Pilot Plant Studies

Introduction

Plant: it is a place where the 5 M's like money, material; man, method and machine are brought together for manufacturing of the products.

Pilot Plant: it is the part of industry where a lab scale formula is transformed into a viable product be development of liable and practical procedure of manufacture.

Scale-up: The Art for designing of prototype using the data obtained from the pilot plant model.
Introduction

The ultimate purpose of all pilot plant and model experiments is framed in a phrase:
"Commit your blunders on small scale and make your profits on a large scale".

A small experiment made upon a few grams of material in the laboratory will not be much use in guiding to the erection of large scale works, but there is no doubt that an experiment based upon a few kilograms will give nearly all the data required.
It is technically possible to transfer any new process whatever from the laboratory directly to large scale production provided that unlimited money is available and also huge risk factor is involved. On the other hand, there is also little doubt that performance data obtained from a correctly designed and operated small scale plant are always more accurate and reliable than data scaled up from the laboratory bench or derived by calculation from generalized correlation.

Pilot Plant:- A pilot plant is a small industrial system which is operated to generate information about the behavior of the system for use in design of larger facilities. Pilot plant is a relative term in the sense that plants are typically smaller than full-scale production plants, but are built in a range of sizes. (A pilot plant is a small scale replica of the full scale final plant that is not yet built to provide design data for the ultimate large one).

Model: A geometrical similar replica of complete prototype on a small scale with equal scale ratios in all direction is termed as a model. Its function is to exhibit the effects of change in shape or operating conditions of a existing plant.

Prototype: A large scale apparatus or full size plant is called prototype.

Scale-up:- The art for designing of prototype using the data obtained from the pilot plant model.

The ultimate goal of any laboratory research activity is the commercial exploitation.

There are two routes to reach this final goal.

They are (1) The calculation approach (2) Experimental approach

The first route is by translating the ideas directly into a commercial feasibility without any experimentation. In this normally an array of differential equations and most often the computer facilities are made use of.

The second route is carried out via Pilot Plant stage.

In Pilot Plant Stage there are usually six systematic steps to be taken to reach the commercial exploitation from research.

Process Research

Preliminary Process Design

Preliminary Process Evaluation

Process Development

Final Process Design and Final Process Evaluation

What is the necessity to build a pilot plant?

Small scale plant is employed in chemical engineering for two main purposes;

The first is as forerunner to a full sized production plant that is not yet built. In this case, the small scale equipment is called a pilot plant and its principal function is usually to provide design data for the ultimate large one, although it may also be required to produce small quantities of a new product for trial.

The second purpose is to study the behavior of an existing plant of which the small unit is a reproduction. In this case, the small scale equipment is what is ordinarily called a model, and its chief function is to exhibit the effects of change in shape or operating conditions more quickly and economically than would be possible by experiments on the full-sized prototype.

Objectives of scale up….

- To provide master manufacturing formula
- To identify the critical features of the process
- Evaluation and Validation
- Guidelines for production and process control
- Review of the processing equipment
- To produce physically and chemically stable product

Analytical Versus Experimental Scale up

With the advent of computers now it looks ugh the need for pilot plants will be negligible. It is theoretically possible. Every now and then we come across reports which will be little the needs of pilot plants. For a completely analytical approach to reach the commercial stage without pilot plant experimentation differential equations can be formulated for the chemical reaction or the combined simultaneous processes of chemical reaction and diffusion. The differential equations describing heat and mass transfer can also be formulated.

In many instances, particularly in heterogeneous catalytic system the equations are difficult to solve without the aid of electronic computers. With the aid of these computers full scale operating conditions will give data which will materially assist the scale up without lot of successive experimentation. This does not imply that the pilot plant is no longer required. The computer only places the use of pilot plant in process design on a more rational and effective basis.

In some cases one may require the scale up of a medium sized plant as one encounters in fine chemical industry. When compared to the cost of the whole plant, the operational cost for the computer itself will be more excessive. Under these conditions the scale up rules through successive experimentation via pilot plants will be more useful.

The computer only aids the successive experimentation in a shorter duration and it cannot replace pilot plants.

