# Sugar syrup decolourisation using a continuous fluidised bed ion exchange process

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# Outline

- The Australian sugar industry and project partners
- Laboratory trials on MIEX® resin decolourisation
  - various process streams
  - colour levels and volumes
  - degree of colour removal required to have an impact
  - resin contact time and treatment cycles needed
- Pilot plant results - 2006 and 2007
- Plans for further pilot work during 2008 crushing season

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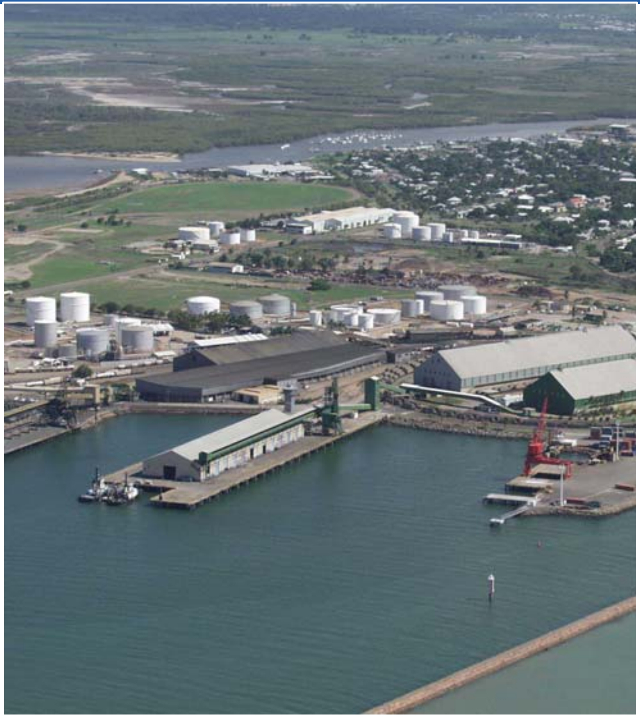
# Australian Sugar Industry - scale



- Cane area: 560,000 ha
- Cane harvested: 36 Mt
- Raw sugar: 4.3 Mt (bulk handled) - 85% exported
- Sales : $1.5b per year (2nd largest export crop)
- Employs 40,000 people

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# Orica Pty Ltd

- **Global business – turnover > US$3 Billion**
- **13,000 employees in over 40 countries**
- **Diverse product range**
  - **Mining Services - explosive/blasting solutions**
  - **Consumer Products - Paints/handyman products**
  - **Chemnet - Chemical Trading in Asia/Pacific Region**
  - **Chemicals Services - manufacturing and services**
    - ***Watercare - conventional and advanced water treatment solutions provider (MIEX®)***

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# Mulgrave Central Mill

Mulgrave Central Mill was established in 1896 and exists as a grower co-operative

- Processes cane from 300 farms across 17,000 hectares
- Annual harvests last 22-24 wks, processing 1.3 Mt of cane to produce 160,000 t raw sugar
- The mill operates a 232 km railway system, allowing cane transport across the district with no impact on local roads
- Self-sufficient for electricity and exports surplus to the grid

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# BSES Limited

BSES is the principal research, development and extension organisation serving the Australian sugar industry

Our role is to deliver realised value across strategic and applied research efforts

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# The BSES R,D&E Team

**160 total staff (approx 100 directly involved in R,D&E)**

***Research Programs***

- ***Plant Improvement***
- ***Cropping Systems***
- ***Technology Support***
- ***Crop Protection/Biosecurity***
- ***Biotechnology***

***Expertise***

- ***Agronomists***
- ***Entomologists***
- ***Plant pathologists***
- ***Plant breeders***
- ***Chemists and chemical engineers***
- ***Agricultural engineers***
- ***Biotechnologists***
- ***Agricultural Extension***

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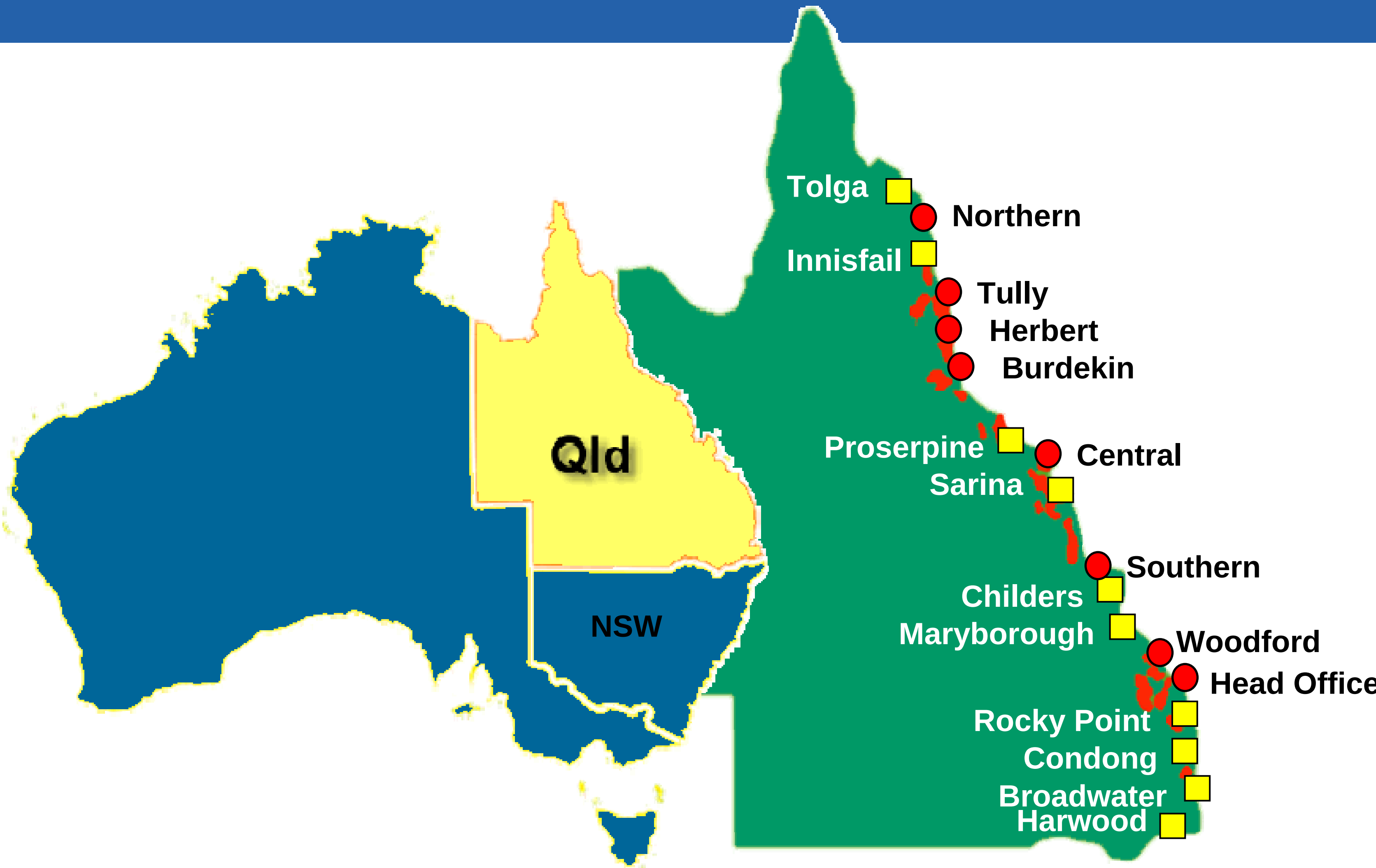
# You can find BSES here

