supply and discharge lines are 12 mm diameter stainless steel pipes.

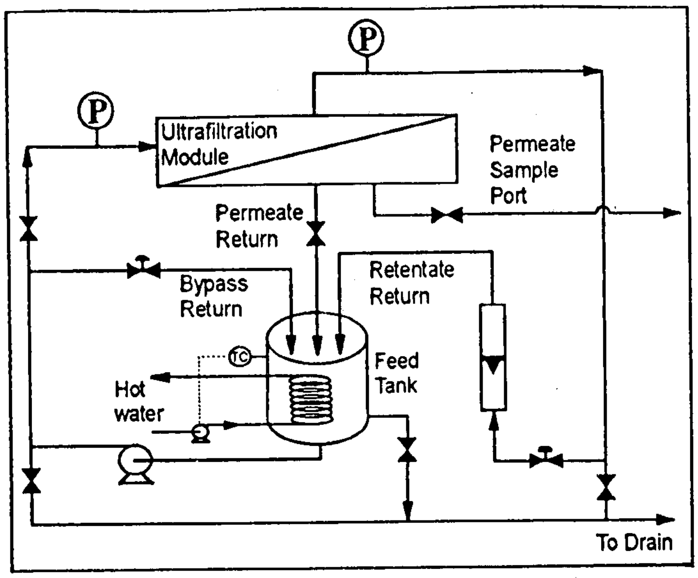

**FIGURE 3** Flow diagram of the refined sugar ultrafiltration rig.

## Experiment A: The ultrafiltration of Johnson filter sweetwater

The purpose of this series of experiments was to assess the ability of the above three membranes to retain colour in Johnson filter sweetwater. Sweetwater from the Johnson filter sweetwater tank was pumped through the particular membrane module using the Johnson sweetwater ultrafiltration rig. Feed pressure was maintained at 400 kPa while feed temperature, brix, pH and ICUMSA colour were allowed to vary with the condition of the Johnson filter sweetwater. Average Johnson filter sweetwater characteristics are presented in Table 2.

**Table 2**
**Average Johnson filter sweetwater characteristics based on weekly average quality control data for 1991 and 1992**

| | |
|---|---|
| ICUMSA colour | 3 514 |
| Brix | 9,7 |
| Temperature (°C) | 80 |

The membranes were exposed to the Johnson filter sweetwater for varying periods of time depending on the degree of fouling and associated flux decline of the particular membrane. Feed and permeate samples were taken periodically and analysed for brix and ICUMSA colour. Values of brix retention and ICUMSA colour retention were calculated by determining the percentage difference between the respective feed and permeate values. The average point retention values are presented in Table 3 while point permeate flux values are presented in Figure 4.

**Table 3**
**Experiment A**
**Average point retention values for the ultrafiltration of Johnson filter sweetwater**

| Membrane | Average point brix retention (%) | Average point ICUMSA colour retention (%) |
|---|---|---|
| M5 Micro-Carbosep 60 | 5 | 50 |
| Modified Membralox with dual layer $ZrO_2$/PAA membrane | 9 | 44 |
| CeraMem LMDA-20-P1 | 3 | 31 |

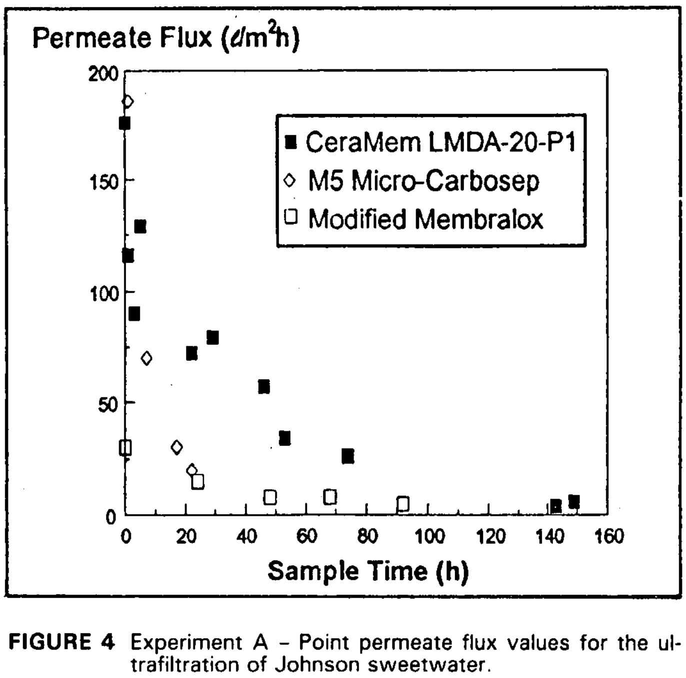

**FIGURE 4** Experiment A – Point permeate flux values for the ultrafiltration of Johnson sweetwater.

The results of Experiment A represent typical values of brix and ICUMSA colour retention for the ultrafiltration of Johnson filter sweetwater using the above three inorganic membranes. It must however be noted that the experiments are not entirely reproducible due to the variable nature of the feed Johnson filter sweetwater.

