

# Cyclic Distillation Technology - A New Challenger in Fluid Separations

Anton A. Kiss<sup>a,b,\*</sup>, Vladimir N. Maleta<sup>c</sup>

<sup>a</sup>The University of Manchester, School of Chemical Engineering and Analytical Science, Centre for Process Integration, Sackville Street, The Mill, Manchester M13 9PL, United Kingdom

<sup>b</sup>University of Twente, Sustainable Process Technology, PO Box 217, 7500 AE Enschede, The Netherlands

<sup>c</sup>Maleta Cyclic Distillation LLC OÜ, Parnu mnt 130-38, 11317 Tallinn, Estonia  
 tony.kiss@manchester.ak.uk

This paper provides a dual academic and industrial view on cyclic distillation. The cyclic mode of operation is based on separate phase movement (SPM) that can be achieved with specific internals. This leads to major advantages: increased column throughput, reduced energy requirements, and better separation performance (exceeding 200% Murphree efficiency). Next to the substantial theoretical developments in terms of process modeling and simulation, there have been also significant advances in the practical demonstration at pilot and large scale. All this contributed to a better understanding of the design, control and optimization of cyclic distillation processes, and to the development of more robust industrial solutions. The application range is now expanding to other areas, such as: oil refining, chemistry, petro-chemistry, pharmaceuticals, biofuels and others.

## 1. Introduction

Although considered by many professionals a mature field, distillation has enjoyed during the past decades a number of major enhancements based on process intensification: e.g. HiGee distillation, dividing-wall column, heat-integrated distillation, membrane distillation, reactive distillation (Kiss, 2013). Among these advanced technologies, cyclic distillation stands out as a new challenger in fluid separations due to a different way of thinking in contacting the liquid and vapor phases (Maleta et al., 2011). In contrast to conventional operation, cyclic distillation uses separate phase movement (SPM) that can be achieved with specific internals and a periodic operation mode (Kiss and Bildea, 2015). One operating cycle consists of two key parts: a *vapor flow period* (when the thrust of rising vapor prevents liquid down flow) followed by a *liquid flow period* (when the liquid flows down the column, dropping by gravity, first to a lock chamber and then moving to the tray below) – see Figure 1 and Figure 2. A cyclic distillation column looks from outside just like a regular tower. However, the cross section view inside a cyclic distillation column shows the absence of downcomers and the presence of different internals (Figure 2, left) that allow an efficient SPM in practical operation. A precise analogy exists between the cyclic and conventional distillation, which basically is reduced to the substitution of time as independent variable (in case of cyclic distillation) for distance (in the case of conventional distillation). Recent articles and a mini-review of cyclic distillation provides more details about the history of cyclic distillation, working principle, design and control methods, benefits and limitations (Patrut et al., 2014; Bildea et al., 2016).

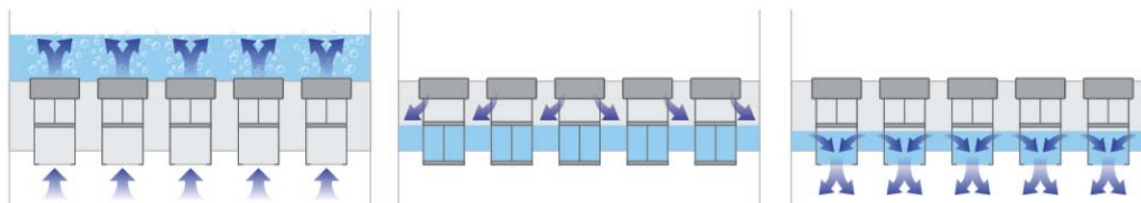


Figure 1: Operation of cyclic distillation systems: vapour flow period (left), first half-liquid overflow: tray – sluice chamber (middle), second half-liquid overflow: sluice chamber - tray below (right)

