

HybridICE[®] filter: ice separation in freeze desalination of mine waste waters

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ABSTRACT

Freeze desalination is an alternative method for the treatment of mine waste waters. HybridICE[®] technology is a freeze desalination process which generates ice slurry in surface scraper heat exchangers that use R404a as the primary refrigerant. Ice separation from the slurry takes place in the HybridICE filter, a cylindrical unit with a centrally mounted filter element. Principally, the filter module achieves separation of the ice through buoyancy force in a continuous process. The HybridICE filter is a new and economical means of separating ice from the slurry and requires no washing of ice with water. The performance of the filter at a flow-rate of 25 L/min was evaluated over time and with varied evaporating temperature of the refrigerant. Behaviours of the ice fraction and residence time were also investigated. The objective was to find ways to improve the performance of the filter. Results showed that filter performance can be improved by controlling the refrigerant evaporating temperature and eliminating overflow.

Key words | filter, HybridICE[®], salt removal, yield

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INTRODUCTION

The purpose of this work was to establish the effect of mass ice fraction, ice bed residence time in the filter, and evaporating temperature of the refrigerant on the performance of the HybridICE[®] filter. In addition, the study aimed to describe the behaviour of ice in the filter in order to find ways to improve the performance of the filter.

The principal problem in a freeze desalination process may be the separation of ice crystals from the concentrated brine (Lu & Xu 2010). Separation devices and processes can be classified as: presses, gravity drainage, centrifuges, filter, and wash columns. The most common technique is the wash column, which exists in three different types, namely gravity, mechanical or hydraulic depending on the transport mechanism of the slurry in the filter (Oord-Knol *et al.* 2002). However, as the name implies, the wash column requires washing with water. Washing ice in the wash columns has several issues that include channelling, viscous fingering, and ice pack clogging (Qin *et al.* 2008). Another issue with wash columns is solid carry over (Rahman *et al.* 2006). Filtration has proven less effective for crystal separation and it cannot be used for washing the crystals. In many cases, screens or filters have a history of freezing-up (brine freezing

in the openings of the lace) (Rahman *et al.* 2006). Other techniques include use of rotating drums (Keus 1996) and horizontal cross filtration (Dickey *et al.* 1998), but these also require the introduction of washing fluid. However, use of a technique whereby compressive force is applied on the ice layer has been successfully achieved without the addition of washing fluid. The Soussloff *et al.* (1966) process actually combined the use of compression of the ice layer and vacuum to effect ice separation from the slurry. The HybridICE filter is an economical, easy-to-operate piece of equipment that separates the ice from the slurry by buoyancy force, without the need to wash with water.

HybridICE freeze crystallization is an emerging technology for treating complex mine and industrial waste water (process flow-diagram shown in Figure 1). An integral part of the HybridICE process is isolating the suspension of ice crystals from the process waste water in a static filter. The HybridICE filter (Figure 2) is a crucial process component in HybridICE technology. In principle, the slurry of brine and ice is pumped into a vertical column, which creates a piston ice extrusion. The filtering process undergoes three basic steps, namely, a zone to allow the ice to settle due to

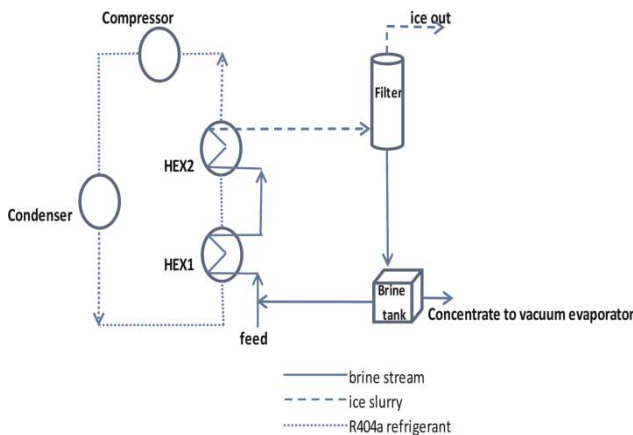


Figure 1 | HybridICE technology process diagram (Mtombeni et al. 2013).



