

Multi-Objective Optimization of a Selexol[®] Process for the Selective Removal of CO₂ and H₂S from Coal-Derived Syngas

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Abstract:

This paper focuses on the multi-objective optimization of a Selexol[®] process for the selective removal of CO₂ and H₂S from coal gasification-derived syngas. A systematic analysis based on a thorough literature review of scheme options, detailed process simulation (including also a properly calibrated PC-SAFT Equation Of State) and design optimization is carried out. More in detail, the design optimization procedure enables the simultaneous optimization of process, utility design, and heat integration, and takes account all of the interactions with the rest of the plant. The multi-objective optimization is carried out with a two-stage approach combining the NSGA-II genetic algorithm with the efficient direct-search method PGS-COM. Results show that in the Selexol[®] process is crucial to optimize the pressures of the flash cascade releasing the CO₂-rich stream to store, because the largest energy penalties are the compression power and the steam necessary for reboiling.

Keywords:

Multi-objective optimization; Selexol[®]; CCS; Syngas; NSGA-II; PGS-COM.

1. Introduction

In coal gasification-based plants requiring deep Acid Gas Removal (AGR) from syngas, benchmark Acid Gas Removal (AGR) processes are Rectisol[®] and Selexol[®] [1], both based on physical absorption. Even though industrial absorption has reached a high level of maturity and technical development, the design criteria for CO₂ Capture and Storage (CCS) purposes are not yet well-known. For these reasons, several researchers are still dealing with their optimization and improvement. For instance, Tock and Maréchal [2] developed a multi-objective methodology with a superstructure approach to optimize processes focused on the production of hydrogen and power with CCS. Their work entails different options from the point of view of input, output and design conditions, including pre and post-combustion CCS. Following the same approach, Urech et al. [3], assessed three different technologies: amines, Selexol[®] and Hot Potassium Carbonate for the CO₂ capture in an IGCC. The focus of Urech et al. is more on the system optimization, and therefore the Selexol[®] AGR flowsheet is not as detailed as it is in the present work. Harkin et al. [4] applied multi-objective optimization to the Selexol[®] section of an IGCC, identifying the reboiler heat duty as a key variable, a conclusion drawn also by Bhattacharyya in [5].

The purpose of this paper is (1) to provide a complete review of the available Selexol[®] process schemes suitable for the selective removal of coal-gasification derived syngas and, similarly to what already performed by Gatti et al. in [6], (2) to identify the optimal process design variables for a wide range of CO₂ capture rates by means of a multi-objective optimization approach.

2. Selection of Selexol[®] scheme for CCS

Selexol[®] is a physical AGR process, deployed by Allied-Signal (Honeywell) more than 50 years ago, now owned by Union Carbide-Dow Chemical Company and licensed by UOP-Honeywell, that uses a mixture of Dimethyl Ethers of Polyethylene Glycol (DEPG) as solvent. The chemical composition of the sorbent is not unique, as several different blends have been developed and commercialized. As reported by [7], the chemical formula of the compounds is $\text{CH}_3\text{O}[\text{CH}_2\text{CH}_2\text{O}]_n\text{CH}_3$, with n ranging from 3 to 12. The Selexol[®] process configuration can be tailored to the specific gas cleaning requirements and therefore different process layouts have been proposed. This application is focused on the selective removal of CO_2 and H_2S . The composition of the syngas entering the AGR unit, as well as the requirements of the clean syngas and CO_2 captured flow are reported in Table 1.

Table 1. Syngas specifications for the process design.

Inlet Stream Properties		Outlet H_2S rich stream to Claus Conditions	
Raw fully shifted syngas produced by GE gasifier fed with Illinois #6 coal		$\text{H}_2\text{S}/\text{CO}_2$ molar ratio	$\geq 1/2$
Composition	Mole %	Outlet CO_2 Conditions	
CO_2	40.93%	Destination: Enhanced Oil Recovery	
H_2S (including COS)	1.12%	State: Supercritical dense at 150 bar	
CO	1.19%	Temperature	
N_2 (including other inerts)	0.44%	CO_2 molar concentration	$> 97\%$
H_2	56.33%	H_2S molar concentration	< 150 ppmv
Total Molar Flow Rate	6.589 kmol/s	Outlet Syngas Conditions	
Total Mass Flow Rate	118.7 kg/s	Temperature	25 °C
Temperature	30 °C	Pressure	30 bar
Pressure	50 bar	H_2S molar content	< 50 ppbv

As far as the selective removal of H_2S and CO_2 from gasification-derived synthesis gas is concerned, five main schemes are available in the literature: LaBarge [8], the two solvent loops scheme [8], the Integrated Sour Shift Concept by UOP [9], the UOP scheme for Urea manufacturing, [9], and the Coffeyville scheme [8].

The most experienced Selexol[®] plant delivering EOR-ready CO_2 is the one operating since 1986 at the ExxonMobil Shute Creek Natural Gas plant of LaBarge, Wyoming, [8]. Its scheme is reported in Fig. 1. This configuration entails many recycle loops to get an effective recovery of the light fuels in the raw gas (mostly CH_4). Moreover, it is particularly well suited for applications featuring high CO_2 and H_2S concentrations in the raw gas, since LaBarge syngas contains about 66%_{MOL} of CO_2 and 5%_{MOL} of H_2S . For these two reasons, it is not well suited for the application considered in this paper (IGCC with CCS).

