

Removal of hazardous gaseous pollutants from industrial flue gases by a novel multi-stage fluidized bed desulfurizer

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ABSTRACT

Sulfur dioxide and other sulfur compounds are generated as primary pollutants from the major industries such as sulfuric acid plants, copper smelters, catalytic cracking units, etc. and cause acid rain. To remove the SO₂ from waste flue gas a three-stage counter-current multi-stage fluidized bed adsorber was developed as desulfurization equipment and operated in continuous bubbling fluidization regime for the two-phase system. This paper represents the desulfurization of gas mixtures by chemical sorption of sulfur dioxide on porous granular calcium oxide particles in the reactor at ambient temperature. The advantages of the multi-stage fluidized bed reactor are of high mass transfer and high gas–solid residence time that can enhance the removal of acid gas at low temperature by dry method. Experiments were carried out in the bubbling fluidization regime supported by visual observation. The effects of the operating parameters such as sorbent (lime) flow rate, superficial gas velocity, and the weir height on SO₂ removal efficiency in the multistage fluidized bed are reported. The results have indicated that the removal efficiency of the sulfur dioxide was found to be 65% at high solid flow rate (2.0 kg/h) corresponding to lower gas velocity (0.265 m/s), weir height of 70 mm and SO₂ concentration of 500 ppm at room temperature.

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1. Introduction

Gaseous pollutants like sulfur dioxide that are once emitted into the atmosphere have no boundaries and become uncontrollable and cause acid deposition. Several toxicological/epidemiological investigations during the last few decades have shown that the effect of this gas is severe. Consequently, in recent years, a number of studies have been undertaken for developing a better understanding of the effects of sulfur dioxide on humans, vegetations, animals and materials. The effects of this gas have motivated the public and private sector industries, government agencies and environmental engineers to work out reliable background information, enforceable regulations and viable control options for sulfur dioxide emission. Furthermore, considering the importance and seriousness of the problem, increasing attention is also being paid to develop processes and equipment for the removal of sulfur dioxide, which can effectively meet the demands of technology and increasingly stringent environmental laws and regulations.

Coal fired power stations, sulfuric acid plants account for more than 50% of sulfur dioxide emissions. This acid gas leads to acid rain and fog, which harm the natural environment and human

life. Being a toxic gas with a strong odor, it can be fatal in cases of acute exposure [1] and its effects on human body are manifested mainly as respiratory odors [2]. Therefore large amount of money are being spent to reduce such emissions and very numerous desulfurization projects have been undertaken worldwide since the 1970s. The technologies for flue gas desulfurization (FGD) can be grossly classified into three different types [3–4]: wet scrubbers, semi-dry processes (such as spray drying and sorbent injection), and dry processes (including regenerable techniques and combined SO_x/NO_x removal systems). Wet scrubbers have been shown to be reliable for a variety of fuels, and have been widely used in industry for achieving SO_x removal [2]. However, this kind of technology generates a large amount of solid waste and requires treatment of wastewater. It also involves a complicated configuration and costly operation [3].

Spray dryers, which are representative of semi-dry FGD processes, have been extensively tested in America and Europe since 1970s [4–8]. Compared to wet scrubbers, this method does not require reheating energy and waste water treatment, thus allowing lower cost of operation [9–11]. However, for achieving an SO_x removal of greater than 95%, the ratio of sorbent to SO_x must be approximately 2.0, which is much larger than that in wet scrubbers [4,12]. To reduce the capital cost of FGD, the so-called sorbent injection processes, including furnace sorbent injection and duct sorbent injection, have been developed. Under a reaction tempera-

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ture of 333 K and a sorbent usually $\text{Ca}(\text{OH})_2$ to SO_2 ratio of 2.5, the overall SO_2 removal in both ducts furnace and bag filters can reach only 70–90% [4,13–14]. This system has been widely employed due to their high desulfurization efficiency, but they are more limited based on stoichiometry.

The desulfurization efficiency and the sorbent utilization of the dry FGD processes so far tested have not reached the level of wet and semi dry scrubbers. The major reason is that the residence time of the SO_x sorbent is very short causing very low desulfurization efficiency in all the processes [15–16]. Accordingly, development of low-cost and high efficiency dry scrubbing technologies is still significant and urgent in both academic circles and industry [4]. Dry scrubbing systems are attractive in principle as compared to wet scrubbers in terms of cost because they do not require water and reheating energy [17–18]. A substantial work on removal of SO_2 in a fluidized bed reactor using sand particles ranging from 500 μm to 590 μm and hydrated lime of 9.1 μm have been reported in the literature [19]. The maximum removal efficiency (97.7%) of sulfur dioxide was achieved in a single stage fluidized bed reactor at a temperature of 700 °C, a Ca/S ratio of 3 and a velocity of 0.8 m/s [19]. At the temperatures at which the process is very efficient (700–850 °C), the reaction produces calcium sulfate, especially in the presence of excessive amounts of oxygen [20]. The efficiency is very less at low temperature making the dry scrubbing ineffective for SO_x removal.