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# Why do we want to remove colour??

- ***Improved raw sugar product***
  - **Lower colour**
  - **Reduced colour development upon storage**
- ***Meet and/or exceed product and value specifications from export customers***

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# Where is the value in reducing colour??

- **Where is the added value??**
  - **Increased $$ value of raw sugar exports**
  - **Access to key markets/customers**
  - **Decreased production costs for refiners**
- **Recent precedents for processes to manufacture low colour factory raw sugars**
  - **WSM process (IEX)**
  - **SAT process (UF)**
  - **Dedini process (IEX)**
  - **Charcoal applications**

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# Ion exchange process in a sugar factory

**Ion exchange processes for factory application have been regularly examined**

- technically feasible and attractive
- economically unsound (usually)

**Major problems include :**

- short cycles due to heavy salt loads in juice streams
- cost of regenerants
- waste regenerant disposal problems (GBRMP)
- speed of resin fouling
- cane quality variation (weather, variety etc)

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# MIEX® - Magnetic Ion Exchange resin

- strong-base macroporous ion-exchange resin
- low particle size (200 µm diam)
- beads contain an evenly dispersed magnetic particulate
- beads act as weak individual magnets in solution
- resin designed to remove dissolved organic carbon (DOC) from drinking water

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# MIEX® Process – drinking water

- Large scale processes already exist
- 16 plants exist worldwide
- 10 more in planning and construction stages
- Largest plant : 112.5 ML per day
- Smallest plant : 0.3 ML per day
- Scalable to large size and can be modular
- Strong IP position – production and application of MIEX® resin
- www.miexresin.com

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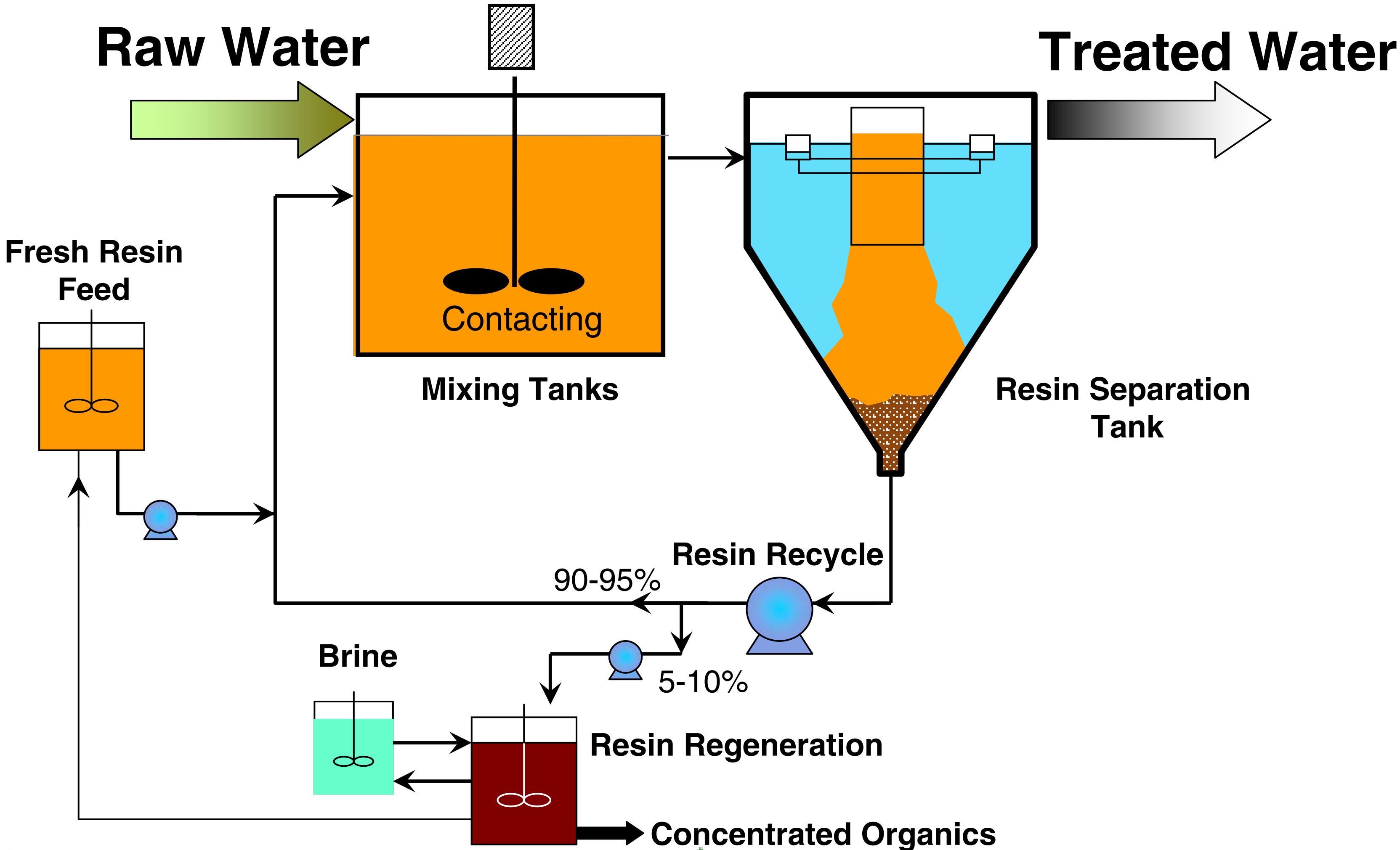
# MIEX® Process – drinking water (CSTR)