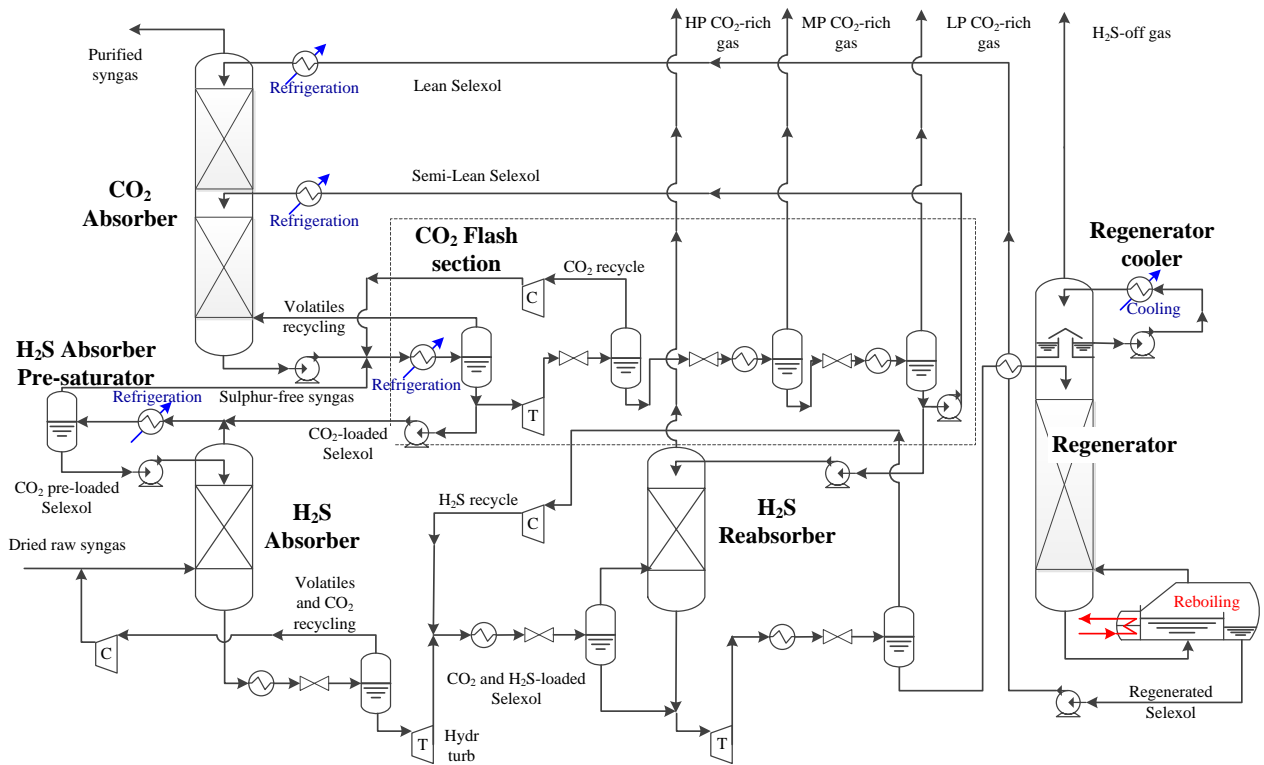


Fig. 1: Selexol® flowsheet of LaBarge plant (adapted from [8]).

Sweny [10] presented a Selexol® variant in which the solvent loops of the H₂S and CO₂ removal sections are separated. However, this option is not attractive because it increases the solvent circulation flow rate and the CO₂ stripped is diluted with N₂, which makes it unsuitable for Enhanced Oil Recovery (EOR, which is the option considered in this paper) or geological sequestration.

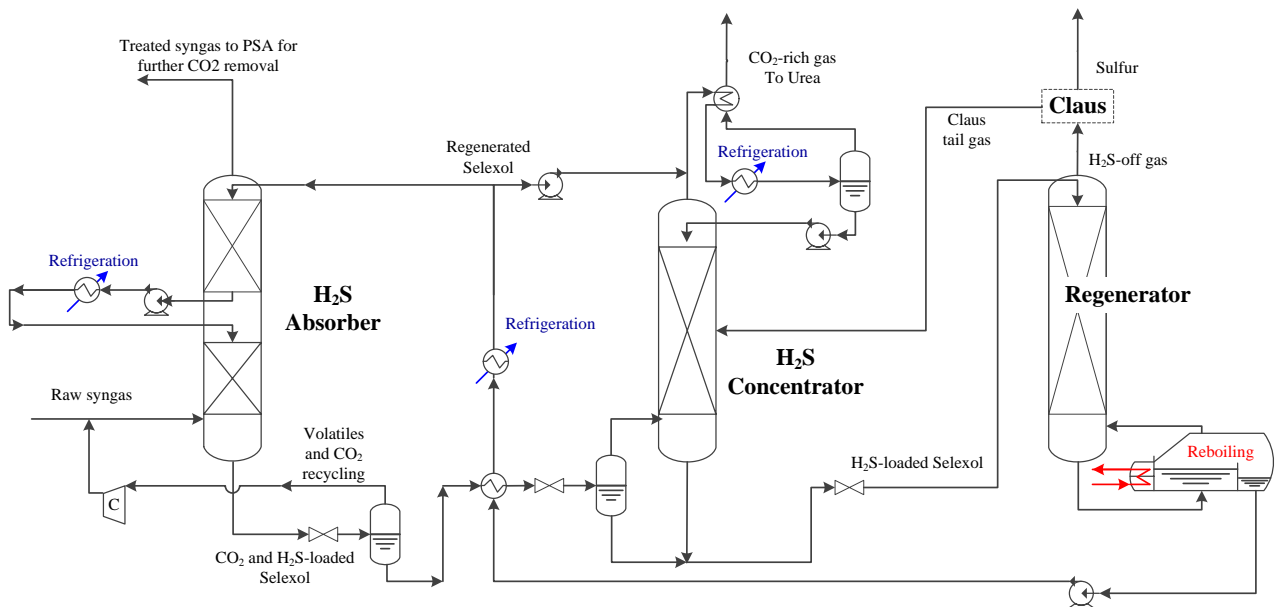


Fig. 2: Selexol® unit designed for co-producing pure hydrogen, sulfur and CO₂ for urea manufacturing, with the contribution of a PSA unit, in a Gasification to ammonia/urea complex (adapted from [11]).

