

Optimisation of the Rectisol™ Design with Packing: the Rectisol™ Demonstration Unit

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The Rectisol™ process is used for more than 80% of the gasification projects for acid gas removal. The Rectisol™ process is an efficient way to remove sulfur compounds, CO₂ and other traces from syngas with chilled methanol as solvent. In order to respond to the customer need for continuous CapEx (Capital Expenditure) and OpEx (Operational Expenditure) improvement, the use of packing to replace trays in the absorber and reabsorber columns seems promising. Nevertheless, as Rectisol™ is a process with a large range of operating conditions (Liquid to gas ratio (L/G), temperature, pressure, liquid loads...), the correlations from literature, mostly established for low pressure distillation, should be out of their domain of validity for some sections. This uncertainty leads to increased design margins to ensure the quality of the product gases. In order to have a better understanding of the performance of packings for Rectisol™ applications, a demonstration unit has been built and connected to a commercial size Rectisol™ unit in order to perform tests with packings. Gas and liquid flows circulating in the Rectisol™ Demonstration Unit (RDU) are directly extracted from and returned to the main plant. The RDU generates performance data on the use of packing in the Rectisol™ columns of an industrial scale application and significantly improves the understanding of the operating limits.

Based on the results of the tests performed on the RDU, optimized design rules for the Rectisol™ process with packing have been defined. This new design allows to significantly reduce the CapEx and OpEx compared to the unoptimized design with packing or tray design and has already been applied in industrial scale Rectisol™.

Keywords: Packing, Rectisol, acid gas removal, pilot plant

1. Introduction

The Rectisol™ (Air Liquide, 2018) gas purification technology is a physical absorption process operated at low temperatures and high pressures. The absorption medium used is a liquid organic polar solvent, methanol. Mass transfer from the gas into the methanol solvent is driven by the concentration gradient of the respective component between the gas and the surface of the solvent, the last being dictated by the absorption equilibrium of the solvent with regard to this component. This means that all gas components detrimental to the downstream process stages, typically hydrogen sulfide, carbonyl sulfide, mercaptans, hydrogen cyanide, ammonia and other harmful compounds as well as carbon dioxide are almost completely removed from gases with a small rate of circulating solvent and thus, low energy requirements. Extremely high purities - e.g. a total sulfur content of less than 0.1 ppmv - can be reached, making the product gas suitable for being fed to most syntheses without further purification steps. The compounds absorbed are removed from the solvent by flashing (desorption) and additional thermal regeneration, so that the solvent is ready for new absorption.

The Rectisol™ technology provides an option to recover the major part of the carbon dioxide contained in the feed gas as a pure CO₂ product stream. Recovery of the full amount of CO₂ (e.g. for sequestration) is also possible with a slightly modified process set-up. Acid gas containing the sulfur components is enriched in the

Rectisol™ to concentrations above 30% of hydrogen sulfide, ensuring an optimized post-treatment is possible. The acid gas is routed to battery limit, usually for processing in a Claus Sulfur recovery unit.