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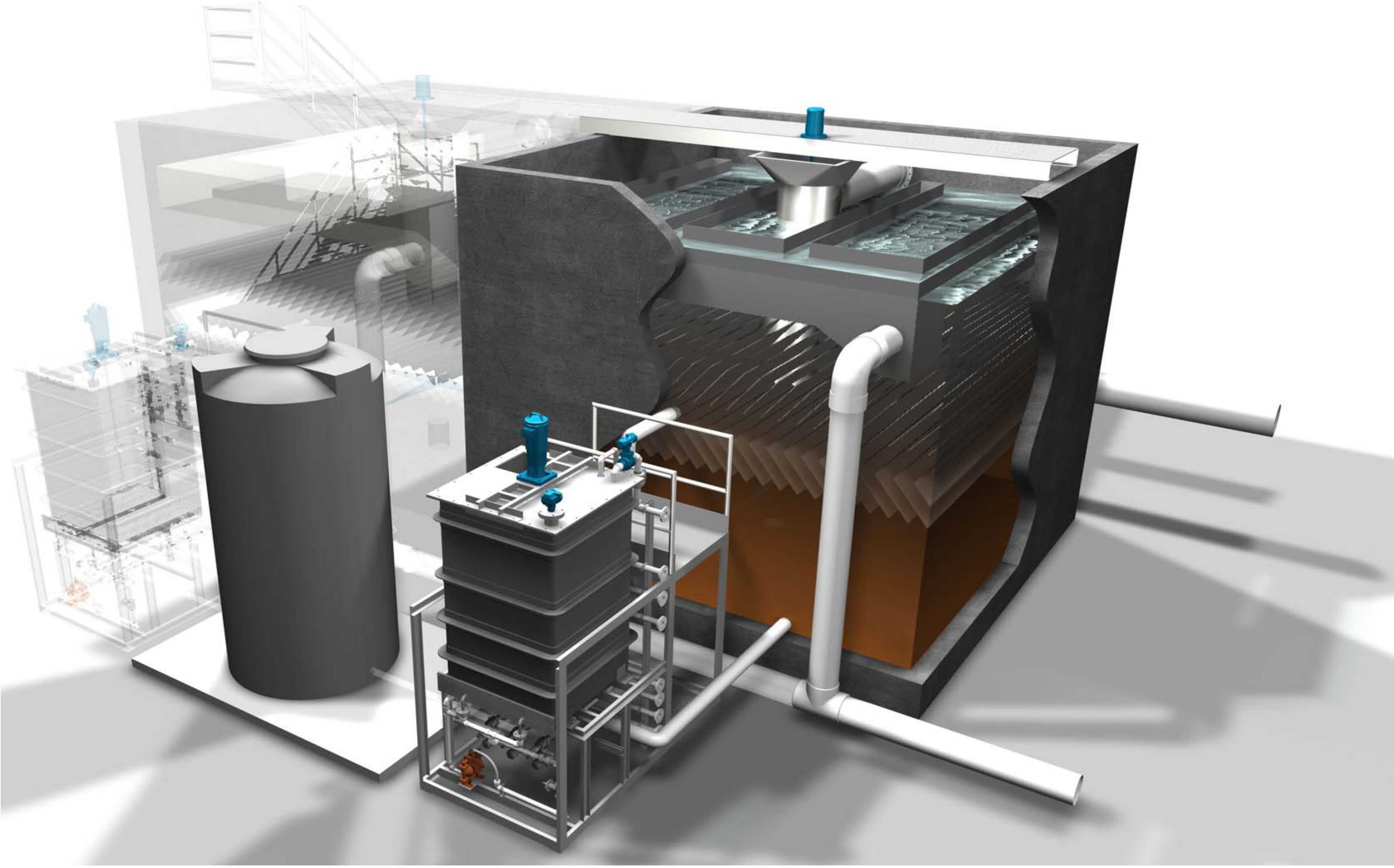
# MIEX® process schematic – alternative design




2.5 MLD: Mt Pleasant, South Australia
Settler
Contactor
Regeneration System
Treated Water Tank

# 112.5 MLD: Wanneroo, Western Australia

# Why does the MIEX® process work so well?

## Rapid decolourisation kinetics

- Smaller beads (200 µm vs 500-2000 µm)
- Higher surface area to volume ratio
- Smaller resin inventories required

## Uniform process stream quality

- Continuous process unlike traditional batched ion exchange applications

## Process flexibility

- Stirred contact tank reactors
- Fluidised bed system (upflow chromatography)

**Would it be effective for sugar colorants?**

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# Could MIEX® work for sugar colorants?