In 1973, Geldart classified powders depending on their fluidisability using air at the ambient temperature. Group B particles are those that have an average particle size exceeding about 100 μm . Many of the gas–solid reactions are operated industrially with different size group of particles. Group A particles are smaller and/or lighter than group B particles. Most manufactured catalysts are in this category, with particle size ranging from about 10 to 130 μm . Group D particles are large, of the order of more than 1000 μm in average particle size. In a fluidized bed, they behave like the group B particles. Due to the high gas velocities required to fluidize group D particles, it is often more economical to process them in spouted beds when lower gas rates are required [21]. Pisani and de Moraes [22] used limestone (24 μm) in a binary fluidized bed reactor with sand particles in the range of 500–900 μm . They operated under bubbling regime under all operating conditions studied. They calculated average reagent residence time in the range of 4–14 min which one of the important findings.

Therefore, it was considered to use a multistage counter-current fluidized bed reactor as a desulfurization apparatus. One of the special features of this type of reactor is to enhance the performance of the reactor and at the same time it reduces the effect of gas bypassing, narrows the residence time distribution of solids and thus approaches plug flow. The aim of the study is to analyze the operation of a three-stage fluidized bed reactor for sorption of SO_2 gas on lime particles for a wide range of operating conditions at room temperature in the fluidization regime.

2. Experimental

2.1. Preparation of adsorbent

The adsorbent used here lime (CaO), was obtained in lumpy form from commercially available chemical raw materials. To avoid being deliquesced, they were stored in desiccators after crushing and sieving to the investigated size. Table 1 lists the physical characteristics of adsorbents used in the present study. The physical

Table 1
Physical characteristics of lime.

Adsorbent	Characteristic	Value
Lime (CaO)	Average particle diameter (μm)	426
	Density (kg/m^3)	2040
	Minimum fluidization velocity at 30 °C (m/s)	0.112
	Specific area of unreacted sorbent (m^2/g)	15

Table 2
Average chemical composition of lime used.

Compound	Weight (%)
CaO	25.0
$\text{Ca}(\text{OH})_2$	64.4
CaCO_3	4.76
MgO	3.22
Impurities	2.62

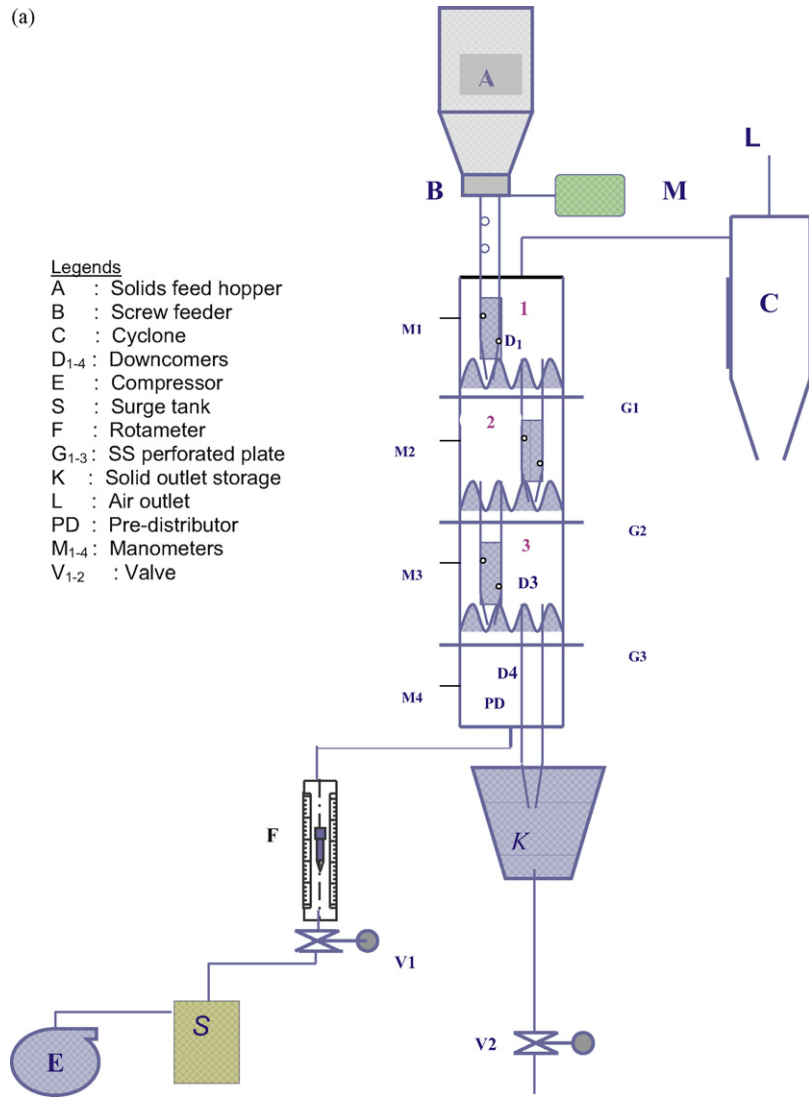
characteristics such as Brunauer–Emmett–Teller (BET) specific surface area was measured with a Micromeritics surface area analyzer that utilized the BET low-temperature N_2 adsorption technique. The chemical properties of the calcium sorbent measured by atomic absorption spectrometer (AAS), thermogravimetric analyzer (TGA) are shown in Table 2.