Figure 2 | HybridICE filter in operation (Adeniyi et al. 2013b).

the buoyancy force, a filtering zone that isolates a substantial amount of process concentrate, and a purification zone that allows the separation of the remaining concentrate by gravity. The HybridICE filter is an approach that filters the ice particles as pure frozen water in a continuous process without requiring washing. This characteristic reinforces the economic feasibility of this freeze crystallization technology (Adeniyi et al. 2013b).

Ice formation thermodynamics

This work focused on the filtering process in the HybridICE filter. However, it was necessary to understanding the thermodynamics of ice formation because the performance of the filter depends on the quality of ice generated in the crystallization process.

Crystallization can only occur if supersaturation is created, which acts as the driving force. Nucleation occurs when the solution is supersaturated and then the crystal can grow to larger sizes (Van der Gun et al. 2001).

Supersaturation describes the difference between the current state of a system and the state that it would be in were the system in thermodynamic equilibrium. Two different properties can be used to define the deviation from equilibrium: concentration and temperature. In terms of concentration, supersaturation (ΔC) is the difference between the actual concentration (C) and the equilibrium concentration (C_{eq}) at that temperature. In terms of temperature, undercooling or supercooling (ΔT) is defined as the difference between the actual temperature (T) and the temperature that a system with this concentration would have if it were in equilibrium (T_{eq}). Crystallization can be achieved by cooling, in which case the driving force is created by withdrawing heat from the solution, a process also known as freeze crystallization. In the HybridICE process, the interest is on ice formation, which begins at the equilibrium freezing point where liquid to solid transformation occurs. The formation of ice in the solution will not start at 0°C because of the presence of salts that act as a freezing point depression substance. Thus, a significant energy barrier must be surmounted by applying a large driving force. The existence of an energy barrier is demonstrated by the continuous withdrawal of sensible heat below 0°C without the occurrence of a phase change. The supercooling results in a thermodynamically unstable state until submicron water aggregates form, leading to the suitable interface necessary for a massive liquid-to-solid transformation (Petzold & Aguilera 2009).

Large crystal sizes are obtained by minimizing the rate of nucleation of new crystals relative to the nucleation rate of existing crystals. The nucleation rate has been observed to be proportional to the number of ice crystals present and also has been seen to increase sharply with overall undercooling in the range of 0.3 to 8°C (Brian 1968).

Figure 3 shows the relationship between temperature and cooling time. Waste water cools rapidly until temperature, T_L ,

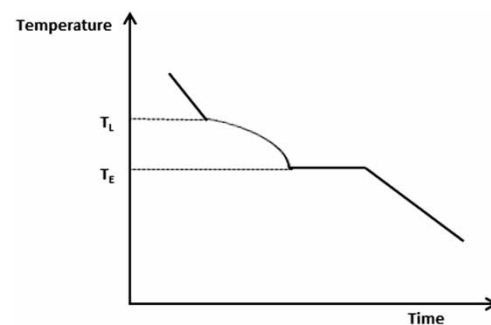


Figure 3 | Temperature plotted as a function of cooling time (University of Cambridge 2011).

the equilibrium freezing point (first ice point), is reached where ice formation begins. Thus, there are two phases formed, solid and liquid. The rate of cooling will now become slower until the eutectic point, T_E , is reached where salts crystals will begin to precipitate with the solid ice.

This therefore implies that the supercooling must be controlled, not only for crystal size, but to prevent salt from crystallizing with the ice and to enhance salt removal in the filter.

MATERIALS AND METHODS

The yield of ice was the mass of ice produced, in kg/min. Salt removal was calculated based on the electrical conductivity of the feed brine C_f and the electrical conductivity of the melted ice sample C_i

$$\text{Salt removal(\%)} = \frac{(C_f - C_i)}{C_f} \times 100\% \quad (1)$$

$$\text{Ice fraction} = \frac{\text{Mass of ice}}{\text{Mass of slurry}} \quad (2)$$

Electrical conductivity measurement was used for the chemical analysis (Wolkersdorfer 2012). The electrical conductivity of water estimates the total amount of solids dissolved in water. The electrical conductivity is a good indicator of the total salinity. The electrical conductivity of the water can be determined in a quick and inexpensive way, using portable meters. The relationship between concentration and electrical conductivity is linear for sodium chloride solution of less than 10% (IC Controls 2005).

