CAUSTIC RECOVERY VIA MEMBRANE MICRO-FILTRATION

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Abstract

Spent caustic from chemical cleaning of evaporators and pre-heaters was cleaned by membrane micro-filtration. A rig supplied by Tongaat-Hulett Technical Management Department (TH-TMD) was refurbished and used for this purpose. It was determined that pretreatment was necessary to improve permeate flux rates and prevent clogging of the membrane. Experiments run over 6-18 hours showed that high trans-membrane pressures improved reduction of permeate viscosity and hence lowered suspended solids content but at the sacrifice of lower overall flux rates. Cross-flow velocity had a favourable influence on flux rates. The best operating point tested was a trans-membrane pressure of 400 kPa and a cross-flow velocity of 1.2 m/s. Chemical cleaning with weak caustic and acid solutions was necessary between runs.

Keywords: evaporator, caustic, membrane, effluent.

Introduction

Four South African sugar mills practise chemical cleaning of evaporators. The spent caustic is allowed to settle in a tank and the resulting sludge is washed, however not all the suspended solids are removed. There have been occurrences when high strength caustic has been dumped into the cooling water ponds due to high viscosities since the pumps could not handle the fluid. Tightening effluent policy coupled with increasing caustic prices has created a need to recover spent caustic and thereby reduce caustic effluent.

Method

The membrane rig supplied by TH-TMD (Figure 1) was used in a cross-flow mode under feed and bleed conditions (batch). A tubular metal membrane with a pore size of 0.1 µm and an effective area of 0.035 m^2 was employed. Initially, 100 litres of spent caustic was used but a single run lasted for a week. Hence a lower volume of 40 litres was used which limited the volumetric concentration factor[1] (VCF) to below 5. The NaOH concentration of the caustic was around 25% when supplied, but a later analysis showed that it had dropped to 14%, by partial conversion to the carbonate.

A schematic representation of the rig is shown in Figure 2. Various trans-membrane pressures (TMPs) and cross-flow velocities were tested. Permeate viscosity was used to monitor the success of the run. A target viscosity of 8 cP was established. Chemical cleaning of the membrane between runs was accomplished using alternating weak caustic and phosphoric acid cycles. Water flux rates were checked to confirm that the membrane was clean. Since the feed tank was heated by a steam coil evaporation did occur. This was compensated for by slow addition of water to the feed tank via a peristaltic pump (not shown in Figure 2).

[1] VCF is the ratio of the initial feed volume to the final volume of the retentate.

It proved necessary to pre-treat the caustic to remove the larger solids particles that would block the pumps, pipes and the membrane. This was done by pressure filtering the caustic through a 60 :m filter (C-massecuite centrifugal screen), a potentially hazardous operation. However, diluting with hot water and allowing it to stand resulted in little or no residue. Hence adding hot dilution water to the caustic tanks and allowing to stand could be used to replace pre-treatment and in-line cartridge filters could be used to remove large particles which would damage the membrane.

Figure 1. The membrane filtration rig.

