

# Seawater Desulphurization of Simulated Flue Gas in Spray and Packed Columns: an Experimental and Modelling Comparison

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Flue Gas Desulphurization (FGD) is a key topic for most of combustion fossil fuel plants and industrial applications. Wet processes are usually preferred when the removal efficiency required to comply with environmental regulation exceed 90 %, but their flexibility makes them useful also for lower performance requirements. Recently, seawater has been considered as a viable absorbent for FGD processes in coastal and naval applications, when SO<sub>2</sub> concentration is below 1000 ppm<sub>v</sub>. This liquid absorbs SO<sub>2</sub> mostly thanks to its natural alkalinity (on average 2.4 mmol/L) and, of course, is also largely available. The main costs of seawater FGD (SW-FGD) plants are related to the pumps needed to supply the required amount of liquid and the cost of wash water restoration before discharge. Corrosion related to the presence of acidic seawater is also a major issue of these units. While thermodynamic limitations cannot be overcome, process design can be optimized to improve mass transfer rates and reduce seawater requirements while keeping a suitable absorber size. In this work, we report the experimental findings achieved by our group on process design and optimization of SW-FGD units. Two different kinds of SW-FGD units were tested and compared: a spray column and a packed column with structured packing.

## 1. Introduction

Flue Gas Desulphurization (FGD) is an effective system to achieve high purification efficiency and the use of seawater (SW) as absorbing liquid has become convenient in coastal and naval applications. In light of the introduction of new regulations concerning marine transport (IMO-Marpol VI regulation 14) and combustion plants (Directive 2016/2284/CE), SW-FGD units need an improvement of their performance. IMO-Marpol regulation has ratified that, since 1 January 2016, the sulphur content in the fuel must not exceed 0.1 % into SECAs (SO<sub>x</sub> emission control areas), while the European community will follow, from January 2016, the Industrial Emissions Directive (IED) on SO<sub>2</sub> emissions, which set the limits at an average value of 200 mg/m<sup>3</sup> (71 ppm<sub>v</sub>) for coal-fired power plants having a power generation capacity higher than 300 MW<sub>th</sub> (Wynn, 2017). The new EU guidelines (BREF standards) impose a significant reduction of SO<sub>2</sub> emissions, with an average value of 130 mg/m<sup>3</sup> (46 ppm<sub>v</sub>), from January 2021 onwards (Wynn, 2017). Similarly, for USA and China the SO<sub>2</sub> emission limits are 136 and 35 mg/m<sup>3</sup> (48 and 12 ppm<sub>v</sub>), respectively (Zhang, 2016).

In this sense, it is worth investigating possible solutions to improve the performances of conventional spray columns for SW-FGD, by improving the spray properties (Di Natale et al., 2016; Manna et al., 2016) and considering the introduction of scrubbers with structured packings (Flagiello et al., 2017; Flagiello et al. 2018). In this paper, we report a comparison of two SW-FGD prototypes, a spray column and a packed column, which were operated in the same operating conditions and compared in terms of SO<sub>2</sub> removal efficiency and exhausted liquid (wash water) pH levels. The spray SW-FGD tests were carried out in a pilot-scale stainless steel column operated in counter-current flow. The model gas flow rate was 130 Nm<sup>3</sup>/h and liquid flow rate

was varied from 500 L/h to 1500 L/h. The packed column was a lab-scale prototype used to treat a simulated flue gas at a flow rate equal to 8.12 m<sup>3</sup>/h and a liquid flow rate variable from 31.25 to 93.75 L/h. The packing selected for this application was a Mellapak™ 250.X to assure high mass transfer rates with low gas pressure drops. Experimental runs were performed in similar conditions in terms of liquid to gas ratio (L/G= 3.84-11.53 L/m<sup>3</sup>), gas velocity (u<sub>G</sub>= 0.29 m/s), on simulated exhausts flue gas containing 700 ppm<sub>v</sub> of SO<sub>2</sub> (as the value deriving from a fuel HFO containing about 3.5 % of sulphur in marine diesel engine applications or a low sulphur grade coal) and at same temperature and pressure (25°C and 1 atm, respectively). Experiments were interpreted starting from our recent findings on SO<sub>2</sub> equilibrium modelling (Flagiello et al., 2018). Spray and packed-bed SW-FGD were then compared in terms of SO<sub>2</sub> removal efficiency and height of transfer units. Finally, the results were used to delineate a comparison between spray and packed SW-FGD units, also in terms of economic constraints.

