

## Effluent Treatment Using Evaporation and Crystallisation Technology

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The authors will demonstrate with the use of case studies how evaporation and crystallisation technology may be used to treat effluent streams. Particular emphasis will be given to the advantages of keeping differing effluent streams separate to ease their treatment. This approach allows the production of valuable by-products that can offset the cost of effluent treatment. Consideration will also be given on how to minimise the energy usage of evaporation plants.

### 1. Crystallisation

The following case studies demonstrate the advantage of altering the conditions under which crystallisation takes place to produce multiple products.

#### 1.1 Case Study 1: Vinasse Concentration with Depotassification

Vinasse is an aqueous effluent containing both organic and inorganic components from alcohol or yeast production using sugar beet molasses as the feedstock. A typical analysis for vinasse from yeast production is given in Table 1.

Table 1: Typical Vinasse Composition

Item	Units	Quantity
Feed vinasse total solids	%wt	6.00
Calcium content of solids	%wt	2.90
Potassium content of solids	%wt	12.50
sodium content of solids	%wt	3.25
Chloride content of solids	%wt	4.10
Sulphate content of solids	%wt	6.50
Organics content of solids	%wt	70.75

Clearly this liquor would cause serious environmental problems if discharged directly into water courses. Treatment by aerobic and/or anaerobic digestion does not produce sufficiently clean effluent for current effluent discharge standards. Evaporation of this liquor produces syrup that than be used as animal food, but the potassium level must be kept below 3%, otherwise it is unpalatable to animals. By addition of sulphate the potassium can be removed from the concentrated vinasse as potassium sulphate crystal,

which may be sold as a fertilizer. Crystallisation needs to take place at low concentration to produce a saleable by-product, because the crystals produced at high concentration and high liquor viscosity are fine ( $<100\ \mu\text{m}$ ). Therefore to enable large crystals to be produced, a two stage crystallisation process is used. The fine crystals produced at high concentration are separated from the product vinasse, and recycled back to the feed. The block flow diagram Figure 1 below demonstrates the concept. Two stage crystallisation enables a saleable product to be produced.

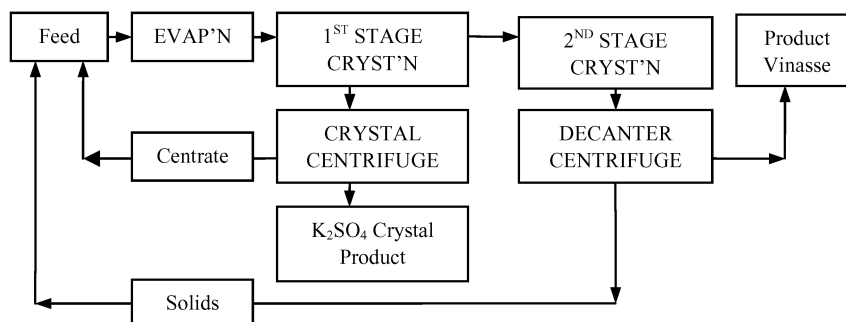


Figure 1. Block flow diagram, vinasse evaporation plant

### 1.2 Case Study 2: Vinasse Concentration with Depotassification and Desalting

This process is a variation on the Case Study 1, specifically for vinasse from yeast plants. It is common in yeast plants to spray NaCl brine solutions onto the yeast rotary vacuum filter, to reduce the water content of the yeast. This high chloride effluent is normally combined with the rest of the vinasse and sent to aerobic or anaerobic treatment. However if an evaporation process is to be used this would cause corrosion problems in the high temperature, high concentration part of the plant. Instead of type 316 stainless steel, exotic alloys such as titanium and Monel would be required. In addition the crystal product would be contaminated with NaCl.