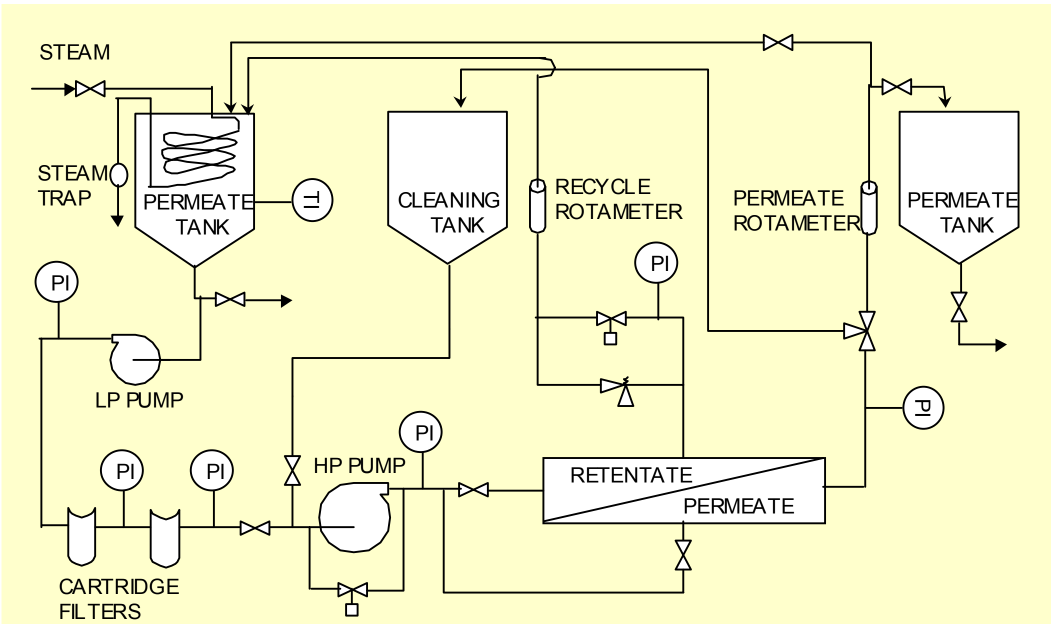

Figure 2. Schematic representation of the membrane filtration rig.

Results

Preliminary testing revealed that TMPs higher than 1 MPa and operation below 85°C caused rapid fouling of the membrane. Hence all runs were conducted with temperatures above 85°C and TMPs ranging from 280-600 kPa. Table 1 shows condensed results from trials at TMPs of 280 and 400 kPa. Figure 3 shows permeate flux rates for the duration of the run. Clearly the higher TMP improved viscosity reduction but caused a lower overall flux rate. It was decided to test varying cross-flow velocities at a TMP of 400 kPa (Figure 4). Table 2 shows condensed results. The higher cross-flow velocities improved flux rates at no sacrifice to viscosity reduction. Thus far the best operating point was at a TMP of 400 kPa and a cross-flow velocity of 1.2 m/s. A single test was run at a TMP of 600 kPa and a cross-flow velocity of 1.4 m/s. The results did not compare well to the best operating point.

Table 1. Results of varying trans-membrane pressure.

Trans-membrane Pressure (TMP), kPa	280	400
Feed Viscosity, cP	11	12
Permeate Viscosity, cP	9	8
Average Flux Rate (8 hour period), l/m^2h	89	78
Cross-flow velocity, m/s	1	1
% Caustic Recovery	52	67
Volumetric concentration factor (VCF)	2.1	3.5

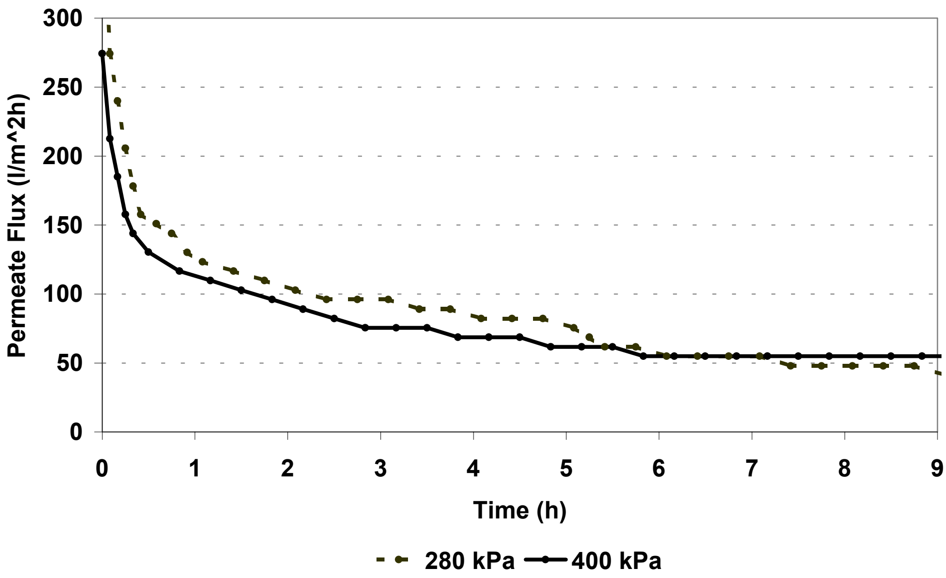

Figure 3. Flux rates at varying TMPs at a cross-flow velocity of 1 m/s.