- Sugar mills have no specific decolourisation process
- Other systems designed to produce "mill white" sugars (WSM, SAT, Dedini) are far more capital intense
- Significant process differences compared to water treatment – heat, density, high colour loadings
- Would there be other mill effects?? e.g. crystallisation rate, filtrability improvements, altered ash levels
- MIEX® may provide a system to tailor-make various types of better quality raw sugars

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# Laboratory trials - resin suitability

***The following questions required answering…..***

1. **What colour (quantity and type) does the resin remove?**
2. **Under what mill conditions can it work?**
3. **Long term stability – 50 Bx, 70°C, 12 weeks (chloride capacity analysed at 0,2,4,8,12 weeks)**
4. **Performance of resin after multiple regeneration cycles (colour loading at 50 Bx, 70°C, regeneration at 25°C with saturated brine)**
5. **Decolourisation of simulated factory process streams – resin loading, contact time**

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# What colour does the resin remove?

| Experiment | Resin (g) | Time (min) | *Decolorisation efficiency (%)* Industry resin | MIEX® |
|---|---|---|---|---|
| Raw sugar syrup (64° Bx) | 1 | 15 | 37 | 72 |
| " | 1 | 15 | 31 | 71 |
| FRL | 1 | 15 | 76 | 88 |
| " | 0.5 | 15 | 57 | 82 |

- **MIEX® removes colour from raw sugar better than equivalent doses of a "normal" ion exchange resin**
- **Similar behaviour on refinery sourced filtered raw liquor (FRL)**

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# What colour does the resin remove?

***Would it remove difficult colorants?***

- **Acid Maillard polymer tested - the most difficult of the HMW colorants to remove during refinery decolourisation[1]**
- **Much better performance at same dose rates**

| Experiment | Resin (g) | Time (min) | *Decolorisation efficiency (%)* Industry resin | MIEX® |
|---|---|---|---|---|
| Acid Maillard sucrose syrup dosed at 1 mg/mL | 3 | 15 | 97 | 99 |
| Acid Maillard sucrose syrup dosed at 0.1 mg/mL | 3 | 15 | 98 | 99.7 |
| " | 1 | 15 | 62 | 99 |
| " | 1 | 5 | 36 | 92 |

**[1] Lindeman P.F. and O'Shea M.G. (2001). *Proc. Aust. Soc. Sugar Cane Technol.*, 23, 322-329**

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# Under what factory conditions can it work?

- **Settling rate tests examining…**
  - **Brix range 25 - 70 Bx**
  - **Temperature range 25 - 65°C**

- **Conclusions**
  - **Fastest settling rates for low Bx and high temperatures as expected**
  - **Suitable up to 50 Brix only (for contactor type application) due to settling rate limitation**

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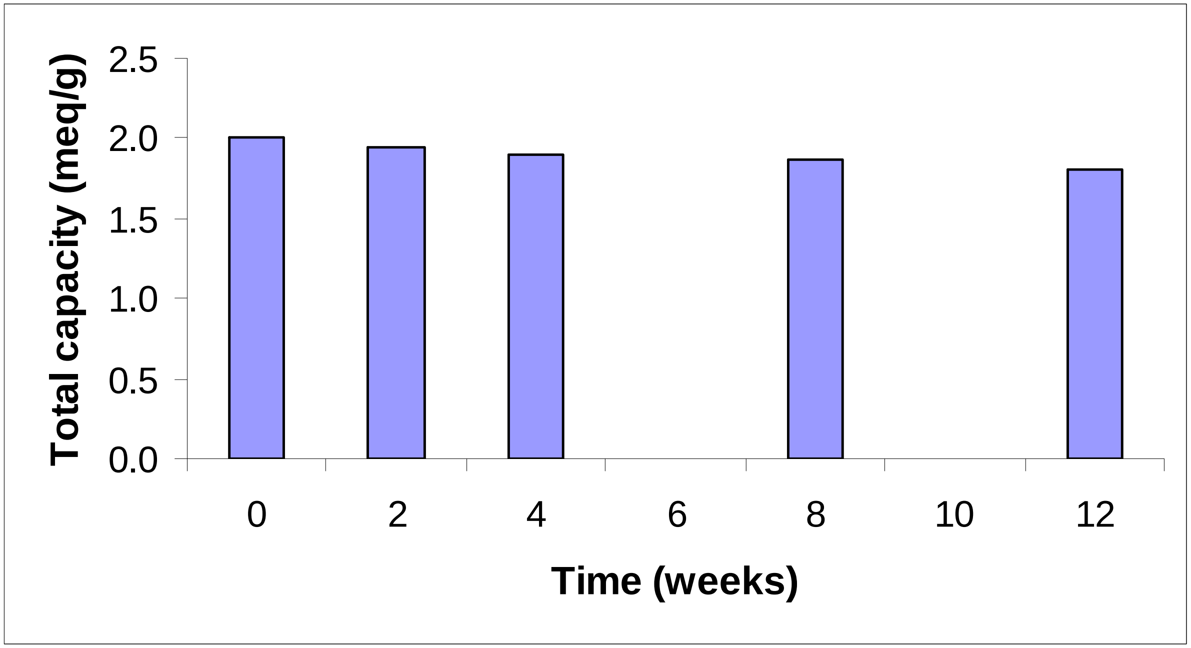
# Long term stability test




- Resin turned black at 1 wk
- Resin capacity not affected
- 12 wk data typical of long term drinking water plant results (1.8 meq/g after 10 months)
- Positive conclusions

# Performance across regeneration cycles

| Cycle | Abs | Colour | RI | Concentration, g/mL | True Reduction, % |
|---|---|---|---|---|---|
| 1 | 0.359 | 9399 | 1.4046 | 0.050 | 42.3 |
| 2 | 0.417 | 9926 | 1.4039 | 0.0499 | 39.0 |
| 3 | 0.425 | 10448 | 1.4075 | 0.0524 | 35.8 |
| 4 | 0.457 | 10803 | 1.4036 | 0.0497 | 33.7 |
| 5 | 0.457 | 11029 | 1.4089 | 0.0535 | 32.3 |
| 6 | 0.444 | 10304 | 1.4064 | 0.0517 | 36.7 |
| 7 | 0.479 | 10933 | 1.405 | 0.0507 | 32.9 |
| 8 | 0.486 | 11040 | 1.4131 | 0.0565 | 32.2 |
| 9 | 0.481 | 10999 | 1.4016 | 0.0483 | 32.5 |
| 10 | 0.467 | 10677 | 1.4016 | 0.0483 | 34.4 |
| *55 Brix* | *0.442* | *16284* | *1.432* | *0.07* | *0* |