The Carbosep membrane achieved the highest average colour retention of 50% while the CeraMem membrane achieved a lower average retention of 31%. This was expected due to the molecular mass cut-off ranges of the two membranes being specified as 10 000 daltons for the Carbosep membrane and between 10 000 and 15 000 daltons for the CeraMem membrane. The modified Membralox membrane achieved an average colour retention of 44%. The Membralox membrane support is actually a microfiltration membrane having a relatively large nominal pore size of 0,02 $\mu$m. The high colour retention can therefore be attributed to the successful formation of the dynamic, dual layer $ZrO_2$/PAA membrane. However, the drawback of this dynamic membrane is that it significantly reduced permeate flux. Initial flux for the Membralox membrane was only 30 ℓ/m$^2$h which decreased to 12 ℓ/m$^2$h after just 24 h. Initial fluxes for the Carbosep and CeraMem membranes were 180 and 176 ℓ/m$^2$h, decreasing to 15 and 75 ℓ/m$^2$h after 24 h respectively. The Carbosep membrane exhibited greater fouling characteristics than the CeraMem membrane which is evident in the difference between the two flux values after 24 h operation as shown in Figure 4.

Average point brix retentions for the three membranes are all below 10% with 3 and 5% being observed for the CeraMem and Carbosep membranes respectively. Brix is a measure of the total dissolved solids in a solution. Hence, the retention of sucrose will be lower than the brix retention values due to the presence of the impurities in the feed solution. The low sucrose retentions are confirmed in Experiment B where solutions of refined sugar were ultrafiltered.

### *Motivation for subsequent experimental work*

The results of Experiment A give a good indication to which membranes achieve better decolourisation of Johnson filter sweetwater based on point ICUMSA colour retention values. From an economic point of view, the colour and brix retention characteristics of a particular membrane would have to be considered together with the particular membrane's permeate flux and fouling characteristics. Hence, high *effective* colour retention, high sucrose recovery, high permeate fluxes and low cost cleaning requirements are the prerequisites for the economically advantageous ultrafiltration of Johnson filter sweetwater.

Johnson filter sweetwater is a recycle stream, as opposed to a product stream, and influences the decolourisation processes downstream of the melter. For colour retention by a membrane process in the Johnson filter sweetwater stream to be *effective* the membrane would have to reduce the colour load to the ion exchange plant significantly and also retain those colourant species which would ultimately be included in the refined sugar crystal.

It already has been shown that the inorganic membranes are capable of retaining ICUMSA colour in Johnson filter sweetwater. Hence, the focus of the investigation was directed at establishing whether the ultrafiltration membrane retains colourants which are included in the refined sugar crystal. The CeraMem LMDA-20-P1 membrane, although displaying the lowest of the three average colour retention values, was selected for subsequent experimental work due to the low permeate flux rates of the modified Membralox and Carbosep membrane modules.

### Experiment B: The ultrafiltration of refined sugar, affinated refined sugar, affination wash liquor and raw VHP sugar.

The aim of Experiment B was to assess the ability of the CeraMem LMDA-20-P1 membrane to retain the colour found in refined and raw sugar solutions. The sugar solutions are described as follows:

- *Refined sugar* is the final refined sugar product marketed by Hulett Refineries and is a mixture of 1st to 4th boiling sugars.
- *Affinated refined sugar* is the affinated product of refined sugar. The purpose of affination is to remove the surface film of colourants surrounding the crystal. The procedure involves the washing of the refined sugar crystals in a saturated refined sugar solution, followed by recovery in a Martin Christ laboratory centrifuge and rinsing with 50 mℓ distilled water in the spinning centrifuge.
- *Affination wash liquor* comprises the saturated refined sugar solution discharge from the centrifuge. This liquor has a high concentration of surface film colourants which are not regarded as having been included.

A volume of 10 ℓ reverse osmosis permeate water was added to the feed tank of the refined sugar ultrafiltration rig and was allowed to heat up to 40°C prior to the required mass of sugar being added to achieve a feed concentration of about 10° brix. This solution was allowed to circulate for about 30 minutes whereafter point feed and permeate samples were taken for subsequent brix and ICUMSA colour analyses. This procedure was repeated for concentrations of 25 and 50° brix for the three sugar solutions. In the case of the affination wash liquor, only concentrations of 10 and 25° brix were investigated.