Example

We want to produce benzyl acetate which is used in the perfumery industry. Small scale experiments indicate the possibility of making this chemical from benzyle chloride and sodium acetate in a batch reactor.

Because of the formation of sodium chloride which is insoluble which is insoluble the consistency increases. Here the important factor is the efficiency of stirring . We want to know whether we can get the same efficiency and yield in a bigger scale as the values obtained in the lab scale experiments ? For this a pilot plant method is most suitable as this is not getting to be expensive. Why should one go along the route of a computer? Process development and pilot plant and scale up are definitely less expensive than the usage of computer for this type of problem.

Process Evolution/ Process Development:

The evaluation of new process starts in laboratory. This is done by carrying out a preliminary engineering study. Flow diagrams are prepared showing material quantities and any information on heat and power requirements that is available.

On the basis of laboratory results complete process is broken down into a series of unit operations or processes and a suitable type of equipment for each step is chosen. At this stage, a rough estimate of capital cost may be attempted.

Having tentatively selected the type of equipment to be used the chemical engineer is able to write down all the quantitative data that will be needed for the design of each component.

Data will include material and heat balance, chemical .physical and thermodynamic properties of raw materials, intermediate compounds and final products, reaction rates, heat and mass transfer coefficients and other rate parameters, power requirements, corrosion rates etc.
The design data required for each piece of equipment can then be classified under six heads:

- Data available from past experience
- Data given in the laboratory reports or which can be derived from the laboratory results.
- Data available in the literature.
- Data obtained from empirical and semi empirical correlations.
- Data which could be obtained by further research in the laboratory.

Sometimes the preliminary engineering study may suggest that no pilot plant experiments at all are needed. The new process may be entirely made up of standard unit operations which are well understood and from which plant can be confidently designed from laboratory data alone. But as a rule one or two critical operations of the new process are selected for study on pilot plant scale.

Case History:

Vinyl chloride from Naphtha

Careful planning and the time involved in a process development program of a project are brought out by the following case history.

This is a new process to an existing product. Various steps involved in the process are

(1) High temperature naphtha cracking which produced acetylene and ethylene.
(2) Vinyl chloride synthesis by the reaction of cracked gas containing acetylene with HCl.
(3) Ethylene dichloride synthesis by reaction of chloride with cracked gas containing ethylene.
(4) Thermal cracking of ethylene dichloride to vinyl chloride and HCl-separation of HCl for reaction in the step 2.
(5) Separation and purification of Vinyl Chloride obtained in above steps to obtain high purity monomer.

- **April 1960:** RAP began the first phase naphtha cracking project using a pilot plant of 40lb/hr acetylene.
- **August 1960:** Kureha began development work by using the above pilot plant.
- **April 1961:** RAP began the second phase project by scaling up the pilot plant to a capacity of 240lb/hr.
- **April 1962:** Both Kureha and Chioda continued research and development of naphtha cracking by scaling up the plant further to 720 lb/hr acetylene.
- **September 1962:** A new company has been licensed to produce in commercial scale.
- **October 1962:** it started constructing the plant of a capacity of 10,000 lb/hr.
- **December 1963:** Completed the above commercial plant.
- **February 1964:** The above plant was put on stream.
- **May 1964:** Reached full design capacity.
- Thus the full development program from idea to commercial exploitation has taken nearly 5.5 years.

- **June 1966:** Russia has signed a contract for this process with these 2 companies.

Thus in 2 years time the process has been accepted by others also.

Process Study

Process study means the examination and improvement of existing full scale process or plant. Here the principal object of using models is to determine the effect of modifications in design or major changes in operating conditions without incurring the expense or risk of making these change on the large scale.

For example, to establish the effect altering the shape or position of a baffle wall in a furnace by means of a full scale trial, it would be necessary to take the furnace out of service, allow it to cool down, break out the old baffle wall, build the new one, and heat up sufficiently slowly to avoid damaging the brickwork. Altogether the furnace might be out of use for a month or more, and again perhaps for an equal period of restoration to its original state if the change should have proved deleterious. The required information could probably be obtained by means of a model in a few days and without interrupting production.

