



uniteltechnologies

Your Partner in the Pursuit of Process Innovations

THE FAIR COLUMN. Reconfigurable for Distillation Pilot Plant Studies

Unitel Technologies has been involved in designing and building a variety of distillation systems over the last several decades. Based upon our extensive project experience, we have developed a highly flexible and reconfigurable design.

Distillation is one of the most important unit operations in the field of chemical engineering. It is a mass transfer process that is uniquely characterized by the McCabe-Thiele diagram to predict the number of theoretical stages for the intended operation. However, theoretical formulas cannot accurately predict efficiencies, capacities and pressure drops in a distillation column. In order to compensate for the lack of vapor-liquid (VL) equilibrium data, potential

azeotrope formations, reactions and difficulty of separations, it is generally necessary to conduct experiments and determine the actual number of mass transfer stages.

Professor James Rutherford Fair was a leading proponent of flexibility in the design and operation of distillation systems. He developed the concept of adding or deleting the number of transfer stages in the column while keeping the heat input to the reboiler and heat withdrawal in the overhead condenser external to the primary column. His design is a radical departure from the rigid limitations of traditional distillation systems.

DISTILLATION



**REACTIVE
DISTILLATION**



**EXTRACTIVE
DISTILLATION**

Column

The number of stages will vary by application resulting in a change in the height of the distillation column. The Fair column has been engineered to specifically address variations in stage requirements for different applications. This is readily achieved by inserting spool pieces of different lengths ranging from 3 ft. to 6 ft.

Reboiler

This section introduces heat into the distillation column. It has been configured for extreme flexibility by making sure that it is separated from the column, thus enabling it to be changed and/or replaced when necessary. The reboiler is equipped with an electric heater that enables operation over a much wider range of temperatures.

Overhead Condenser/Reflux

A special spool piece located at the top of the column enables the vapors to exit and the refluxed liquid to be directed back into the column. Vapors flow into a vertically mounted condenser through a pipe that is comprised of an appropriate number of spool pieces. This design enables the condenser and reflux pumps to be located near ground level while the top section of the column can be moved up and down by removing and/or adding the appropriate spools.



Professor James R. Fair

Professor James R. Fair was the John McKetta chair in Chem. Eng. at the University of Texas. He established the UT Separations Research Program (SRP) and was internationally recognized as a pioneer and leader in the field of distillation. His industrial career included work at Shell followed by several years at Monsanto where his final position was Director of Corporate Technology.

FAIR COLUMN SPECIFICATIONS

- Maximum column temperature 200°C (390°F) / full version
- Maximum column pressure 10 barg (145 psig)
- Standard column diameter 6 inches
- Standard vapor line diameter 1 inch
- Standard spool pieces 36 inches & 72 inches
- Column internal options Structured packing
 - Non-reactive packing
 - Reactive packingSieve valve
- Reboiler Electrically heated
 - 25 kW
 - 50:1 turndown