The experimental operating conditions were:

Feed temperature = 40°C
Trans-membrane pressure = 400 kPa
Feed volumetric flow rate = 21 ℓ/min
Feed linear flow rate = 1,5 m/s

The results of Experiment B are presented in Table 4.

**Table 4**
**Experiment B**
**Average point ICUMSA colour and brix retention values for the ultrafiltration of refined sugar, affinated refined sugar, affination wash liquor and raw VHP sugar at three sugar concentrations**

| Feed solution type | ICUMSA Colour Retention (%) | | | | Average brix retention (%) |
|---|---|---|---|---|---|
| | Average | 10° Brix | 25° Brix | 50° Brix | |
| Refined sugar | 34 | 29 | 48 | 26 | 1 |
| Affinated refined sugar | 52 | 48 | 62 | 47 | 1,5 |
| Affination wash liquor | 59 | 60 | 58 | | 1,8 |
| Raw VHP sugar | 48 | 50 | 40 | 54 | 2,5 |

The highest ICUMSA colour retention value was achieved in the affination wash liquor. This can be attributed to the high concentration of colourants having a positive retention effect on the concentration polarised layer. The concentration polarised layer is the dynamic layer of retained solute species which forms at the surface of the retention layer of the membrane due to the selective passage of certain solute molecules through the membrane and the retention of others. The affination wash liquor had an average ICUMSA colour value of about 110 units compared to about 52 units for the refined sugar solution and 37 units for the affinated refined sugar solution.

Average colour retention in the VHP sugar solution was relatively high although slightly lower than the affination wash liquor, 48% as opposed to 59%. This is probably due to the VHP sugar having a relatively higher proportion of very low molecular weight plant derived colourants which may pass through the membrane.

The average colour retention value achieved in the affinated refined sugar was 52% as opposed to 34% in the refined sugar. Hence there is a higher proportion of retained solute species contained in the crystal itself as opposed to the crystal and surface film together.

In both the refined and affinated refined sugar solutions, colour retention was greatest for the 25° brix case. This can again be attributed to the concentration polarised layer exhibiting more favourable retention characteristics at that refined sugar concentration under the particular operating conditions. Retention over the three concentrations in the VHP sugar did not vary significantly, being slightly lower for the 25° brix case.

Average point brix retention values ranged from 1 to 1,8% for the refined sugar solutions. The average point brix retention value for the VHP sugar solution was 2,5%.

Permeate fluxes were similar for the refined sugar, affinated refined sugar and the affination wash liquor solutions for each of the three sugar concentrations respectively (Table 5). Permeate flux values for the VHP sugar at the same concentrations are significantly lower. This can be attributed to the high concentration of impurities in the VHP sugar and the resulting membrane fouling.

**Table 5**
**Experiment B**
**Point permeate flux values for the ultrafiltration of refined sugar, affinated refined sugar, affination wash liquor and VHP sugar at three concentrations**

| | Permeate Flux (ℓ/m$^2$h) | | |
|---|---|---|---|
| | 10° Brix | 25° Brix | 50° Brix |
| Refined sugar | 143 | 81 | 18 |
| Affinated refined sugar | 137 | 74 | 24 |
| Affination wash liquor | 139 | 75 | |
| VHP sugar | 81 | 35 | 4 |

From the results of Experiment B it can be concluded that ultrafiltration has the capacity to retain colour in solutions of refined sugar and therefore has the ability to retain those colour molecules that were *included* in the refined sugar crystals. Further deductions from these results were not attempted because experimental conditions will vary considerably as the feed sugars vary in concentrations and types of impurities.

### Experiment C: Johnson filter sweetwater feed and permeate crystal quality analyses

Crystal quality experiments were performed to determine whether the ultrafiltration of Johnson filter sweetwater removes those colour molecules which will ultimately include in the refined sugar crystal, thereby resulting in crystals of lower colour than those produced from the original feed Johnson filter sweetwater.

The potential influence of ultrafiltered Johnson filter sweetwater cannot be predicted on the basis of ICUMSA colour due to the fact that it does not indicate the relative fraction of *potentially included* colour. No reliable analytical technique is available which can accurately determine the amount of colour which will transfer to the sugar crystal. This is due to the highly complex nature of sugar colour. It was decided, therefore, to investigate the quality of the crystals which could be produced from solutions of Johnson filter sweetwater feed and corresponding ultrafiltration permeate solutions.