2.2. Experimental apparatus and procedure

Fig. 1 is the schematic of the multi-stage fluidized bed reactor developed and used in this study. The configuration of this staged gas–solid fluidized bed reactor is similar to that of the sieve trays distillation column. The reactor consisted of a three-stage fluidization column having provision of solid feeding from the top and air supplying system from the bottom along with other auxiliary equipments used for experimentation. Each stage of the column was made up of perspex cylinder of 0.10 m internal diameter and 0.305 m long. The stainless steel plates (G_1 , G_2 and G_3) were used as internal baffles sandwiched between the flanges of two stages and each plate was drilled with perforations having 8.56% total grid openings. The grid plates were fixed with fine wire mesh (100 mesh size) to prevent weeping of the solid particles through the openings. Each section was provided with a downcomer of perspex cylinder of 0.025 m internal diameter (D_1 , D_2 , D_3 and D_4) and the downcomers were fitted to the gas distributor by special threading arrangement having the provision for adjusting the weir height as desired. The downcomers were further fitted with a cone at the exit end in order to reduce the up flow of the gas through the downcomer and consequently, widening the stable operating range. Pressure tapings were provided just below the grid plate and the near the air out let and was provided with weir mesh filter to prevent any solid particle from the entering the tapings. Four manometers were provided to measure the pressure drop at every stage as well as the total pressure drop.

In order to generate synthetic air– SO_2 mixture, in composition similar to that of the exhaust of sulfuric acid plants, copper smelters was made by mixing compressed air from an air compressor and SO_2 gas from an SO_2 cylinder. Provision was made to feed the air– SO_2 mixture at the base of the fluidized bed reactor. The air– SO_2 mixture was generated by mixing air and SO_2 in an air-jet ejector assembly. Compressed air from the compressor was used as the motive fluid in the ejector to aspirate and thoroughly mix air with SO_2 from the SO_2 gas cylinder. The ejector was mounted

Fig. 1. (a) Schematic diagram of the experimental set-up of a three-stage counter-current fluidized bed reactor. (b) Photographic view of the experimental set-up of a three-stage counter-current fluidized bed reactor.



with a downward slope of 30° with an air nozzle perfectly aligned along the axis of the ejector throat to ensure an axially symmetrical jet. The air nozzle was fixed at a projection ratio (which is the ratio of the distance between the nozzle tip and the beginning of the parallel throat to the throat diameter), of 3.78, which was determined experimentally for obtaining the highest possible mass ratio of aspirated gas. Compressed air at the desired pressure and flow rate was forced through the air nozzle and regulated by a valve. Simultaneously the SO₂ was routed at a controlled rate through SO₂ gas regulator and into the ejector. The air–SO₂ gas mixed intensely in the mixing throat of the ejector and the mixture was fed into the gas chamber fitted at the bottom of the column. Pre-calibrated rotameter (F) were used to measure the gas flow rate.

A conical hopper was attached at the bottom of column for storage of solids coming out from the bottom stage through the downcomer. The gas leaving the column from the top stage was passed through a standard air cyclone (C) fabricated in the laboratory with diameter of 100 mm and then into the exhaust system.

The solids from the screw feeder were fed to the first stage downcomer of the reactor. Necessary precautions were made to ensure that no air from outside intruded into the column during operation.

Experiments have been conducted by setting the gas flow rate of $208.3 \times 10^{-5} \text{ m}^3/\text{s}$ and $291.6 \times 10^{-5} \text{ m}^3/\text{s}$. Corresponding to gas flow rate of $208.3 \times 10^{-5} \text{ m}^3/\text{s}$ and $291.6 \times 10^{-5} \text{ m}^3/\text{s}$, solid flow rate of 2 kg/h and 4 kg/h have been used. The weir height of the downcomer was kept at 30 mm and 70 mm and the gap between the downcomer bottom and the grid plate were kept 15 mm and 30 mm respectively. For each gas flow rate, the inlet SO₂ loadings were varied from 500 ppm to 1500 ppm in three stages. The study was carried out at room temperature and a pressure of 1 atm absolute. Percentage removal of SO₂ has been calculated for each experimental run by the formula.

$$\eta_{\text{SO}_2} = \frac{\text{SO}_{2\text{Inlet}} - \text{SO}_{2\text{Outlet}}}{\text{SO}_{2\text{Inlet}}} \times 100$$

Likewise for stage *i*,

$$\eta_{\text{SO}_2} = \frac{C_{i+1} - C_i}{C_{i+1}}$$

where *C_i* and *C_{i+1}* are outlet and inlet sulfur dioxide concentrations in gas.