Dickey *et al.* (1989) described a method of determination of ice fraction through density measurements. This method is limited by the procedure of measurement of the slurry density since density and solute concentration in the slurries are not available for many solutes of interest. Oosthuizen (2000) described another method known as the calorimetric method. This is done by stirring an ice slurry sample of known mass and temperature with hot water of a known mass and temperature. This method is also limited by the many avenues for loss that can affect the accuracy of the result. Kousksou *et al.* (2009) described the ice-filtration method, which involves taking a sample of the slurry and passing it through a filter. This process is simple, fast and minimizes the loss of ice so it was used for mass ice fraction measurement in this work.

Feedstock water

The brine used for the experiment was a sodium chloride solution prepared with tap water and sodium chloride salt (up to 4% by mass). The initial electrical conductivity of the prepared solution was 50.9 mS/cm.

Apparatus

The apparatus used for the experiments included the pilot HybridICE plant (situated at the Soshanguve Campus of the Tshwane University of Technology) and the laboratory scale 'baby' HybridICE unit. The operation of both is the same but the pilot plant handles a higher volume of process water. For the pilot plant, cooling of the process water to generate ice slurry using a suspension freeze crystallization technique takes place in two heat exchangers in series employing R404a as the refrigerant, which is not in direct contact with the process water. The heat exchangers are configured in series to facilitate nucleation in the first heat exchanger and ripening in the second heat exchanger. The baby unit uses only one heat exchanger. The outer part of the filter in the baby HybridICE is made of perspex, which allowed the formation of the ice bed to be clearly seen. The mass ice fraction was determined by filtration using a coffee-plunger. Ice bed residence time in the filter refers to the time taken for the ice bed to move through the filter. It is an indication of the time spent by the ice bed in the filter. The effect of residence time was measured by improvising a bucket with an opening to allow for draining of ice. This was because it was difficult to measure and vary the residence time in the filter on the pilot plant. The only difference was that the ice bed was in motion in the filter while it was static in the bucket, but the draining effect was the same, largely due to gravity. The plan was to determine the effect on the purity of the ice if the ice bed was allowed a longer residence time in the filter. The purity of the feed solution and the generated ice was measured by the value of the electrical conductivity.

EXPERIMENTAL

Salt removal and yield behaviour with time

Brine was fed to the HybridICE pilot plant at a flow-rate of 25 L/min. The evaporating temperature of the refrigerant was set at -10°C for each of the heat exchangers. Ice was collected at the filter chute after harvesting with the scraper

ejector mechanism. Samples of ice produced were taken, allowed to melt and reach room temperature before measuring electrical conductivity. Ice was also collected and weighed to determine the yield of ice. The mass ice fraction was determined by filtration. A sample of slurry was taken from the filter bypass and weighed; the slurry was then slowly filtered using the coffee plunger, and the ice part of the slurry was weighed.

Effect of evaporating temperature of the refrigerant

The above procedure was repeated when the evaporating temperatures of the refrigerant were varied as follows:

1. When the refrigerant evaporating temperature of the two heat exchangers were set at the same values.
2. When the refrigerant evaporating temperature in the first heat exchanger was kept constant and that of the second heat exchanger was varied.
3. When the refrigerant evaporating temperature in the first heat exchanger was varied and that of the second heat exchanger kept constant.

Effect of residence time of the ice bed in the filter

The bucket was filled with wet ice and allowed to drain over time. Ice samples were taken every 20 min. Electrical conductivity measurements were taken on the ice samples after allowing the ice to melt and reach room temperature.

Ice behaviour in the filter

The principal force behind the separation of ice from the slurry in the filter is buoyancy force. Ice, being less dense than the cold brine, floats on the cold brine in the static filter. Ice behaviour refers to the way the ice bed is formed inside the filter. Ice behaviour in the filter was studied using the baby HybridICE apparatus.

