A Semitechnical Evaporator for Investigations in Salt Technology

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ABSTRACT

A number of sound reasons can be advanced for the desirability of setting up a semitechnical evaporation facility for carrying out process investigations on salt production. Chiefly these arise from the cost and inconvenience of carrying out prolonged and carefully programmed experiments on a production plant which is not often sufficiently flexible in its range of attainable operating conditions or sufficiently instrumented to provide the scope of variables and measurements necessary to research work. A great deal can, and should, be done in the laboratory on quite a small scale, but rarely is it possible to apply results with confidence directly to a production scale. It is suggested that the manufacturing plant is better used as a final confirmation of the best results obtained from experiments derived from a suitably designed semitechnical evaporator.

A semitechnical scale evaporator capable of producing up to about 200 lb/hour of salt has been built at the Winnington Works of Imperial Chemical Industries Limited. In designing this facility attempts were made to match those parameters of the full-scale plant judged to be most important. The result was an evaporator in which the vertical height of the growing region and the calandria tube size were approximately the same as in the full-scale plant, but in which the horizontal dimensions were reduced prorata to the salt make.

Experience with this equipment has shown that the design criteria adopted were justified and since it was commissioned it has been used successfully for the purpose envisaged.

INTRODUCTION

Much of the research which has led to advances in the technology of salt has been done on the small scale with ordinary laboratory apparatus. But progress towards the final utilization of a new effect on the production scale depends very often on carrying out experiments on increasingly larger scales and over longer periods of time. A large part of research into saltmaking processes involves vacuum evaporation, and laboratory work in glass apparatus is convenient up to evaporator capacities of say 20 litres or salt production rates of a few pounds per hour. There is no denying that much useful work has been carried out in apparatus of this kind, but seldom is it possible to apply the results so obtained directly to normal production without carrying out trials on the full scale.

A few years ago that part of my Company which concerned itself with the manufacture of salt found that the carrying through of process investigation programmes was being hampered by the fact that the production plants were working to full capacity and there was little opportunity to carry out research work on them. Thought was therefore given to means of furthering the research and development programmes without disrupting the production schedules more than was absolutely necessary.

It was also considered that there were a number of other valid reasons for experimenting at a stage intermediate between the laboratory and the production plant. A semitechnical evaporator facility seemed to present the best answer to this problem and a decision was taken to design and build a suitable unit.

JUSTIFICATION FOR SEMITECHNICAL EQUIPMENT

When a new and promising-looking effect has been discovered in the laboratory -- let us say a new agent for preventing the build-up of salt scale in an evaporator -- the need often arises to carry out experiments under conditions encountered on the production scale.

There are serious limitations to the idea of immediately doing plant trials. The following are the most important.

1. The programming of plant trials is often difficult in view of competing (and almost invariably dominating) production requirements. Programmed experiments are often required to be postponed, and what is perhaps worse, trials are sometimes required to be terminated prematurely. This can be wasteful of research effort and frustrating for the personnel involved.

2. A production plant is normally designed to operate over a fairly limited range of conditions and the controls and instrumentation are of a sophistication, accuracy and availability appropriate to the steady manufacture of a standard product or at most a few related products. It is essential in research and development work to strive for conditions which give unequivocal results and to design experiments where the maximum information is obtained with the appropriate accuracy. This implies steady conditions of operation at any particular setting of the controls, and measuring values of the operating conditions over a wide range. It is very rarely that these are available on saltmaking plants, and indeed this is hardly to be expected.

3. We all know that even the best run plants have occasional stoppages often for reasons quite removed from the evaporator itself. If short, these are rarely catastrophic in production, but they can be so for an experimental run where steady conditions are being sought. Sometimes the experiment has to be abandoned altogether with consequent wastage, and almost always a stoppage makes the analysis of results difficult. Of course, a new development, to be successful, must be shown to be capable eventually of surviving the normal vicissitudes of real-life plant operation, but at this stage uncontrolled variations are inappropriate.

4. Plant experimental trials, especially prolonged ones, can be very costly if the rate of production or product quality are adversely affected. The use of a new additive may result in a contaminated salt unsuitable for sale in its most profitable market.