2.3. Sampling and analysis

Prior to sampling of the gas, the reactor was operated for quite some time till all the stages of the reactor were identical in their operation and the pressure drops across each stage was almost equal indicating steady and stable operation of reactor. Samples were made at iso-kinetic conditions. Samples at the inlet of the column and outlet of each stage were drawn at flow rate of 1.0 LPM. Under steady state operating conditions the SO₂ gas samples were collected at each point with the help of midjet impinges and aspirator bottles. The gas samples were analyzed for sulfur dioxide by the "Tetrachloro-Mercurate Method" [IS: 5182 (Part-VI)]. The method consists of passing a portion of the gas sample, through a solution of absorbing medium (sodium tetrachloromercurate) and analyzing the resulting solution spectrophotometrically by UV–visible recording spectrophotometer.

Absorbing solution of 0.1 M sodium tetrachloromercurate was prepared by dissolving 27.2 g (0.1 mole) mercuric chloride and 11.7 g (0.2 mole) sodium chloride in 1.0 l of distilled water. This solution could be stored at room temperature for several months. The indicator, *p*-rosaline hydrochloride of 0.04% (w/v), acid bleached was prepared by dissolving 0.20 g of *p*-rosaline hydrochloride in 100 ml of distilled water and filtered the solution after 48 h. This solution

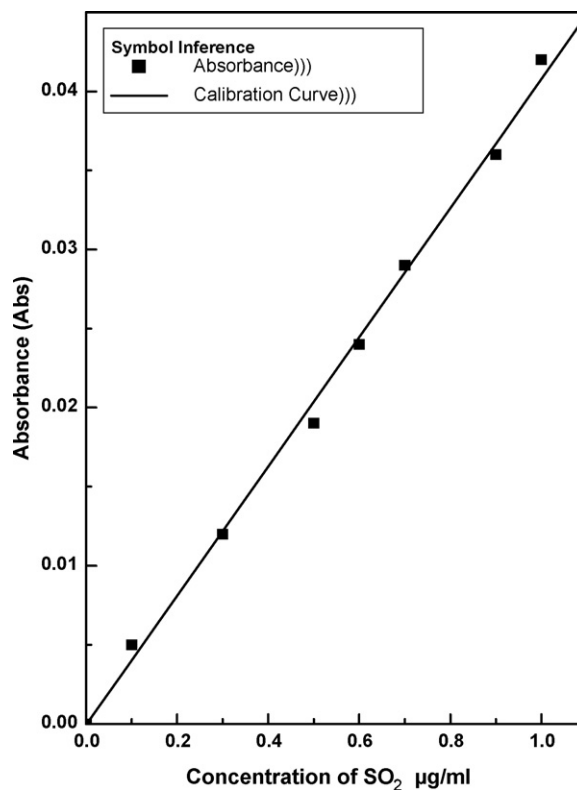


Fig. 2. Calibration curve of UV spectrophotometer for SO₂ concentration.

was stable for at least three months when stored in the dark and kept cool. The *p*-rosaline was required to have an assay of 95% or better and an absorbance maximum at 560 nm. 20 ml of this solution was pipetted into a 100 ml volumetric flask to which was added 6 ml of concentrated hydrochloric acid. This solution should be pale yellow with a greenish tint. It could be stored at room temperature in an amber bottle for a week or for about two weeks if refrigerated.

When SO₂ from the air stream was absorbed in a sodium tetrachloromercurate solution it formed a stable dichlorosulfito-mercurate complex. The amount of SO₂ was then estimated by the color produced when *p*-rosaline hydrochloride was added to the solution. The color was estimated by using a spectrophotometer for which a calibration curve was already prepared (Fig. 2). The amount of SO₂ in air sample was obtained from the differences in spectrophotometric values of the blank and test samples and reported in ppm using the calibration curve. The measurement has been reported to the nearest 0.005 ppm at concentrations below 0.15 ppm and to the nearest 0.01 ppm for concentrations above 0.15 ppm. Ozone and nitrogen dioxide interfere if present in air samples at concentrations greater than SO₂. Interference of nitrogen dioxide was eliminated by including 0.06% sulfamic acid in the absorbing reagent. This may, however, result in a different calibration curve of lower sensitivity and in greater losses of SO₂ on shortage of the sample for more than 48 h after *o*-toluidine subsequent to sample collection. Heavy metals, especially iron salts, interfere by oxidizing dichlorosulfito-mercurate during sample collection. This interference was eliminated by including ethylenediaminetetraacetic acid in the absorbing reagent. Sulfuric acid or sulfate do not interfere. If large amounts of solids materials are present, a filter may be used advantageously upstream; however a loss of SO₂ may occur. In the adsorption experiments, detailed studies are going on to determine the effect of gas and solids flow rates, inlet loading of sulfur dioxide on the percentage removal of sulfur dioxide using lime as the scrubbing medium. The

range of concentration for SO₂ monitoring was 500–1500 ppm and the accuracy was ±10 ppm.