Model theory indicates the necessary conditions foe reproducibility. A working model of a production plant can furnish valuable advance data on the effect of proposed changes on operation and hookup. For this reason it is often wise to keep the pilot plant in commission long after its original process development function has been accomplished. As the detailed design and construction of the full scale plant proceed, the pilot plant is then available to solve last minute problems.

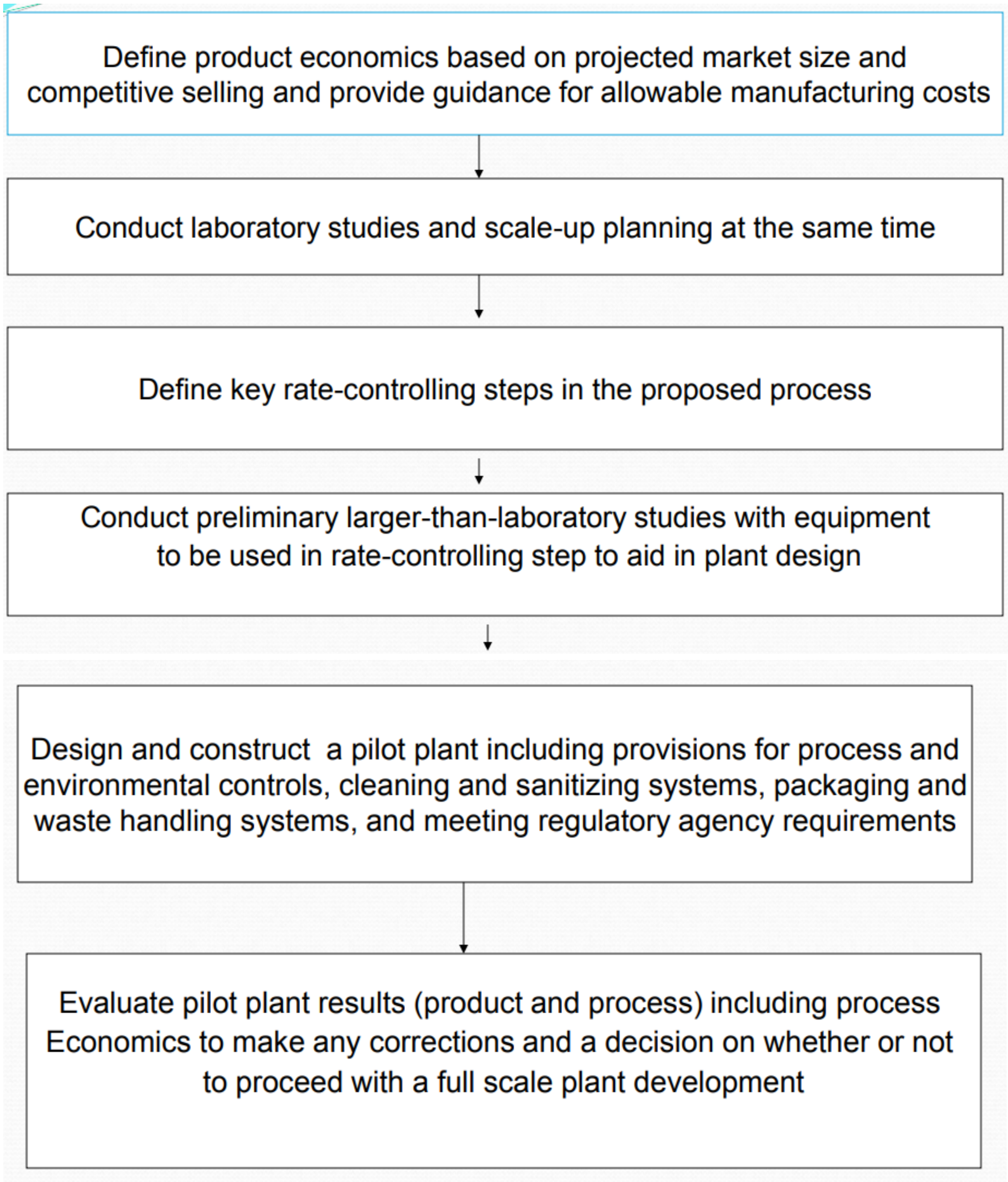
Classification of design data

The design data required for each piece of equipment can be classified under six heads-

- Data available from past experience.
- Data given in the laboratory report or which can be derived from the laboratory results.
- Data available in the literature.
- Data which can be approximated sufficiently closely for design purpose by means of thermodynamic relations, the theorem of corresponding states, or some of the many empirical or semi empirical correlations that have appeared in recent year.
- Data which could be obtained by further research in the laboratory.
- Other practical felt to bee necessary for design purpose.