5. The trying out of a new idea sometimes involves the necessity for extra-auxiliary equipment and modifications to the plant. This auxiliary equipment often has to be large enough to take the whole make of the plant. Even minor modifications to a large plant can be expensive. Radical alteration is rarely permissible without a good guarantee of success.

6. Promising results in the laboratory are rarely immediately reproduced on the full scale. The optimum effect is sometimes realized only after a number of changes in conditions. There is therefore a danger that promising ideas will be prematurely abandoned if disappointing results are shown in the first plant runs.

A suitably designed semitechnical model is capable of bridging the gap between the laboratory findings and the application of an effect on the full scale. It can be designed so that the above limitations are avoided. As much sophistication as seems necessary can be introduced into instrumentation and controls and a great deal of flexibility of operating conditions can be achieved. The operation of the equipment can be quite independent of the production facilities and the quantities of salt produced are trivial. The internal geometry of the evaporators can be altered at will and the equipment can be devoted to one project for prolonged periods -- an important point where contamination by active trace materials is involved.

If the model is designed so that hydrodynamic and thermal conditions obtaining in it are not too far removed from those of the full-scale equipment, there is a good chance that results obtained with it will be reproduced when bulk manufacture is embarked upon. I do not believe, however, that one can expect to do away with plant trials altogether. Final confirmation of the best results obtained from the semitechnical plant should be carried out on a production scale. But one has at least avoided using the latter to carry out what is, in effect, exploratory work.

GENERAL DESIGN PRINCIPLES

We considered that what we wanted was a plant designed so that the maximum flexibility was achieved with the minimum change in the units comprising the plant. It was thought that a "kit of parts" could be provided so that a change from one type of evaporator to another would involve the changing over of only a small unit of the whole. It was hoped that it could be so designed that numerous variations in plant geometry and operating conditions could be achieved with little difficulty. We wanted the rearrangement of piping, etc., to be as easy as possible and changes to involve the minimum of fabrication of parts. We wanted the mounting structure to be as adaptable as possible. We did not finally achieve all these things, but we went a long way towards it.

We chose as our basic arrangement a natural circulation internal calandria evaporator, a type which is fairly common at our Works. We wished this to be capable of easy conversion to a forced circulation, external calandria type evaporator and to other arrangements of our own conception as the need arose.

The first problem that has to be tackled is to decide what design characteristics of the full-scale plant one should attempt to maintain on the semitechnical model. Considerable thought was given to this point, as it is crucial to the question of whether results obtained on the smaller scale will be representative of those which would have been obtained on the full scale. Clearly a choice has to be made of those dimensions which are to be scaled down. To some extent any decision will be a compromise, and there is no certainty that eventual modifications will not make one's original choice inappropriate.

Although it is desired to scale down the plant, the product is not being scaled down and the properties of the plant liquors have to remain the same. A straightforward geometrical model did not seem to be inappropriate to provide the salt product with the same immediate environment for most of its life in the evaporator. It was considered that a distorted model element was likely to be much more satisfactory as the retention of the same tubes as are used on the full scale would at least provide the same hydrodynamic and heat transfer conditions for the salt magma travelling through the calandria.

The following conditions were chosen as being sufficiently controlling to be maintained as nearly as possible the same at the two scales.

1. area intensity, i.e., salt production per unit area of brine surface;
2. volume intensity, i.e., salt production per unit volume of "growing area";
3. heat transfer conditions in calandria; and
4. impeller tip speed (in the internal calandria version).

A salt production rate of 40 kilograms per hour was chosen as being adequate for test purposes, but not too high to involve much expense in disposal. In order to match the area intensity of the chosen full-size evaporator, at this production rate a body diameter of about 18 inches would be required. This seemed to be about right for what we had in mind.

In order to preserve the same order of volume intensity -- and in this way approximately the same conditions of supersaturation -- it was decided to maintain the same height of brine above the tube plate and tube length and to scale down the volume of the portion below the calandria in proportion to the scaling down of cross-sectional area.

