



RESEARCH ARTICLE

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SULFURE DIOXIDE DEEXPERIMENTAL ASSESSMENT OF SULFURE DIOXIDE REMOVAL FROM GAS STREAM FOR INTENSIVE ALGAE CULTURE

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ABSTRACT

In this research, several SO₂ removal alternatives were evaluated for the treatment of gaseous effluents generated by the "Carlos Manuel de Céspedes" power plant in Cienfuegos to be use in gaseous stream to *Ulva lettuce* intensive culture. A low-cost sensor was used to monitor the gas concentration during the proposed methods. The sensor was previously calibrated and a linear regression model with a coefficient of determination of 0,9926 was obtained. The alternatives evaluated were adsorption with activated carbon, absorption with calcium hydroxide and absorption with seawater. The removal was evaluated at three levels SO₂ concentration. In addition, the parameters pH, salinity and alkalinity were determined to a seawater sample from the Bay of Cienfuegos and with which the experiments were carried out. The values obtained were 8,17±0,003; 26,50±0,10 ups and 171,67±1,03 mg/L respectively, which confirms its good performance for gas removal. The three methods studied showed satisfactory results for SO₂ removal. The seawater absorption experiment was the most efficient alternative, achieving the lowest final concentrations in the shortest time.



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I. INTRODUCTION

Sulfur compounds are one of the most important acidifying agents and appear to be key factors in regulating the ionic composition and nutrient efficiency of natural environments. However, anthropogenic emissions of SO₂ originates mainly in process that involve combustion processes. [1],[2]. This pollutant is produced by sulfur-containing fossil fuels used for thermal power generation, heating, cooking, and transportation. Oil refineries and ore smelters are additional sources. In the atmosphere, SO₂ is converted to SO₃ by reaction with oxygen. SO₂ and SO₃ react with the moisture in the air to form H₂SO₃ and H₂SO₄, and producing acid rain [3].

In Cuba net emission inventories showed the importance of SO₂ as a pollutant because of the use of old technologies in industry and transportation leads to higher fuel consumption levels. Alejo, D [3] obtain that an average concentration of 4 ppb for the period February to April 2010 in Santa Clara City and in Havana, a study of air pollution due to vehicular traffic showed average SO₂ concentrations between 5 and 15 ppb, with peaks of up to 60 ppb [4].

In the province of Cienfuegos, SO₂ emissions are 43,205 tons per year, which represents 78.8% of the main pollutants in this city. Ninety-nine percent of these emissions come from boilers and furnaces, including the power plant and the oil refinery, which emit the most with more than 42 thousand tons per year [5].

For this reason, there is a need to control SO₂ and to invest in technologies that remove the gaseous emissions in the industry and the transportation

This work aims to compare SO₂ removal with low-cost system according three experimental methods in laboratories. Gas desulphurization by mean absorption in alkaline solution of Ca(OH)₂, alkaline seawater solution and adsorption with activated carbon.

II. THEORETICAL REFERENCE

II.1 METHODS OF GAS DESULPHURIZATION.

Flue gas desulphurization is carried out by mean non-regenerative processes and regenerative processes, the former being the most widespread in the world - in both processes, alkaline solutions or suspensions are used. In non-regenerative plants, the reagent used to retain SO₂ is disposed of; low-cost absorbent reagents are usually used, mainly aqueous limestone suspensions, Ca(OH)₂ and seawater [6].

Wet technology based on calcium hydroxide has its value in the best reactivity of Ca(OH)₂ than limestone slurry, but it is still more expensive raw material. The main drawback of these plants is that the manipulation and management of large volumes of waste sludge is necessary, although in some of these technologies reusable by-products are obtained that improve the operating costs.

Seawater is naturally alkaline and has a high SO₂ neutralization capacity. The seawater process is very similar to that of the Limestone with Forced Oxidation (LSFO) process, except that there is no dissolution or precipitation of solids [7].

On the other hand, in regenerative processes, the reagent is recovered after SO₂ elimination, usually thermally; however, the technological complexity of the design and the costs are high. Both technologies, regenerative and non-regenerative, are in turn divided into dry or wet. In this sense, about dry regenerative process, there are several studies that show the effectiveness of activated carbon in the removal of polluting gases, specifically. SO₂ [8-10].

