

## Carbon Dioxide Capture from Model Marine Diesel Engine Exhaust by means of $K_2CO_3$ -Based Sorbents

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The on-board ship installation of a high-efficiency and low-cost  $CO_2$  separation unit seems to be an attractive option to comply with recent regulations aimed at reducing the carbon footprint derived from the maritime sector. Our research group is currently developing a capture and storage scheme based on the use of potassium carbonate for  $CO_2$  conversion into solid potassium bicarbonate.

In this paper, we summarize preliminary  $CO_2$  capture tests on both raw and alumina-supported  $K_2CO_3$  carried out in a fixed bed apparatus under typical model marine diesel engine exhaust composition ( $CO_2$  5 %,  $H_2O$  5 %,  $N_2$  90 % by vol.) and temperatures (60 - 105 °C). Carbonation data for the parent bulk  $K_2CO_3$  showed a maximum capture capacity of 0.138 mmol  $g^{-1}$  corresponding to a nearly 2 % sorbent utilization factor. An increase in the operating temperature produced a reduction of the carbonation capacity and faster capture kinetics. The alumina-supported sorbent tested at 60 °C displayed enhanced  $CO_2$  capture capacity with a maximum conversion degree of 43 %. This testifies the positive effect derived by the dispersion of the active phase onto a substrate with large surface area in the  $CO_2$  capture process, likely for a remarkable reduction of diffusion limitations during the carbonation reaction in the case of nano-sized potassium carbonate with respect to granular sorbents.

### 1. Introduction

The reduction of  $CO_2$  emissions deriving from human activities is nowadays required to face global climate change (Figuerola et al., 2008). Besides electricity and heat generation systems that produce nearly two-thirds of global  $CO_2$  emissions, about 23 % of carbon footprint derives from the transport sector (International Energy Agency, 2014). Contextually, the European Union set the target of 40 % cut in maritime shipping emissions to be achieved in 2050 (European Commission, 2011). In order to cut  $CO_2$  emissions, the International Maritime Organization (IMO) introduced different measures, among which the Energy Efficient Design Index, EEDI (International Maritime Organization, 2014). Ship design architecture, design of auxiliary systems, alternative propulsion systems and routing can reduce energy consumption and consequently  $CO_2$  emissions (Di Natale and Carotenuto, 2015). In the framework of the carbon capture and storage (CCS) approach, post-combustion purification systems for  $CO_2$  capture from flue-gas have the greatest near-term potential to mitigate  $CO_2$  environmental impacts, as they can be retrofitted to existing emission sources (Erto et al., 2015). Chemical scrubbing of  $CO_2$  in aqueous amine solutions (mainly monoethanolamine, MEA) represents the most widely investigated technology in the CCS field, but the process suffers drawbacks related to the considerable amounts of thermal energy required for absorbent regeneration, solvent degradation and equipment corrosion (Boot-Handford et al., 2014). In 2012, Det Norske Veritas (DNV) and Process Systems Enterprise Ltd. (PSE) developed a design concept for the application of CCS on-board ships based on  $CO_2$  chemical absorption into amine solutions (DNV-GL, 2013).

Our research group is currently developing a CCS scheme for on-board naval installation based on the use of potassium carbonate that converts  $CO_2$  into potassium bicarbonate. The process can be operated at low temperature, about 50 °C, as a tail-end unit after a  $SO_2$  scrubbing system. The exhaust sorbent can be regenerated in the temperature range 100 - 200 °C (Zhao et al., 2013) to recover  $CO_2$  as an almost pure gas,

stored as a liquid in cryogenic tank and disposed, or sold, at docks. The use of alkali metal-based sorbents (e.g.  $K_2CO_3$ ) is considered a cost-effective and an energy-efficient technology for  $CO_2$  capture when compared with the conventional MEA process (Zhao et al., 2013).

In this paper, we report experimental results on  $CO_2$  removal from a model marine diesel engine exhaust by means of  $K_2CO_3$  both raw and supported onto porous alumina.  $CO_2$  carbonation tests were carried out in a fixed bed column integrated in a lab-scale unit in the temperature range 60-105 °C. The obtained results allowed a preliminary assessment of the potentiality of this purification technology for  $CO_2$  capture in the maritime sector. This research activity aims at the development of a  $CO_2$  capture unit to be integrated into depuration systems that we are developing to cut gaseous pollutants and particulate matter emissions deriving from marine diesel engine exhaust (Di Natale et al., 2013).