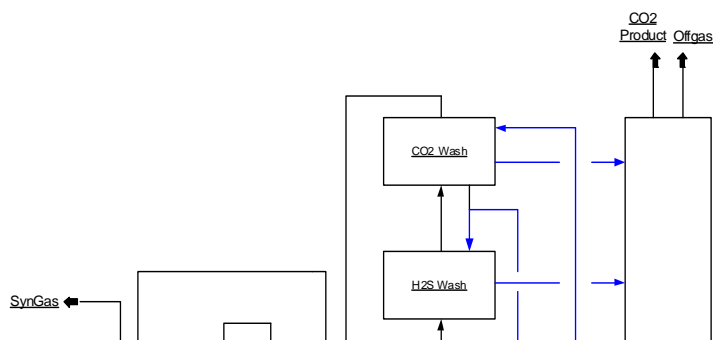


Figure 1. Rectisol™ configuration downstream entrained flow gasification

The absorber operates at a high pressure (Typically >30 bar), where hoop stress and column shell thickness are considerable. A reduction in column diameter would reduce the hoop stress, and thus the thickness and cost of the column and its structure, which is worth 5 to 7 times more than the internals. It follows that total installed cost optimization requires reducing the column diameter even if it leads to increasing the total volume of the internals. A high capacity packing, which will result in a smaller column diameter and thickness, is consequently essential in reducing the costs. The absorber design is purely driven by CapEx considerations. The reduction of the diameter is also an important factor for the Chinese market, indeed the column diameter is limited by the road infrastructure and for large scale Rectisol™ without the Air Liquide optimized design, the absorber may have to be split in two columns due to this limitation.

The other column of interest in the Rectisol for packing application is the reabsorber (part of the cold flash regeneration in Figure 1). The reabsorber operates at a low pressure (~2 bara) and a high liquid load in some sections. The purpose of this column is to enrich the methanol with H₂S while releasing a stream of pure CO₂ by re-absorbing any H₂S released through flashing and stripping in the bottom of the column. A reduction in the pressure drop of the reabsorption section yields a reduction in power or utilities consumption, especially refrigerant. A 60% reduction in the pressure drop of the reabsorption section would generate annual OpEx savings equivalent to the cost of the column internals. The design of the reabsorber is consequently primarily driven by OpEx considerations. A packing which generates a low pressure drop per theoretical stage is required to achieve these OpEx savings.

2. Materials and methods

2.1 Process and instrumentation

The Rectisol™ Demonstration Unit (Figure 2) is divided into two sections: one cooling section (S-2) to control the gas temperature and one test section (S-1) which allows tests to be performed with different column internals. The column internal diameter is 40 cm and the maximal height of packing to be tested in the test section is 4 m. A flash vessel (F) upstream of the test column prevents flashing at the inlet of the packed section. Various instruments are available to measure the hydrodynamic parameters and those necessary for the calculation of the Height Equivalent to a Theoretical Plate (HETP) and to check the stability of the RDU. The RDU is able to be operated at pressures ranging from atmospheric to the main plant absorber pressure. The RDU is operated under real industrial conditions using slipstreams meaning operating conditions and composition from a real plant, the pressure of the test is the same as the pressure of the section of the industrial plant. This allows the real conditions to be represented including the impact of all the compounds present in the raw gas from the gasification units.

In order to be able to determine the efficiency of the packings tested in the RDU, the composition of the gas and liquid phases has to be known. In addition, samples are taken directly from within the packing allowing the measurement of the column concentration profile. Online gas analyses allow the measurement of both H₂S and CO₂ by using a Gas Chromatograph equipped with TCD (Thermal Conductivity Detector) and FPD (Flame Photometric Detector).

Regarding the liquid phase, two sampling points for offline analyses are available:

- one for the liquid inlet which is on the line between the flash vessel and the column inlet
- one for the liquid outlet downstream of the test column.

The sample of chilled methanol laden with CO₂ and H₂S is a challenging point. Depending on the campaigns, the solvent stream is saturated, then the changes in the temperature or pressure may lead to a stripping into the sampling line and therefore may alter the sample composition. Various methods have been developed and tested.