## 2. Background on absorption process

Design equations for absorption columns are available since many years (see for example Coulson & Richardson's, 2002). The correlations require the knowledge of thermodynamic and mass transfer parameters and can be resumed in the definition of the effective absorption height and the gas pressure drops in wet conditions:

$$Z = HTU_{OG} \cdot NTU_{OG} = \int_{y_{SO_2}}^{y_{SO_2}^o} \frac{G}{SK_{ov,SO_2} a_e (1 - y_{SO_2})(y_{SO_2} - y_{SO_2}^*)} dy_{SO_2} \quad (1)$$

$$\frac{\Delta P_{wet}}{Z} = f(u_L, u_G, \Phi, Y) \quad (2)$$

The effective height of the absorption column, Z [m], is written according to the number of transfer units (NTU<sub>OG</sub>) and the height of transfer units (HTU<sub>OG</sub> [m]), where y<sup>o</sup><sub>SO<sub>2</sub></sub> is the inlet SO<sub>2(g)</sub> molar concentration [mol/mol], y<sub>SO<sub>2</sub></sub> is the final SO<sub>2(g)</sub> molar concentration [mol/mol], y\*<sub>SO<sub>2</sub></sub> is the SO<sub>2(g)</sub> molar concentration at equilibrium with liquid phase [mol/mol], (K<sub>ov,SO<sub>2</sub></sub>)<sup>-1</sup> is the overall resistance to mass transfer [kmol/m<sup>2</sup>s], a<sub>e</sub> is effective wet surface [m<sup>2</sup>/m<sup>3</sup>], S is the column section [m<sup>2</sup>], G is the gas molar flow rate and L is the liquid molar flow rate, both expressed in [kmol/s].

While Eq(1) is a standard equation, valid for any type of contact device, the gas pressure drops highly depend on the system and Eq(2) is written in a general form by simply recalling the dependence on main parameters, such as operating conditions and section of the column (liquid velocity, u<sub>L</sub> [m/s] and gas velocity, u<sub>G</sub> [m/s]), physical and transport parameters of liquid and gas (Φ), and finally, the packing geometric parameters (Y) used. The number of transfer unit (NTU<sub>OG</sub>) is a purely thermodynamic parameter that depends on the driving force, given by the L/G ratio [mol/mol], and the equilibrium data y\*<sub>SO<sub>2</sub></sub>.

$$NTU_{OG} = \int_{y_{SO_2}}^{y_{SO_2}^o} \frac{dy_{SO_2}}{(1 - y_{SO_2})(y_{SO_2} - y_{SO_2}^*)} \quad (3)$$

The equilibrium data (y\*<sub>SO<sub>2</sub></sub>) can be expressed as a function of the liquid molar concentration of total sulphur (x<sub>S(IV)</sub>) and the slope of the equilibrium curve:

$$y_{SO_2}^* = F_{eq} (x_{S(IV)}) \quad (4)$$

in which x<sub>S(IV)</sub> [mol/mol] is the liquid concentration of total sulphur (SO<sub>2</sub>, HSO<sub>3</sub><sup>-</sup> and SO<sub>3</sub><sup>2-</sup>) in equilibrium with gas phase (y\*<sub>SO<sub>2</sub></sub>) and F<sub>eq</sub> is an equilibrium function of gas concentration.