UOP [9], and [11] proposed a Selexol® variant, depicted in Fig. 2, in which the absorption of H₂S and CO₂ takes place in the same column. The process is designed to remove at least 99.5% of the H₂S from syngas, while producing a CO₂-rich stream suitable for urea manufacture. In the

intercooled absorber the syngas is washed with a refrigerated solvent at 0 °C and 63 bar. The loaded solvent exits at the bottom of the column and is flashed and heated before entering the H₂S concentrator. This column has the aim of extracting only CO₂ by re-capturing the stripped H₂S by means of a stream of solvent fed at the top of the column. This process scheme is not ideally suited for CCS applications where large amounts of CO₂ must be removed from the raw gas.

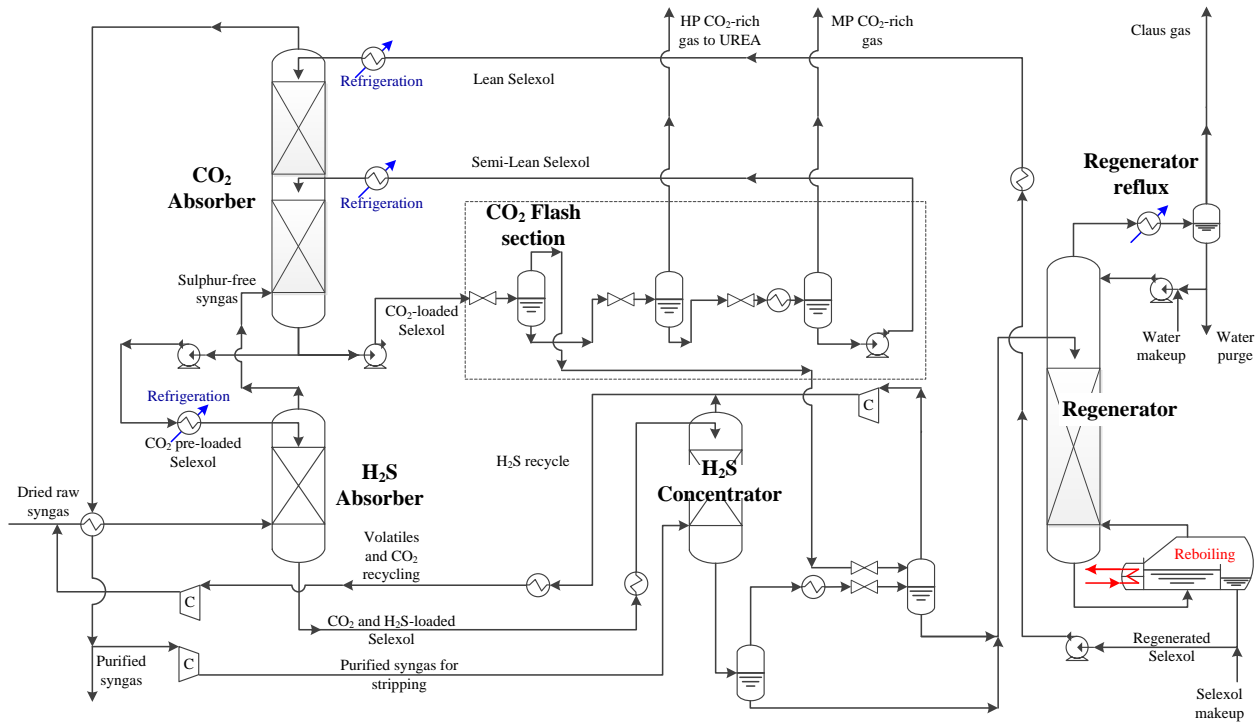


Fig. 3: Selexol® flowsheet of Coffeyville plant (adapted from [8]).

The Integrated Sour Shift Concept by UOP [9] is not considered for our application, since we assume to operate on a fully shifted syngas.

A particularly interesting Selexol® scheme is the one of the Urea and Ammonia production plant in Coffeyville (Kansas, USA), provided by UOP. The scheme, detailed by Kubek [8], is reported in Fig. 3. This process performs a two-stage selective removal of H₂S and CO₂ and differs from the scheme of Fig. 1 only in the arrangement of the H₂S enrichment section and for the absence of the H₂S pre-saturator. This difference may be due to the higher CO₂/H₂S molar ratio produced by the Coffeyville gasifier (almost 70, with a CO₂ content of 41%_{MOL}) compared to LaBarge's gas.

On the basis of the advantages and disadvantages of the reviewed schemes and the specifications of the application considered in this study, the scheme represented in Fig. 4 was derived. Its absorption and regeneration sections are similar to those of the Coffeyville [9] Selexol® scheme, whereas the H₂S concentration section includes only a single thermal and flash desorption stage (H₂S enrichment box in Fig. 4). This is a simplification which does not affect considerably the selectivity of the system, while avoiding the complexity of the H₂S concentrator. Another difference with respect to the Coffeyville scheme is the number of CO₂ flashes (here chosen equal to four to reach a satisfactory CO₂ recovery).