To avoid these problems, on a recent plant that we installed in Turkey, these two streams were kept apart, see block flow diagram, Figure 2. A separate evaporator body is dedicated to the high chloride effluent and is used to produce crystalline salt, which is discarded. As the high chloride effluent evaporator is operating at low temperature, cheaper duplex stainless steel construction is used instead of titanium and Monel. To maintain the energy efficiency of the evaporation plant, the vapour side of the evaporators is combined into a quintuple effect configuration, with four evaporator bodies operating on vinasse, and one on high chloride effluent. Separating the feed streams enable cheaper materials of construction to be used and prevents contamination of the products.

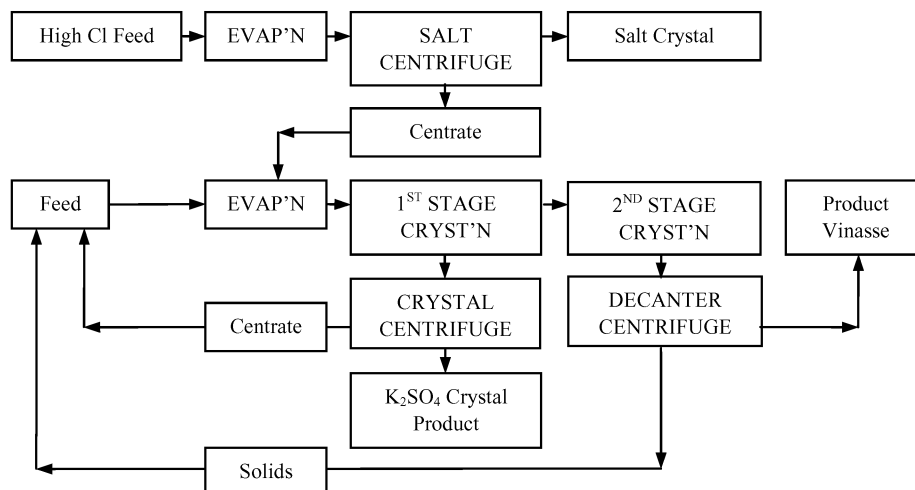


Figure 2. Block flow diagram, vinasse evaporation plant with desalting

### 1.3 Case Study 3: Production of Fertilizer from Straw Fly Ash

This plant treats the fly ash produced from burning straw in a power station. The ash is high in potassium, chloride and sulphate and produces a solution containing principally 27% KCl and 3%  $K_2SO_4$  when subjected to hot leaching followed by precipitation of heavy metals and filtration. The published solubility data by Seidell (1958) is given below.

Examination of the solubility data indicates that on cooling of the hot feed brine,  $K_2SO_4$  crystal is produced, until the transition line concentration is reached, where a mixture of  $K_2SO_4$  and KCl is precipitated. A mixed product is produced which is difficult to sell as a by-product.

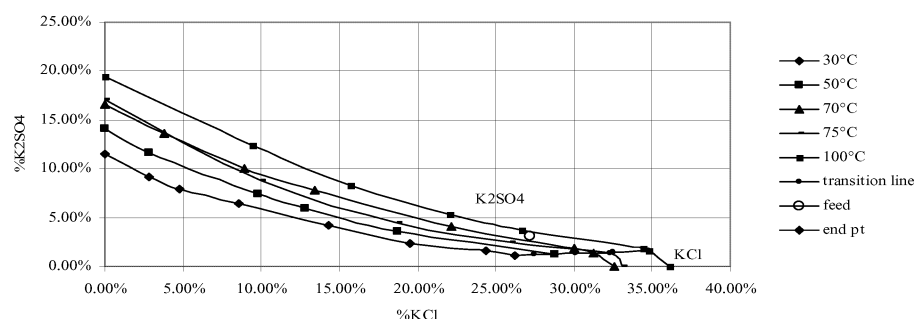


Figure 3. KCl- $K_2SO_4$  solubility in water

By using a cold leach initially, a stream containing 25% KCl can be produced, for sale as a liquid fertilizer. Alternatively it can be concentrated and crystallised to produce

solid KCl fertilizer. The remaining solids can then be subjected to a hot leach to produce a stream containing 15%  $K_2SO_4$  and 2.3% KCl. On cooling this stream produces only  $K_2SO_4$  crystals. Potassium sulphate is of higher value than potash and does not require drying, so the revenue produced from the plant is higher and the running costs are lower. Two stage crystallisation enables higher value products to be produced.