III. MATERIALS AND METHODS.

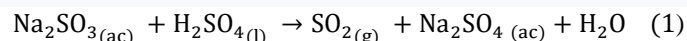
III.1 LOW-COST MONITORING SYSTEM

The principles of low-cost systems have already been described in previous work [46,47]. Here, only the principle of the SO₂ and CO₂ sensors measurements is described in more detail. The low-cost monitoring system that was used in the experiments consisted of an Arduino MEGA2560 as the computer unit and interface board, a real-time clock, and an SD card for data storage and the sensor The Arduino Mega 2560 contains an ADC with a 10-bit resolution. This ADC, with a reference voltage of 5 V, is capable of detecting voltage variations of about 4 mV.

The SO₂ sensor type A from the manufacturer Alphasense, is inserted in an analog conditioning board (analog front end), which is designed to accommodate 4 different sensors (part number 810-0023-00). The configuration used includes sensors for NO₂, OX (O₃ + NO₂), CO, and SO₂. The Analog Front End (AFE) has an internal power supply of 3.3 V and only requires an input voltage between 3.5 and 6.5 V (DC). The conditioning board provides 8 analog outputs (2 for each sensor; more precisely, the “working electrode” and the “auxiliary electrode”) that must be connected to analog-digital converters (ADC). In addition to the CO₂ sensor type a COZIR was coupled to the instrument.

Calibration have been carried out on SO₂ and CO₂ sensor allowing for obtaining reliable values of the readings of the

sensors. Inexpensive calibration of the SO₂ sensor by generating SO₂ gas inside a syringe according the reaction 1.



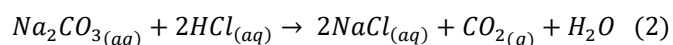
The SO₂ gas in the syringe was introduced in three successive steps and at each gas injection, the sensor signal jumped. The sensor reading followed the jumps of a staircase function, while the room temperature and relative humidity inside the box remained constant (i.e., 25°C and 65% RH).

González, R [11] pointed out that the voltages of the working and auxiliary electrodes for air without pollutants (W_{E0} and A_{E0}) can be obtained from a zero air calibration process. Then, the voltage values of the working electrode (W_E) can be calculated according the equation proposed by the manufacturer.

III.2 Experimental study of gas desulfurization.

The removal alternatives analyzed are adsorption with activated carbon, adsorption with a calcium hydroxide solution Ca(OH)₂ (ac) and adsorption with seawater. The gas flow is simulated for the study with an air flow where the SO₂ and CO₂ compositions are adjusted to the maximum values close to the detection limit of the sensor in order to obtain an initial gas mixture closer to the flue gas composition.

To obtain the CO₂ gaseous, it is necessary to adjust the initial composition of the mixture., reaction 2 is the following inside the syringe



The different volumes of SO₂ were obtained according to the stoichiometric reaction 2.1 and other two molar ratios of Na₂SO₃ and H₂SO₄

III.3 Design of the experimental system.

The evaluation of the gas desulfurization methods was carried out by means of a full factorial design as shown in Table 1 and indicated by the codes (-1, 0, 1). Factor A refers to the method used for SO₂ removal: adsorption with activated carbon A1, adsorption with calcium hydroxide A2 and adsorption with seawater A3. Factor B refers to the SO₂ concentration to be removed: ambient concentration B1, 10 ppm B2 and 20 ppm B3. The response variable is the SO₂ concentration expressed in ppm. To obtain the different levels of SO₂ to be removed, 8.6 mL, 16 mL and 22.9 mL previously diluted in a 1.2-liter container were injected into the chamber. These volumes correspond to the concentrations 5.79 ppm; 10.81 ppm and 20.85 ppm. A fixed amount of CO₂ corresponding to 470ppm was added to the system. The results were processed in the Statgraphic statistical program.

Table 1: Designing the experiment

A: Removal methods	B: Inicial SO ₂ concentration
A1 (-1)	B1 (-1)
A2 (0)	B1 (-1)
A3 (1)	B1 (-1)
A1 (-1)	B2 (0)
A2 (0)	B2 (0)
A3 (1)	B2 (0)
A1 (-1)	B3 (1)
A2 (0)	B3 (1)
A3 (1)	B3 (1)

Source: Authors, (2024).

III.4 SO₂ REMOVAL EXPERIMENT.

The configuration used for the activated carbon adsorption experiment is shown in Figure 1 and used a 2 cm diameter glass column with 1 cm of glass wool was placed at the bottom followed by 8 cm of activated carbon. The carbon used has a granulometry of ± 3 mm (for laboratory use only) and was activated at 120 °C for two hours prior to conducting the experiments.