3. Results and discussion

The SO₂ scrubbing in the multi-stage fluidized bed by lime adsorbent is conducted to study the effect of solid flow rate, inlet concentration of SO₂, weir height and gas velocity on the removal efficiency of SO₂. A stage wise analysis has also been performed to find out the best suitable operating parameters.

3.1. Effect of gas flow rate at different SO₂ loading on the percentage removal of SO₂

The percentage removal efficiency of SO₂ (η_{SO_2}) at different superficial gas velocity for a weir height of 70 mm and solid rate of 4 kg/h have been plotted in Fig. 3. At different inlet SO₂ concentration. It has been observed that with increase in SO₂ concentration the removal efficiency decreases rapidly from 55% to 38%. This may be due to the fact that as the SO₂ concentration increases, the adsorbed SO₂ molecules on the adsorbent (lime) forms a monolayer of products. This gives the additional resistances to penetrate gas molecules inside the solid particle. Thus as concentration of SO₂ increases, the efficiency of removal decreases. Also it may be seen that a lower gas velocity at a particular solid velocity had higher sulfur dioxide removal efficiency of 65% at 70 mm weir height and 62% at 30 mm weir height. The increases in efficiency for higher weir height may be due to the fact that as the height of weir is more the residence time of the solid particles in the fluidized bed is more and get more contact time to absorb the SO₂ on lime. This resulted in higher adsorption and increases in efficiency of the system under all other operating conditions

constant. As observed in Fig. 3, increasing inlet sulfur dioxide decrease the removal efficiency which may be due to an increase in sulfite concentration thus resulting in decrease of the sorbent activity.

It is interesting to note that as the gas flow rate increases for both weir height of 70 mm and 30 mm, the percentage removal of sulfur dioxide decreases. This is quite obvious as with increases in gas velocity with in the bed particle population decreases, solid hold-up decreases [24] as a result decreasing the removal efficiency. It can be seen that the removal efficiency decreases from 50% to 35% at a SO₂ concentration of 1500 ppm.

3.2. Effect of solid flow rate at different SO₂ loading on the percentage removal of SO₂

Effect of solid flow rate on percentage removal of SO₂ at various inlet concentration of SO₂ have been shown in Fig. 4. It can be seen from this figure that the percentage removal efficiency of SO₂ (η_{SO_2}) at constant weir heights of 70 mm and 30 mm significantly affected by the solid flow rate. For 70 mm weir height the efficiency of removal ranges between 65% and 53% for 4 kg/h of solid flow rate. However for 2 kg/h of solid flow rate the efficiency of removal ranges from 55% to 38%. This indicates higher solid flow rate augment the removal process. Similar trend has been observed for weir height of 30 mm, where efficiency of removal ranges between 62% and 48% for 4 kg/h of solid flow rate and 49% to 32% for 2 kg/h solid flow rate. It may be seen that a higher solid flow rate (4 kg/h) at a particular gas velocity had higher sulfur dioxide removal efficiency then lower solid flow rate (2 kg/h) due to higher solid holdup. As observed in Fig. 4, increasing inlet sulfur dioxide decrease the removal efficiency due to decrease of the sorbent activity.