An equilibrium model for the determination of the solubility dataset and NTU<sub>OG</sub> evaluation was proposed by Flagiello et al. (2018) and applied to SO<sub>2</sub> absorption in seawater solutions. The model provides mass and charge balance equations coupled liquid-gas phase equilibria and chemical equilibrium reactions in liquid phase (Flagiello et al., 2018). The Electrolyte Non-Random Two Liquid model (Chen et al., 1986) was adopted for the estimation of the activity coefficients for non-dilute liquid solutions. The values of the constants for liquid-gas equilibrium and the chemical reactions in liquid phase are available in Flagiello et al. (2018).

The height of transfer unit (HTU<sub>OG</sub>) is a kinetic parameter whose assessment requires the knowledge of mass transfer rates into the gas (k<sub>y,SO<sub>2</sub></sub> [kmol/m<sup>2</sup>s]) and liquid (k<sub>x,SO<sub>2</sub></sub> [kmol/m<sup>2</sup>s]) films, the effective wet surface (a<sub>e</sub>) and the solubility data. The overall resistance to mass transfer for dilute systems can be written according to the two-film theory (Whitman, 1962) as the sum of the resistances to mass transfer in liquid and gas films:

$$HTU_{OG} = HTU_G + \lambda HTU_L = \frac{G}{Sk_{y,SO_2} a_e} + \lambda \frac{L}{SEk_{x,SO_2} a_e} \quad (5)$$

$$\lambda = \frac{G}{L} \left( \frac{\partial F_{eq}}{\partial x} \right)_{x=x_{S(lv)}} \quad (6)$$

here  $HTU_G$  [m] and  $\lambda HTU_L$  [m] are the contributions to  $HTU_{OG}$  related to the gas and the liquid phases respectively,  $\lambda$  is the stripping factor that is defined as a relationship between the slope of the equilibrium curve [mol/mol] and the operating line (L/G [mol/mol]), while  $x_{S(lv)}$  is the liquid concentration of total sulphur at liquid-gas interface [mol/mol].  $E$  represents the enhancement factor for mass transfer in liquid film (its value is equal to the unit for physical absorption). The enhancement factor offers a useful indication on the limiting step of the process, as it is expressed as the ratio between the mass transfer rate in the liquid phase in the case of chemical reaction and the physical mass transfer rate (Levenspiel, 1999). In the case of  $SO_2$  absorption, the chemical reaction for  $SO_2$  hydrolysis is instantaneous, hence the final concentrations may be approximated by the thermodynamic equilibrium concentrations and the removal process is limited only by gas resistance to mass transfer.

### 3. Experimental

#### 3.1 Spray column

Pilot-scale tests were performed in a spray column constructed in AISI 316 steel (3 mm thick) to reduce corrosion issues (i.d.: 0.4 m; length: 4 m) operated at 25 °C and 1 atm. The system is similar to the unit used in former experiments reported in Di Natale et al. (2015), in which the flow sheet of the spray column prototype is also available. The column was equipped with four PNR<sup>®</sup> full spray nozzle (BRB 2117 B1 model). The droplets were distributed according to a Rosin-Rammler distribution with an average droplet size of 500  $\mu$ m and a shape factor of 3. The average upflow gas velocity was about 40% of the average droplet terminal velocity and the effective spray height was 3.5 m. The seawater solution is supplied by a centrifugal pump (Grundfos Lenntech) and controlled by a digital flowmeter (ASA srl) at the top of the column in counter-current flow to gas inlet. To assure a good distribution of the sprayed water on the entire column section, the four nozzles were arranged on the three vertices and in the center of an equilateral triangle. Liquid discharges (wash water) were sent to an accumulation tank, whose content was periodically sampled to measure pH value. The carrier gas is compressed and filtered air (ULPA filter), mixed with controlled amounts of  $SO_2$  contained in pressurized cylinders ( $SO_2$  at 100 % vol.) to achieve the desired column inlet concentration (700 ppm<sub>v</sub>). The model gas was controlled by two ASA srl digital flowmeters, can be heated up to 90 °C with electrical resistances (maximum power consumption 6 kW) and humidified with a steam generator (maximum power consumption 5.5 kW), so to enter in the column with a relative humidity close to saturation at the temperature desired. The regulation of gas and liquid flow rates were managed by a PLC control. In addition, the pilot plant was equipped with temperature and pressure sensors at the input and output of the column, connected to the PLC controller. Removal tests of sulphur dioxide in the spray column were carried out with a constant gas flow rate equal to 130 m<sup>3</sup>/h (0.29 m/s) while the liquid flow rate was varied between 500 and 1500 L/h.