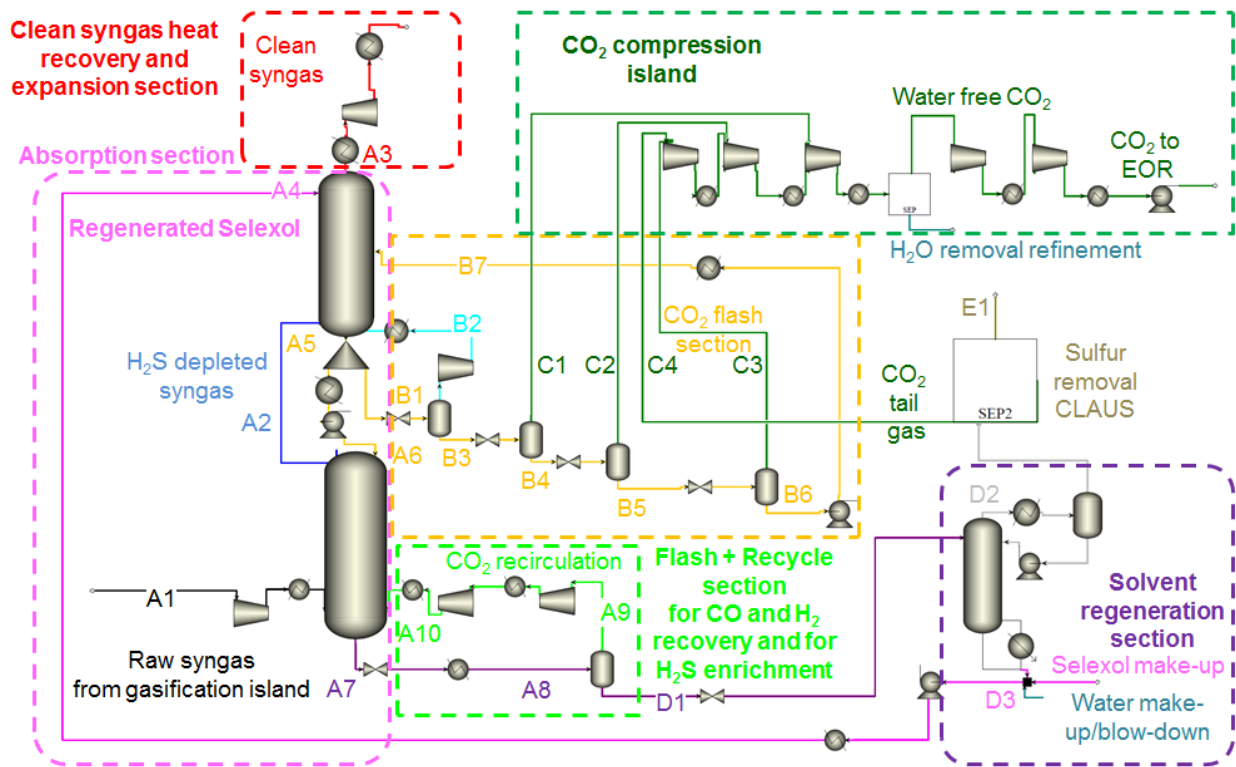


Fig. 4: Selexol® flowsheet implemented in Aspen Plus® and adopted as Reference configuration for the combined, selective removal of CO₂ and H₂S.

As described by Fig. 4, in this process, the raw syngas (A1) enters the absorption section (water saturated), then is optionally compressed and cooled prior to be desulfurized in an H₂S absorber. The partially purified syngas (A2) is then sent to the CO₂-removal column where a bulk CO₂ capture is performed. Then, the clean syngas (A3) goes through an expansion and cooling duty recovery step, before being sent to the downstream conversion and utilization island. The lean solvent (A4), which is made of 35%_{MOL} water and 65%_{MOL} DEPG, meets first the CO₂ absorber and then, at its exit is split in two fraction: a limited amount (A5), after proper cooling, washes the H₂S out of the syngas in the H₂S absorber, whereas the largest fraction (B1) enters the CO₂ flash section, where CO₂ is desorbed in a series of three flashes (C1, C2 and C3) preceded by a high-pressure flash for H₂ and CO recovery (B2), and the semi-lean solvent is cooled down and recycled back to the absorption column (B7) to remove CO₂ again. The H₂S enrichment and the solvent regeneration sections are arranged like in the H₂S-removal only version. The CO₂ tail gas released by Claus is supposed to be recycled to the CO₂ compression island which comprises a five stages intercooled compressor featuring an H₂O removal drum at 30 bar.

3. Optimization Framework

The optimization problem is the following: for a given raw syngas shifted for IGCC applications, identify the process design variables, thermal integration and utility designs that are Pareto-optimal for:

- Minimizing the Equivalent Electricity Consumption (ELEC), i.e. the overall thermodynamic penalty index
- Maximizing the CO₂ removal efficiency, i.e. CO₂CL, defined as the ratio between the amount of CO₂ captured and the CO₂ in the raw gas

while satisfying the set of thermodynamic, technological and environmental constraints related to its design and operation, as well as the specifications on composition, temperatures and pressures of outlet streams.

The performance index proposed, ELEC takes into account all the interactions of the detailed AGR unit with the rest of the plant.

ELEC is calculated as in (1), as the sum of the following contributes:

- $\dot{E}_{Process}$, electric power absorbed by the process compressors and pumps,
- $\dot{E}_{CO_2\ compr}$, electric power absorbed by the compressors and pumps of the CO₂ compression section,
- \dot{E}_{Fuel} , chemical exergy (LHV basis) of the fuel species (mainly CO and H₂) co-captured with acid gases and sent either to storage or to the CLAUS,
- \dot{E}_{Refrig} , electric power absorbed by the refrigeration cycle (cold utility), see *Fig. 6*,
- $\dot{E}_{Cool\ wat}$, electric power required to circulate the cooling water (cold utility),
- $\dot{E}_{Reboiler}$, electric power loss due to the extraction of steam from the steam cycle for the reboiler/s (hot utility), see *Fig. 6*.

$$ELEC = \dot{E}_{CO_2\ compr} + \dot{E}_{Process} + \dot{E}_{Reboiler} + \dot{E}_{Refrig} + \dot{E}_{Cool\ wat} + \dot{E}_{Fuel} \quad [kW] \quad (1)$$

The optimization should include as a third objective function also the CAPEX. However, Gatti et al. in [6] showed that, for a given pinch point temperature difference between the hot and cold streams ΔT_{min} (5 °C in this study), if CAPEX is included as a third objective, the Pareto frontier would have the same trend of the bi-objective one. Therefore, results are presented in the form of a Pareto frontier where the conflicting objectives are ELEC and CO₂CL.