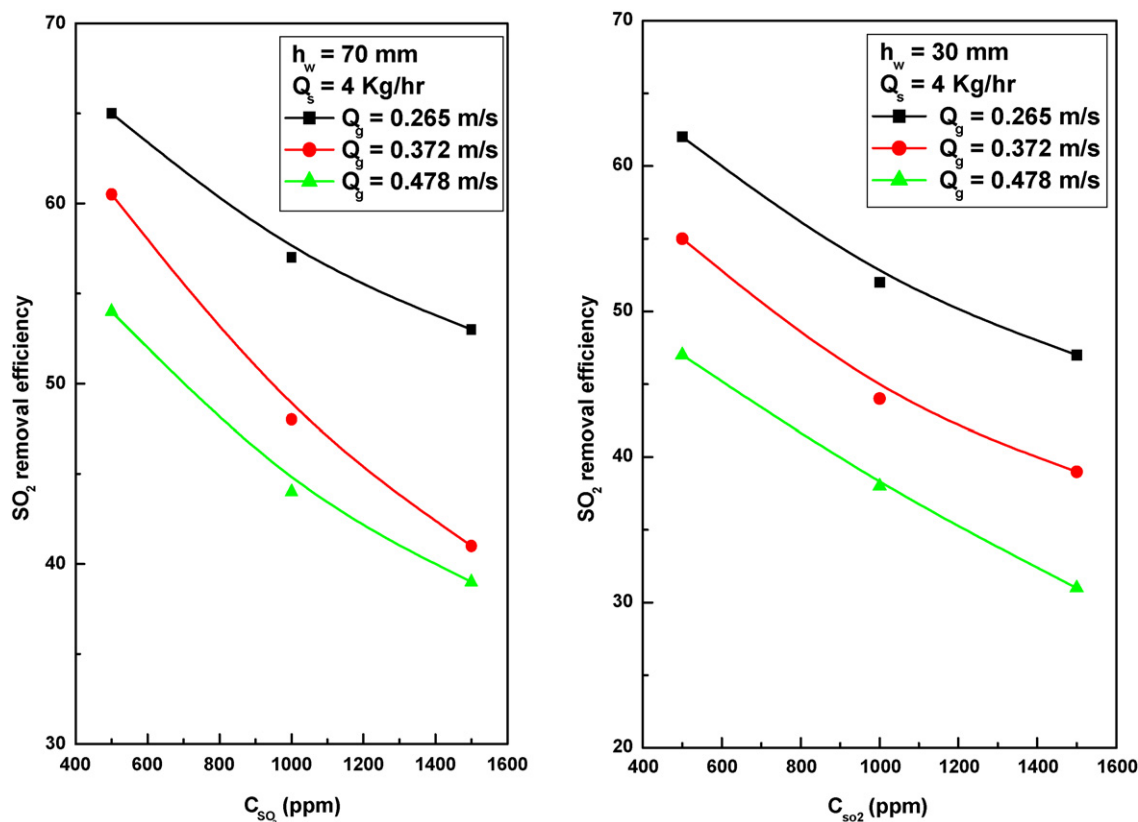


Fig. 3. Effect of gas flow rate on SO₂ removal efficiency of (a) 70 mm weir height, (b) 30 mm weir height.

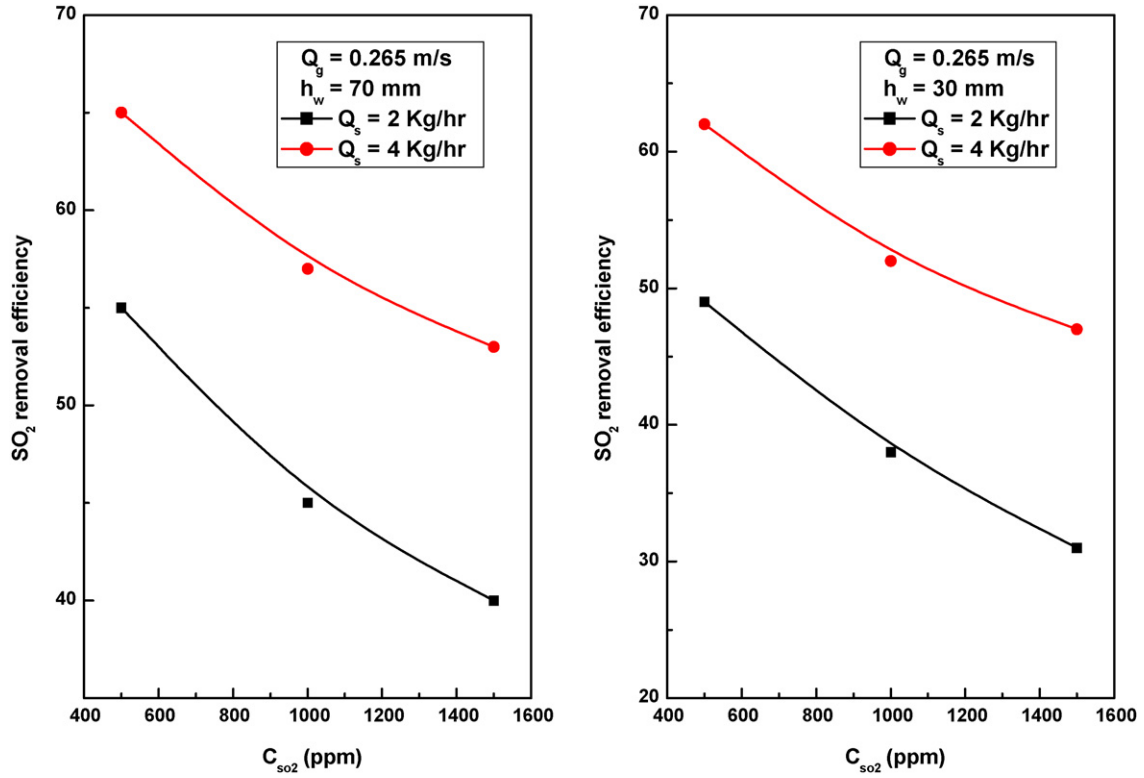


Fig. 4. Effect of solids flow rate on SO₂ removal efficiency (a) 70 mm weir height, (b) 30 mm weir height.