## 2. Materials and methods

### 2.1 Sorbents

The sorbent material used in carbonation tests is a commercially available potassium carbonate (Carlo Erba Reagents). The raw  $K_2CO_3$  (termed  $K_2CO_3$ -raw) was mechanically sieved in the range 300 - 630  $\mu m$  so to obtain sorbent particles ensuring low pressure drops ( $\approx 10^{-3}$  bar) across the fixed bed reactor. The as-received sorbent textural properties were obtained by  $N_2$  adsorption at -196 °C in a volumetric apparatus (Sorptomatic 1990). The porosimetric analysis highlighted a prevailing macroporous nature with a mean pore diameter ( $d_{pore}$ ) equal to 119 nm. Moreover, the total pore volume ( $V_p$ ) and the surface area ( $S_{BET}$ ) are 0.17  $cm^3 g^{-1}$  and 5.9  $m^2 g^{-1}$ , respectively. An alumina-supported  $K_2CO_3$  sorbent (termed  $K_2CO_3$ -sup) was also synthesized to verify the effectiveness of dispersing nano-sized  $K_2CO_3$  onto a large surface area substrate in the  $CO_2$  capture process. To this aim, a commercial  $\gamma-Al_2O_3$  (1 mm diameter spheres, supplied by SASOL) was adopted as substrate. The nitrogen porosimetric analysis of the bare support revealed a mesoporous structure with  $d_{pore} = 10$  nm,  $V_p = 0.47$   $cm^3 g^{-1}$  and  $S_{BET} = 166$   $m^2 g^{-1}$ . A functionalized sorbent with 20 %wt.  $K_2CO_3$  loading was prepared via incipient wetness impregnation: an aqueous solution of anhydrous potassium carbonate (active phase concentration and total solution volume equal to 0.625  $g mL^{-1}$  and 4 mL, respectively) was added dropwise under stirring to 10 g of  $\gamma-Al_2O_3$ . Finally, the sorbent was dried in a fixed bed reactor at 115 °C under a  $N_2$  flow (0.45  $L min^{-1}$ , evaluated at 25 °C and 1 bar).

### 2.2 Carbonation tests and data analysis

Figure 1 depicts a schematic representation of the lab-scale apparatus adopted for  $CO_2$  capture runs.