RESULTS AND DISCUSSION

As shown in Figure 4, the ice mass fraction increased with time but tended to fluctuate between 0.15 and 0.25 as time increased. This arose because the brine was being processed in a continuous mode at a constant flow-rate and refrigerant evaporating temperature. As the first ice point was reached, the heat removed led to the formation of ice and the temperature would remain constant. If the evaporating temperature

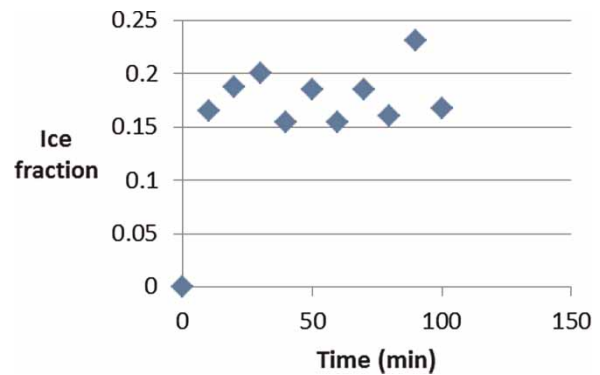


Figure 4 | Ice fraction formation over time.

of the refrigerant was adjusted, the driving force would remain constant. If the ice fraction must increase significantly then the evaporating temperature of the refrigerant or the brine flow-rate must be lowered.

Ice yield increased with an increase in the ice fraction; this was the reason behind the pattern in Figure 5. As more ice was formed in the slurry, more ice was available to be separated in the filter leading to a greater yield of ice from the filter. Although it was desirable to have a high yield from the process, behaviours of the slurry with an ice fraction higher than 25% made the separation of ice from the slurry in the filter difficult. When the ice slurry had between 0 and 25% ice fraction, it flowed like conventional chilled water; thus the ice could be easily separated from the slurry by buoyancy in the filter. At ice fractions higher than 25%, the slurry behaved like paste which made separation in the HybridICE filter difficult (Environmental Process Systems Limited). Moreover, when the ice fraction was higher than 25%, there was a tendency for the salt content of the ice crystals in the slurry to increase due to salt becoming trapped within the ice crystals.

Figure 6 shows that salt removal increased with residence time; the result also implied that ice purity is affected by its

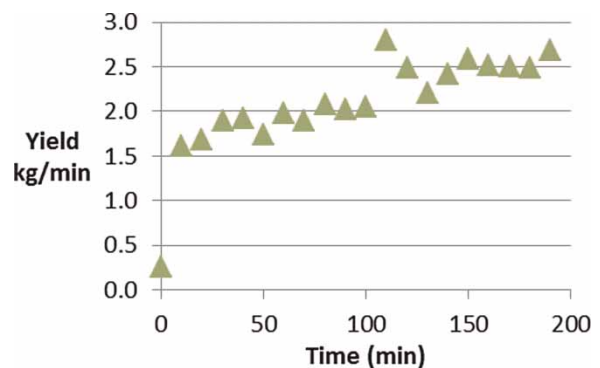


Figure 5 | Ice yield as function of time.

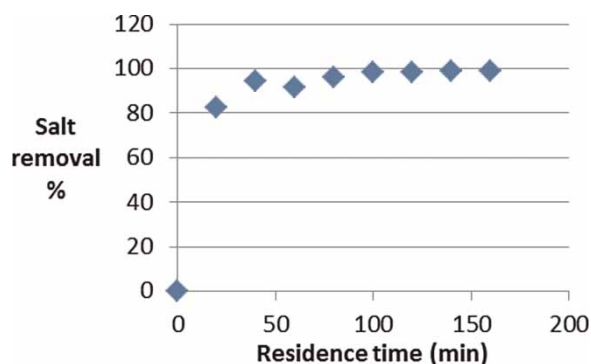


Figure 6 | Salt removal plotted as a function of residence time.

'wetness'. The wetter the ice, the more impure it is, because the salt content is rejected into the solution when ice is formed. When the ice crystal is wetted by the concentrated solution, causing the ice surface to be surrounded by the mother liquid, it reduces its purity despite the fact that the ice crystal itself is pure. This is because when retained mother liquor of low purity dries on the product, contamination results. The extent of the contamination depends on the amount and degree of impurity of the mother liquor retained by the crystal (McCabe *et al.* 1993). Oswald ripening occurs in the ice bed; this is the phenomenon in which smaller particles in solution dissolve and deposit on larger particles in order to reach a more thermodynamically stable state wherein the surface to area ratio is minimized. This phenomenon increases with an increase in the ice bed residence time in the filter which leads to bigger ice crystals. The longer the ripening, the bigger the crystals and the narrower the crystal size distribution, therefore enabling better filtrability. Bigger ice crystals exhibit lower interfacial surface tension, due to the lower surface area to volume ratio of the ice crystals, and thus so they easily reject the concentrated solution. This implies that the more the ice bed is allowed to grow with increased residence time, the purer the ice. Increases in residence time also increase the effect of the gravity force in draining the ice bed.