Table 2. Condensed results at different cross-flow velocities.

Cross-flow velocity, m/s	0.7	1.0	1.2
Trans-membrane pressure, kPa	400	400	400
Average flux rate (over an 8 hour period), l/m^2h	60	79	93
Feed viscosity, cP	10	12	12
Permeate viscosity, cP	9	9	8
% Caustic recovery	67	68	63
Volumetric concentration factor	3.2	3.5	2.9

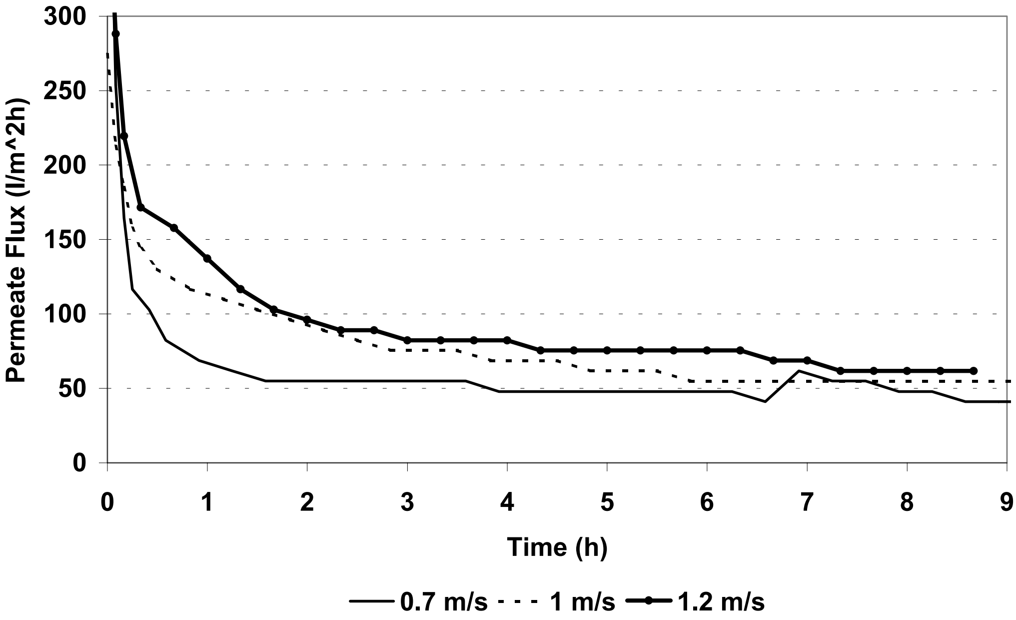

Figure 4. Flux rate curves for varying cross-flow velocities at a TMP of 400 kPa.

Discussion

The best operation point within the range of parameters tested for a batch process was determined. These results may not be directly applicable to a full-scale unit as batch results cannot be used for a continuous plant. However, as caustic is used in batch cycles, being stored in tanks between cycles, one tank at a time could be processed in a batch mode. In addition, lower permeate flow rates can be used over a longer period minimizing the membrane area required. A movable rig or a system of pipes and valves can be used in this regard. The 8 and 11 hour average flux rates of 93 l/m^2h and 82 l/m^2h provide a guide to the expected outlet flows and membrane cleaning intervals.

The caustic recoveries for each run were influenced by the volumes of permeate and feed since the feed and permeate caustic strengths were only slightly different. It was necessary to maintain a small volume in the system to allow for a constant cross-flow velocity. Hence higher recoveries are possible if a larger amount of feed is used and thus higher volumetric concentration factors could be achieved.

Conclusion

Spent mill caustic can be partially recovered using a tubular micro-filtration membrane under cross-flow. An eight-hour average flux of 93 l/m^2h is possible at a trans-membrane pressure of 400 kPa and a cross-flow velocity of 1.2 m/s at 85^{o}C. Pretreatment of the feed and chemical cleaning between runs are necessary steps.

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