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## Synthesize and Characterization of Rice Husk Silica to Remove the Hydrogen Sulfide through the Physical Filtration System

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### ABSTRACT

The main goal of this study was to synthesis and characterization of silica prepared from rice husk and its application to remove Hydrogen sulfide. Polyvinyl chloride filters with 50 cm in height and 7.5 cm in diameter have been used in this study. Rice husk silica (RHS) and regenerated rice husk silica (RRHS) were used as packing material in this work. System was studied at a constant empty bed residence time (EBRT) of 60 sec and different inlet concentration of H<sub>2</sub>S from 15 to 450 mg m<sup>-3</sup>. Brunauer-Emmett-Teller method showed rice husk silica has a very high surface area (226.3 m<sup>2</sup> g<sup>-1</sup>) with median pore radius of 2.4 nm and mesoporous structure. The chemical composition analysis showed that the silica consisted of SiO<sub>2</sub> up to 97.35%. The removal efficiency (RE) more than 98% were observed for both packed filters at a loading rate of 0.98 g H<sub>2</sub>S m<sup>-3</sup> h<sup>-1</sup> and 15 mg m<sup>-3</sup> inlet concentrations of H<sub>2</sub>S. The maximum removal capacity of both packed filter were obtained 7.02 m<sup>-3</sup> h<sup>-1</sup> at a loading rate of 9.0 m<sup>-3</sup> h<sup>-1</sup> and 150 mg m<sup>-3</sup> inlet concentration. The result from the analysis indicates that at a 1.0 L min<sup>-1</sup> flow rate and different inlet concentration of H<sub>2</sub>S there is no significant difference between the RHS packed filter and the RRHS packed filter in the amount of removal efficiency and removal capacity (p<0.01). Also the result showed pressure drop was undetectable amount (zero) in both packed filter. The results of this study show the best performance of RHS and RRHS packed filters in low concentration of H<sub>2</sub>S.

**Key words:** Air pollution, regenerated rice husk silica, removal efficiency, removal capacity, empty bed residence time, scanning electron microscope

### INTRODUCTION

Air pollution has become a worrisome over the decades. The effects of air pollution on human health and the environment have received an increasing amount of consideration from industrialization (Seyyednjad *et al.*, 2011; Moussavi *et al.*, 2007). One of the main concerns in the some industries such as petroleum and natural gas industry is to achieve the best efficient operation of gas treatment (Chavez and Guadarrama, 2006).

In recent years, the influence of even low concentrations of air pollutants on human health has re-emerged as an important scientific issue. They also have reported that there were several studies that have shown various acute and chronic health impacts to air pollution such as due from

hydrogen sulfide (Ballesta *et al.*, 2008). Hydrogen sulfide is a major air pollutant when it is emitted to atmosphere because  $H_2S$  is not only a malodorous but also a corrosive gas and a source of acidic rains (Stepova *et al.*, 2009). Waste gases containing reduced sulfur compounds such as Hydrogen Sulfide ( $H_2S$ ), dimethyl sulfide ( $Me_2S_2$ ) and methyl mercaptan ( $MeSH$ ) have an unpleasant odor even at extremely low concentration (Hartikainen *et al.*, 2000). The malodor caused by  $H_2S$  has been an environmental and public nuisance in recent years. These nuisances happen mostly from the odors of emanating from food processing industry, common effluent treatment systems, night soil and solid waste processing plants.  $H_2S$  can poison several different systems in the body, although the nervous system is most affected. Exposure to lower concentrations can result in eye irritation, a sore throat and cough, shortness of breath and fluid in the lungs. Long-term, low-level exposure may result in fatigue, loss of appetite, headaches, irritability, poor memory and dizziness. At 150-250 ppm, the olfactory nerve is paralyzed after a few inhalations and the sense of smell disappears. Concentrations over 1000 ppm cause immediate collapse with loss of breathing (Shareefdeen *et al.*, 2010). In actual situations such as industrial emissions, the strong odor threshold and vapor density makes the bothersome compounds travel several kilometers from the emission site (Jeong *et al.*, 2008). As information about the hazardous effects of contaminants increases, industry is facing new regulations for contaminated air discharged from different activities. Those regulations are more and more restrictive and legal limits on emissions are continuously being lowered. The release of malodorous chemicals is a public health concern but it is also related to personal comfort (Barona *et al.*, 2004).

