

## CHAPTER 3

# VARIOUS CHEMICAL PROCESSES

### **CHOICE OF PROCESS:**

Nitrations, as technical process have evolved from batch type operations using stoneware vessels and hand operations to automatically controlled continuous type processes carried out in gleaming stainless steel vessels. The high heats of reaction and dilution involved in nitration, originally absorbed by placing the stoneware vessels in a water bath, are now taken up by coils or jackets cooled by refrigerated brine. Controls have evolved from none at all to the completely automatic systems that may be so elaborated as to permit operation from remote locations. The use of remote control is very much desired in the design of equipment for the manufacture of hazardous, explosive compounds, which often are results of nitration processes.

Both batch and continuous process have their relative merits and drawbacks.

The *advantages of batch process over continuous process* are:

- *Flexibility:*

Batch process equipment possesses general usefulness because each batch of material passing through the process is separate, or nearly separate, from batches which have preceded or which will follow it. It is usually easier to introduce process variations into a batch process than into a continuous process. Furthermore, batch, processing equipment is often of such general applicability that a given plant may be readily converted production of one nitrated material to another. The batch process because of the operating flexibility conveniently does beginning production of new compound or pilot production, even though the use of the continuous may be planned for the completely developed process.

- Labour Usage:  
For high rates of production, when large batches are used the labor efficiency of the batch process may be just as good as that for a continuous process. This appears to be true for large-scale industrial production of nitrotoluenes.

The *advantages of continuous process over batch process* are:

- Lower Capital Costs:  
For a given rate of production the equipment needed for a continuous process is smaller than for a batch process. This is usually the most striking difference between the two types of process. Since it necessary to accumulate material in a continuous process anywhere the vessel are designed with capacities dictated by the rate of the reaction process step, which they must accommodate. Alternatively because of the relatively small size of continuous process equipment, it is often possible and advantageous to use materials of construction, which would be excessively high in cost for batch sale equipment. Thus e.g. corrosion resistant alloys such as the appropriate stainless steel may be used for a continuous plant, whereas ordinary mild steel may be dictated fore a batch plant because of cost.
- Safety:  
Because of the relatively small size of the continuous process equipment, there is less material in the process at anytime than at certain times in a comparable batch process. For example, at the completion of batch process nitration and during its normal; separation of the product from spent nitrating acid; the entire batch of an often hazardous compound will be present in the equipment. In the continuous process, only as much material need be present in hazardous conditions as needed to gain sufficient reaction or process time. In the case of high explosives made by nitration, such as nitroglycerine this inherent safety factor of a continuous process is very attractive.

- Labour Usage:

In the nitration field, a continuous process is usually a more efficient labour user than a batch process. This is particularly true for small or medium scale production and for hazardous products. Since continuous processing minimizes the amounts of material in process on the average, it is often possible to handle operations at one place that require physical separation in a batch process and hence require additional labour. The discrepancy in labour efficiency tends to disappear as the scale of operations increases.

## **DESCRIPTION OF PROCESS:**

The flow sheet for the manufacture of mononitrotoluenes is as shown in flow sheet. The fresh 60% HNO<sub>3</sub> acid and 96% sulfuric acid are mixed with recycle acid in a jet mixer and the mixed acid is sent to the first nitrator where toluene is fed. The toluene and mixed acids that is coming into the nitrator are vigorously agitated. The reaction mixture flows by gravity to a second nitrator and finally to a separator, where the nitrotoluenes and spent acid are continuously separated. A part of spent acid is recycled and rest goes for sulfuric acid concentration.

The crude mononitrotoluenes with residues are then washed in three stages, in Holley-Mott type of washers; with water, then with sodium carbonate solution, and again with water. The washings are discarded. The washed mononitrotoluenes are then collected in the intermediate storage tank as shown in flow sheet.

Upto this point the process is continuous but a batch topping still is used to separate the three isomers from the paraffin and unreacted toluene, and from heavier residue. When the storage tank has collected an amount of washed mononitrotoluene equivalent to a day's out put from the washing units, the mixture is pumped, to still with a short plate column; the forerunnings are taken off as a paraffin fraction and the next fraction is collected in a tank as a mixture of the ortho-, meta-, and para-mononitrotoluenes. The residue from the batch still is collected and redistilled to increase, the recovery of the pure mononitrotoluenes.

The mixture of isomers from the batch still are passed to a continuous still of the packed column type, the ortho isomer is withdrawn from the top of the column, while a mixture of the meta and para-isomers are withdrawn from the bottom. This mixture is then cooled to crystallize out the desired para mononitrotoluene, which is recovered by a centrifuge.