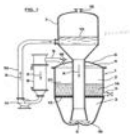

DESIGN OF INDUSTRIAL OSLO CRYSTALLYSER

A CASE STUDY

SALT PRODUCTION

Keywords: Sodium chloride, Oslo crystallizers, large particle size

SUMMARY

The design of an industrial OSLO crystallizer is based on experiments in the lab, in asmall scale pilot plant, in a semi industrial plant and in an industrial plant that has been operating for 20 years .The objective was to produce spherical particle of salt with a size of about 5 mm; the known state of the art allowed only to produce particle size of about 2mm maximum. A development was made for 2 years in lab and small pilot scale; an industrial plant was started in 1987

This paper describes the main problems encountered at all stages of development and how they were solved: choice of the main operating parameters, validation on all the steps, resolution of hydrodynamics problems and practical controls of nucleation, control of scaling

Most of the solutions adopted for production of large particle size of sodium chloride are also valid for other salts production and the OSLO technology can be applied for other salt production ; in some cases, very large particle size obtained can compete with usual crystallization + drying + size enlargement by compaction technology.

INTRODUCTION

Production of large spherical particle size of sodium chloride has been requested for market applications: a size of 5mm diameter was desirable

Known technology tells that a maximum size of 2 mm diameter can be reached (1), (2),(3)

In particular, the paper (2) describes 2 design of OSLO crystallizer:

- one classical design with only one dimension of fluidized bed of crystal : it was not possible to obtain particle size larger than 0.8 mm and there was large circulation of liquid in the bed mixing large and small particles destroying the classifying effect of the crystallizer
- One modified design called the «double circulation crystallizer”; two separate beds of crystals are formed; in both beds, a relatively intense circulation of brine and solid occurs.

In this second case, particle size of about 2 mm could be produced; it is not clear if it was produced at industrial capacity.

In addition, the author visited a large producer of sodium chloride who had large OSLO crystallizers to produce industrially large particle of sodium chloride: the normal salt had a particle size of 1.5 mm maximum.

So it became clear that an internal development was necessary in order to meet the market demand .

LABORATORY DEVELOPMENT

This laboratory development was made in a center of research

The research consisted mainly to determine the following parameters:

- The rate of growth of crystal versus the super saturation and dependence upon the temperature
- The width band of maximum super saturation and the limit of homogeneous nucleation
- The practical super saturation that can be operated safely ; in our case, a practical super saturation of maximum 0.5 g/kg of brine was chosen at a temperature around 100 °C
- The limit of crystallization of some impurities: as the purification of brine was not total for economic reason, there was a risk in some conditions of precipitation of calcium sulfate (CaSO4) and glauberite (CaSO4.Na2SO4). Limits were determined in order to control the purge of the system

SMALL PILOT DEVELOPMENT

A small pilot was operated for a while in order to solve the following problems:

- Choice of operating conditions :
    - Fluidization velocity : range of 200 to 500 m/h