#### 3.2 Packed-bed column

Experiments were performed in a packed-bed column made of plexiglass (i.d.: 0.1 m; length: 1.6 m) operated at room temperature (25 °C) and 1 atm. The column was filled with four modules of structured packing (Mellapak<sup>™</sup> 250.X, provided by Sulzer Chemtech), with a total packing height of 0.892 m. Mellapak<sup>™</sup> 250.X are made in Hastelloy C-22 alloy, which was selected to prevent acid corrosion effects during  $SO_2$  chemical absorption. The lab-scale system was already used for similar experiments and is fully described in Flagiello et al. (2018). The model flow gas was made by mixing compressed air with  $SO_2$  available in a pressurized cylinder at 2 % vol. in  $N_2$ , so to reach a concentration of 700 ppm<sub>v</sub> of  $SO_2$ ; the relative humidity was about 20-25 %. Feeding gas section was controlled by SMC Corporation digital flowmeters. The gas (8.12 m<sup>3</sup>/h or 0.29 m/s) was injected at the bottom of the column with a distance of 25 mm from the gas distributor (a mesh with rhombic grid) and 100 mm from the packing bottom. The absorbing liquid (varied between 31.25 to 93.75 L/h) was supplied at the top of the column, in counter-current flow to the gas, by a centrifugal pump (Grundfos Lenntech) and the liquid flow rate was controlled by a flowmeter (Cryotek Eng). The seawater solution was fed by a PNR<sup>®</sup> full spray nozzle (1212 B31 model) with a complete opening of the liquid jet equal to 45° and it was positioned to 35 mm from the packing top. The wash water was temporarily stored in the bottom section of the

column where samples were taken for measuring the solution pH value. The gas pressure at top and bottom of the column was measured by a differential manometer (FLUKE Corporation). In addition, a two-channel digital thermometer (PCE Instruments) allowed temperature measure at two column levels (i.e. at inlet and outlet of the column, after a demister).

### 3.3 Analytical methods

The gas measurement for the spray column was made by MGA 3000C Multi-Gas Analyser with NDIR detectors for SO<sub>2</sub>/CO/CO<sub>2</sub> and galvanic cell for O<sub>2</sub>, while for the packed-bed column, the quantitative analysis of SO<sub>2</sub> concentration was made by ABB O2020<sup>®</sup> Advanced optima process gas analyser. The analysers were daily calibrated using the same reference gas cylinders. The pH values of the absorbing and the exhaust solution were measured by Thermo Scientific ORION Start, A111 pH Meter model. The chemical characterization of the seawater was performed by ionic chromatography (Metrohm AG, 883 Basic IC PLUS), and reported in Table 1.