Column Internals

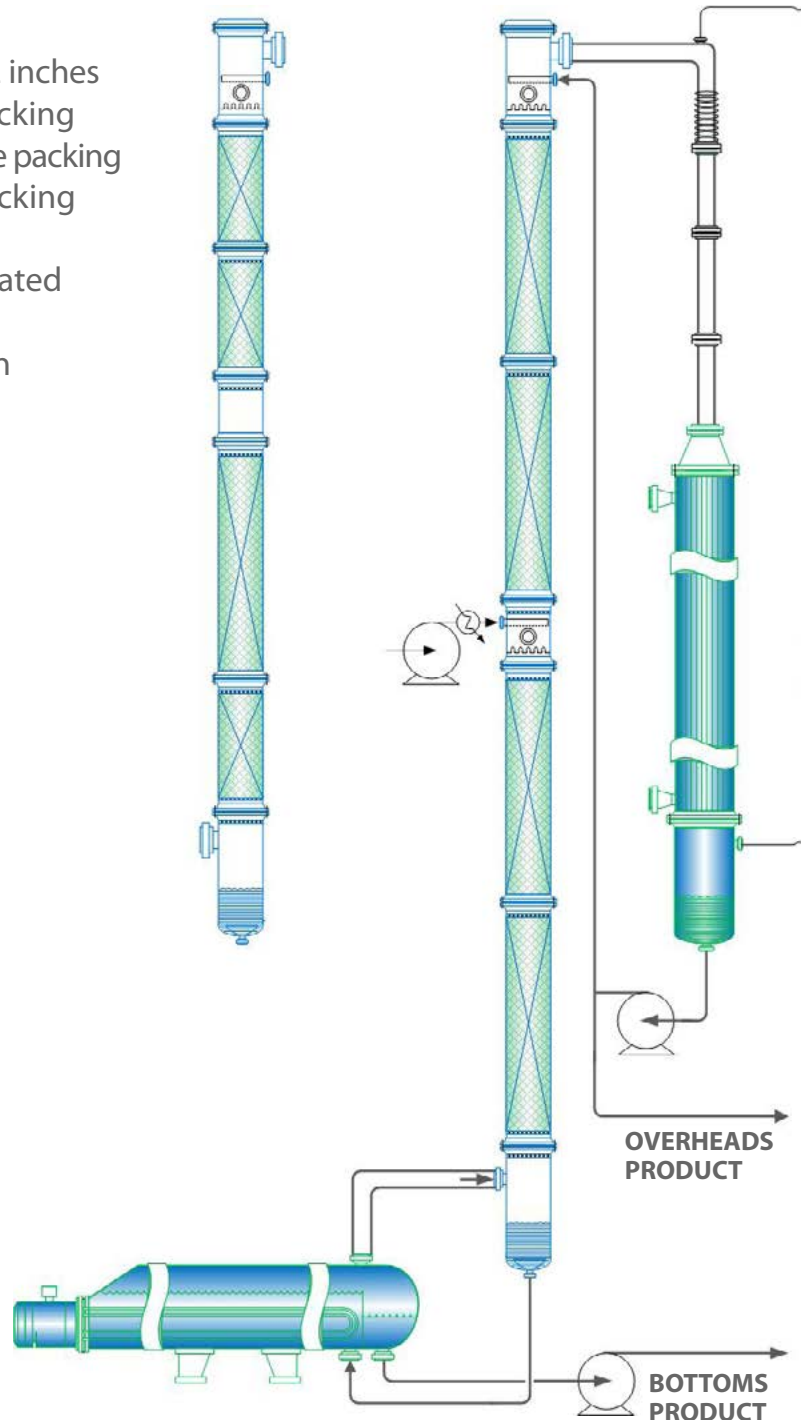
Column internals – trays or packings -- used to enhance component separations.

Trays

- Valve trays have perforations that are covered by liftable caps.
- Sieve trays are simply metal plates with holes in them.

Packings

- Random packings are passive devices designed to increase the interfacial area for vapor-liquid contact.
- Structured packings consist of thin corrugated metal plates, gauzes or wire mesh arranged such that they force fluids to take complicated paths in addition to increasing interfacial area.



Two separate common configurations

RECONFIGURABLE FOR REACTIVE DISTILLATION PILOT PLANT STUDIES

Reactive distillation is a unit operation that combines reaction and distillation.

This process intensification technology offers the benefits of lower energy and capital costs.

Reactive distillation includes heterogeneous catalyst reactions, homogeneous catalyst reactions and thermal non-catalytic reactions. These reactions typically occur in the liquid phase, specifically applicable to equilibrium limited reactions in which one of the reaction products is the more volatile component. The design of a reactive distillation process has to be optimized

in order to achieve stable operation.

Most reactive distillation processes have to be developed in pilot plants and demonstration units prior to commercialization.

The complexity of this technology includes catalyst optimization, catalyst location, gas and liquid traffic, gas-liquid separation and last but not least the enthalpy exchange protocol. Modeling is useful, but real data is necessary before commercial implementation.