Information falling under this head can be determined only in a pilot plant.

Steps In Scale-up(Scale up techniques)



Scale up and Major factors:

A pilot plant is unlikely to yield the maximum possible amount of information unless the critical components at least are designed and operated in accordance with model theory.

The first step is to derive the similarity criteria which govern the operation or processes to be studied on the small scale.

These may be obtained either by dimensional analysis or from the fundamental differential equation of the process.

A study of similarity criteria will reveal the conditions under which the model should be tested in order that the results may simulate those obtained under given conditions on the large scale.

It will also show whether there are likely to be appreciable scale effects, in which case it will be necessary to apply corrections to the pilot plant results before they are used for full scale design.

Finally, such a study will reveal those awkward cases in which two or more similarity criteria are incompatible and where consequently, it is not possible to simulate large scale results in a small apparatus with any certainty. Even in these cases, experiments with small scale unit over a sufficiently wide range of conditions can give valuable information for the design of the full sized plant.

The safest method of evaluating scale effects and allowing for incompatibility of similarity criteria is to scale a process up by easy stages through two or three pilot plants of increasing size. The effects if any of change of scale on rate parameters and yields can then be observed directly and extrapolated to the full scale. A graduated succession of pilot plants is part of the traditional technique of process development a technique that is both costly and slow. The Principal aim of model theory is to allow of larger steps in development and reduce their number and duration, and it is to be hoped that in the future the need for more than one pilot stage between the laboratory and the full scale will become increasingly rare.

Example:

Scale up methods eliminates the factor of safety. Scale up models simulating the performance of large scale prototype have been successfully used in the design or ship hulls and aircraft wings. In mechanical, civil aeronautical engineering the usefulness of models have been recognized and made use of very extensively. Although the chemical engineers have used this without their knowledge since the time of Reynolds and Prandtle only in recent years the application of model theory has found its way into the field of chemical reactors and process equipment.

To illustrate the above statement we will consider the case of a pressure drop of a fluid moving downward through a fixed bed of catalyst particles. It is desired to know what pressure drop through this bed will be when the bed is large enough to handle 1000 times as much vapour and the catalyst particle are of the same size as in the pilot reactor. Measurement of the delta P as a function of vapor rate can be easily made on pilot reactor. The delta P is calculated as function ‘f’ Fanning friction factor from the equation and the velocity of the vapor is expressed as Nre .A plot is made f Vs Nre. Now the pressure drop for larger bed can than be calculated by calculating the Nre for larger bed finding the ‘f’ for that Nre and using that friction factor to get delta P.

Limitation of Scale-up

Example : Incomplete Data (Furnace Oil)

To encounter poor technique which might give incomplete data or sometimes erroneous data.
Case history is discussed to appreciate this kind of limitation:
It was desired to remove Mercaptan sulphur compound RSH from furnace oil.The chemistry of the process is

$RSH+NaOH \rightarrow RSNa+HOH$

The Mercaptans are objectionalable in the furnace oil.
Using a caustic reagent it can be extracted in an extractor.
the concentration level of disulphide sulphur should not be disturbed.
This reaction results in formation of mercaptide (sodium)
This is further treated in a regenerator where a reverse reaction takes place when the caustic was regenerated
Small quantities of phenols and cresols present in the raw oil charge are expected to react and accumulate in the caustic stream.
An portion of this caustic is rejected from the system to keep the phenolates and cryslates from building to too high a level of concentration in the circulating stream.
Very small loss to the removal of these materials is expected.
To prove this a model was constructed and tested.
The results obtained as compared with expectations are:

	Raw Feed	Expected	Obtained
Mercaptan sulphur RSH mg/100 CC	80	0	0
Disulphide sulphur RSSR mg/100CC	3	3	50
Acid oil lost %	-	0.2	5.0

Result :
Merceptan sulphur has been removed from the oil as desired.
Unexpected results obtained:
Diusulphur concentration in the product was very high
Loss in the acid oil was also very high.
These are the sufficient reasons to kill the project in the initial stage itself.
The logistic conclusion would be to abandon the project.