    - Definition of polish of surface
    - Observations of hydrodynamic behavior

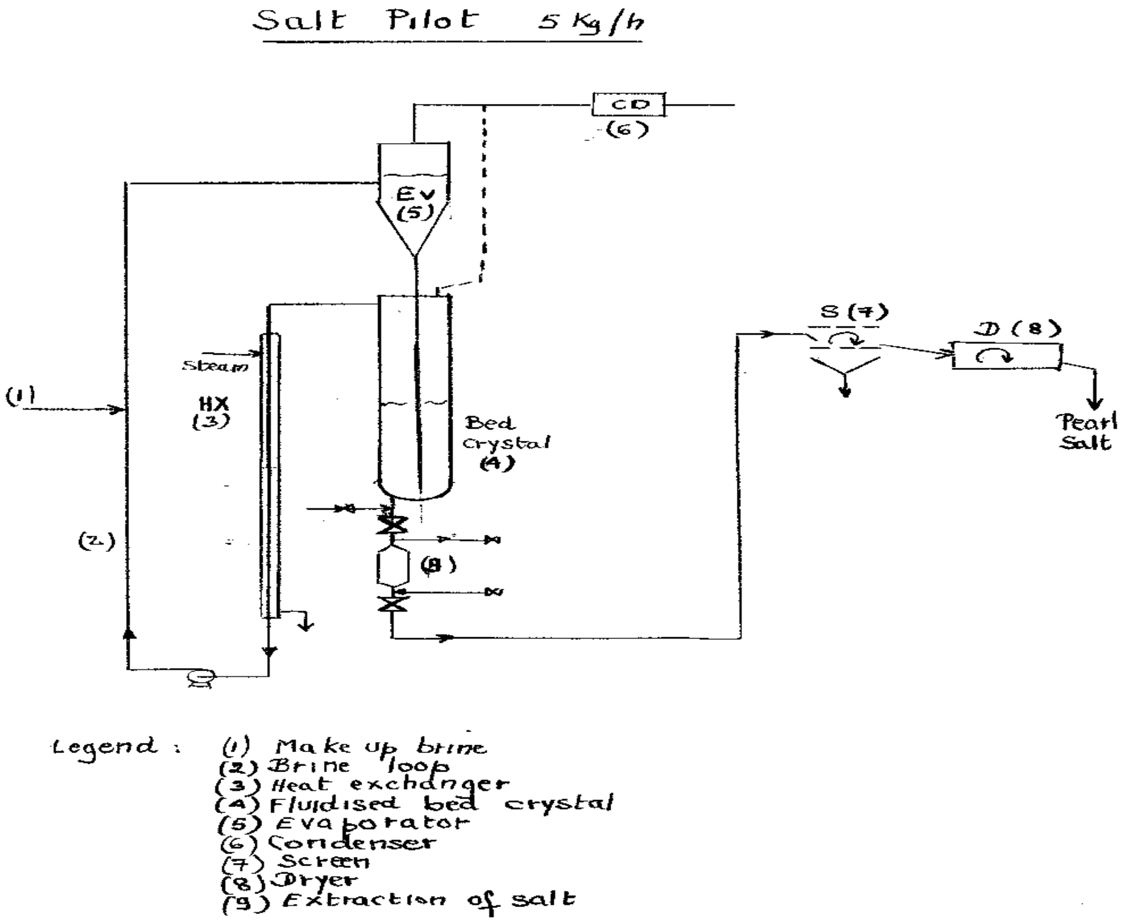

Figure 3

It was very clear that in order to reach a particle size of 5 mm, technical solutions had to be found for particle size control and to extraction of production.

The production was extracted in small batch through a small vessel and a sequence of valves

The seeding was made with current salt produced in salt plant with a size of 500 to 1000 microns; this type of seeds has the advantage to limit the residence time of the crystallizer and stabilize it and allow to extract if needed the very small particle size resulting from uncontrolled nucleation

The pilot plant was able to run continuously for 1 to 2 weeks without trouble

INDUSTRIAL PLANT

An industrial plant was built and has been operating for 20 years (1987 to 2007)

The drawing of the process is shown below in Figure 4

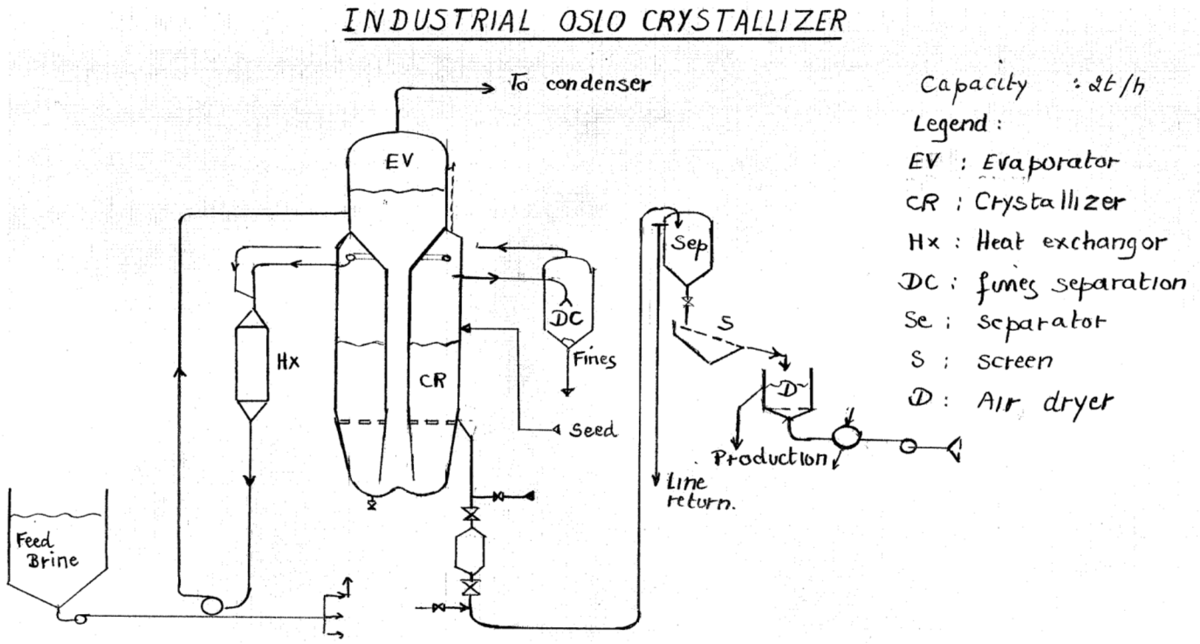

Figure 4

Problems at the start-up:

Shape of particle .Some hydrodynamics problems were encountered at the start-up: the shape of particles was not spherical or near spherical like in pilot; it looked like particles were eroded by strong vortices at the bottom of the bed

Size was limited to less than 1.5 to 2 mm which is the indication of a strong nucleation

Control of shape

Extensive investigation on large cold models with Plexiglas lead us to believe that to obtain round near spherical particle, the large vortices at the bottom at the bed would always be responsible for large attrition

So a completed new solution was adopted: a perforated distributor of super saturated brine at the bottom of the fluidized bed; this distributor has to satisfy a few requirements:

- Distribute evenly the brine
- Scaling has to be avoided
- The jets of brine from the distributor have to create a very small erosion of salt particles

This problem was solved with the help of cold models. A patent was filed. (4)

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Control of particle size

The perfect control of particle size was realized through a set of actions listed below. All the actions serve the purpose to limit or to cancel all sources of uncontrolled nucleation; however In a large industrial equipment like this crystallizer, there is always some possibility for some uncontrolled nucleation

- The surface of the crystallizer was perfectly polished
- The interface of brine and vapor in the evaporator had to be self-cleaning by limited local condensation
- The large vortices , responsible of a large attrition of particles have been suppressed by the distribution plate
- The diameter of the holes are such that there is almost no attrition by the jets from the distributor
- If there is some uncontrolled nucleation , the small nuclei have a size below the cut off size of the rising velocity of brine in the crystallizer ;so these small nuclei circulate with brine in the external loop , they grow at some rate of growth until they reach a size close to the cut off of the bed ; so it is important to capture them before they reach that size : this is the role of an external decanter of fines :
    - The cut off size is about 350 microns
    - All particles smaller than 350 microns must be removed and destroyed with a probability >99.99%
    - The flow rate going to the fines decanter is such that this specification is met
    - The size of seeds must be >350 microns in order to control the particle size
- The choice of operating super saturation has to limit the risk of nucleation

RESULTS

After resolution of start-up problems, the industrial plant was able to operate continuously by campaigns of 2 or 3 weeks

The initial load of salt for start-up could be a load of fines salt, a load of large particles from previous campaigns or compacted salt

The capacity of 2 t/h was reached

The shape of particles size is almost nearly spherical and average size is about 5 mm; a production of 8 mm size has been tested, but this is the limit because of limitation due to the surface of crystal offered to crystallization; it would need larger volume and weight of crystal to make the production but it is possible

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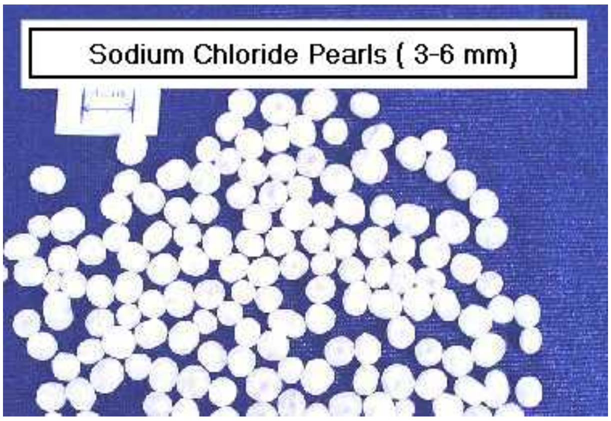

Figure 5

Real capacity is in the range of 100 to 150 kg/h.m^2 for sodium chloride. This is a practical limit but is the consequence of particular case of sodium chloride: high rate of growth of crystal but very limited width of super saturation band.

APPLICATION TO OTHER SALTS

This technology has pushed up the apparent limitations of existing technology; it can be applied to many other salts if there is a need for market .

Orientation tests in small pilot have been conducted to a limited range of salts and have confirmed the expectations

In particular, when we need large particle size, a full comparison in term of investment and operating has to be carried out case by case between:

- A crystallizer + separation +dryer +compaction
- A full size OSLO crystallizer + a very simple dryer

In some case there is room for this technology especially in the dry blend of fertilizers

INFORMATION

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