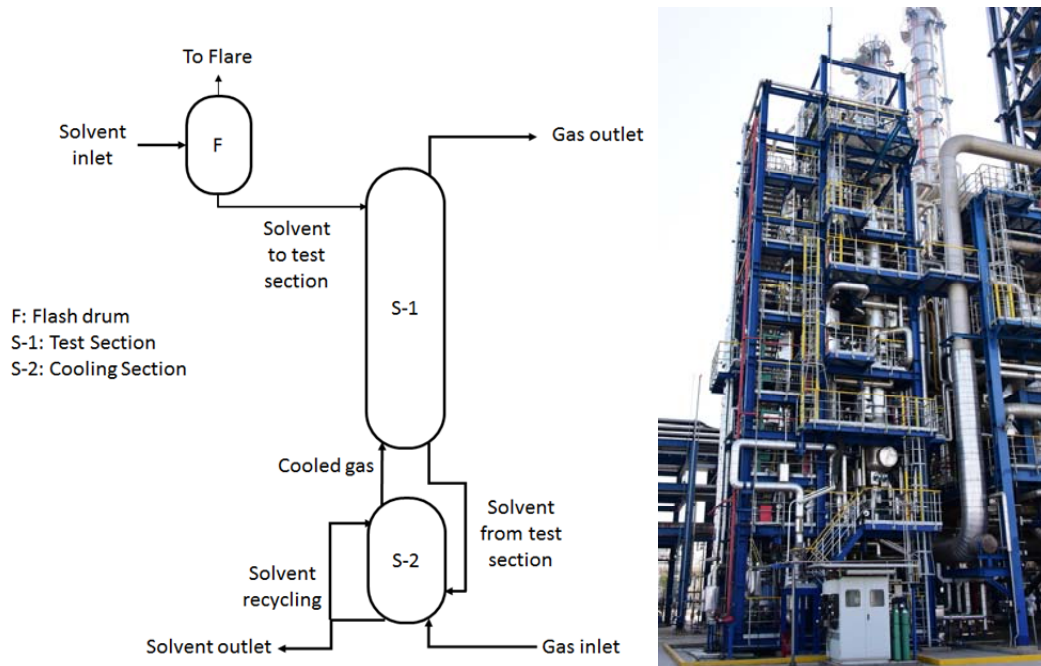


Figure 2. Block flow diagram (left) and picture (right) of the Rectisol™ Demonstration Unit

2.2 Method

Each packing has been tested on 7 campaigns, the present paper focuses only on 3 campaigns, each campaign corresponds to specific gas and liquid feeds from the industrial Rectisol™ plant. The table 1 represents the tests performed on the packings for the different campaigns. In the tests, flooding is defined as the point where liquid entrainment is observed in the gas outlet line, this definition differs from vendor definitions which consider that the flooding of the packing corresponds to a certain pressure drop (Lockett M.J. et al., 2006). These tests allow a better estimation of the maximum capacity that can be used for the design of the column and to compare the real measurements to vendor curves.

Table 1. Information obtained on the packings for each campaign

	Campaign C1			Campaign C2			Campaign C3		
	Flooding	Pressure drop	HETP	Flooding	Pressure drop	HETP	Flooding	Pressure drop	HETP
Packing 1	X	X	X						X
Packing 2	X	X	X	X	X		X	X	X
Packing 3	X	X	X	X	X		X	X	X

In order to study the impact on main design parameters on packing efficiency, tests are performed for different L/G and different flow parameters. The tests performed on the RDU provide raw data for each sensor (flow, temperature, pressure, density...) which need to be evaluated to give a set of data corresponding to a steady

state period. In order to be able to have a complete gas composition profile, the minimum time period is 2 hours. Longer time periods were preferred to assess the stability of the gas compositions. Figure 3 presents the workflow of campaign execution.