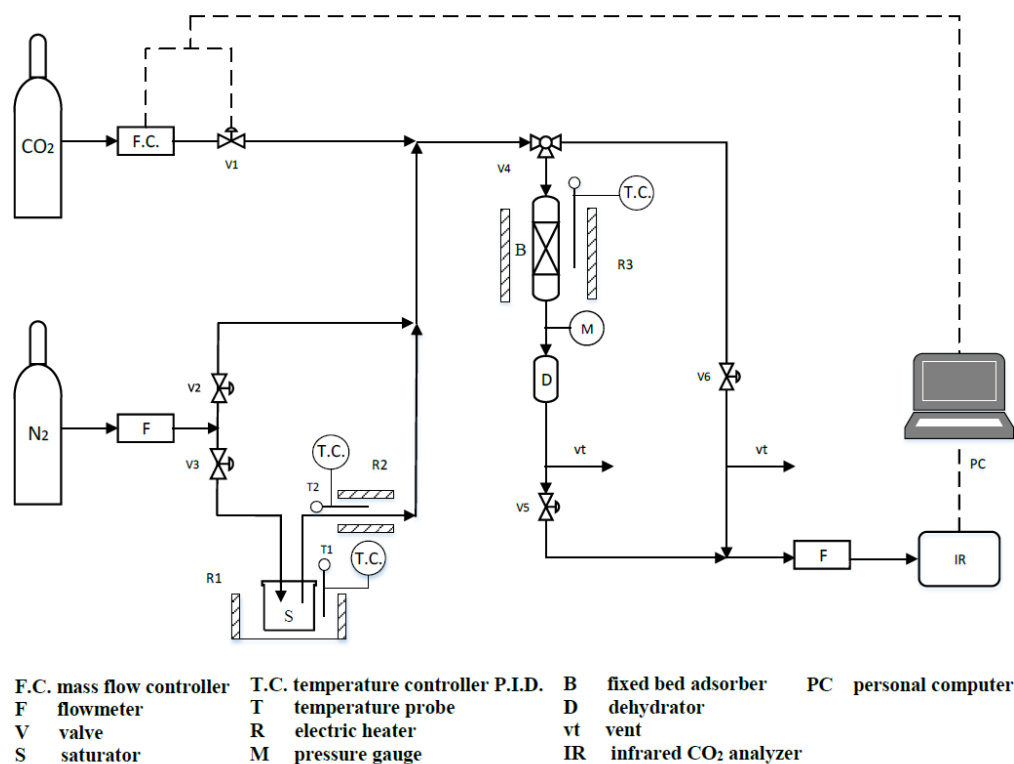


Figure 1: Layout of the experimental apparatus

The carbonation reactor is a stainless steel fixed bed column (length = 11 cm; inner diameter = 2 cm), equipped with a 35  $\mu\text{m}$  porous septum. The fixed bed temperature was controlled by means of a heating system arranged coaxially with the carbonation unit. It is made up of two 320 W cylindrical shell band heaters (Watlow) enveloped in a thermal insulating layer of stone wool and connected to PID controller (i/16 series Omega). The fixed bed temperature was measured by means of a K-type thermocouple. Pure carbon dioxide was fed to the reactor by means of a mass flow controller (series EI Flow Bronkhorst 201-CV), and mixed with a pre-humidified nitrogen stream ( $\text{N}_2$  flow rate controlled by a flow meter) to generate a gas mixture of desired  $\text{CO}_2$  concentration. The humidity level of the feed gas was set by fluxing the  $\text{N}_2$  stream in a thermostatically-controlled water saturator set at 33  $^\circ\text{C}$ . Carbon dioxide concentration was measured by a continuous NDIR gas analyzer (AO2020 Uras 26 provided by ABB); data acquisition were performed by interfacing the gas analyzer with a PC via LabView™ software. A  $\text{CaCl}_2$  trap was placed at the fixed bed outlet to remove water prior to  $\text{CO}_2$  concentration measurements.

Continuous carbonation tests were performed by feeding the column with a 1.2  $\text{L min}^{-1}$  gas mixture with 5 %  $\text{CO}_2$ , 5 %  $\text{H}_2\text{O}$  and 90 %  $\text{N}_2$  to simulate the typical exhaust composition of a marine diesel engine (Environmental Protection Agency, 2000). The column was loaded with a known sorbent amount (20 g for  $\text{K}_2\text{CO}_3$ -raw and 12.5 g at 20 %wt. potassium carbonate loading for  $\text{K}_2\text{CO}_3$ -sup). The effect of operating temperature on the  $\text{K}_2\text{CO}_3$ -raw capture performances was investigated at 60, 75, 90 and 105  $^\circ\text{C}$ . A preliminary carbonation test for the supported sorbent was carried out at 60  $^\circ\text{C}$ , which resulted the best operating temperature (among those investigated) for  $\text{CO}_2$  capture, as obtained from tests conducted on the raw  $\text{K}_2\text{CO}_3$ . Dynamic carbonation tests allowed to calculate the amount of  $\text{CO}_2$  captured per unit mass of  $\text{K}_2\text{CO}_3$  at any time  $t$ ,  $\omega(t)$  [ $\text{mmol g}^{-1}$ ], via the following material balance over the fixed bed reactor:

$$\omega(t) = \frac{Q_{\text{CO}_2}^{\text{in}} \rho_{\text{CO}_2}}{m M_{\text{CO}_2}} \int_0^t \left( 1 - \frac{Q_{\text{CO}_2}^{\text{out}}(t)}{Q_{\text{CO}_2}^{\text{in}}} \right) dt \quad (1)$$