It was considered that the most satisfactory way of simulating the heat transfer conditions, that is the same temperature and velocity of brine in the tube, was to keep the same diameter of tube but to use a reduced number in proportion to the different rates of make. It was calculated that 15 tubes would be about the right number and at an outside diameter of two and one half inches these could just be fitted into a tube plate of 18-inch diameter. The linear ratio of central downcomer to shell diameters on the full-scale plant was used to determine the diameter of the downcomer in the pilot plant.

In order to simulate the performance of the large scale impeller it was necessary to choose criteria of similarity from those normally recognized as important. Tip speed was in fact chosen as the most appropriate for this problem. It was also necessary, as I said earlier, to keep the velocity of brine in the tubes at about the same value, and it was considered that an impeller of the same shape as that used on the full-scale plant rotating at the same tip speed would satisfy this condition.

There were other parts of the semitechnical equipment which needed special consideration, although it was recognized that some changes might have to be made with experience in order to achieve optimum arrangements for any particular requirement. Thought had to be given to the steam disengaging height, the shape of the shell below the calandria, the elutriation column, salt withdrawal, ease of access to sight glasses and valves, interchangeability of parts and pipework, adequate sampling points and facilities for introducing materials into the evaporator at different points.

SEMITECHNICAL EVAPORATOR AND ITS OPERATION

A semitechnical facility was eventually designed and erected in a suitable building at the Winnington Works of Imperial Chemical Industries Limited. An artist's sketch shown in Fig. 1 gives a general impression of the size and shape of the plant. Plenty of room was allowed above and below the calandria section for longer tubes and longer elutriation equipment. What finally emerged was a self-contained unit with the following characteristics.

The evaporator is a long, thin shell (Fig. 2) in which the vertical height is of the same order as on the full scale, but in which horizontal dimensions have been reduced in relation to the salt production rate. It is designed to operate at temperatures between 35 and 140°C. It is made of mild steel, but is lined internally with neoprene. The calandria section comprises a four and one half-foot long section containing the copper or copper/nickel alloy tubes and tube plates, which are in monel-clad mild steel. Other calandria sections can be inserted to simulate other evaporators, or the calandria can be removed completely and replaced by a hollow shell fitted with connections to an external calandria and pump, so that forced circulated conditions can be obtained. Below the calandria, the cylindrical portion above the conical part can be altered in length so as to allow changes in volume. The stirrer consists of a four-bladed impeller mounted on a monel shaft passing through the bottom cone. Some further details of the shaft and its sealing are dealt with later.

The impeller is capable of stepless variations of speed in the range 50-1,000 rpm. It rotates in an extension of the calandria downcomer (called the skirt) which may be varied in length. The vertical position of the impeller blades can also be varied. A large door is fitted into the side to allow ready access to the impeller and skirt.

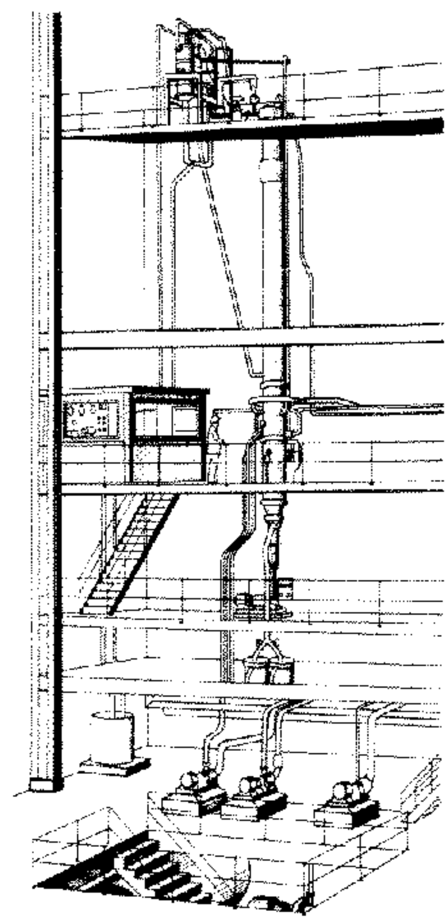

Figure 1. General Impression of Equipment.