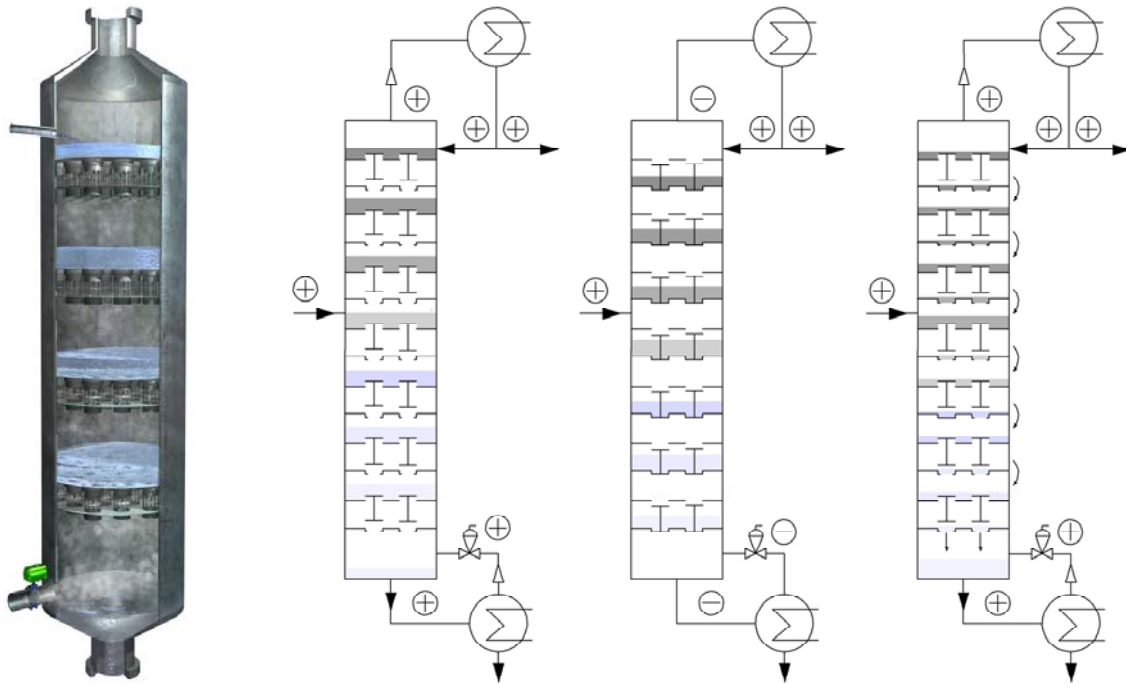


Figure 2: Cross section of a cyclic distillation column (left). Schematics illustrating the operation principle of cyclic distillation system: vapour flow period, followed by 1<sup>st</sup> and 2<sup>nd</sup> half-liquid overflow (right).

**2. Process theory**

The theory of the cyclic distillation processes has been developed along several decades of engineering. The best established design algorithms for cyclic distillation columns include analytical and numerical methods of increased complexity and usability, developed during 1977-2014 (Nielsen et al., 2017). Figure 3 illustrates the modeling of a classic theoretical stage and the operating lines in case of classic vs cyclic distillation – where  $L$  and  $G$  are the liquid and gas (vapor) flow rates,  $H$  is the liquid holdup on the tray (mol),  $\tau$  is the vapor period duration (s), while  $x$  and  $y$  are the liquid and vapor composition, respectively (Maleta et al., 2011).