## 2. Evaporation

The following case studies demonstrate various methods employed to reduce or recover the heat used for evaporation. These are inter-effect heat recovery systems, use of electricity rather steam, waste heat, and thermal recompression.

### 2.1 Case Study 4: Integration of Steam Stripping with a Black Liquor Evaporator

Black liquor is the aqueous effluent produced in Kraft pulp mills from the pulping process. In large integrated pulp mills the black liquor is concentrated and burnt in a recovery boiler to produce high pressure steam, which is let down through a turbine to produce power to run the mill and low pressure steam to run the evaporator. The energy balance over the mill requires the use of multiple effect evaporators, typically sextuple effect. In addition the black liquor readily fouls the heat transfer surfaces at high concentration and it is common to provide standby evaporator bodies for the final concentration stage.

Methanol is present in the feed and this degasses in the evaporator to produce a polluted condensate that cannot be discharged without further treatment. The most polluted condensate from the evaporator is kept separate from the cleaner condensate and steam stripped to remove the methanol as vapour. The methanol vapour is fed to the recovery boiler where it is burnt and the energy recovered. However the stripper steam requirement upsets the mill energy balance. To overcome this problem, the stripping column can be integrated into the evaporator as shown in Figure 4 so that no extra energy is required. Part of the vapour from an evaporator body is used as stripping steam in the bottom of the column. The energy in the steam is recovered at the top of

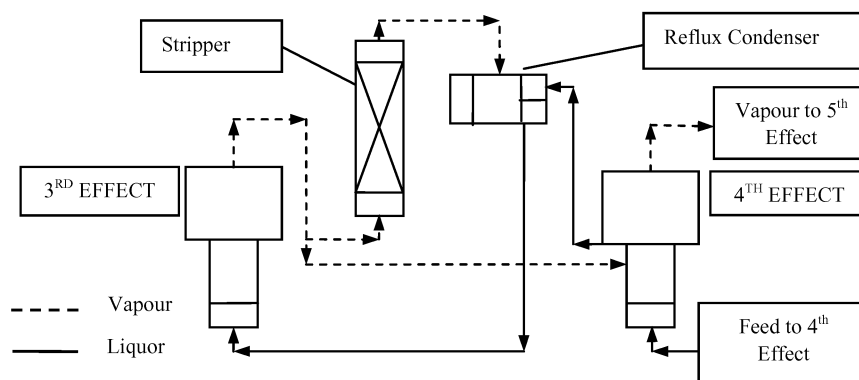


Figure 4. Condensate stripper flow diagram

the column by using black liquor as coolant in the reflux condenser. The heated black liquor is fed to the evaporator body and so the energy is recycled back to the evaporation plant. Wellman provided a nine body sextuple effect evaporator with an evaporation capacity of 540 te/h with such a system for a plant in Portugal.

## **2.2 Case Study 5: Conversion of Distillery Waste Evaporator from Multiple Effect to Mechanical Vapour Recompression (MVR)**

Wellman supplied a quadruple effect rising film evaporator on a wheat liquor distillery waste evaporator. The concentrated liquor is dried and is used for animal feed. As the potassium levels are low, there is no need for potassium sulphate crystallisation in this application. Recently the owners of the plant asked Wellman to convert the plant to MVR in order to reduce energy costs.

An MVR evaporator uses a compressor or fans to compress the vapour so that it may be reused as heating steam. The temperature difference available over the heat exchangers is limited due to the compression ratio that can be produced by these machines. In practical terms this means that more heat transfer surface is required for MVR evaporation plants for the same duty. So we need to supply additional heat transfer surface when converting to MVR. For this application we supplied an additional heat exchanger and converted the existing rising film evaporators to falling film. The conversion improved the heat transfer coefficient and also gave more stable operation.