where:  $Q_{\text{CO}_2}^{\text{out}}(t)$  and  $Q_{\text{CO}_2}^{\text{in}}$  [ $\text{L s}^{-1}$ ] represent the  $\text{CO}_2$  volumetric flow rates at the bed outlet and inlet, respectively;  $m$  [g] is the  $\text{K}_2\text{CO}_3$  mass loaded into the column;  $\rho_{\text{CO}_2}$  [ $\text{mg L}^{-1}$ ] represents the  $\text{CO}_2$  density (at 20 $^\circ\text{C}$  and 1 bar);  $M_{\text{CO}_2}$  is  $\text{CO}_2$  molecular weight [ $\text{mg mmol}^{-1}$ ]. If  $t=t^*$ , where  $t^*$  [s] is the saturation time for which the  $\text{CO}_2$  outlet concentration is approximately equal to 99 % of its inlet value,  $\omega(t)$  coincides with the saturation capacity  $\omega^s$ .

The carbonation kinetics was conveniently expressed in terms of time evolution of the  $\text{K}_2\text{CO}_3$  conversion degree  $x(t)$  [-] corresponding to the molar fraction of active phase reacted with  $\text{CO}_2$ . Considering a 1:1 stoichiometry for the reaction between  $\text{CO}_2$  and  $\text{K}_2\text{CO}_3$  (Zhao et al., 2013),  $x(t)$  can be expressed as:

$$x(t) = \omega(t) M_{\text{K}_2\text{CO}_3} \quad (2)$$

where  $M_{\text{K}_2\text{CO}_3}$  is the molecular weight of  $\text{K}_2\text{CO}_3$  [ $\text{g mmol}^{-1}$ ]. The experimental value of  $x(t)$  corresponding to saturation conditions is named  $x^{\text{max}}$ .

Kinetic differences in the  $\text{CO}_2$  capture process for the sorbents tested under different operating conditions were also interpreted in light of a simple pseudo-first order model (termed PFO) applied to  $x(t)$  vs  $t$  patterns:

$$x(t) = x^{\text{max}} [1 - \exp(-kt)] \quad (3)$$

where  $k$  [ $\text{s}^{-1}$ ] is a pseudo-first order kinetic constant and it was obtained as best-fitting parameter via the least square method.

Finally, an initial carbonation rate  $\Phi$  [ $\text{s}^{-1}$ ] can be computed from the PFO model as:

$$\Phi = \left. \frac{dx(t)}{dt} \right|_{t=0} = k x^{\text{max}} \quad (4)$$

### 3. Results and discussion

Table 1 summarizes the main  $\text{CO}_2$  capture data obtained from the experimental carbonation tests performed onto both raw ( $\text{K}_2\text{CO}_3$ -raw) and alumina-supported potassium carbonate ( $\text{K}_2\text{CO}_3$ -sup) together with the kinetic parameters derived from the PFO model applied to dynamic removal data. Figure 2(a)-(b) depicts the time dependence of the carbonation degree  $x(t)$  in the different operating conditions.

Table 1: Main parameters obtained from the carbonation process of  $K_2CO_3$ -raw and  $K_2CO_3$ -sup sorbents

Sorbent	Carbonation temperature	$\omega^s$ [mmol g <sup>-1</sup> ]	$x^{max}$ [-]	$t^+$ [s]	k [s <sup>-1</sup> ]	$\Phi$ [s <sup>-1</sup> ]
	[°C]					
$K_2CO_3$ -raw	60	$1.38 \times 10^{-1}$	$1.9 \times 10^{-2}$	500	$7.7 \times 10^{-3}$	$1.5 \times 10^{-4}$
	75	$1.01 \times 10^{-1}$	$1.4 \times 10^{-2}$	480	$8.2 \times 10^{-3}$	$1.1 \times 10^{-4}$
	90	$2.90 \times 10^{-2}$	$4.0 \times 10^{-3}$	40	$1.0 \times 10^{-1}$	$4.0 \times 10^{-4}$
	105	$2.60 \times 10^{-2}$	$3.6 \times 10^{-3}$	40	$1.2 \times 10^{-1}$	$4.3 \times 10^{-4}$
$K_2CO_3$ -sup	60	3.1	$4.3 \times 10^{-1}$	900	$3.5 \times 10^{-3}$	$1.5 \times 10^{-3}$