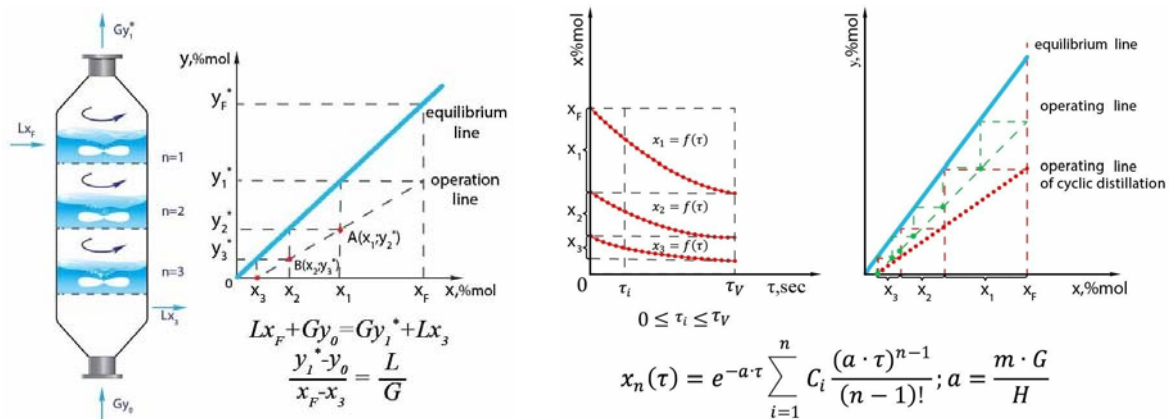


Figure 3: Conventional theoretical stage and operating lines (left), operating lines for cyclic mode (right)

More recent studies proposed iterative numerical solutions of the cyclic distillation simulation model with simultaneous tray drainage. Figure 4 shows the iterative procedure, where  $M$  and  $x$  represent trays holdup and composition and the superscripts  $(V)$  and  $(L)$  refer to the end of vapor- and liquid-flow periods, respectively (Bildea et al., 2016). Note that  $\Phi^{(V)}$  and  $\Phi^{(L)}$  are the model equation used to calculate the state of the system at the end of the vapor- and liquid-flow periods. More recently, the driving force based design has been adapted from conventional continuous distillation to cyclic distillation (Nielsen et al., 2017).

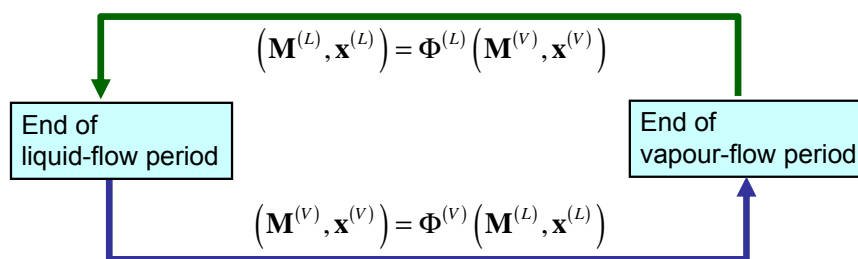


Figure 4: Iterative solution of the cyclic distillation simulation model with simultaneous tray drainage.

### 3. Techno-economical advantages

The cyclic mode of operation leads to major economic advantages (reported theoretically and experimentally):

- 20-50% lower investment cost (due to lower column height; smaller column diameter; smaller area of the heat exchangers; less steel construction; less space used).
- 20-35% lower hot utility usage (due to high mass transfer efficiency; reduction of the reflux rate)
- 20-35% lower cold utility usage (lower reflux rate)
- Improvement of product quality (high mass transfer efficiency; higher concentration of key product)
- Increase of product yield (high mass transfer efficiency; more concentrated impurities or other fractions)
- Enhanced process sustainability (less GHG emissions due to lower energy usage)

The technological advantages of cyclic distillation include:

- Mass transfer efficiency of 1 cyclic distillation tray is equal to 3 classic trays
- Reduction of the residence time of liquid in the column, and uniform arrangement of liquid on the tray
- Ability to control the amount of liquid on the tray, and the reaction time (in case of reactive distillation)
- Any geometric configuration of the trays (allows the possibility to build dividing wall columns with trays)
- The separation efficiency does not depend on the column diameter, thus easy industrial scale-up.
- Placement of any type of packing between the trays further increases the mass transfer efficiency
- The pressure drop in the column does not depend on the liquid load in the column, since the amount of liquid on the trays is constant, and only the frequency of cycles is changed (range: 1-30 m<sup>3</sup>/m<sup>2</sup>hr liq. load)
- The vapor velocity in the column typically ranges 0.2-20 m/s, as it depends on the pressure in the column.
- The operation remains stable and efficient in case of changed concentrations of the key components

However, in all fairness, cyclic distillation also has some important limitations: it is difficult to apply to vacuum systems, and the performance enhancement critically depends on the separation of the V/L periods.