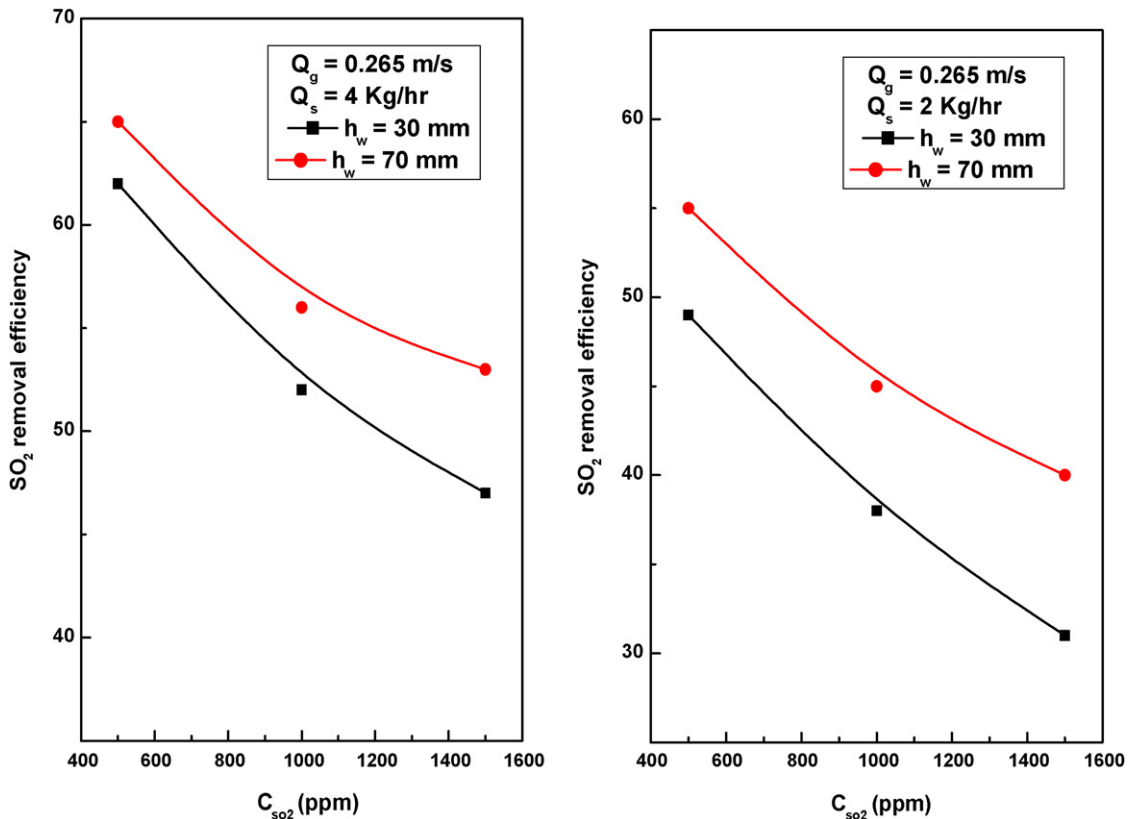


Fig. 5. Effect of weir height on SO₂ removal efficiency (a) 4 kg/h solid flow rate, (b) 2 kg/h solid flow rate.