The Fair Column can be easily reconfigured for reactive distillation studies. Spool

pieces of appropriate lengths can be installed for optimizing reaction residence times.

Reactive distillation is the preferred choice:

- When the reaction equilibrium limitation has a high level of exothermicity that necessitates heat removal by cooling a conventional reactor.
- When the main reaction is comprised of serial reactions and the product is either the light or heavy component in a separation.
- When an azeotrope formation is prevented by a chemical reaction.

CATALYST LOADING

The key in developing a reactive distillation technology is the design of the internal elements that house the catalyst. Many catalysts, particularly in the resin family, are small and subject to swelling resulting in a compact mass. This usually causes an excessive pressure drop that retards the upward flow of vapor and downward flow of the liquid. Some methods for optimizing catalyst loading are:

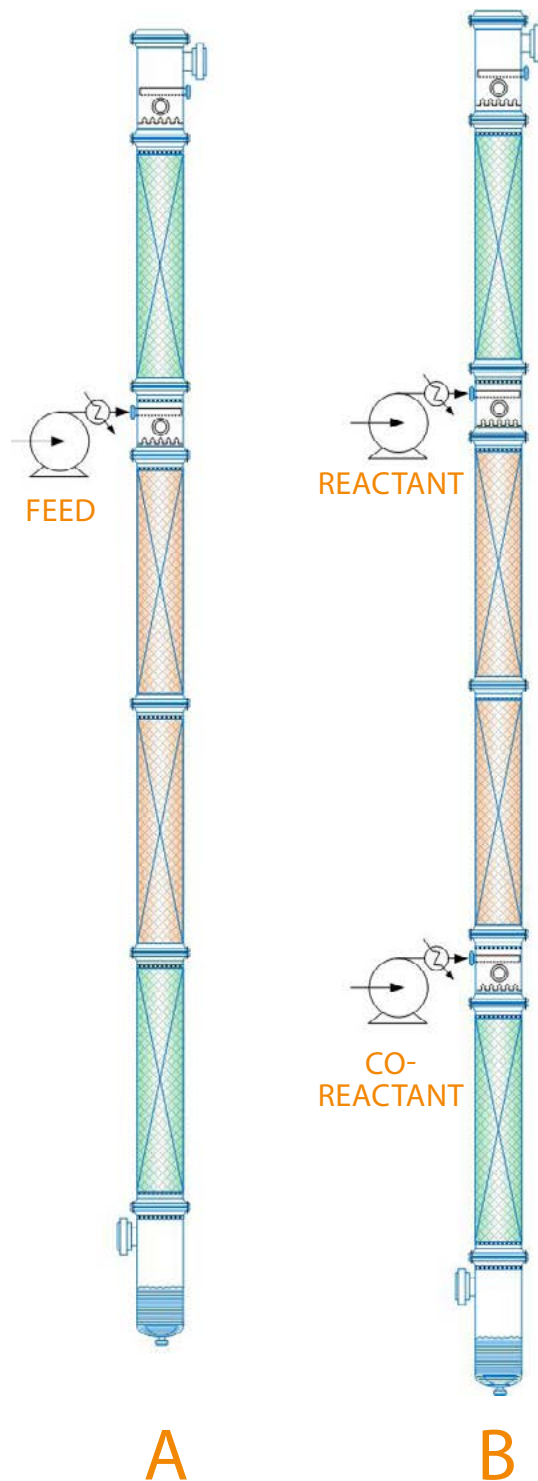
- Bale packing: the catalyst is sealed within pockets in a fabric belt, which is then wrapped in an open mesh knitted non-reactive wire.
- Structured packing: constructed using corrugated screen envelopes with catalyst filled inside. Each envelope comprises of two layers of crimped screen.
- Catalyst bags: the catalyst is loaded inside wire mesh bags similar to tea bags. These bags can then be randomly loaded or contained within plates of structured packing.
- Random packing: the catalyst may consist of extrudates or rings that are dumped in the column so that reactions and distillative mass transfer can occur simultaneously.
- Catalyst container held on a tray: comprises of rectangular metal screen troughs filled with catalyst. These troughs must be located within a froth zone when the column is operational.
- Sandwich containers: these metal screen tubes are tall enough to extend between trays.
- Conventional distillation trays with catalyst placed in the downcomer: the reaction zone is in the downcomer and the distillation zone is in the trays.
- Integrated tray: this configuration has a distillation section, a mixing section and a reaction section all integrated for simultaneous catalytic reaction and distillation.