The optimization strategy proposed by Gatti et al. [6] for the multi-objective optimization of the Rectisol[®] process is adopted. More in detail, the problem has been solved by adopting the black-box framework represented in *Fig. 5*. The main steps are: (a) a multi-objective optimization algorithm sets the design variables of the process (the absorption pressure, the pressure of the four CO₂ flashes and of the solvent regenerator, the temperature of the solvent streams entering the columns (A4 and A6 in *Fig. 4*), the temperature of raw syngas at H₂S absorber inlet (A1) and the temperature of the loaded solvent entering the regenerator (A8)) and utility (i.e., refrigeration cycle: the flow rate of ammonia, the intermediate throttling stage pressure, and the temperature of the evaporator and condenser); (b) the Selexol[®] process is simulated in Aspen Plus (for the given design variables); (c) the heat integration and the design of the refrigeration cycle are optimized with a simultaneous heat-cascade-based methodology (for the given configuration of the refrigeration cycle); (d) the equipment units are preliminarily sized according to engineering rules of thumb and the capital cost of the Selexol[®] plant section, including the utilities, is estimated.

The problem must be solved with a direct-search derivative-free multi-objective algorithm because:

- the feasible region (solution domain) is disconnected
- the evaluation of many solutions fails (no objective function value) because of the convergence issues of the Aspen Plus simulation;
- the results (objective functions) are noisy because affected by the numerical noise generated by the iterative solvers of the process simulation tool;
- the heat integration technique leads to discontinuities/non-differentiabilities in the solutions as a result of the activation of pinch points;

The multi-objective optimizer is a 2-step sequential method including: (i) the multi-objective evolutionary algorithm NGS-II by Deb et al. [12] available in Matlab; followed by, (ii) PGS-COM, a single-objective direct-search algorithm developed by Martelli and Amaldi [13], and used to further refine the solutions found by the NGS-II. As a key advantage, both algorithms are coded to take advantage of multiple-core machines (parallel computing) by distributing the evaluation of the black-box functions to the available processors. This algorithm capability is valuable because of the long computational time of the process simulations (see next section).

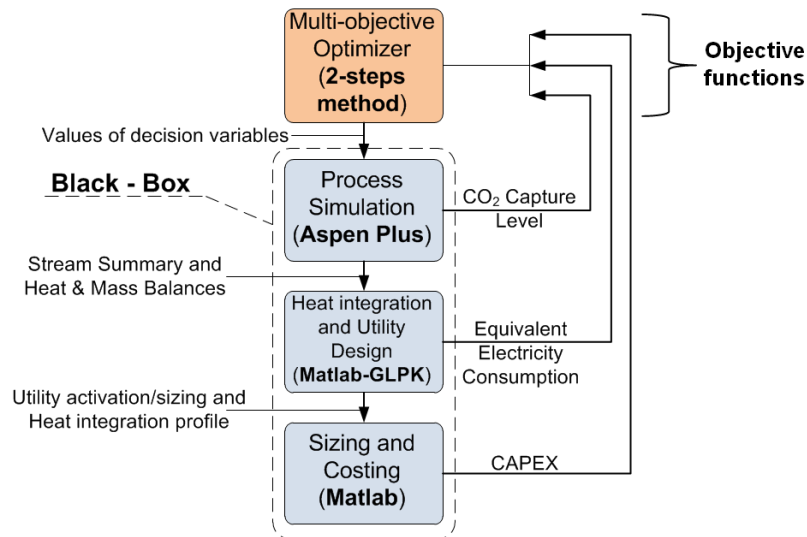


Fig. 5: Multi-objective optimization framework.

4. Thermodynamic model and simulation assumptions

The process was modelled with Aspen Plus[®] (v7.3.), by using the available PC-SAFT equation of state with properly calibrated binary interaction parameters. Results were validated with the absorption and regeneration section data of two reference plants (for confidentiality reasons, data and calibrated binary interaction parameters cannot be published). In any case, the EOS is consistent with the PC-SAFT model recently presented by Aspentech in [14].

The absorption and regeneration columns of this process were modelled with the theoretical equilibrium stage approach (usually, in Selexol[®] packed column are adopted). However, it should be noted that this assumption, which is likely to lead to an overrate of the absorption load of the solvent, affects only the sizing of the column, where an assumption of a proper absorption efficiency is required to estimate the actual height from the ideal one. The other key process modelling assumptions are reported in Table 2.

Table 2. Assumptions for the process simulations carried out in Aspen Plus[®].

Process assumptions	
Pressure loss $\Delta p/p_{in}$	2 %
Polytropic efficiency of syngas and CO ₂ compressors	84 %
Isoentropic efficiency of expanders	88 %
Polytropic efficiency of refrigerator compressors	82 %
Mechanical/electric efficiency of the driver	92 %

Due to the presence of multiple recycle loops and absorption columns, with a highly non-ideal solvent, the flowsheet was simulated by adopting a sequential approach, which is more robust, even though more time consuming, than an equation oriented one.

It is worth noting that the average computational time required to reach convergence of the process flowsheet model is about 50 s on a 12 core PC featuring 2.8 GHz/core and 24 GB of RAM.

5. Heat integration and utility design strategy

The process simulation sets the heat duties of the cold/hot streams. Then, the heat integration methodology and algorithm must determine the optimal heat recovery (heat which is directly exchange between hot and cold streams of the process), the energy requirement from utilities (heat which must be supplied/removed by external systems, like refrigeration cycle, cooling water, etc), and the design/size of the required utility systems. To this aim, the energy integration methodology and algorithm proposed by Maréchal and Kalitventzeff [15] was used. The algorithm, based on a reformulation of the LP transshipment problem of Papoulias and Grossmann [16], determines the

solution which minimizes the overall exergy consumption of the utilities without detailing the heat exchanger network between hot and cold streams.