Optimum temperature range

The first ice point of the saline solution used for the experiment, as read through the data acquisition system on the pilot plant, was about -1.7°C . Supercooling reduces with an increase in the evaporating temperature of the refrigerant.

Figures 7 and 8 show the behaviour of salt removal and yield when the refrigerant evaporating temperatures in each heat exchanger were the same. Salt removal increased with an increase in evaporating temperature while yield was

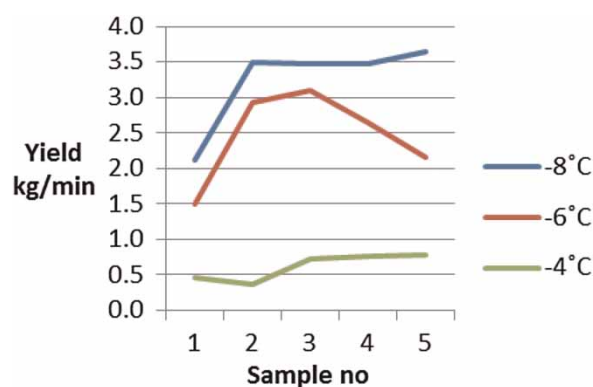


Figure 7 | Ice yield plotted at different evaporating temperatures.

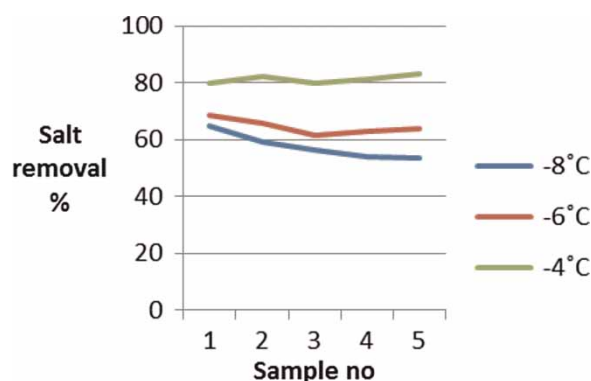


Figure 8 | Salt removal at different evaporating temperatures (Adeniyi *et al.* 2013a).

reduced. Ice formation is a function of supercooling, which is driven by the temperature difference between the refrigerant and the first ice formation temperature (first ice point) of the brine solution. At the first ice point, ice formation begins. For this to be achieved, the temperature of the refrigerant must be lower than the first ice point. The lower the refrigerant temperature, the faster the first ice point is reached and the higher the rate of ice formation. The rate of ice formation is reflected in the yield of ice from the filter, which is the mass of ice harvested per minute. An increase in the rate at which the ice bed is formed reduces the residence time of the ice bed in the filter. More ice is formed rapidly at higher temperature differences due to the higher driving force (supercooling) but also because more salts are trapped in the ice crystals. The more the difference between the evaporating temperature of the refrigerant and the first ice point of the brine, the higher the tendency for the concentrated mother liquor to be trapped between the ice crystals. It was also noted that there were no significant differences in salt removal and yield at evaporating temperatures of -8 and

-10°C . This was because there were no significant differences in the temperature gradient at these evaporating temperatures.

Figures 9(a) and 9(b) are bar charts showing the behaviour of the salt removal and ice yield when the evaporating temperature of the refrigerant in the first heat exchanger was kept constant while the refrigerant evaporating temperature in the second heat exchanger was varied. The bar chart is used for easy comparison of data.