Figure 3 shows diagrammatically a simplified arrangement of the pieces of equipment and pipework for the internal calandria system. The condenser is a jet type in which vapour and river water are in contact. Vacuum is raised by a two-stage steam jet ejector, to which air is leaked in order to control the amount of vacuum.

Brine may either be taken direct from the normal brine supply into the Works via a stock tank, or the latter can be filled with special brines from a road tanker. Additives can be admitted to the evaporator from a small tank via an accurate metering micropump. Facilities are provided for automatically controlling steam supply, evaporation temperature, condensate removal, evaporator brine level and river water flow to the condenser. The flows of water, brine and steam; the temperatures of brine, steam and differential temperature between condenser effluent and cooling water; and the pressures of steam and vapour above the brine are all measured on recording instruments. The brine level is also automatically recorded.

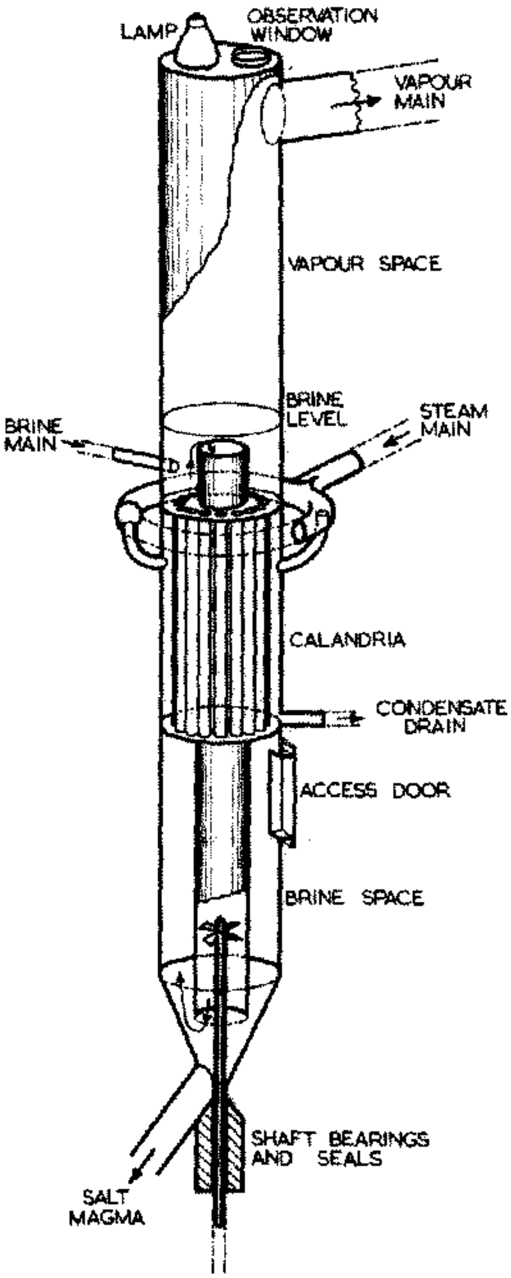

Figure 2. Evaporator Body -- Internal Calandria.

Figure 4 shows the corresponding simplified layout for the external calandria (forced) system.

The plant was normally operated around the clock for continuous periods of up to four weeks with boil-out intervals of 60 hours. The routine operation was carried out by process operatives working to a detailed set of instructions and they were supervised during the day by a staff supervisor.

Sampling was an important duty of the operatives, and some simple analysis was carried out by them in the laboratory, which also housed most of the recording instruments.

The design of the sealing arrangement for the impeller shaft was found to be a crucial matter. The original design allowed a leakage of air in an amount which would not have been very important on the full-scale plant, but with the model, it led to continuous instability and uncontrolled variations. It was replaced by a much more satisfactory arrangement which is shown diagrammatically in Fig. 5. The seals do not now rub on the monel shaft itself, but on a "Colmonoy" hard facing alloy (chromium boride in nickel) deposited on the hollow sleeve carrying the shaft itself. It is in the sleeve, too, which is in contact with the bearings. Improved sealing was also achieved by the use of a bucket-seal kept filled with lubricating oil. Oil fed to the seals was cooled. Additional cooling could be brought into use, if necessary, by means of a cooling water jacket built around the gland housing. Provision was also made to lead away any spent gland lubricant which contained air bubbles. This in fact was never required. A small stream of distilled water could also be injected into the region where the impeller shaft entered the evaporator. This was very effective in preventing scaling and damage to the shaft at this point. This new sealing arrangement ensured that air leakage was reduced to a minimum and much steadier running was achieved.