The Fair Column can be easily reconfigured for reactive distillation studies. Spool pieces of appropriate lengths can be installed for optimizing reaction residence times.

Two potential designs are shown on the right.

- In arrangement A, the “orange” represents the reactive distillation sections and the feed is introduced at the top of this stage. A rectifying section is included above the feed introduction point while the stripping section is located below the reactive stage. A typical example of this configuration involves the dehydration of methanol to make DME.
- In configuration B, the “orange” represents the reactive distillation sections. In this mode, one of the reactants is fed at the top of the reactive section while the other coreactant is fed at the bottom of the section. The reaction occurs over the catalyst where the two reactants flow in a countercurrent mode. The preferred product may leave the top of the column, but more often than not, it leaves at the bottom. Esterification is a common application in which the organic acid is fed at the top and the alcohol from the bottom.

The catalyst in the reactive section may be placed on top of the trays, as encapsulated packing -- Katapak or woven inside Teflon fabric bags that may be shaped like tea bags and/or in longer lengths that are then rolled and pushed into the columns.



Some common applications for reactive distillation are:

- | | | |
|----------------------------------|----------------------|--------------------------|
| • Esterification | • Alkylation | • Carbonylation |
| • Transesterification/Hydrolysis | • Metathesis | • Polymerization |
| • Etherification | • Disproportionation | • Chlorination/Amination |
| • Hydrogenation | • Hydration | • Chiral Separation |
| • Hydrodesulfurization | • Dehydration | • Acetalization |

RECONFIGURABLE FOR EXTRACTIVE DISTILLATION PILOT PLANT STUDIES

Extractive distillation is a process for distilling and separating mixtures that possess a low value of relative volatility, often approaching one. Such mixtures cannot be separated by simple distillation because the volatilities are almost identical, thus causing them to evaporate at nearly the same temperature and at a similar rate.

Extractive distillation uses a separation solvent that is non-

volatile, has a high boiling point and is miscible in the mixture without forming an azeotrope with either feedstock component.

The solvent interacts differently with the components of the mixture, causing a change in the relative volatilities. This change in volatility enables the three part mixture to be separated by normal distillation. The component

with the greatest volatility separates out as the overhead product.

The bottom product typically comprises of a mixture of the extractant and the component with the higher volatility. The bottom product can be readily separated using conventional distillation and the extractant can be recycled.

The initial seminal work on developing the technology of extractive distillation and solvent selection was conducted by Professor Lloyd Berg at Montana State University.

Extractant Selection & Screening

The purpose of screening extractants is to determine whether extractive distillation is a promising separation technique and to rank several solvents in order to select the most effective compound.

Estimating effectiveness of solvents involves determining coefficients of the solutes in the presence of solvents. This is typically done by using the UNIFAC method which uses the functional groups present on the molecules that make up the liquid mixture to calculate activity coefficients. The primary effect of adding a solvent to the original binary mixture is to alter the ratio of activity coefficients. Thus, for a given binary system, the ratio of vapor pressure can be expected to remain relatively constant and the effectiveness of a solvent can be measured by enhancement of the activity coefficient.