- **5 min contact, 65°C, 55 Bx raw sugar syrup**
- **Relatively constant decolourisation efficiency after first few cycles (around 34%)**
- **Morphology unchanged during regeneration regime**
- **No osmotic shock to resin beads (breakage)**

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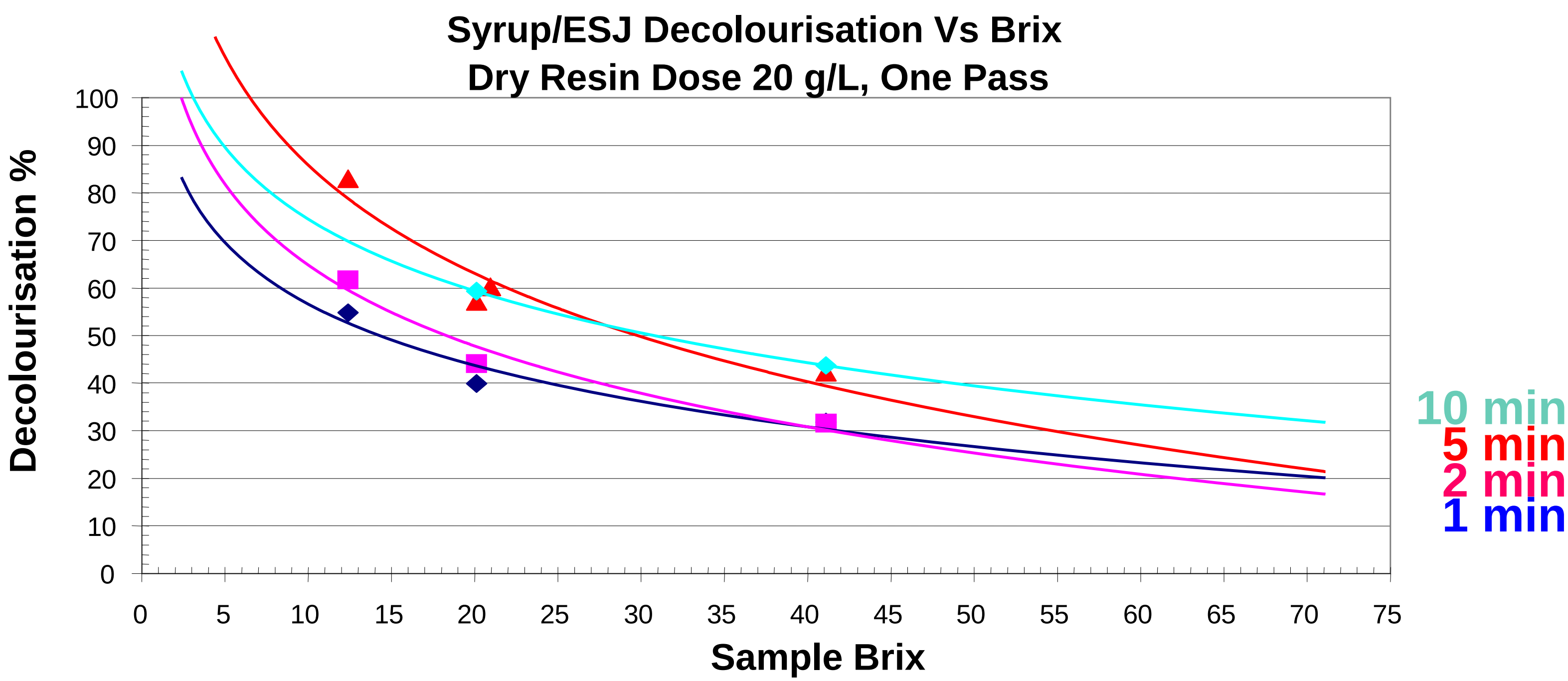
# % Decolourisation vs Brix (contact time)




**Decolourisation (relative)**

- **Exhibits logarithmic behaviour**
- **Increases with time up to 5 min**
- **Reduces with increasing Brix**
- **Improves with multiple passes (not shown)**

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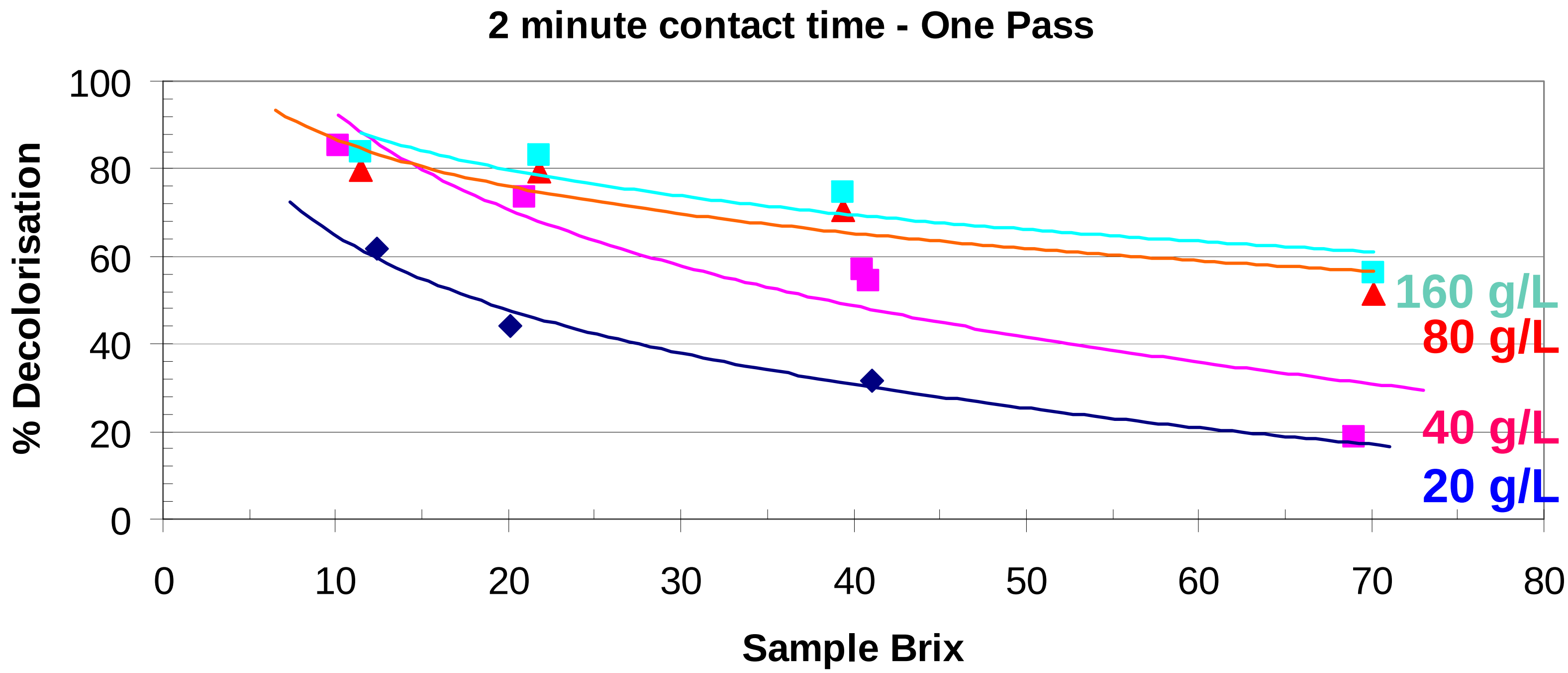
# % Decolourisation vs Brix (resin dose)