There was a steady increase in salt removal as the refrigerant evaporating temperature in the second heat exchanger increased when the evaporating temperature of the refrigerant in the first heat exchanger was kept at -4°C . Average salt removal was almost the same for lower refrigerant evaporating temperatures (-10 and -8°C) in heat exchanger 2 at refrigerant evaporating temperatures of -4 , -6 and -8°C in heat exchanger 1. This was because the brine was brought closer to ice formation as refrigerant evaporating temperatures in the first heat exchanger decreased due to a higher temperature gradient for cooling. It was interesting to note that salt removal was almost as high when the refrigerant evaporating temperature in heat exchanger 1 was -8°C as it was when it was -4°C at a refrigerant evaporating temperature of -4°C in heat exchanger 2. Moreover, salt removal was highest when the refrigerant evaporating temperature in heat exchanger 1 was -6°C and the refrigerant evaporating temperature in heat exchanger 2 was kept at -4°C . This was because less salt was trapped in the ice when the refrigerant evaporating temperature in heat exchanger 2 was closer to the first ice point.

Ice yield steadily decreased as the refrigerant evaporating temperatures increased in heat exchanger 2 when the refrigerant evaporating temperature in heat exchanger 1 was at -4°C . This maintained the trend as shown in Figure 7

when the refrigerant evaporating temperatures in each of the heat exchangers were the same. However, it was not the case when the refrigerant evaporating temperatures of heat exchanger 1 were -8 and -6°C , respectively. Average ice yield reduced with an increase in evaporating temperature (lower supercooling) while salt removal increased with an increase in evaporating temperature. This followed the same trend when evaporating temperatures in each of the heat exchanger were the same.

Figures 10(a) and 10(b) show the trend for ice yield and salt removal when the refrigerant evaporating temperature of the first heat exchanger was kept constant while that of the second heat exchanger was varied.

For every value of refrigerant evaporating temperature in heat exchanger 1, the trend was similar in the average salt removal for the variation of the refrigerant evaporating temperature in heat exchanger 2. As the refrigerant evaporating temperature increased in heat exchanger 2, salt removal increased.

Average ice yield was lowest at the highest refrigerant evaporating temperature in heat exchanger 1 for all values of refrigerant evaporating temperature in heat exchanger 2. The trend was almost the same as recorded when each heat exchanger had the same refrigerant evaporating temperatures.

Ice yield and salt removal were high at the refrigerant evaporating temperature of -6°C in the heat exchangers. This was about 4.3°C less than the first ice point of the process saline water.

In summary, for a process solution with a first ice point of -1.7°C , the evaporating temperature of the refrigerant in each heat exchange should be set at the same value and should be between -6 and -8°C for optimum ice yield and salt removal in the filter.

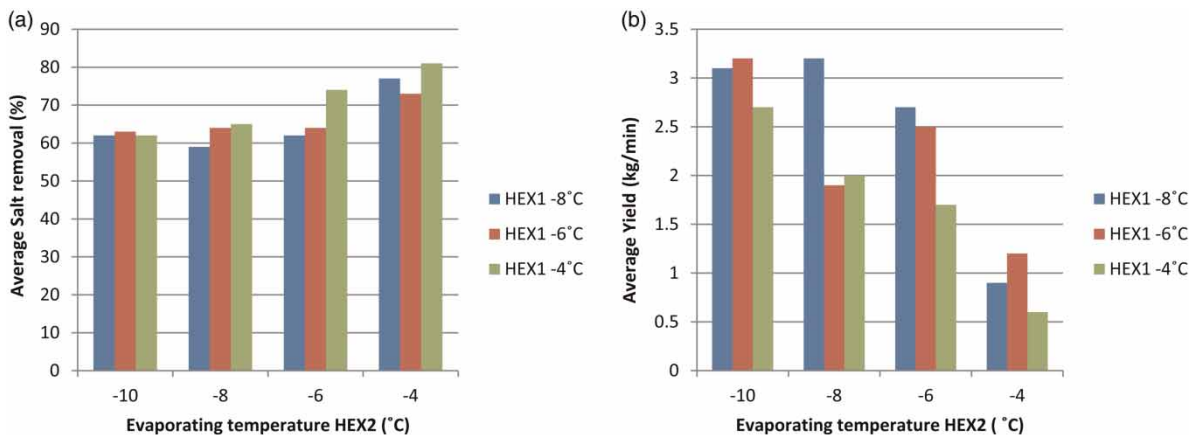


Figure 9 | Average salt removal and ice yield plotted against evaporating temperature HEX2 ($^{\circ}\text{C}$).