### 4. Industrial equipment

The industrial interest in cyclic distillation has been renewed due to the availability of design and control methods (supported by increased computing power) and the introductions of special trays that allow better control of phase movement. Due to these developments, during the past decade cyclic distillation turned into a real challenger of the status quo in distillation at industrial scale, being ready to make a difference in many processes and getting accepted as a proven technology. Along with the scientific research carried out at several universities in EU, cyclic distillation sparked the interest of large companies e.g. BASF (Buetehorn et al., 2015), software vendors, and technology providers e.g. MaletaCD (Maleta et al., 2015). Although all simulation studies predicted enhanced separation performance, when applying conventional internals in practice the fluid dynamic limitations restrained the industrial breakthrough of cyclic distillation technology. However, this barrier has been removed with the development of new internals (Maleta et al., 2011). Also, the cyclic operation uses reliable stop valves (butterfly valves with pneumatic drive) from leading manufacturers, which can withstand up to 20 million cycles (e.g. first industrial columns operated well for more than 10 years). The current leader offering cyclic distillation technology (MaletaCD) has developed some novel engineering solutions for more efficient contact devices (e.g. Maleta trays with lock chambers allowing the liquid to follow the path: tray - lock chamber - tray below) – see Figure 5 and Figure 6. Practical implementation at pilot and large scale (see column illustrated in Figure 7) has showed the following benefits in practical operation:

- High tray efficiencies (140-200% Murphree efficiency), thus reduced equipment cost.
- Higher throughput and equipment productivity than conventional distillation.

- Reduced energy requirements (20-35% savings), thus lower operating costs.
- Increased quality of the products due to the higher separation efficiency.



Figure 5: Trays suited with sluice chambers, especially designed for cyclic distillation columns



Figure 6: Top view of an installed cyclic distillation tray column with a diameter of 1.7 m (left), and visualization of cyclic distillation trays suited with solid catalysts for catalytic distillation applications (right)



Figure 7: Cyclic distillation (dividing-wall) column prepared for transportation to be installed on a chemical site

Pilot distillation systems are available at MaletaCD for testing various feedstocks in the temperature range of 50-320°C and pressure range 5 kPa – 1 MPa for absorption, distillation and reactive distillation. The pilot column can work in three modes: stripping column, rectification column, and full column (with both stripping and rectification sections). The stripping and rectification parts have 20 trays each, being equivalent to a full conventional column with 120 trays. The services offered include a wide range: process simulation, pilot plant testing, technology development, basic and detail design, industrial automation and control solution, column and mass transfer equipment manufacturing, revamping and optimization of the existing columns, installation / supervision, start-up and staff training, project management, warranty (e.g. 10 years) and service.

## 5. Industrial applications

So far cyclic distillation has been used mostly in the food industry (for concentrating alcohol) but the application range is expanding to other areas with potential use, such as: oil refining, chemistry, petrochemistry, pharmaceuticals, biofuels, etc. Moreover, cyclic distillation offers new opportunities by applying the same principles of the cyclic operation mode to other intensified processes, such as catalytic distillation (CD) or dividing-wall column (DWC) - see Figure 8. Today, cyclic distillation technology works in stripping columns, rectification columns, and tray dividing wall columns with a diameter ranging from 400 to 1700 mm. Cyclic distillation technology is employed in the production ethanol food grade, biofuels, recovery of methanol from water and/or acetone, distillation of various chemicals (ethers, propylene, propanol, hexane, formalin, aniline, cyclohexane, butyl acetate, ethylbenzene, methyl acetate, BTX), isopropyl alcohol dehydration, industrial solvents distillation (white spirit), cleaning of raw coal benzole, fractional of kerosene (with implementations in Ukraine, Republic of Belarus and Saudi Arabia).

Since 2006, MaletaCD has built and installed commercial scale plants with cyclic distillation columns of 5 to 42 trays and column diameters of 0.4-1.7 m. More recently, MaletaCD built in 2014 an industrial scale dividing wall column using cyclic operation (42 trays, 1.5/1.7 m diameter, and a capacity of 25 m<sup>3</sup>/hr) for a plant processing kerosene and white spirit (see Figure 8). Figure 9 illustrates the industrial implementation of cyclic distillation technology in a plant producing ethanol in the food industry, as well as the first use of cyclic distillation dividing-wall columns for fractionating kerosene.