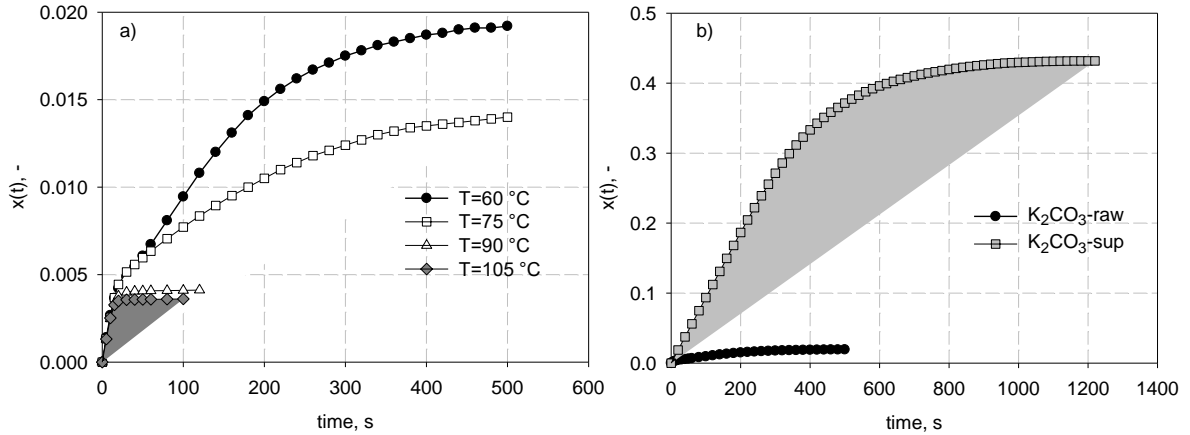


Figure 2: Time evolution of the carbonation degree  $x(t)$  for a) raw  $K_2CO_3$  at different temperatures and b) raw and alumina-supported  $K_2CO_3$  at 60 °C

Data obtained for the raw  $K_2CO_3$  testify the significant effect exerted by the process temperature on the sorbent capture. In fact, the experimental value of the carbonation degree obtained under saturation conditions  $x^{max}$  monotonically decreases as the process temperature is raised: at 60 °C  $x^{max}$  is about 5-times the values retrieved at 90 and 105 °C. This behavior can be related to the exothermic nature of the carbonation process for the tested sorbent (Zhao et al., 2013). On contrary, the dynamic carbonation profiles obtained for  $K_2CO_3$ -raw generally highlight a faster  $CO_2$  capture process when the temperature increases. Tests carried out at 60 and 75 °C show that saturation conditions are attained in times shorter than 500 s, whereas a plateau value of the carbonation degree is reached for  $t < 50$  s for higher temperatures (Figure 2 (a)). These observations are also corroborated by the kinetic parameters retrieved from the PFO model (Table 1). Contextually, the pseudo-first order kinetic constant  $k$  derived at 105 °C is approximately 16- and 15-times greater than the values obtained at 60 and 75 °C, respectively. Similarly, the initial adsorption rate  $\Phi$  both at 90 and 105 °C increases about 3-times with respect to the values obtained at lower temperatures. Coherently with experimentally observed  $x(t)$  patterns, negligible differences in  $k$  and  $\Phi$  values can be inferred for tests performed at 90 and 105 °C. Faster carbonation kinetics at higher temperatures can be likely related both to a quicker diffusion process of  $CO_2$  molecules in the pore network of  $K_2CO_3$  and to the already described decrease of  $x^{max}$  (smaller number of reactive sites to be converted) (Ruthven, 1984). As a general comment, carbonation data obtained for the raw potassium carbonate highlight a poor exploitation of the active phase, with a maximum conversion degree equal to nearly 2 % in the best case (i.e. at 60 °C). The experimental evidences suggest that  $K_2CO_3$  characterized by large particle sizes experiences strong diffusion limitations during the conversion process: the formation of a less porous product layer ( $KHCO_3$ ) hinders the  $CO_2$  molecules to access to the potassium carbonate active sites (Guo et al, 2015). Therefore, a large particle volume fraction is left as an unreacted core of potassium carbonate, which results in a very low carbonation degree. In turn, this makes unpractical the use of raw  $K_2CO_3$  for reducing  $CO_2$  emissions on-board ships, because large-volume reactors would be required with associated problems of space allocation and high additional fuel consumption. Consequently, we investigated the use of  $K_2CO_3$  supported onto a large surface area alumina to overcome the aforementioned drawbacks. The sorbent obtained at 20 %wt. loading of the active phase was tested under the same feed composition adopted for  $K_2CO_3$ -raw at 60 °C, the latter chosen as the best T-level on the basis of the previously analyzed carbonation data.