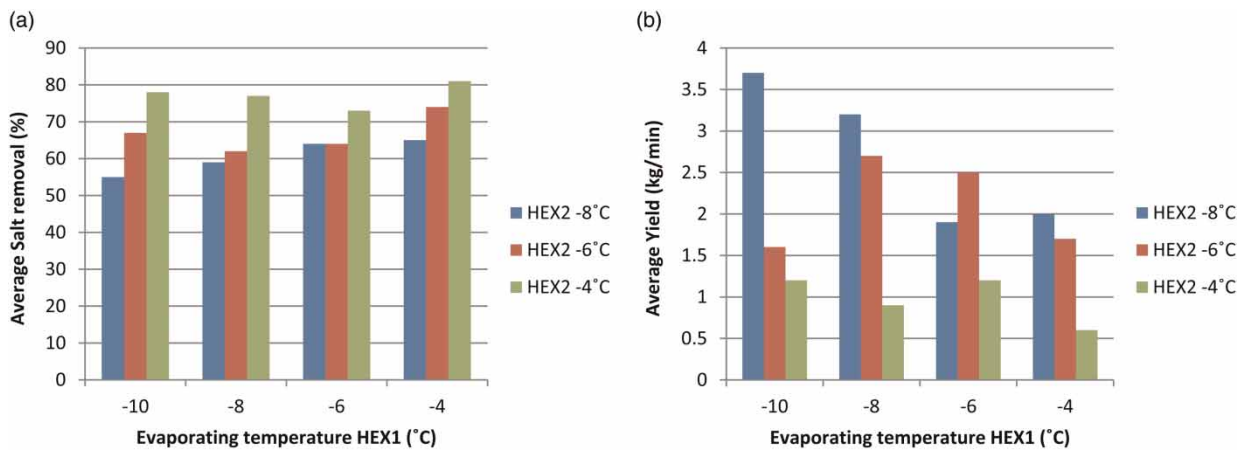


Figure 10 | Average salt removal and ice yield plotted against evaporating temperature HEX1 (°C).

Ice behaviour as observed on the baby HybridICE apparatus

The baby HybridICE apparatus is as shown in Figure 11. As the ice slurry flowed into the filter, the ice rose through the slurry to float at about the perforation in the filtering medium. An interface was created between the slurry and the ice at the perforation on the filtering element. It was observed that ice crystals showed a tendency to form around the filtering element and then grow. This caused a blockage in the perforation that impeded the outflow of the concentrated brine. The difference in the inflow of the slurry and the outflow of the concentrated brine caused the formation of a 'stagnant section' which grew with the increase in height of the ice bed, to ultimately result in an overflow. Three sections were eventually created in the filter; these were the ice bed, the slurry and the stagnant section. The degree of mixing between the ice bed and the concentrated brine in the stagnant section had a direct effect on the yield and purity of the ice from the filter. The



Figure 11 | Ice behaviour in the filter.

stagnant section needed to be kept at a minimal height so there was a need to create alternative openings for the excess concentrated brine to leave the filtration section to reduce the size of the 'stagnant section' to the minimum and avoid overflow (Adeniyi *et al.* 2013a).

CONCLUSION

The following conclusions were reached from the results of the experiment.

1. The performance of the filter is defined by the ice yield and salt removal.
2. The performance of the filter depends on the quality of the slurry generated in the heat exchangers. The quality of the slurry is defined by the ice fraction and purity of ice.
3. The lower the evaporating temperature of the refrigerant, the higher the ice fraction of the slurry and the higher the yield of ice harvested from the filter.
4. The lower the evaporating temperature of the refrigerant, the lower the purity of the ice in the slurry and the lower the salt removal from the ice in the filter.
5. For optimum ice fraction and purity of ice in the slurry, the refrigerant evaporating temperature in the heat exchangers should be set at a value lower than the first ice point of the process solution by a value between 4 and 6 °C.
6. For a saline solution with a first ice point of -1.7 °C, the refrigerant evaporating temperature should be the same and set at a value between -6 and -8 °C in each of the heat exchangers used for the cooling of the process water.

7. Purity of the ice harvested from the filter increases with an increase in the residence time of the ice bed in the filter.
8. Overflow must be eliminated from the filter to increase salt removal and ice yield.
9. The performance of the filter can be improved by controlling the refrigerant evaporating temperature in the heat exchangers and eliminating overflow in the filter.

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