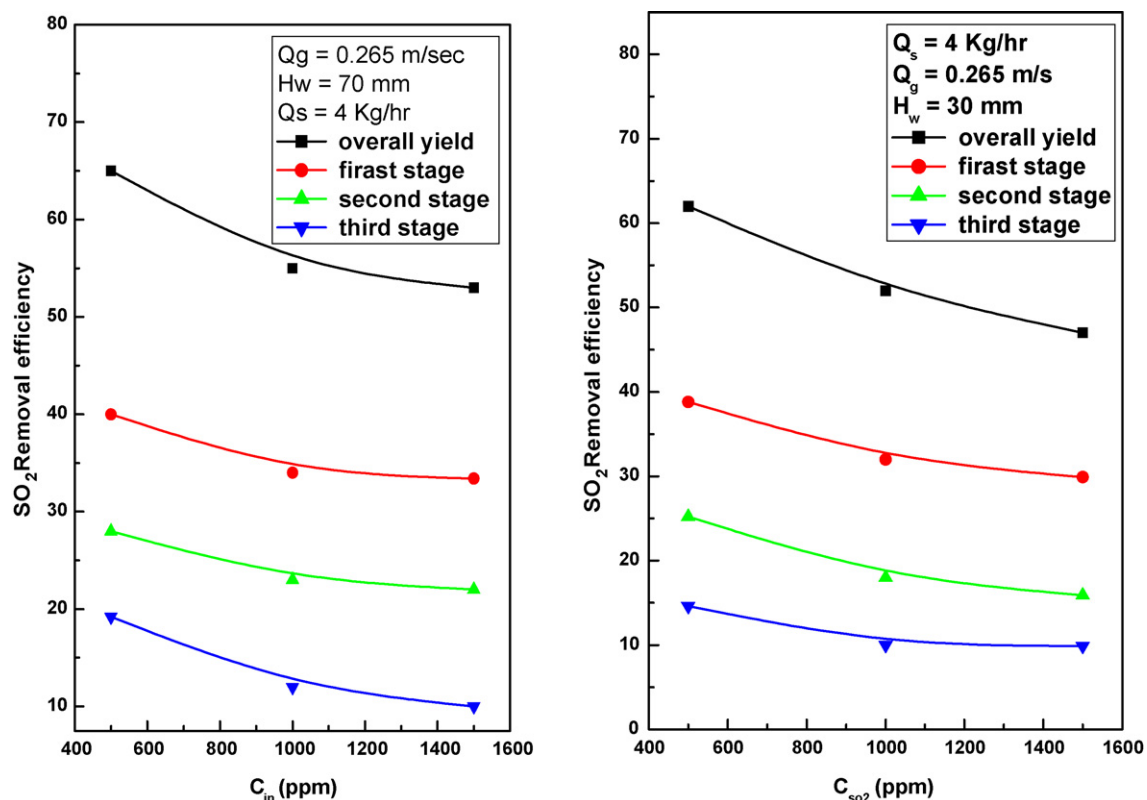


Fig. 6. Stage wise removal efficiency of SO₂ (a) 70 mm weir height, (b) 30 mm weir height.

3.3. Effect of weir height at different SO₂ loading on the percentage removal of SO₂

The percentage removal efficiency of SO₂ (η_{SO_2}) at different inlet SO₂ loading and for a constant weir height have been plotted against gas flow rates in Fig. 5. It can be seen from these figures that the percentage removal of SO₂ at the higher weir height was maximum as the solid reactant available was maximum. The increases in weir height from 30 mm to 70 mm increases the efficiency nearly 8% at a concentration of 1500 ppm. However the increases in removal efficiency at lower concentration is less may around 4% for higher solid flow rate i. e. 4 kg/h. At lower solid flow rate the difference in removal efficiency is much more than at higher solid rate. This is due to the fact that the solid rate to the fluidized bed has strong affect on SO₂ removal efficiency.

3.4. Effect of temperature on the percentage removal of SO₂

The experiment was carried out at the ambient temperature ranging from 30 °C to 40 °C. No discernible difference in the sulfur dioxide removal efficiency was observed due to change in temperature from 30 °C to 40 °C. This was similar to that observed by Ho and Shih [23] in a fixed bed.

3.5. Stage wise percentage removal of SO₂

Fig. 6 indicates the percentage removal efficiency of SO₂ at different stages at a particular solid flow rate and gas flow rate. It may be seen that at the first stage, the solid reactant was fresh for which maximum percentage of the inlet sulfur dioxide was absorbed and reacted at the surface of the calcium sorbent. As a result, the removal efficiency was higher than other two stages. At the second stage, a low porosity product layer (CaSO₃·1/2H₂O)

formed gradually at the surface of the sorbent with a conversion of 12% which was similar to that observed by Ho and Shih [23] in a fixed bed. Since this low porosity product is formed, the diffusion resistance of SO₂ from the emulsion phase to the inner un-reacted calcium sorbent gradually might have increased. Thus, the removal efficiency kept decreasing. At the third stage, the removal efficiency of SO₂ was quite low. Considering the flooding aspect, the variables are suitably chosen to operate in various stages of fluidized bed. The solids were fed into the column at the rate from 0.035 kg/m² s to 0.142 kg/m² s with gas flow rate varying from 0.32 kg/m² s to 0.59 kg/m² s. The weir height of the downcomer was kept at 30 mm and 70 mm and the gap between the downcomer bottom and the grid plate were kept 15 mm, 25 mm and 35 mm respectively. The pressure drops across each stage and across the entire column have been recorded. In the experiment, for a given apparatus conditions, the effect of gas flow rate and solid flow rate on pressure drop and solids hold-up have been observed. The details of pressure drop characteristics of the multi-stage fluidized bed reactor in each stage have been extensively studied and discussed elsewhere by Mohanty et al. [25].

4. Conclusions

The removal of gaseous reactive pollutants depends on type of reactor, operating conditions and chemical nature of adsorbent. In this study a multistage fluidized bed reactor have been designed and fabricated which was employed for sorption of sulfur dioxide on the CaO to remove SO₂. Experiments were carried out under various operating conditions It was found that the effect of the superficial gas, solid velocity and weir height had a significant effect on the removal efficiency of sulfur dioxide. However, the attrition was negligible under above stable operating conditions. The bed was operated in bubbling regime of fluidization.

The maximum removal efficiency of 65% has been observed at high solid flow rate (2.0 kg/h) corresponding to lower gas velocity (0.265 m/s), wier height of 70 mm and SO₂ concentration of 500 ppm at room temperature. Furthermore, as the superficial gas velocity was increased, the sulfur dioxide removal efficiency and calcium conversion decreased. Thus a multistage fluidized bed reactor can replace the other air pollution control devices such as wet scrubber and semi-dry system due to their simplicity and higher residence time. However the detail studies at higher temperature (80–120 °C) and scale up of this type of fluidized bed reactor requires further study.

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