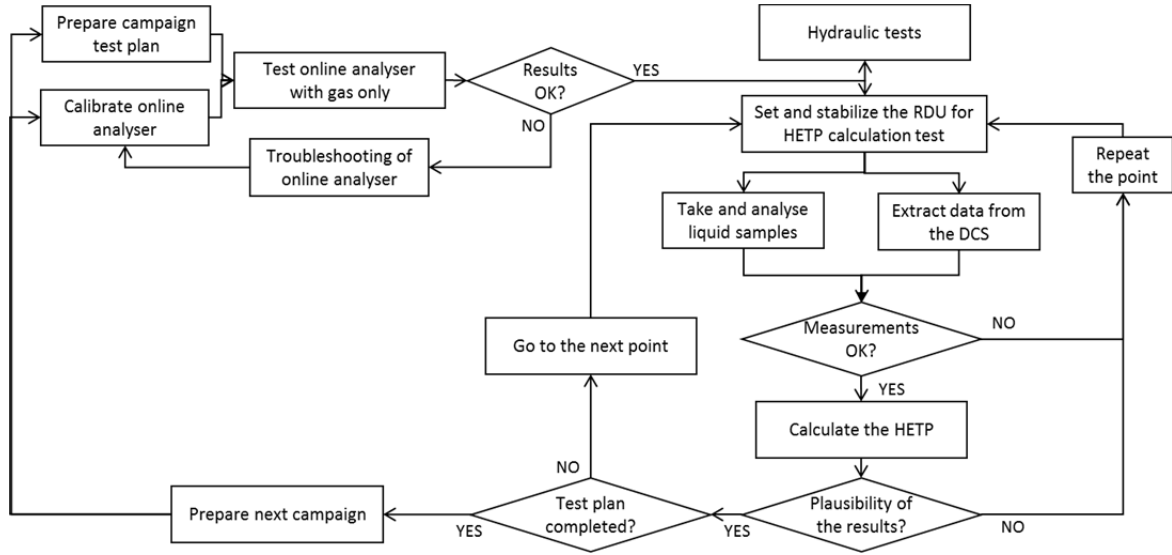


Figure 3. Workflow RDU campaign execution

To calculate the HETP with AspenPlus using EO modeling, the equilibrium approach using Murphree efficiency has been used to take into account the non-idealities in a real column. This method is common for trays sizing. This approach allowed calculation of a non-integer number of stages required for the separation by considering an efficiency of the stages. The Murphree efficiency considering CO_2 transfer is defined in equation 3. In case of H_2S absorption, the equation is the same but using H_2S properties instead of CO_2 .

$$Eff_{\text{CO}_2,j} = \frac{y_{\text{CO}_2,j} - y_{\text{CO}_2,j+1}}{K_{\text{CO}_2,j}x_{\text{CO}_2,j} - y_{\text{CO}_2,j+1}} \quad (1)$$

With K_{CO_2} the equilibrium K value for CO_2 defined by a proprietary thermodynamic model, x_{CO_2} the CO_2 liquid mole fraction, y_{CO_2} the CO_2 vapor mole fraction, Eff_{CO_2} the CO_2 Murphree efficiency and j the stage index.

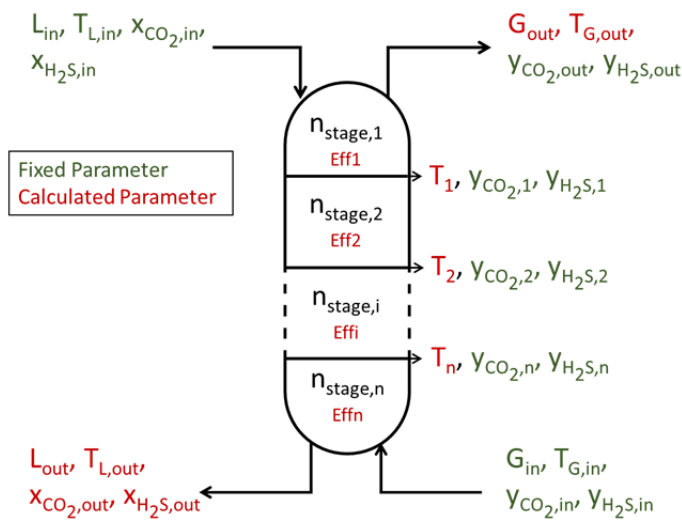


Figure 4. Simplified scheme of the RDU with the corresponding AspenPlus EO Modelling parameters