The crystal quality experiments were carried out at the SMRI using their pilot vacuum pan and established colour transfer procedures described by Lionnet and Reid (1993). Initially, several problems had to be overcome. The first was related to the dilute nature of the Johnson filter sweetwater (average brix = 10). The SMRI pilot pan requires a minimum of 20 kg brix for a full boiling and a minimum of 13 kg brix for a half boiling. To satisfy the minimum, half boiling requirements, a mass of about 130 kg Johnson filter sweetwater and ultrafiltration permeate would have to be collected. Assuming a flux rate of 100 ℓ/m$^2$h for the CeraMem LMDA-20-P1 module, a collection time of 10 h would be required. This is not feasible due to the fact that the Johnson filter sweetwater will get cold and degrade over the sample time. In addition, the pan requires a feed solution concentration of about 60° brix. To concentrate the minimum mass of 130 kg Johnson filter sweetwater or permeate from 10 to 60° brix would require the evaporation of 100 ℓ of water. Using the SMRI pilot evaporator (Lionnet and Reid (1993)), which has a specified evaporation rate of 12 ℓ/h, this would take longer than 8 hours. It is well known that sugar solutions form colour when exposed to heat for long periods of time, hence the need to avoid long evaporation times.

As a compromise it was decided to increase the concentration of the Johnson filter sweetwater feed and permeate solutions by the addition of first boiling sugar prior to evaporation. This resulted in colour being added to the respective feed and permeate samples, although this was minimised by the addition of first boiling sugar which constitutes the purest sugar produced by the refinery. Average colour of the first boiling sugar was 20 ICUMSA units.

The refinery ultrafiltration rig was used and 38 ℓ samples of feed and permeate was collected. The samples were collected simultaneously and at the same rate due to the varying nature of the Johnson filter sweetwater. Following collection, they were transported to the SMRI where they were brixed up by the addition of 12 kg first boiling sugar to increase the concentration from about 10° brix to about 32° brix. The samples were then stored overnight in a cold storage room. The following day the samples were concentrated to about 60° brix using the SMRI pilot double-effect evaporator. Typical evaporation times were about 4 hours. The samples were then stored overnight prior to being boiled in the SMRI pilot pan. Typical boiling times were 4 to 5 hours for each sample whereafter the massecuite was struck from the pan and centrifuged in a Martin Christ laboratory centrifuge to recover the crystals. These were allowed to air dry prior to being affinated according to the ICUMSA method of successive washes in saturated solutions of refined sugar, 95% methanol and 100% methanol. The purpose of this was to remove all surface film colourants leaving behind only those colourants included in the sucrose crystal itself. A measure of the affinated crystal colour is equivalent to a measure of the included colour.

The affinated feed and permeate sugar crystals were analysed for ICUMSA *colour and colour differences* were determined. Three experiments were performed, the results of which are presented in Table 6.

**Table 6**
**Experiment C**
**Johnson filter sweetwater feed and permeate crystal quality analysis**

| | Experiment C1 | Experiment C2 | Experiment C3 |
|---|---|---|---|
| Feed crystal ICUMSA colour | 17 | 26 | 31 |
| Permeate crystal ICUMSA colour | 13 | 19 | 25 |
| Colour difference | 24% | 27% | 19% |

Lower levels of included sugar colour were observed for affinated permeate crystals than for affinated feed crystals in all three cases. Colour difference values ranged from 19 to 27%, that is, permeate crystal quality was improved by 19 to 27%.

The mechanisms for the inclusion of colour in refined sugar crystals are highly complex and are dependent on the types and concentration of molecules present. Short range forces determine whether the *potentially included* molecules do get included in the forming crystal. The results of the crystal quality experiments do not establish the types and quantities of molecules which were retained; however it can be concluded that ultrafiltration of Johnson filter sweetwater produces a permeate of higher quality, that is, having a lower concentration of *potentially included* colour molecules, than the original feed.

## Conclusions

(a) From Experiment A it is evident that a range of ICUMSA colour retentions can be achieved in Johnson filter sweetwater depending on the type of membrane used, the feed quality and the experimental conditions. For colour retention in Johnson filter sweetwater to be *effective*, the membrane is required to reduce the colour load to the ion exchange plant significantly and to retain those colourant species which would ultimately be included in the refined sugar crystal.

(b) The ultrafiltration of refined sugar solutions demonstrated that the ultrafiltration membrane is capable of retaining those molecules which are included in the refined sugar crystal.

(c) In Experiment C, crystals produced from the Johnson sweetwater ultrafiltration permeate contained up to 27% less colour than the original feed Johnson sweetwater. Ultrafiltration therefore retains a significant amount of those colourants which will ultimately include in the final refined sugar crystal.

(d) It is proposed that future investigations include ion exchange of feed and ultrafiltered Johnson filter sweetwater prior to crystal quality analysis. This will demonstrate whether ultrafiltration prior to ion exchange results in crystals of lower colour than ion exchange treated Johnson filter sweetwater, that is, whether ultrafiltration prior to ion exchange is advantageous from a colour removal point of view. From this, the overall effectiveness of the ultrafiltration of Johnson filter sweetwater can be established.

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