The old quadruple effect plant required 0.29 tonnes of steam for 1 tonne of evaporation. The MVR plant requires no steam, but the fans absorb 29 kW per tonne of evaporation, representing a 40% saving on energy costs, which justified the extra capital cost.

## **2.3 Case Study 6: Pot Ale (Malt Distillery Waste) Evaporator using Waste Heat**

Pot ale is the waste left over from distillation to produce malt whisky. Wellman provided a small double effect evaporation plant to concentrate the waste for use as animal feed. The feed stock for malt whisky plants is barley, which produces a waste liquor easier to concentrate than wheat feedstock liquors. There was insufficient boiler capacity to run a conventional multiple effect evaporator and an MVR plant would have required a new transformer and electricity supply. The batch still condensers on the distillery were converted using tube inserts to produce hot water. The hot water is stored in a tank and is flash cooled in a two stage flash tank to provide heat to run the evaporator. The cooled water is returned to the condensers. The hot water tank is used as buffer storage to even out the supply of hot water to the evaporator. PLC control is used to match the evaporator demand with hot water availability. The flow diagram for the plant is shown below in Figure 5. This plant demonstrates that low grade heat can be usefully employed to save investment in additional utility supply.

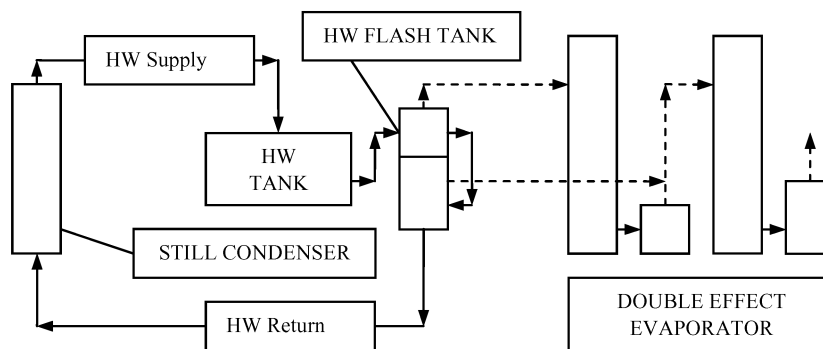


Figure 5. Flow diagram of an evaporator using hot water as the heat supply

#### 2.4 Case Study 7: Integrated Evaporator Treating Two Effluent Streams

Wellman supplied an evaporator to a catalyst manufacturer to handle two separate waste streams in one evaporation unit. Sodium disilicate was concentrated in a single effect thermal recompression evaporator. Thermal recompression uses a large steam ejector to compress process vapour using high pressure steam. Excess vapour from the sodium disilicate evaporator was used to run a triple effect evaporator concentrating sodium/ammonium nitrate solution. The sodium/ammonium nitrate was pre-concentrated in a stainless steel cooling tower before being fed to the evaporator to further reduce energy usage. Combining the two duties in one evaporation plant enabled greater energy savings to be made.

### 3. Technical and Economic Criteria in Specifying Evaporation and Crystallisation Plant

The following criteria need to be considered when specifying evaporation and crystallisation plant:

1. Utilities (steam cost, pressure and availability, power cost and availability, cooling water availability and maximum supply and return temperatures, availability of low grade heat);
2. Physical properties (feed composition, temperature, specific heat, thermal conductivity, heat of crystallisation, viscosity, boiling point elevation solubility data, presence of volatiles, foaming, presence of fouling compounds such as calcium);
3. Product specifications (maximum liquor operating temperature, product purity, crystal size);
4. Materials of construction, compressor type (centrifugal, fans, Roots blowers), site conditions, turndown required.

### 4. References

Seidell, A., 1958, Solubilities of Inorganic and Metal Organic Compounds. 4<sup>th</sup> ed. USA: American Chemical Society.