Figure 1: Experimental setup used for activated carbon adsorption.

Source: Authors, (2024).

The setup in Figure 2 is the one used for SO₂ removal with Ca(OH)₂ and seawater. The box was also connected to a closed loop system. This circuit contained a pump, a wash bottle, an empty wash bottle to trap the water droplets present in the air, a U-shaped glass tube containing 10 g of silica gel to dry the air. After several minutes, zero air was obtained and the pump was turned off. A known amount (470ppm) of diluent was then added to the system.

Seawater from the Bay of Cienfuegos, where the "Carlos Manuel de Céspedes" power plant is located, was used for desulfurization with seawater. This sample was taken in October 2022, against the current and at a depth of 1 m, according to the Cuban standard NC 25:1999 "System of Standards for the Protection of the Environment. Hydrosphere. Specifications and Procedures for the Evaluation of Aquatic Objects for Fishing". [12]



Figure 2: Absorption with seawater and Ca(OH)₂.

Source: Authors, (2024).

The parameters included in the characterization were pH, salinity and alkalinity of the seawater, parameters that influence the absorption of SO₂ in this sorbent.

IV. RESULTS AND DISCUSSIONS

IV.1. ADSORPTION WITH ACTIVATED CARBON.

The results of the adsorption removal process with activated carbon are shown in Table 2 for the three levels evaluated, where it can be observed that in all cases gas removal is achieved, and that as the SO₂ concentration inside the chamber increases, the final concentration (cf) is higher than that obtained in the previous level and the time to reach the minimum SO₂ concentration in each experiment is greater when the initial concentration (ci) of the gas is at its maximum.

Table 2: Results of the adsorption removal process with activated carbon.

Levels	c _i (µg/m ³)	c _f (µg/m ³)	Time (s)
ambient air	0,030±0,002	0,008±0,007	898
10 ppm	9,061±0,222	0,030±0,045	926
20 ppm	19,139±0,667	0,111±0,028	1200

Source: Authors, (2024).

There have been several studies on the SO₂ removal capacity of activated carbon due to its high efficiency where the removal occurs by passing SO₂ to H₂SO₄ in the presence of water vapor and oxygen in the gas stream. The H₂SO₄ is retained on the carbon and subsequently regenerated. These studies demonstrate that the adsorption capacity of activated carbon is not only related to its surface area and pore size, but also to its surface chemical properties, its structure resistant to acidic or basic media and its stability at elevated temperatures. Finally, during this experiment the CO₂ sensor showed constant readings of around 470 ppm and no variation in gas concentration was observed, which is very positive for the intensive cultivation of marine macroalgae, which requires the supply of CO₂-rich gases.

IV.2. ABSORPTION WITH CALCIUM HYDROXIDE CA(OH)₂.

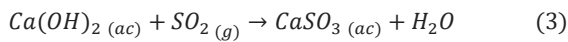
During the SO₂ removal process with the Ca(OH)₂ solution as absorber, as in the previous experiment, it can be observed that in all cases the removal of the gas is achieved, and that since the initial concentration of SO₂ inside the chamber is higher, the final concentration (cf) is higher than that obtained in the previous level as shown in Table 3. The time required to remove 9,626 ppm of SO₂ was less than in the previous level, while the time required to remove 18,972 ppm was higher than in the other experiments.

Table 3: Results of SO₂ removal with calcium hydroxide.

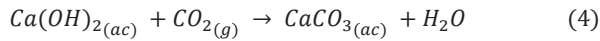
Levels	c _i (µg/m ³)	c _f (µg/m ³)	Time (s)
ambient air	0,026±0,006	0,003±0,003	920
10 ppm	9,626±0,327	0,059±0,005	909
20 ppm	18,972±0,838	0,065±0,028	1220

Source: Authors, (2024).

The behavior observed in the evaluation of this removal alternative can be explained by Equation 3. The resulting aqueous mixture from the adsorption, formed by CaSO₃, is usually treated when this method is used on an industrial scale.



During the desulfurization process using the inorganic absorber, the CO₂ sensor recorded an initial concentration of approximately 530 ppm. After the time required for SO₂ removal, a final concentration of 225 ppm CO₂ was obtained, corresponding to a loss of 305 ppm. This behavior can be explained by the consequent formation of calcium carbonate (CaCO₃), which is confirmed by chemical reaction 4. Despite the efficiency of the method to remove SO₂ gas, the removal of CO₂ is not convenient, since CO₂ is necessary for the photosynthetic process of macroalgae.