**Decolourisation (relative)**

- **Increases with increasing resin dose**
- **Reduces with increasing Brix**
- **80% decolourisation only achieved for < 20 Bx (2 min)**
- **Calculate resin requirement for % decolourisation, brix and contact time combination**

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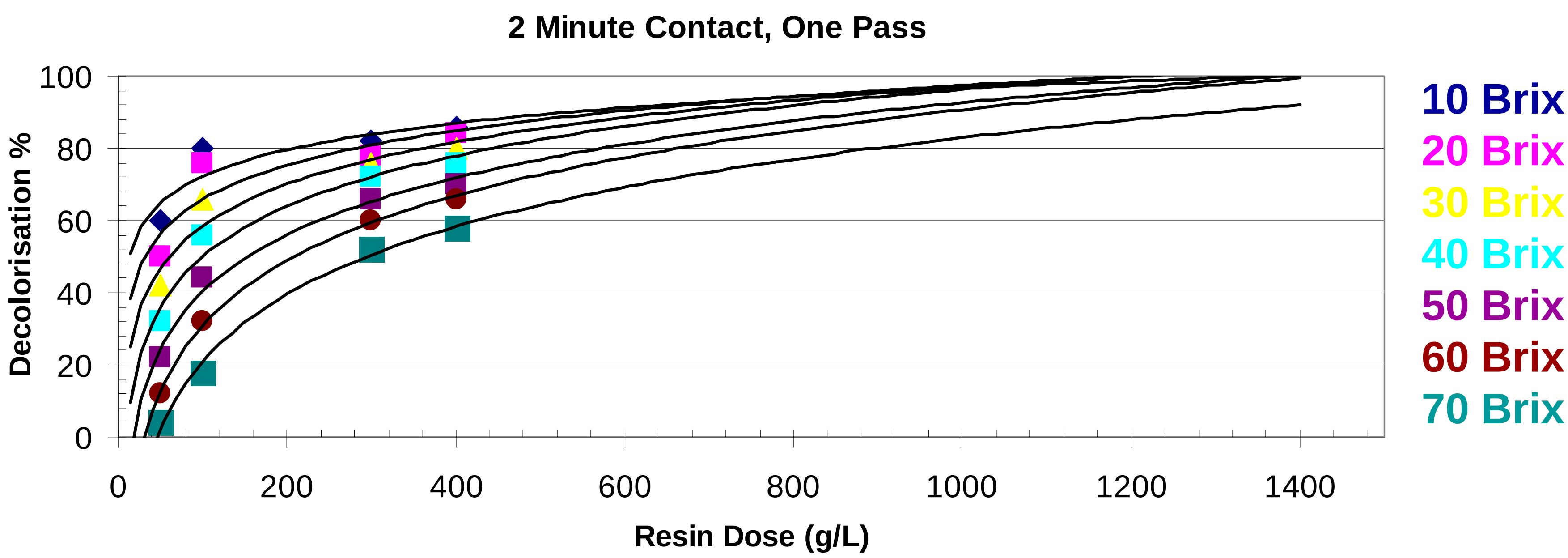
# % decolourisation vs resin dose (Brix)




- Log relationship between % decolourisation and resin dose
- Multiple passes would provide higher decolourisation rates
- Resin dose rates here unrealistic (too high)