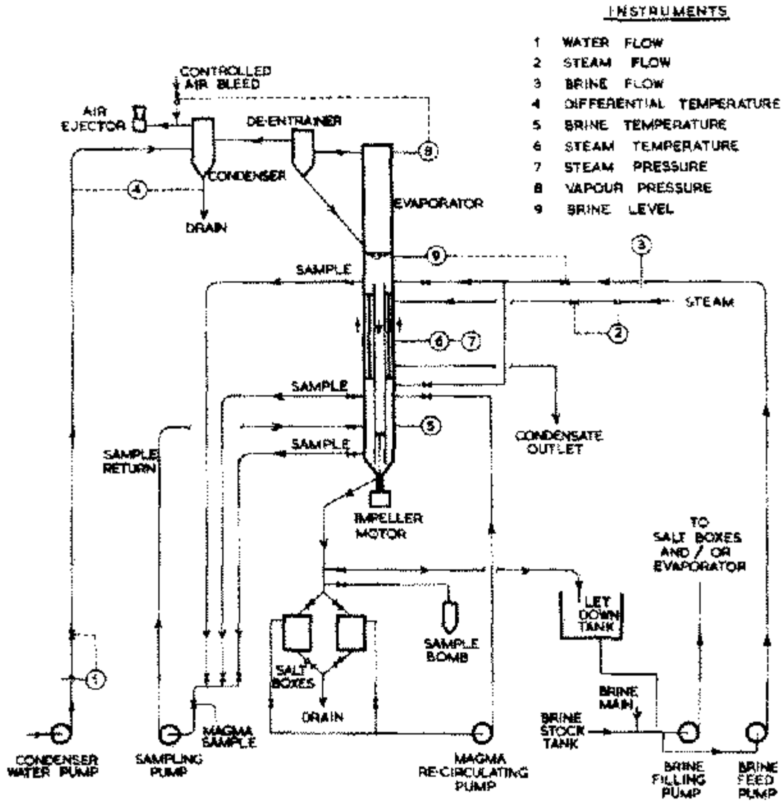

Figure 3. Internal Calandria Circuit Diagram.