IV.3. CHARACTERIZATION OF SEAWATER

The different parameters of the seawater from the Bay of Cienfuegos (pH, salinity and alkalinity) used for the desulfurization process studied are shown in Table 4.

Table 4: Seawater characterization parameters.

pH	Salinity (Cl ⁻) ups	Alkalinity (CaCO ₃) mg/L
8,17±0,003	26,50 ± 0,10	171,67±1,03

Source: Authors, (2024).

The pH value obtained was 8.17±0.003 and the temperature indicated by the instrument was 28 °C. Despite the fact that the glass electrode used in the analysis is sensitive to the sodium element, the results obtained were in agreement with those reported by Seisdedo and Moreira [13], measured at the same sampling point and during the same climatic season, which was 8.18. According to the Cuban standard NC 25: 1999 [12] and according to the pH value measured, the water is of good quality with a pH value between 8.1 and 8.3. Zhang and Millero [14] studied the oxidation reaction of S(IV) to S(VI) as a function of pH (4.0-8.5). These studies showed a strong dependence, demonstrating that at pH above 7, there is the tendency for the rate of the reaction to slow down. These researchers also showed that, when the pH is higher than 7.5, the SO₂ dissolved in seawater is mainly present as SO₃²⁻ ions.

The salinity value obtained (26.50±0.10 ups), expressed as Cl⁻ ions, was similar to that reported by Seisdedo and Moreira [13] in the rainy period, which was 27.10 ups. In addition, considering the Cuban standard NC 25: 1999 [12] and the value obtained, the water is of good quality, since the salinity value is between 26 and 35 ups. The knowledge of this parameter is very important. In the studies carried out by Clarke and Radojevic [15], they demonstrated the catalytic effect of Cl⁻ ions in the oxidation reaction of S(IV) to S(VI). At pH 8.17 the total alkalinity value obtained is 171.67±1.03 mg/L, expressed as CaCO₃. These results confirm the alkaline capacity of seawater, making it suitable for SO₂ removal.

IV.4 Absorption with seawater.

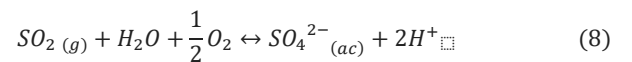
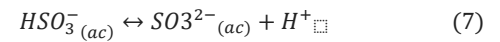
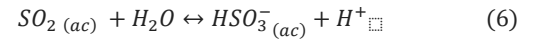
Table 5 shows the results obtained during the SO₂ removal using seawater from the Bay of Cienfuegos, where it is observed that in the third concentration level evaluated, a final SO₂ concentration was obtained that was lower than the previous level. In addition, the time taken to reach the minimum SO₂ concentration in each experiment was greater when the initial concentration (ci) of the gas was at its maximum.

Table 5. Results of SO₂ removal with seawater.

Levels	ci (µg/m ³)	cf (µg/m ³)	Time (s)
ambient air	0,022±0,002	0,001±0,001	600
10 ppm	9,808±1,177	0,027±0,045	828
20 ppm	18,830±0,374	0,018±0,001	1108

Source: Authors, (2024).

Gas desulfurization with seawater is explained by the reactions in equations 5 to 8. Dissolved SO₂ exists primarily as SO₃²⁻ ions because the pH of seawater is above 7.5. The absorbed SO₂ is then converted to SO₄²⁻ ions, Equation 8, which restores the oxygen and pH levels of the water before discharge to the sea.



In this experiment, the CO₂ sensor shows constant readings of about 450 ppm and no variation in gas concentration was observed, which is very positive since macroalgae, like all photosynthetic organisms, use CO₂ as a carbon source.

IV.5. STATISTICAL ANALYSIS.

In summary, the results obtained during the evaluation of the three desulfurization alternatives were satisfactory, as the SO₂ concentration decreased in all cases. Figures 3 and 4 show the main effects and estimated response surface plots, respectively, as a result of the experimental design, confirming this behavior.

In ambient air, total removal of the ambient concentration of the gas is achieved. At the second and third concentration levels studied, absorption with seawater showed the best results, achieving the lowest final concentration and also the fastest. Clarke and Radojevic, [15] demonstrated that the increase in the SO₂ removal rate with seawater is due to the catalytic effect of the Cl⁻ ion. This may be related to the speed at which SO₂ removal occurs compared to with the other methods.