The main areas of uses cyclic distillation technology in petroleum refining include: high-quality fractionation of gas; high-quality narrow fractions during vacuum / atmospheric distillation of petroleum; second refining of all types of petroleum cracking; combining reaction and distillation processes (catalytic distillation); obtaining isomers of high added value: 1,2,3-trimethylbenzene; mesitylene (1,3,5-trimethylbenzene); durene (1,2,4,5-tetramethylbenzene); isodurool (1,2,3,5-tetramethylbenzol) and others. However, some potential limitations of the process include: crude oil processing; liquid load on the tray does not exceed 30 m<sup>3</sup>/m<sup>2</sup>hr.

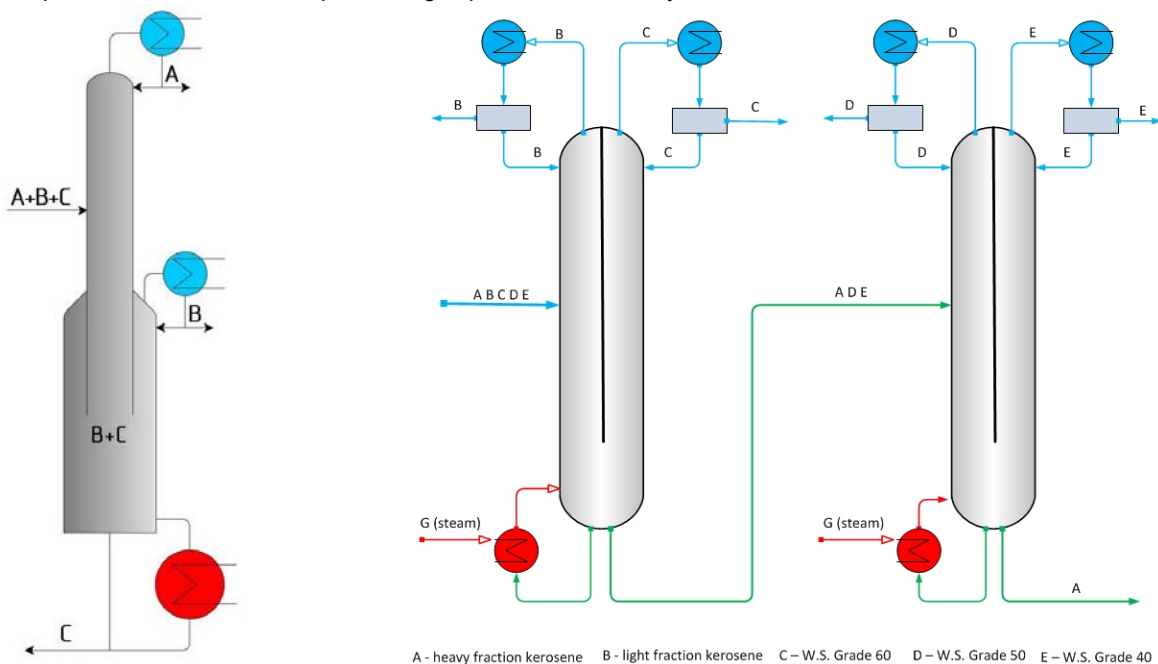


Figure 8: Cyclic distillation dividing-wall column with coaxial partition – any geometric shape is possible for cyclic distillation (left). Novel process for fractionation of kerosene using cyclic distillation DWC (right).



Figure 9: Cyclic distillation column as part of a plant producing ethanol in the food industry (left), and two cyclic distillation dividing-wall columns installed for fractionation of kerosene (right).

## 6. Conclusions

Cyclic distillation can significantly improve fluid separation process, providing key benefits such as: reduced energy requirements, lower capital costs, increased column throughput and better separation performance. The separate phase movement also provides more degrees of freedom that contribute to good process control and simple operation. Yet, similar to the history of other process intensification technologies (e.g. DWC), the chemical industry seems to be reluctant in adopting new technologies due to perceived issues such as: difficult process control, unavailability of adequate models in process simulators, or reliability of moving parts to sustain the cyclic operation. That is why it is crucial to have industrial champions leading by example and willing to pave the way by implementing cyclic distillation and addressing all the perceived and real issues.

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