Table 1: Chemical characterization of the seawater

pH	Cl <sup>-</sup> [g/L]	SO <sub>4</sub> <sup>2-</sup> [g/L]	HCO <sub>3</sub> <sup>-</sup> [mg/L]	CO <sub>3</sub> <sup>-</sup> [mg/L]	NO <sub>3</sub> <sup>-</sup> [mg/L]	Br <sup>-</sup> [mg/L]	F <sup>-</sup> [mg/L]	Na <sup>+</sup> [g/L]	Mg <sup>2+</sup> [mg/L]	K <sup>+</sup> [mg/L]	Ca <sup>2+</sup> [mg/L]
8.1±0.1	20.03	2.81	653	16.20	4.21	0.06	0.07	9.59	26.18	2.34	111.80

## 4. Experimental results

Figure 3 reports the experimental results of the absorption tests carried out with different water flow rates (i.e. feed liquid-to-gas ratio, L/G) in spray and packed-bed column with a model flue gas containing 700 ppm<sub>v</sub> of SO<sub>2</sub> at 25°C and 1 bar. Experiments are presented in terms of SO<sub>2</sub> removal efficiency and wash water pH as function of volumetric L/G ratio (Figure 1).

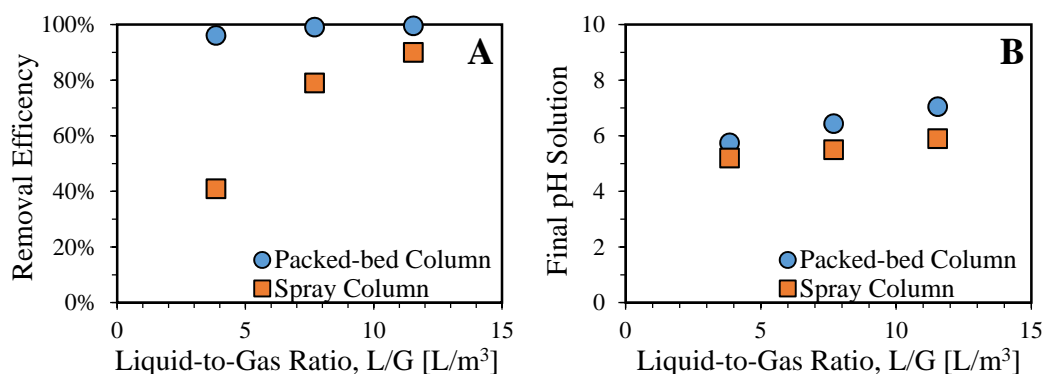


Figure 1: Experimental results in spray and packed-bed columns in terms of SO<sub>2</sub> removal efficiency (A) and wash water pH (B) as a function of the liquid-to-gas volumetric ratio.

The experiments in Figure 1A show that the structured packing column allows achieving higher SO<sub>2</sub> removal efficiency than the spray system in the entire L/G range investigated. In particular, in the packed-bed column, the SO<sub>2</sub> removal efficiencies are all very high and equal to 96.1-99.6 %, so the liquid flow rate does not produce significant effects (except for the L/G range 3.34-7.69 L/m<sup>3</sup>). Differently, the absorption efficiency in spray column shows a marked increasing trend with L/G values (41.8 to 90 %), confirming a high dependence on the liquid flow rate. As a comparison, we can notice that the packed column provided a 96 % of removal efficiency with L/G equal to 3.84 L/m<sup>3</sup> while a similar efficiency required more than 11.53 L/m<sup>3</sup> in the spray column. This is likely to be related to a better distribution and utilization of absorbing liquid in the packed column as well as to a higher effective wet surface.

The final pH values of absorbing liquid, reached during the SO<sub>2</sub> capture (see Figure 1B), are slightly acidic, confirming that the SWS units are mostly consuming the natural alkalinity of seawater (cf. Table 1), which determines low costs for the restoration of the wash water. In principle, the pH values for the packed column should be lower than those of the spray column, due to their higher removal efficiency. However, these discrepancies may be related to changes of the wash water pH in the storage tank and in the tube lines of the spray column.

In Figure 2, the operating lines (molar balance equation on total sulphur) for both the columns and the corresponding equilibrium data (Flagiello et al., 2018) are reported.