But it was not abandoned and further study was done and it revealed that an important reaction has been overlooked.
$4RSH+O2 \rightarrow 2RSSR+2HOH$

Thinking that there is no possibility from oxygen to get in the system.
However in the small scale oxygen had crept in the feed oil in the form of air (dissolved in raw oil).
The long time hold up time in the laboratory unit when compared to the prototype unit gave the reaction time needed to convert the mercaptans to RSSR and increased the acidic oil. This completely obscured the basic intent of the process.
In the bigger unit this trouble has not been noticed because the holding time was less because of the larger quantity and there was no chance for oxygen to get into the system.When the sample taken from the bigger scale unit was used in the smaller unit this trouble was not noticed. When once the hidden ills have been recognized it is possible to correct them immediately
Trace Impurities when it is not properly recognized and correctec for can play vital havoc.]
In small scale experiments due to short run time impurities are literally neglected.This poses very serious threat to scale up problem.
In a catalytic reforming unit using platinum catalyst vergin naphtha of low octanes are passed in the vapour phase through a fixed bed at a temperature of 900-950 F and pressure of 300-600psi.
A large volume of H2 was circulated with the naphtha charge to prevent coke deposition on the catalyst.
In the feed stock the impurities that are normally encountered are sulphur and nitrogen compounds, which can be measured in term of 0.1 to 0.5%.Since their presence are known in substantial level of concentration the difficulties arising out of them can be recognized and suitable remedies can be easily carried out.
But there are certain other impurities which may be present in ppm and ppb range which cannot be assessed in scale unit.A small scale programme was started using synthetic level of impurities. Deleberately 100-1000ppm of tetraethyl lead and triphenyl arsine were added as impurities.the higher level of concentration was added as impurities.The higher level of concentration was choosen to hasten the action of impurities with respect to time.
Graph
The action of these impurities are clear from the graph.
It is also clear that unless the experiments are carried out for longer duration it is not possible to detect the effect of these impurities. Small scale experiments are run only for one or atleast only to a few hours.
The level of concentration in these runs is very high compared to what might be experienced in practice. Nevertheless it shows that in practice we may got this difficulty after about 300-400 hours of operation instead of `150 hours that we have in higher degree of impurities.
Because of the difficulty of detection in small scale work it is much more difficult to handle.
What we must be aware of in the scale up is the existence of this problem. This emphasizes the correctly planned process development and the successive experimentation for its successful commercial exploitation.
Deviations from small scale experiments which cannot be otherwise explained mey be the result of the impurities.

LIMITATION DUE TO INCOMPLETE OBSERVATION: Desalting of Crude Oil.

Lack of observation on the part of the operating group and the lack of communication on the part of the process development group rise to may difficulties.

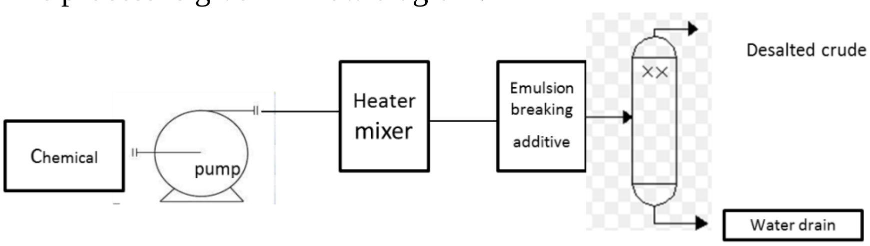
The observation may be most trivial and least suspected. Still if they are properly recognized and interpreted and discussed it may save many men hours and sometimes the project itself.