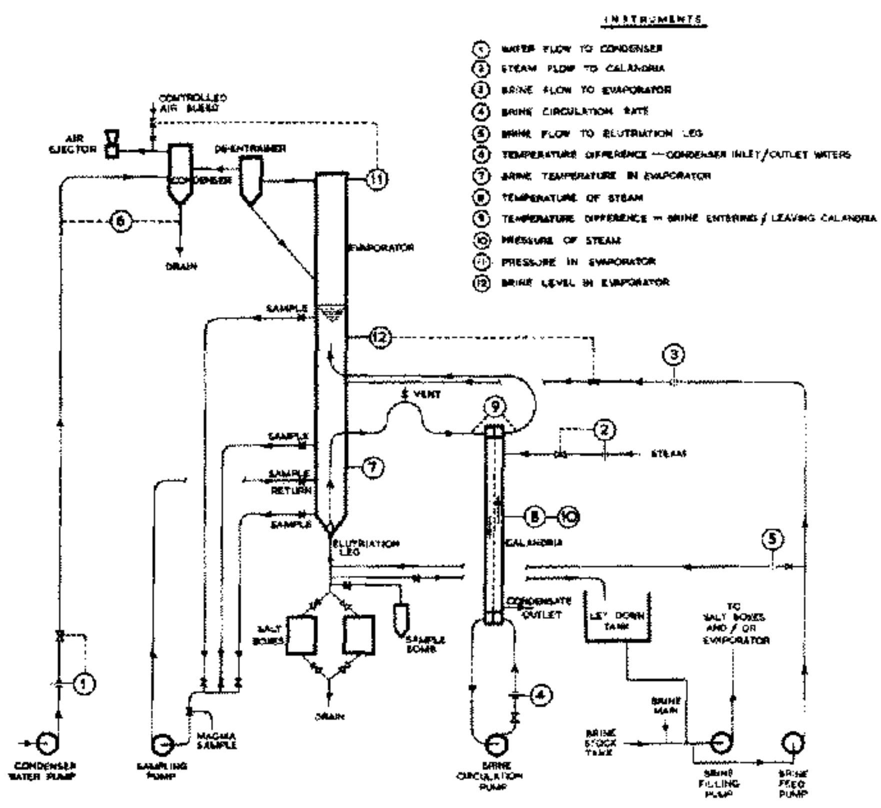

Figure 4. External Calandria Circuit Diagram.

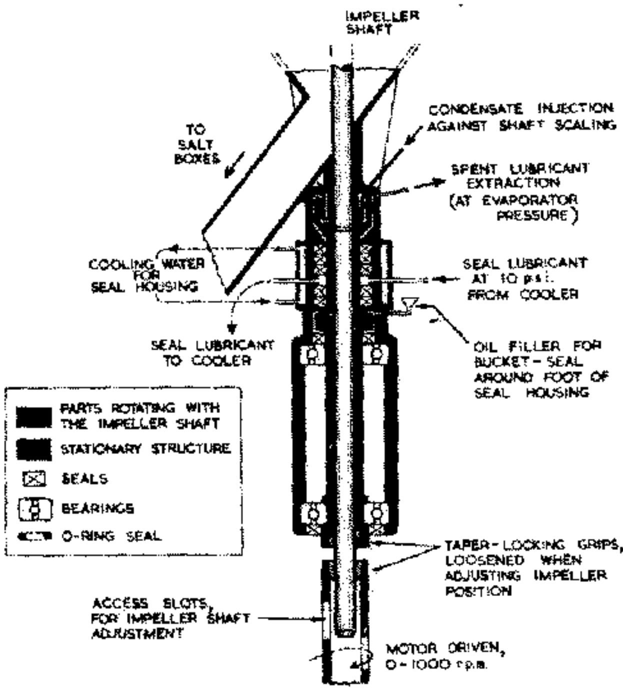

Figure 5. Shaft Sealing Arrangement.

CONCLUSIONS

Were our views on the design principles justified? The many experimental runs now carried out have convinced those using it that in operation it behaves in a very similar way to a large plant. The salt product obtained has been very close in size and crystal form to those which were produced under apparently similar conditions on the full scale. Even the dendritic salt produced was a very good match, and we know that evaporator conditions have to be controlled very carefully and within quite narrow limits to achieve this. All this indicates that nucleation rates, supersaturation and growth rates are similar on the two scales, but we have no direct proof of this. Certainly the heat transfer conditions were close to those which one would get on the corresponding large plant.

In general, we feel we were right in our approach, at least there is not much to show that we were wrong. There is a suspicion that turbulence in the semitechnical equipment is not great enough to match that in the full-scale plant and this could be important where crystal breakage is a significant factor in determining the ultimate properties of the product.

Many useful trials have been conducted with the equipment using different brines, different calandria systems, different additives and different evaporator conditions. We have been able to do statistically-designed experiments to find out the optimum operating conditions for dendritic salt production and have produced results which have helped to explain laboratory findings and which have led to improvements in the commercial product. An interesting series of experiments was carried out to define the operating limits for producing pure salt from raw brine, with and without additives for reducing calcium sulphate occlusion.

There are, not surprisingly, a few disadvantages in doing experiments on a semitechnical scale. Smaller size generally means that any salt scaling has a disproportionate effect on running and shorter runs are generally necessary. The effect of any air leakage is very large and as I have indicated a lot of attention has to be paid to gland sealing. The smaller volume leads to a greater effect being noticed when disturbances occur, for example, sample removal, cooler brine feed than normal and so forth.

Provided the equipment has plenty of work to do, I believe it is a useful tool to a saltmaker who is interested in carrying out process investigations on an evaporator and who is keen to explore the possibilities of introducing new additives and operating conditions into his process.