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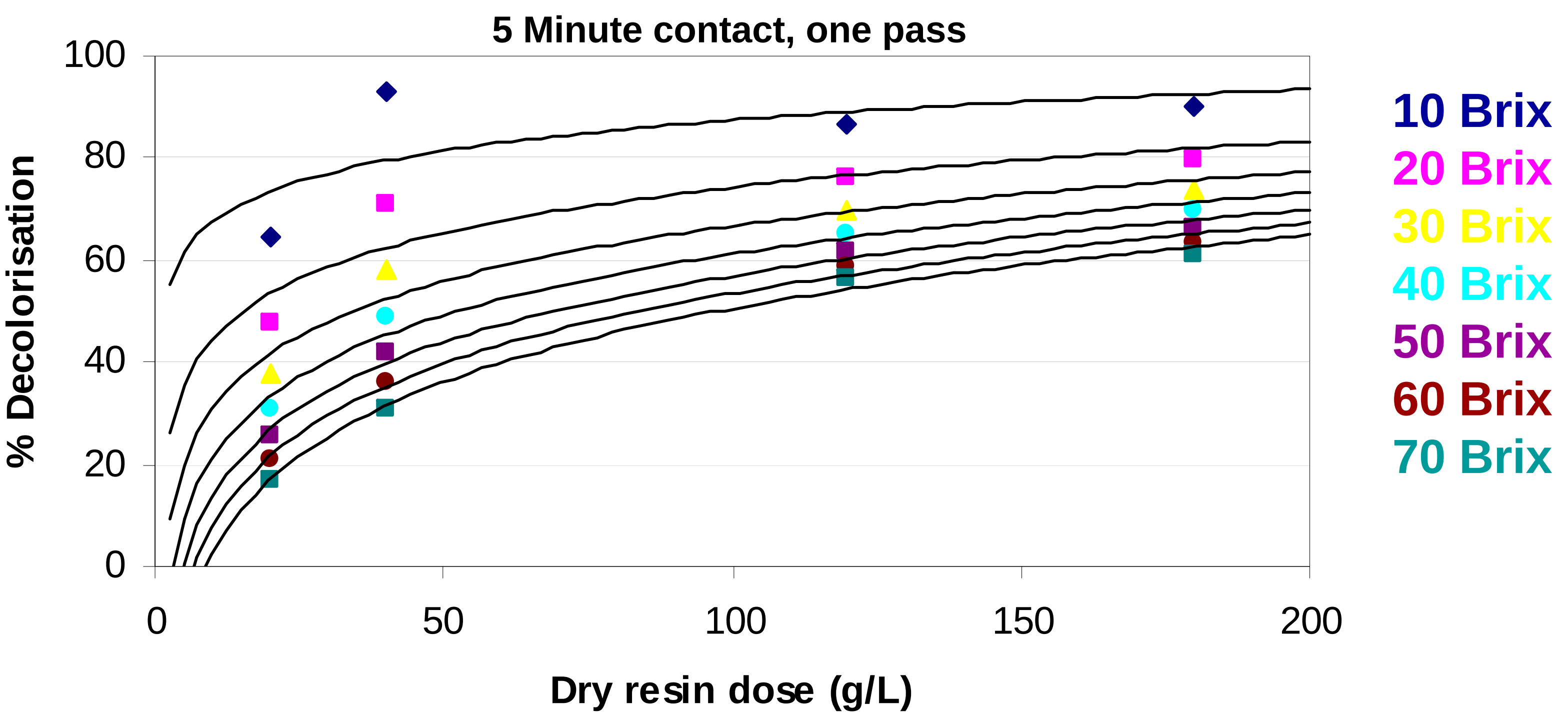
# % decolourisation vs resin dose (Brix)




- Lower resin doses and 5 min contact time
- This provides a basis for choosing factory conditions
- But we still need to understand syrup colour to sugar colour relationships, and define target sugar colour

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# 2006 and 2007 pilot trials

- Pilot apparatus – fluidised bed system or upflow chromatography column
- 2.4m high, 1.9m diam
- Distribution plate, gravel bed and sparger in base
- Designed upflow 10m/hr in column and 5m/hr in disengagement zone
- Plumbed into Mulgrave tank farm and can be integrated into normal production

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# Typical operating conditions

| | |
|---|---|
| Column height | 5000 mm |
| Syrup flow rate | 20 $m^3/h$ |
| Settled bed volume (calculated) | 3.98 $m^3$ |
| Fluidised bed volume (calculated) | 9.24 $m^3$ |
| Resin inventory (as suspension) | 3800 L (initial) |
| Operating temperature | 60 – 70°C |
| Approximate resin mass in column | 836 kg (initial) |
| Average contact time | Around 5 min at 20 $m^3/h$ flow |
| Average syrup brix | 40 – 45 |

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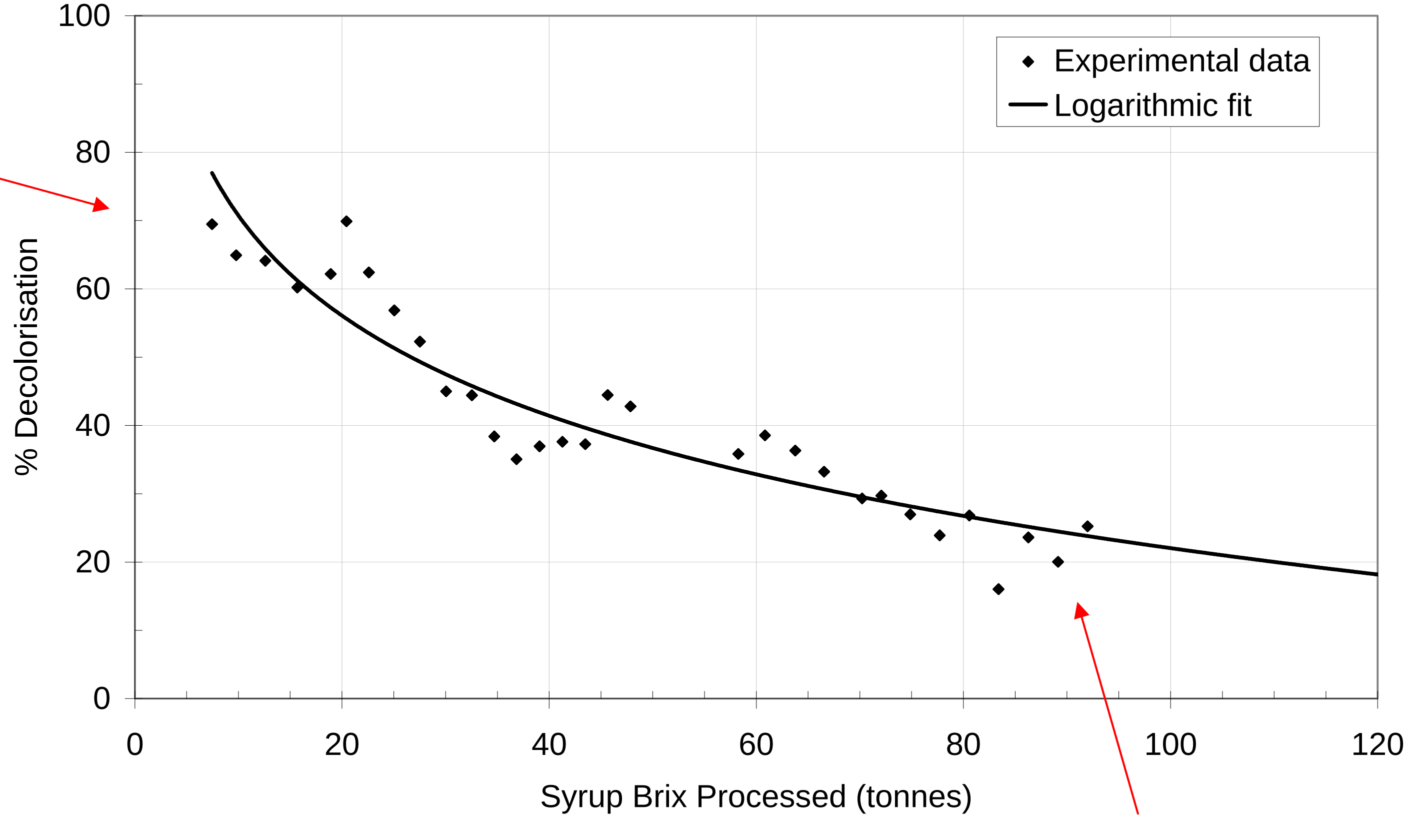
# Typical exhaustion experiment