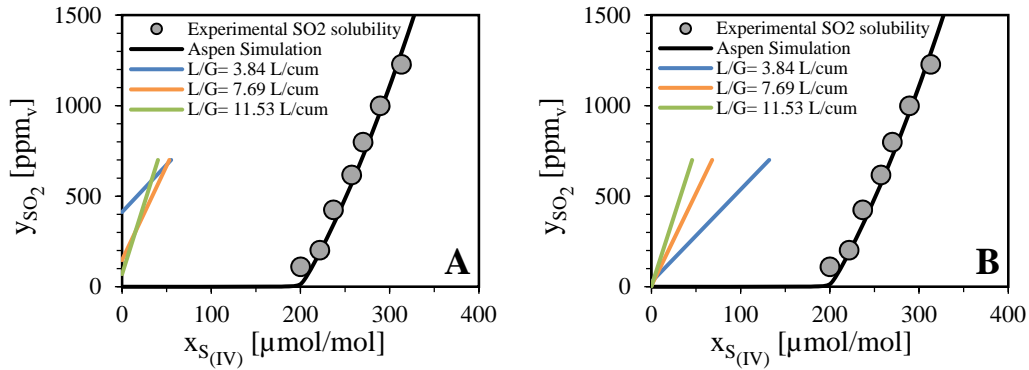


Figure 2: Equilibrium data and operating conditions as a function of the L/G ratio for SO<sub>2</sub> absorption at 700 ppm<sub>v</sub> in spray column (A) and packed-bed column (B)

Figure 2 shows that, in the investigated operating conditions, the equilibrium data are in the  $y^* \approx 0$  region, hence SO<sub>2</sub> absorption is thermodynamically favoured, due to high L/G values and a large excess of alkalinity in the aqueous phase. In these cases, the enhancement factor (E) in Eq(5) is very high, hence the liquid resistance ( $\lambda HTU_L$ ) can be considered as negligible and the SO<sub>2</sub> absorption is limited only by gas mass transfer resistance ( $HTU_{OG} = HTU_G$ ). Moreover, from the data in Figure 2 it is possible to calculate the NTU<sub>OG</sub> values using the Eq(3) and the overall resistance to mass transfer ( $HTU_{OG}$ ) with Eq(1), for the two columns. For the investigated L/G values, the packed-bed column showed a slight reduction of the  $HTU_{OG}$  with L/G (0.27, 0.19 and 0.16 m). In this evaluation, the mass transfer contributions in the column sections above and below the packing are neglected. For the spray column, the  $HTU_{OG}$  values resulted much higher (6.63, 2.24 and 1.52 m) with a strong decrease between the first values, corresponding to L/G of 3.84 and 7.69 L/m<sup>3</sup> (500 and 1000 L/h). In our opinion, this effect depends on the amount of liquid dispersed over the scrubber wall. The experimental results confirm that the packing allowed a reduction of the  $HTU_{OG}$  (or  $HTU_G$ ) of one order of magnitude, thanks to its optimal water distribution and to the higher mass transfer rates. In fact, using Eq(5), the gas mass transfer coefficient ( $k_{y,SO_2}a_e$ ) can be estimated for the two units. For the three L/G ratios, the values for the packed-bed column are equal to  $4.40 \cdot 10^{-2}$ ,  $6.55 \cdot 10^{-2}$  and  $7.50 \cdot 10^{-2}$  kmol/m<sup>3</sup>s while for the spray column the effective mass transfer coefficients are about ten times smaller. However, for a correct estimation, the actual droplets concentration along the height of the column (depending on the amount of liquid loss on the scrubber walls) and, consequently, on the actual value of  $a_e$  should be determined. Hence, the comparison with model results can be made only for packed-bed column. To this aim, Table 2 shows a comparison between the experimental values of  $k_{y,SO_2}a_e$  for the packed-bed column and the results of predictive models available in the open literature.