The utilities considered here for the proposed Selexol[®] process are: the evaporator of the ammonia refrigeration cycle; a closed loop of cooling water which is heated from 15 to 25 °C; saturated steam for reboiling optionally available at 0.5, 1.5, 3 and 10 bar. ΔT_{\min} at reboiler is 20 °C, whereas for all other streams is set at 5 °C.

The refrigeration cycle, shown in Fig. 6, is a reverse Rankine cycle with multiple (3) throttling and compression stages. Ammonia was selected as a working fluid because feature the best performances (COP) at working temperatures across near to 0 °C. The evaporation temperatures and compressor pressures are optimized, whereas the ammonia flowrate results from the heat integration with the process (step (c) of the optimization strategy).

Fig. 7 reports a diagram which explains the method followed for the estimation of the mechanical work loss (w) due to steam extraction, from the steam cycle, for reboiling (solvent regeneration).

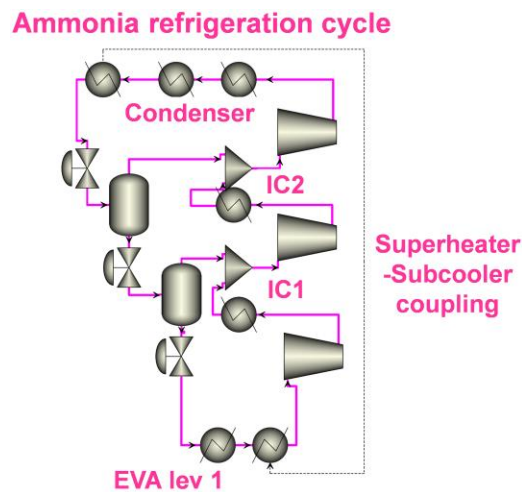


Fig. 6: Ammonia refrigeration cycle configuration.

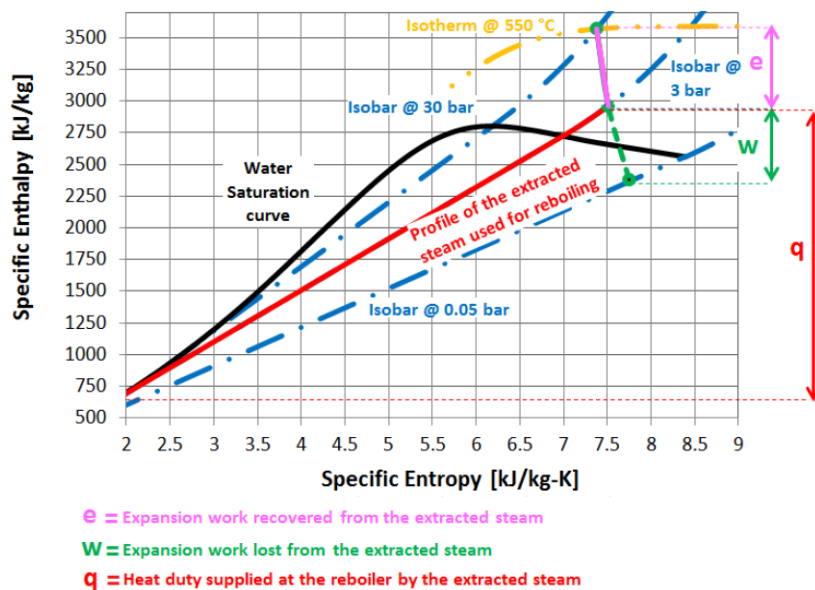


Fig. 7: Representation of the mechanical work loss (w) due to stream extraction for reboiling (case at 3 bar).

6. Heuristically-designed Selexol[®]

Before carrying out the optimization it is worth evaluating a design of the process made on the basis of heuristic criteria (“rules of thumb”), in order to measure the performance improvement introduced by the optimization. To this purpose, we heuristically tuned the Selexol[®] design

variables reported in Table 3 so as to meet the specifications of Table 1 and reach a CO_2CL equal to 90%. The utility and heat integration are instead optimized, as it can be seen from Fig. 8 where the Composite Curves, and Fig. 9 where the Exergy Grand Composite Curves (see Gatti et al. [6]) are reported. The most significant performance indexes are shown in Table 3.

The electric consumption of the reference Selexol[®] for the CO_2 compressor, syngas and solvent compressor, and refrigeration cycle, equal to 482 kJ/kgCO_2 , is in line with the benchmark values reported by Romano et al. [17] and backcalculated from EBTF [1], 431 kJ/kgCO_2 . The 50 kJ/kgCO_2 difference is mostly attributable to the different H_2S molar content of the EBTF stream, which is one order of magnitude lower than the one of this paper (1.1 %_{mol} vs 0.1%_{mol} at AGR inlet).

The compression and the refrigeration electric power consumptions are in line with literature data.