Example:

Inorganic salts from crude oil have to be removed to prevent corrosion and plugging of heat exchangers.

The salts normally present in a crude oil can be retained in a refinery charge as crystalline suspensions and as entrained solution with water. These salts break down during processing to form acids which increases corrosion.

The process is given in flow diagram:



The important problem here is to make the water separate from the crude oil water emulsion which is formed in the mixing step.

This may often required additive of some sort to break the emulsion.

This was achieved in the pilot plant and so a prototype was planned and built. An inorganic alkali was used as the emulsion breaking agent.

The plant performed very well for about the month, with 98% efficiency then suddenly a difficulty developed.

The crude oil and water would not separate. Practically all the water was leaving the desalter emulsifier with crude oil. The success of entire project was in doubt.

A thorough check up of the small scale and prototype data revealed that everything was behaving as planned and the temperature, pressure, flow-rates, injection of water, alkali reagent and mixing intensity were all same. However the pH was found to be different. Instead of the required value of pH 8-9 it was in the range of pH 10-11.

Further intensive check revealed that the crude oil had abnormally low salt content which resulted in less release of HCL by hydrolysis of salt with the resultant alkalinity of waste water. When the pH was corrected, everything functioned normally.

The operating group had noted the differenece in pH but did not realize the implications. A carefully planned discussion between the operating and process development group might have prevented this situation.

Trace Impurities-catalytic Reforming Unit

- Trace Impurities when it is not properly recognized and corrected for can play vital havoc.
- In small scale experiments due to short run time impurities are literally neglected. This poses very serious threat to scale up problem.

Catalytic reforming is a chemical process used to convert petroleum refinery **naphthas** distilled from crude oil (typically having low octane ratings) into high-octane liquid products called reformates, which are premium blending stocks for high-octane gasoline.

- In a catalytic reforming unit using platinum catalyst vergin naphtha of low octanes are passed in the vapour phase through a fixed bed at a temperature of 900-950 F and pressure of 300-600psi.
- A large volume of H2 was circulated with the naphtha charge to prevent coke deposition on the catalyst.
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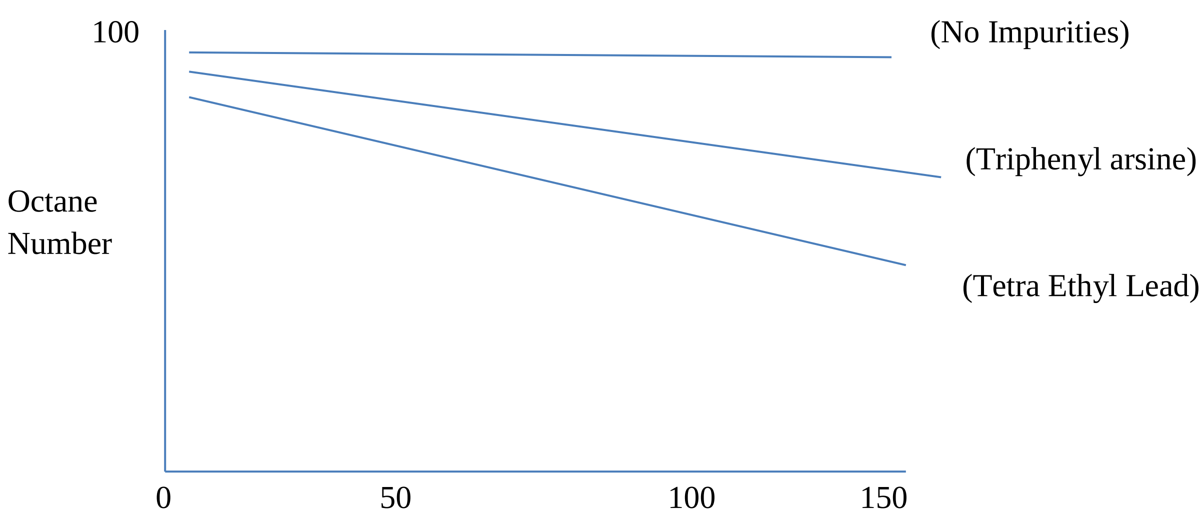

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