A comparison of  $x^{\max}$  and  $\omega^s$  data for  $K_2CO_3$ -raw and  $K_2CO_3$ -sup (see Table 1) remarks the effectiveness of the dispersion of  $K_2CO_3$  on alumina, determining a significant increase in the  $CO_2$  capture capacity. In fact,  $\omega^s$  for the supported sorbent is  $3.1 \text{ mmol g}^{-1}$ , a value about 23-times greater than the one obtained for the unsupported sorbent. Moreover, a maximum conversion degree of 0.43 is reached. It should be highlighted that the contribution of the raw substrate in the  $CO_2$  capture process was practically negligible under the tested experimental conditions ( $5 \times 10^{-3} \text{ mmol g}^{-1}$ ) with respect to the  $K_2CO_3$ -sup capture capacity. The dynamic  $CO_2$  capture data reported for  $K_2CO_3$ -raw and  $K_2CO_3$ -sup (cf. Figure 2 (b)) show that the supported sorbent requires a longer time to reach saturation conditions with respect to the parent  $K_2CO_3$  ( $t^* \approx 900$  and  $500$  s for  $K_2CO_3$ -sup and  $K_2CO_3$ -raw, respectively). This is mirrored by the  $k$  value derived for  $K_2CO_3$ -sup that is almost halved when compared to the value obtained for  $K_2CO_3$ -raw. The generally slower capture process observed for the supported sorbent, mainly in the late stages of the process, could be related to a greater number of active sites of potassium carbonate to be converted into potassium bicarbonate. On the other hand, in the early stages of the process a higher number of reactive centers for  $K_2CO_3$ -sup with respect to  $K_2CO_3$ -raw is likely to determine a higher driving force for the carbonation reaction, thus producing a faster capture rate (cf. the initial slopes of  $x(t)$  curves in Figure 2 (b)). This pattern is also witnessed by the initial carbonation rate  $\Phi$ , being one order of magnitude greater in the case of  $K_2CO_3$ -sup with respect to  $K_2CO_3$ -raw.

#### 4. Conclusions

This work belongs to a wider research framework aimed at developing high-efficiency depuration processes of marine diesel engine exhaust, which includes the abatement of  $NO_x$ ,  $SO_2$  and PM (Di Natale et al., 2015). The depuration train developing at our research facilities includes a final stage for reducing  $CO_2$  emissions, based on the use of potassium carbonate as a selective reactant.

In this paper, we reported the results of experiments on  $CO_2$  capture from a mimicking marine diesel engine effluent on both raw and alumina-supported potassium carbonate. Carbonation tests for the raw  $K_2CO_3$  indicated a higher reactivity of the sorbent at  $60 \text{ }^\circ\text{C}$ , due to the exothermic nature of the carbonation process. On the other hand, a low maximum conversion degree was obtained (2 %), likely due to strong diffusion limitations occurring for coarse particles, thus making unsupported  $K_2CO_3$  not suitable for  $CO_2$  emissions reduction in the maritime sector. The dispersion of the active phase onto a large surface area and porous alumina support allowed to significantly enhance the sorbent exploitation, with a maximum conversion percentage equal to 43 %. The preliminary results of  $CO_2$  capture performances obtained for the supported sorbent are very encouraging in the light of on-board ships installation of a carbonation reactor, based on this class of sorbents, after a  $SO_2$  scrubbing unit.

Future research efforts should be devoted at investigating different classes of substrates and functionalization conditions (i.e. active phase loading) allowing a maximization of the reactant conversion degree, also to reduce the carbonator size (at fixed removal efficiency), the latter being a critical aspect for on-board integration of this purification unit. Regeneration studies in steam will be also performed, in order to ensure a proper re-usability of the sorbent under multiple carbonation-decarbonation cycles and for recovering almost pure carbon dioxide for storage purposes.

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