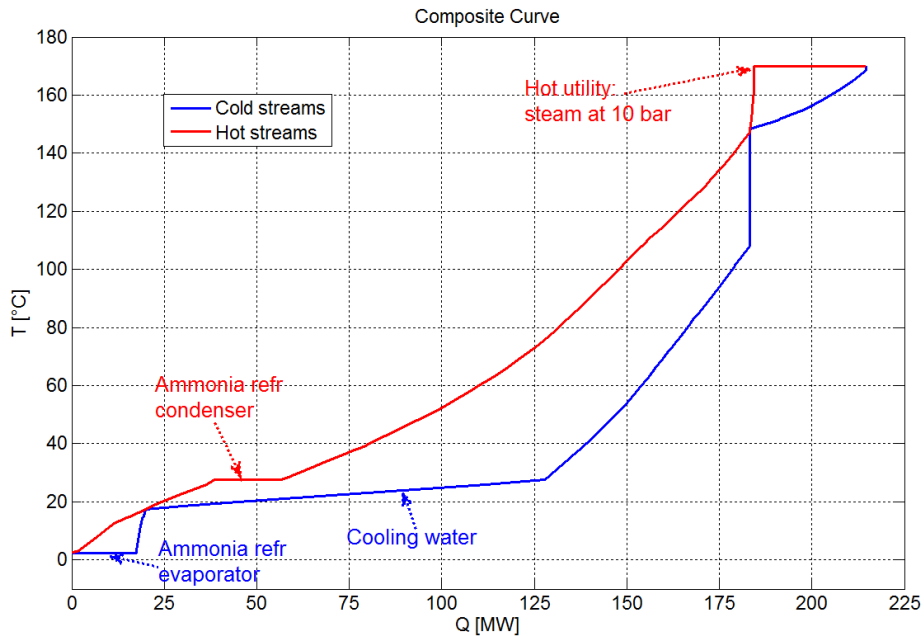


Fig. 8: CC of a Selexol[®] design for a combined $\text{CO}_2\text{-H}_2\text{S}$ selective capture.

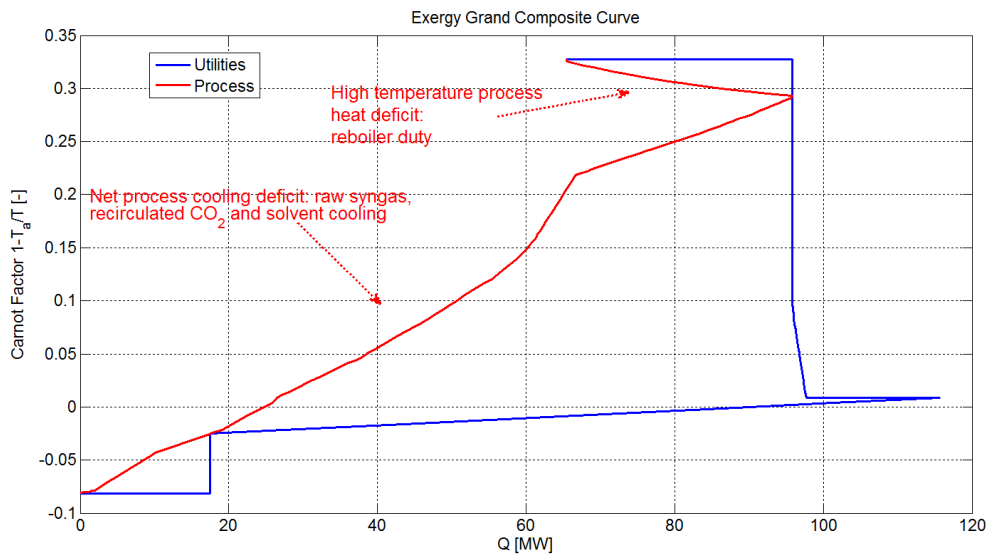


Fig. 9: EGCC of a Selexol[®] design for a combined $\text{CO}_2\text{-H}_2\text{S}$ selective capture.

Table 3. Main performance indexes of the Selexol[®] reference design.

Coal type Syngas application		Illinois #6 coal IGCC syngas
Solvent flow-rate	kg/s	225
Absorption pressure	bar	60.0
Solvent temperature	°C	5
Pressure of four CO ₂ flashes	bar	16-10-5-1.2
split fraction of CO ₂ loaded solvent to H ₂ S absorber	-	1/6
stage location of recirculated solvent feed in CO ₂ absorber	-	5
number of stages of H ₂ S absorber (integer)	-	8
number of stages of CO ₂ absorber (integer)	-	9
CO ₂ Captured	kg/s	107.0
CO ₂ Capture Level	%	90.1%
Overall Reboiler duty (thermal)	MW	30.3
Reboiler steam pressure	bar	1.9
Net Refrigeration duty (thermal)	MW	17.5
Syngas & Solvent Compression power	MW	23.2
CO ₂ Compression power	MW	25.4
Electric equivalent of Reboiler duty	MW	10.5
Refrigeration electric power	MW	2.9
Cooling water consumption (electric)	MW	1.7
Chemical exergy of co-captured fuel	MW	0.2
Overall equivalent electricity consumption (ELEC)	MW	63.9
Specific Electric Equivalent Consumption (SELEC)	kJ/kg _{CO2}	597.2
Specific Electric Equivalent Consumption of the syngas & solvent compressors, CO ₂ compressors and refrigerator only	kJ/kg _{CO2}	482.2

7. Optimization results

The multi-objective optimization was quite computationally expensive: despite the use of a 12-core workstation, completing 6000 design evaluations (black-box function evaluations) took about 15 hours. However, the non-dominated solutions (estimates of the Pareto frontier) reported in Fig. 10 show that a satisfactory convergence of the algorithms was reached. Fig. 11 describes the ELEC (left) and CAPEX (right) breakdown of the optimized solutions for four values of CO₂CLs.

The optimization shows that the improvement with respect to a heuristically designed solution is considerable, since ELEC is reduced by 23% for the case at 90% and by 18% for the case at 98%, whereas the effect on CAPEX is less important, because a better integration leads to higher heat exchanger network costs (3% saving at 90% and 7% higher cost at 98%).

It is worth noting that the most significant design variables (i.e. that change the most with the CO₂CL) are: the solvent circulation flow rate, the absorption pressure, the CO₂ flash pressures, and the split fraction of CO₂ loaded solvent sent to H₂S absorber.