To fit the concentration profile, individual Murphree efficiencies are defined, for each one corresponds to a section between two consecutive gas sampling points. Aside from the Murphree efficiencies, all the other calculated parameters are compared to the measured ones to confirm the accuracy of the modelling. The HETP of the packings is defined as following:

$$HETP = \frac{h_{tot}}{\sum_{i=1}^n Eff_i \cdot n_{stage,i}} \quad (2)$$

With h_{tot} , the height of the packing section, Eff_i , the Murphree efficiency of the section i and $n_{stage,i}$ the number of stages of the section i . The error on the HETP calculated is estimated by error propagation of the errors of all sensors, taking into account both the variations of the RDU and the main plant, and the error from the instruments themselves. The relative error derived ranges from 5 to 10% of the HETP depending on the campaign.

3. Results

In Figure 5, the experimentally determined pressure drop curves gives the hierarchy in the capacity of the packings for the different campaigns. For campaign C1, packing 1 present the highest capacity as the RDU can be operated under stable condition at a F-Factor of $3.1 \text{ Pa}^{0.5}$, whereas the packing 2 has been stably operated until $2.8 \text{ Pa}^{0.5}$ and the packing 3 until $1.9 \text{ Pa}^{0.5}$. For the campaigns C2 and C3, the hydraulic tests have not been performed with the Packing 1 (cf. Table 1). The packings 2 and 3 have been stably operated for F-Factor respectively up to $1.3 \text{ Pa}^{0.5}$ and $1.7 \text{ Pa}^{0.5}$, tests have not been further continued due to operating window limitation.

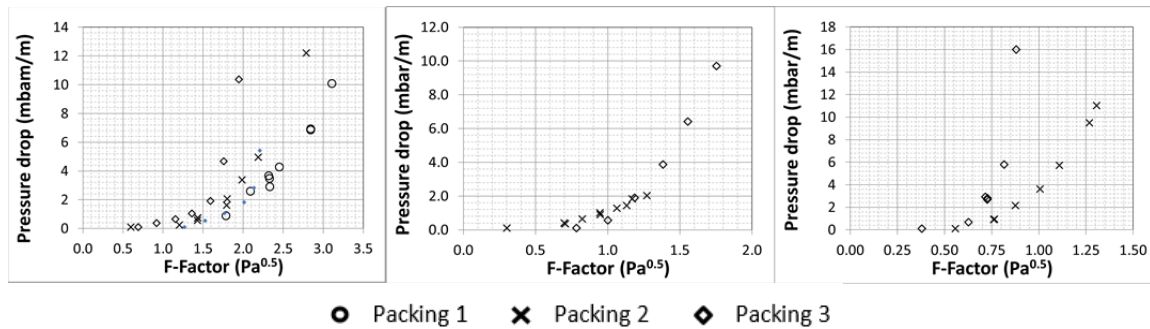


Figure 5. Pressure drop vs F-Factor for the campaigns (left: campaign C1, center: campaign C2, right: campaign C3)

From the three tested packings, Packing 1 is the least efficient. Packings 2 and 3 have similar efficiencies for campaign C2. For campaigns C1 and C3, Packing 3 is more efficient than Packing 2.

The impact of the F-Factor at design L/G ratio on the HETP differs between the campaigns (Figure 6). For the campaign C1, the increase of the F-Factor decreases the HETP, for Packing 1, the increase of the F-Factor from $2 \text{ Pa}^{0.5}$ to $3 \text{ Pa}^{0.5}$ reduced the HETP by 50%. For Packing 2, the impact is less important than for Packing 1, whereby an increase of the F-Factor from $0.6 \text{ Pa}^{0.5}$ to $2.8 \text{ Pa}^{0.5}$ divides HETP by 2.