**70% initial decolourisation**




**20% final decolourisation (60 bed volumes)**

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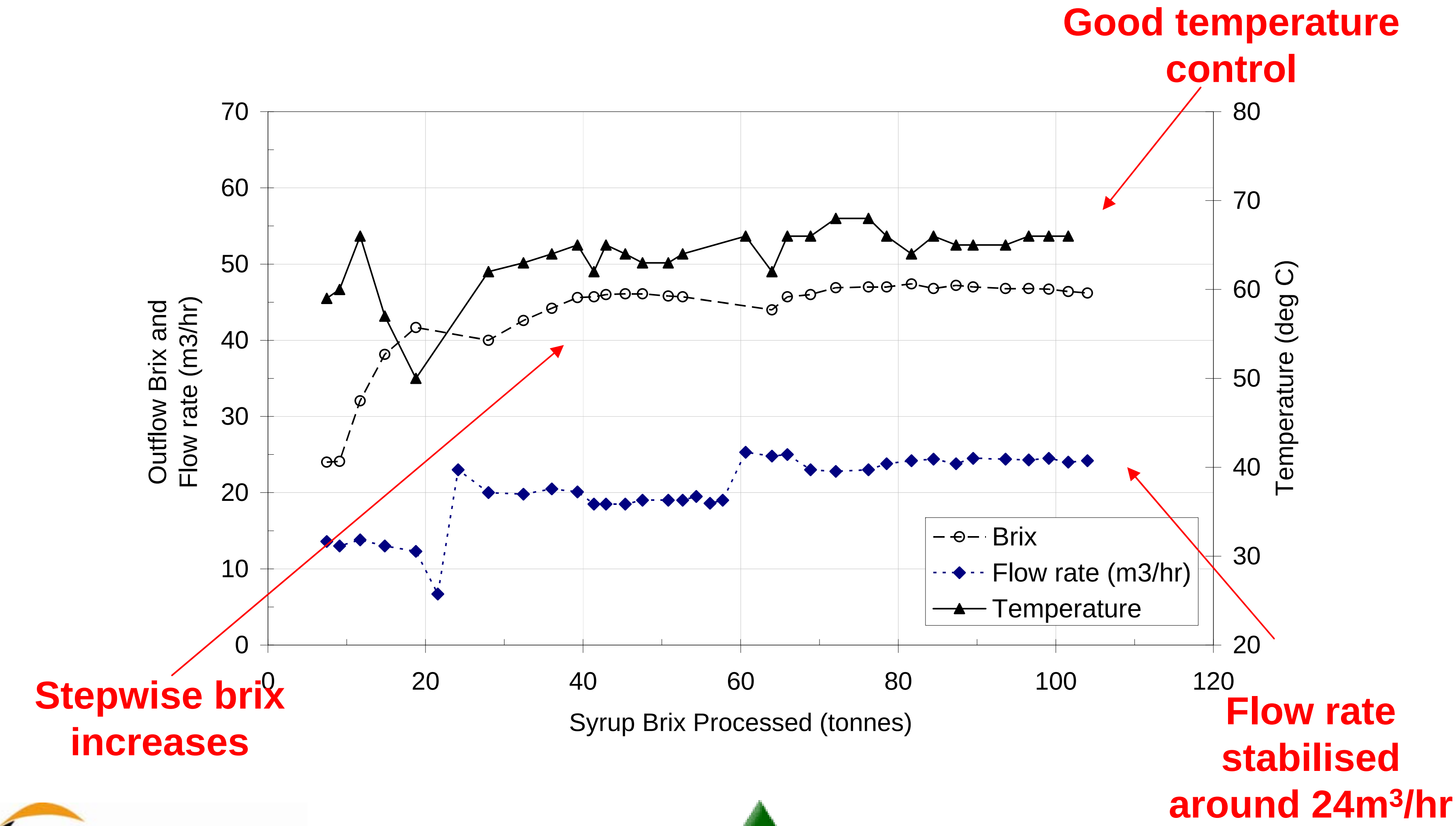
# Typical exhaustion experiment

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# Take home messages

- **Higher brix streams than 45 Brix cannot be treated under these conditions (viscosity, resin settling rates, loss of resin at overflow)**
- **Reproducible decolourisation behaviour across short (2hr) and long runs (12hr) – results not shown**
- **Pilot results confirmed laboratory studies**
  - **Logarithmic relationships between % decolourisation and both resin concentration and syrup brix**
- **One-off pan boiling experiment showed addition of decolourised syrup will result in lower raw sugar colour**
  - **Needs confirming on full pan scale**

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# Plans for 2008

***Process Optimisation***

- Demonstrate on factory scale, that reductions in sugar syrup colour will reduce raw sugar colour
- Test suitability on remelt and recycle streams as potential large benefits are available
- Examine CSTR design as well as fluidised bed

***Future Process Design***

- Based on results, design large scale automated process for a market ready technology package

***Commercial Framework and Product Support Requirements***

- Establish necessary delivery protocols, accreditations and early commercial arrangements for market entry

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# Acknowledgements

| | |
|---|---|
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