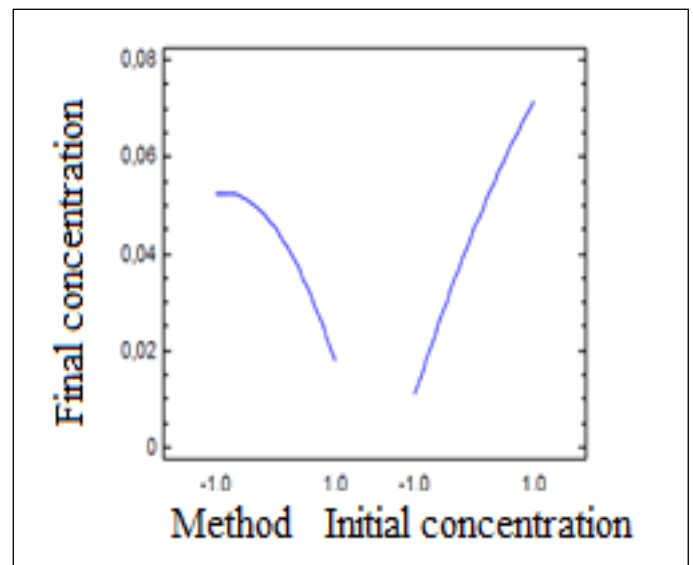


Figure 3: Main effect plot. Source: Authors, (2024).

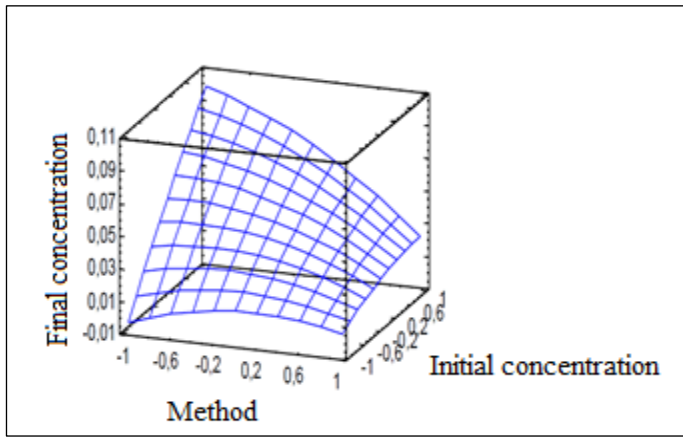


Figure 4: Estimated response surface plot.
Source: Authors, (2024).

Table 6 shows the results of the ANOVA, which tests the statistical significance of each effect by comparing its mean square to an estimate of the experimental error.

In this case, three effects have a P-value less than 0.05; indicating that they are significantly different from zero at the 95% confidence level. This observation is supported by the Pareto plot (Figure 5). This graph shows that the removal method, the initial SO₂ concentration and the interaction between these two factors correspond to the three effects that have a significant impact on the response variable, since they exceed the standardized effect line.

Tabla 6: Variance Analysis.

Source	Square sum	GI	Medium Square	Reason - F	Value-P
A: Method	0,005270	1	0,005270	8,97	0,0074
B: Initial concentration	0,016562	1	0,016562	28,19	0,0000
AA	0,000613	1	0,000613	1,04	0,3197
AB	0,005376	1	0,005376	9,15	0,0070
BB	0,000101	1	0,000101	0,17	0,6825
blocks	0,000374	2	0,000187	0,32	0,7309
Total error	0,011162		19		0,000587
Total (corr.)			0,039460		26

Source: Authors, (2024).

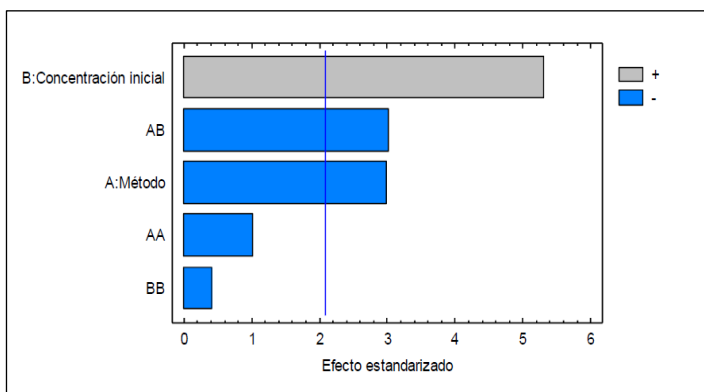


Figura 5: standardized Pareto diagram.
Source: Authors, (2024).