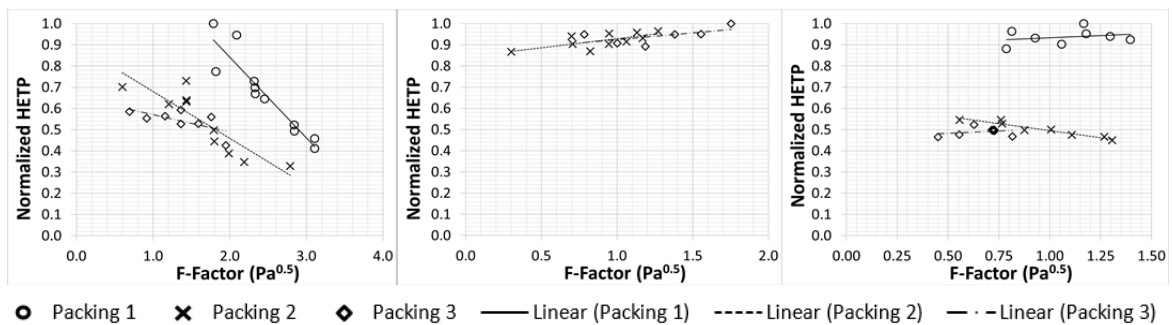


Figure 6. Normalized HETP vs F-Factor for the tested packings at design L/G (left: campaign C1, center campaign C2, right campaign C3)

For the campaign C2, the opposite trend is observed, the increase of the F-Factor increases the HETP but the impact on the HETP is less than 15% on the tested F-Factor range for Packings 2 and 3.

The impact of the L/G ratio for C2 and C3 is negligible (less than 8% and no trend). In the case of the C1 campaign, the impact of L/G ratio is more important. Indeed, depending on the packing tested, a reduction of the L/G ratio by 20% increases the HETP up to 70% (Figure 7).

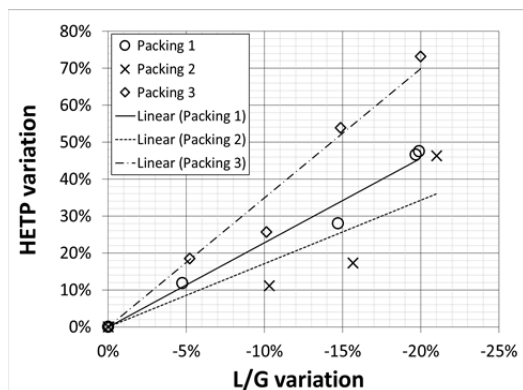


Figure 7. Impact of L/G ratio on HETP for the C1 campaign

4. Conclusions

The results of the RDU show that the use of packing instead of trays to optimize the Rectisol™ design is advantageous. First, the highest capacity packings allow the reduction of the CapEx of the column by improving the maximum design F-Factor, thus reducing the column diameter and wall thickness. Regarding the influence of Chinese road infrastructures on column diameter, the use of packing allows to design a Rectisol™ unit with a capacity up to 1 million Nm³/h without splitting the absorber. This represents a reduction of 50% of the absorber CapEx. Furthermore, the reduction of the pressure drop generated by packing compared to trays allows the reduction of the OpEx of the Rectisol™. The reduction of the pressure drop allows an OpEx reduction up to 4%.

The study done on the impact of the different operating conditions increases the in-house knowledge and provides important information for Rectisol™ optimization. The optimization potential leads to further reduction of both CapEx and OpEx among others by modification of the L/G ratios in several column sections through better known efficiencies. The results of the RDU have been implemented in commercial proposals and Air Liquide is operating Rectisol™ plants with packings.

The next step is to model the mass transfer in greater details using a rate based model within AspenPlus. It is expected that a rate based model might be able to describe the basic phenomena with better accuracy than the currently applied Murphree approach. (Afkhamipour and Mofarahi, 2013). In addition, a mass transfer model would be able to extrapolate to operating conditions which were not tested in the RDU. Nevertheless, the development of a suitable mass transfer model requires access to rigorous information on interfacial area and gas and liquid side diffusion coefficients. The lack of accurate data on these individual contributions on overall mass transfer may lead to inaccuracy in the modelling.

References

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