Some common extractive distillation applications:

Application	Extractant
BTX aromatics purification	Sulfolane, n-methyl pyrrolidone
Cycloparaffin separation	Triethylene glycol
n-propylalcohol from butanol	Sulfolane
Butadiene from C ₄ olefins	Sulfolane
Acetic acid from water	Tributyl amine
Methanol & methanol acetate	Water
Propylene & propane	Acetonitrile

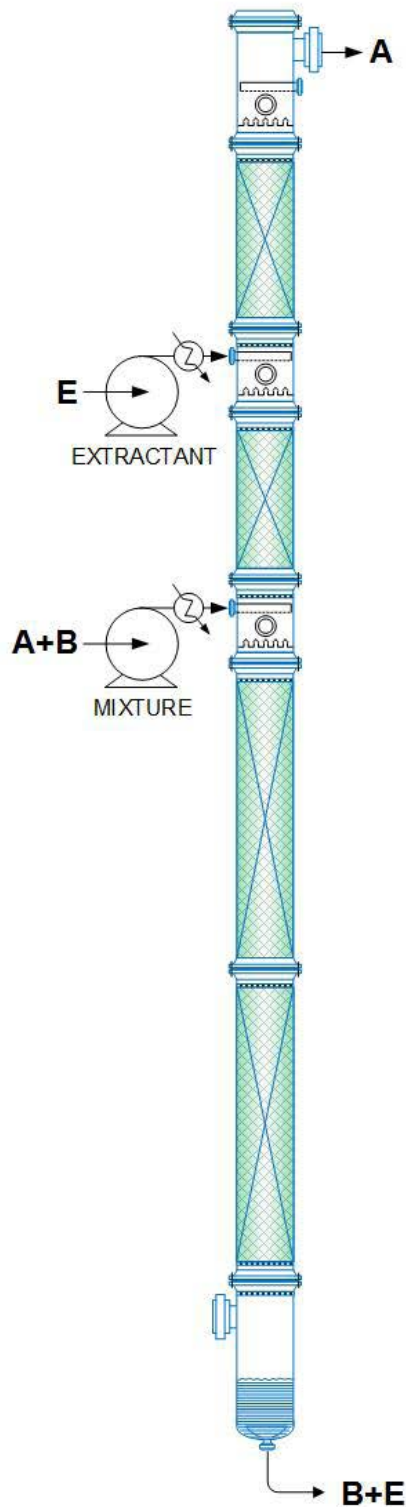
Extractive distillation uses a separation solvent (extractant) that should follow some of the rules shown below:

- Significantly enhance the natural relative volatility of the key components.
- Be miscible within the compounds to be separated.
- Be easily separable from the bottom product for the usual case of a heavy solvent.
- Be stable at the temperatures of distillation and solvent separation.
- Be non-reactive with the materials of construction.
- Possess good viscosity characteristics.

The figure on the left illustrates a typical application. The mixture to be separated is introduced in the lower section while the extractant is introduced a couple of trays above that.

As the non-volatile extractant flows down the column, it preferentially associates with the more polar compound and exits at the bottom of the column. The column reflux stream knocks back any entrained extractant.

Spool pieces of various lengths can be utilized for optimizing the design. In extractive distillation hydrogen bonds are an important factor. It's critical that the extractive agent be easily separable from the components.



The criteria for successful extractive distillation agents is that they boil higher than the components being separated, form no minimum azeotropes with the components and be a highly hydrogen bonded liquid.

These liquids can be classified into two major groups:

Class I: Liquids capable of forming three dimensional networks of strong hydrogen bonds – e.g. water, glycol, glycerol, amino alcohols, hydroxylamine, hydroxyl-acids, polyphenols, amides, etc.

Class II: Other liquids composed of molecules containing both active hydrogen atoms and donor stems (oxygen, nitrogen and fluorine) – e.g. alcohols, acids, phenols, primary and secondary amines, oximes, nitro compounds with alpha-hydrogen atoms, nitriles with alpha-hydrogen atoms, ammonia, hydrazine, hydrogen fluoride, hydrogen cyanide, etc.

If you are interested in learning more about **The Fair Column and its applications**, please contact Unitel Technologies:

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