In addition, the R-squared statistic indicates that the model, as fitted, explains 71.71 % of the variability in the final SO₂ concentration. The standard error of the estimate indicates that the standard deviation of the residuals is 0.0242. The mean absolute error of 0.0143 is the average value of the residuals.

The estimated coefficients of the model for each parameter are shown in Table 7.

Table 7: Model Estimated coefficients.

Coefficient	Estimate
π	0,045
A: Method	-0,02
B: Inicial Concentration	0,03
AB	-0,02

Source: Authors, (2024).

Finally, equation 4.7 shows the fitted model.

$$c_f = 0,045 - 0,02 \cdot M + 0,03 \cdot c_i - 0,02 \cdot M \cdot c_i \quad (9)$$

Where:

cf: is the final SO₂ concentration, expressed in ppm.

M: is the removal method

ci: is the initial SO₂ concentration, expressed in ppm.

IV.6. ALTERNATIVE'S ANALYSIS.

The adsorption capacity of activated carbon makes it an efficient alternative for SO₂ removal. Fortunately, its graphitic structure makes it very stable under a wide range of conditions. It resists high temperatures and sudden temperature changes, is unaffected by acids, alkalis and many different solvents, although it reacts with strong oxidizing agents. Based on these properties, there are methods that allow its reactivation or regeneration, which can eliminate adsorbates of different types and substances deposited on the carbon surface. The use of activated carbon in Cuba is basically limited by its high cost in specialized markets. In addition, its high operating costs limit its use in developing countries.

The high reactivity of Ca(OH)₂ also makes it an efficient alternative for the desulphurization of gaseous effluents. However, the use of this reagent leads to an important problem, since the oxidation of the SO₃²⁻ ion precipitates SO₄²⁻ ions, which are difficult to remove. This process results in a lower acidity of the medium, which favors the development of algae in gas bioremediation processes. However, the amount of solid waste generated as a result is excessive. In addition, the use of this reagent requires a greater economic investment.

The use of seawater as an absorbent in desulphurization systems proved to be a simple, efficient and economical method. Due to the proximity of the "Carlos Manuel de Céspedes" power plant to the bay, the use of this novel technology represents an additional advantage, since it does not require the purchase and transportation of raw materials. In addition, it does not generate solid waste to be deposited for subsequent treatment. The liquid effluent is mainly seawater, which can be returned to its source without any environmental impact.

Based on the results of this research, a multidisciplinary team will decide on the most feasible alternative for use on an industrial scale. It is important to point out that the methods evaluated will be carried out as primary treatment of gaseous effluents. Subsequently, it is proposed to carry out a secondary treatment based on bioremediation with macroalgae *Ulva lactuca* type, which would improve the removal efficiency of SO₂ gas, as well as other combustion gases generated by the "Carlos Manuel de Céspedes" power plant in the city of Cienfuegos.

It also reinforces the importance of the low-cost SO₂ sensor, which was an essential tool in the evaluation of scrubbing

methods. This research has shown that in addition to its use in air quality monitoring, it can be used in this type of research.

V. CONCLUSIONS

The removal of SO₂ using an adsorption column with activated carbon proved to be an efficient alternative at the concentration levels evaluated. The removal of the different SO₂ concentrations using the adsorption column with calcium hydroxide showed a higher removal efficiency than the adsorption with activated carbon. The pH, salinity and alkalinity values determined in a seawater sample from the Bay of Cienfuegos were 8.17±0.003; 26.50±0.10 ups and 171.67±1.03 mg/L, which confirms the adequacy for SO₂ gas removal. The removal of SO₂ using an absorption column with seawater proved to be the most efficient alternative among the methods evaluated, at the three concentration levels, since the lowest final concentrations were obtained in the shortest time. The experiments performed demonstrated that low-cost calibrated sensors can be used as a scientific tool to evaluate gaseous effluent removal methods.

VI. AUTHOR'S CONTRIBUTION

Conceptualization: Liset Roche Delgado, Mayra C. Morales Pérez, Rosa Amalia González Rivero, Arianna Álvarez Cruz.

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Discussion of results: Liset Roche Delgado, Mayra C. Morales Pérez, Rosa Amalia González Rivero, Arianna Álvarez Cruz.

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Writing – Review and Editing: Liset Roche Delgado, Mayra C. Morales Pérez, Rosa Amalia González Rivero, Arianna Álvarez Cruz, Agustín A. García Rodríguez.

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