Table 2: Comparison of the experimental and modelling  $k_{y,SO_2}a_e$  for the packed-bed column

L/G Ratio [L/m <sup>3</sup> ]	Experimental $k_{y,SO_2}a_e$ [kmol/m <sup>3</sup> s]	Bravo et al. (1985) [kmol/m <sup>3</sup> s]	Bravo et al. (1992) [kmol/m <sup>3</sup> s]	Brunazzi et al. (1997) [kmol/m <sup>3</sup> s]	Olujic et al. (2004) [kmol/m <sup>3</sup> s]
3.84	$4.40 \cdot 10^{-2}$	$3.71 \cdot 10^{-2}$	$6.39 \cdot 10^{-3}$	$1.25 \cdot 10^{-2}$	$1.32 \cdot 10^{-2}$
7.69	$6.55 \cdot 10^{-2}$	$3.94 \cdot 10^{-2}$	$8.95 \cdot 10^{-3}$	$1.81 \cdot 10^{-2}$	$1.72 \cdot 10^{-2}$
11.53	$7.50 \cdot 10^{-2}$	$4.13 \cdot 10^{-2}$	$1.09 \cdot 10^{-2}$	$2.27 \cdot 10^{-2}$	$2.01 \cdot 10^{-2}$

The modelling results are not able to accurately describe the experimental  $k_{y,SO_2}a_e$ , except at low liquid flow rates by Bravo et al. (1985) correlations. It is worth noting that some predictive models used (Bravo et al., 1992; Brunazzi and Paglianti, 1997; Olujic et al., 2004) have corrective parameters provided by the authors, for the packing-types studied. In the light of these results, no predictive models allow the correct estimation of mass transfer rates, so it is clear that a fluid-dynamic characterization of the Mellapak™ 250.X is necessary.

## 5. Conclusion

Experimental results showed that the packed column with structured packing allows achieving higher SO<sub>2</sub> removal efficiencies than the conventional spray systems currently used on FGD units, with significant water

and energy savings. For marine applications, the packed-bed column allows to reach an SO<sub>2</sub> emission target of 20 ppm<sub>v</sub> (equivalent to 0.1 % sulphur in the fuel) with L/G values just above 3.84 L/m<sup>3</sup>, while for the spray column neither of the investigated L/G ratios allowed the compliance with regulations. The reduction of equipment size is a crucial issue in the naval sector, where the achievement of increasingly smaller but at the same time more performing technologies is trying to reach. This new type of SWS is a valid alternative also to reduce plant volumes and costs related to the water pumping, maintenance and wash water treatment. Also for stationary combustion plants, in order to comply with current regulations (IED valid until 2021) a higher SO<sub>2</sub> removal efficiency must be reached (equal to 90 % for 700 ppm<sub>v</sub> of SO<sub>2</sub>). In this case, the target efficiency can be achieved using 11.53 L/m<sup>3</sup> in spray column and less than 3.84 L/m<sup>3</sup> in the packed column (realizing a water saving of more than 70 %). Such a reduction is likely to be limited to small to medium size units, where the requirements for high water dosage and geometric constraints on the scrubber section favour the loss of water against the scrubber walls.

The water saving associated to packed-bed columns results particularly useful in terms of space and weight reduction, due to the parallel reduction of pumps, pipings and other auxiliaries (as the wash water treatment plants). It is also beneficial in terms of CAPEX and OPEX of the FGD plants. For example, the costs of centrifugal pumps depends by a factor between 0.6 and 0.7 on the water flow rate. Hence, the 70 % water savings is associated with about 50 % reduction of CAPEX and a 70 % reduction of OPEX for pumping. A similar reduction is expected for the pipings and wash water treatment CAPEX. These savings can sustain the utilization of packed units in place of spray ones. Of course, a detailed technical-economic analysis is required for each special case, but we envisage that use of packed columns can provide a significant improvement in many of the industrial and the marine applications of seawater scrubbing.

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