In Fig. 11 the ELEC terms are the ones already explained in par. 3, whereas the CAPEX fractions reported in in Fig. 11 are: hexch, i.e. the capital cost of the heat exchanger network; refr_cycle, i.e. the capital cost of the refrigeration cycle; expanders, i.e. the capital cost of the expanders of the Selexol[®] process; compr_pumps, i.e. the cost of compressors and pumps of the Selexol[®] process, including CO₂ compressors; separators, i.e. the cost of flash vessels; columns, i.e. the cost of the absorption and regeneration columns, including the internals.

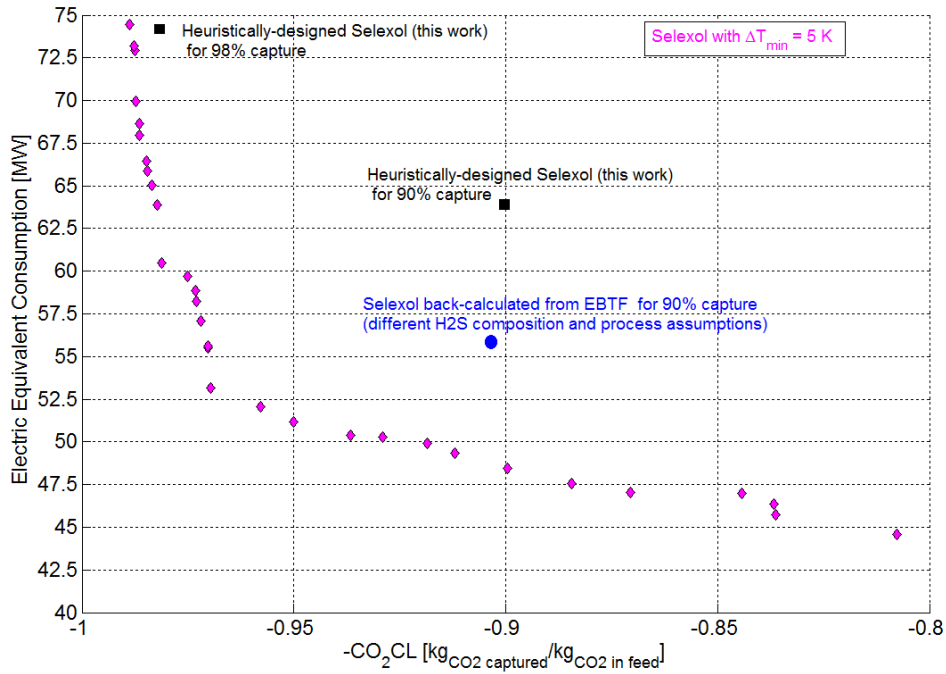


Fig. 10: Pareto frontier after multi-objective optimization. Heuristic designs are also reported (in black the ones evaluated consistently with this paper, in blue the one retrieved from the literature). Notice that negative CO_2 capture levels (x axis) are plotted in order to convert its maximization problem into a minimization problem (required by the used optimization algorithms).

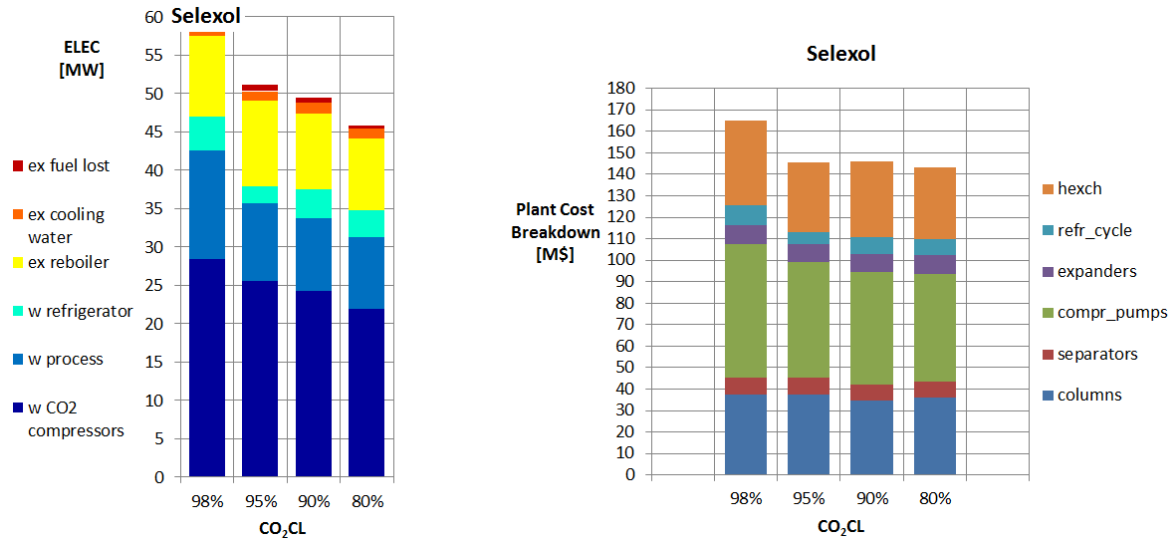


Fig. 11: Equivalent Consumption breakdown for the IGCC optimal solutions resulting from multi-objective optimization and Capital Cost breakdown.

8. Conclusions

This paper presents the methodology and results of the multi-objective optimization of a Selexol[®] process tailored to CCS for an IGCC syngas. The process configuration is selected after screening the most interesting alternatives. The bi-objective optimization is performed via a two-step methodology, recently introduced, by exploiting parallel computing. A heuristic-based design of the process is proposed and evaluated. The Pareto curves obtained confirm the potential of the proposed approach to improve significantly the solutions compared to the heuristic designs, reporting performance improvements up to 23% in terms of Electric Equivalent Consumption. Finally, a breakdown of the performance and costs is provided for four significant solutions, to show the evolution of the cost and energy penalty contributions.

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