There are different methods to remove these compounds. Some of the most important ways of removing them are by using the surface adsorption through activated carbon, burning, catalytic oxidation, wet scrubbers and oxidation by heating. However, these methods are often expensive and produce other pollutants as well. That is why  $H_2S$  removal in industries is restricted (Massoudinejad *et al.*, 2008). Rice husk is a major agricultural by product obtained from the production of rice. For every four tons of rice produced, one ton of waste rice husk is generated. Rice husk has a chemical composition that typically corresponds to the following: cellulose (40-45%), lignin (25-30%), ash (15-30%) and moisture (8-15%) (Kutty, 2001). The expensiveness of adsorption/catalyst technologies prompts a growing research interest into the production of low-cost alternatives to adsorbents from a wide range of waste precursors (Ros *et al.*, 2006). Cost for producing the adsorbent from rice husk is only about one percent of the cost to prepare commercial activated carbon (Balalai *et al.*, 2000).

The main objective of this study was to investigate the removal efficiency of  $H_2S$  by using rice husk silica. There were two specific objectives in the study whereby to characterize rice husk silica incinerated at  $800^\circ C$  in order to determine of  $H_2S$  removal efficiency (%) and to determine of  $H_2S$  elimination capacity ( $g\ m^{-3}\ h^{-1}$ ) by using rice husk silica in a physical filtration system.

## **MATERIALS AND METHODS**

**Preparation of packing material:** This study was a lab-scale study that has been carried out in the laboratory of Environmental Studies (Environmental Science Department) at Universiti Putra Malaysia in 2010.

Rice Husk Silica (RHS) and Regenerated Rice Husk Silica (RRHS) were used as packing material in physical filter for the removal of  $H_2S$ . Rice husk silica was prepared according to Jamwal and Mantri 2007 method. The rice husk was first washed with tap water to remove the dirt and other contaminants present in them and then was dried in the oven at  $110^\circ C$  for about 24 h. The

washed and dried rice husk was then subjected to acid leaching. This process was done by reflux in 3% (v/v) hydrochloridric acid (HCl) and 10% (v/v) sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) for 2 h, at a ratio of 50 g husk L<sup>-1</sup>. Then, the husk was thoroughly washed with distilled water and then dried in an air oven at 100°C for four h. Finally, the cleaned husk was burned inside a muffle furnace at temperature of 800°C. Incineration was done in a porcelain crucible for four h in static air. In order to investigation of reuse capability of Regenerated Rice Husk Silica (RRHS) to removal of hydrogen sulfide, saturated rice husk silica was burned (incinerated) inside a muffle furnace at 800°C. Incineration was done in a porcelain crucible for 4 h in static air.

**Experimental setup and operation:** A laboratory scale physical filter used in this study was shown in Fig. 1. This filter was constructed using PVC cylinder with 50 cm in height (packed with height 22 cm) and 7.5 cm in diameter. Hydrogen sulfide gas cylinder with 4000 ppm and 150 bars inside pressure was used. The malodorous gas (hydrogen sulfide) passed through the packing materials down flow. An air pump has been used to dilution of gas for produce different concentration of H<sub>2</sub>S in mixing chamber. System operated at a constant empty bed residence time (EBRT) of 60 second and different inlet concentration of hydrogen sulfide. H<sub>2</sub>S concentration at the inlet was started from 15 mg m<sup>-3</sup> and was increased in weekly increments from 15 mg m<sup>-3</sup> to a final concentration of 450 mg m<sup>-3</sup> (15, 75, 150 and 450 mg m<sup>-3</sup>).

**Data analysis:** Inlet and outlet hydrogen sulfide concentrations were measured by using hydrogen sulfide detector model ppb RAE 3000, U.S.A. Removal Efficiency (RE), Elimination Capacity (EC) of hydrogen sulfide and pressure drop (mm H<sub>2</sub>O) has been used as performance of filter as stated below:

$$RE = \frac{C_{Gi} - C_{Go}}{V_f} \times 100$$

$$EC = \frac{C_{Gi} - C_{Go}}{V_f} \times Q$$

where, Q is the gas flow rate (m<sup>3</sup> h<sup>-1</sup>), V<sub>f</sub> is the volume of the filter bed (m<sup>3</sup>), C<sub>Gi</sub> and C<sub>Go</sub> are the inlet and outlet hydrogen sulfide concentration (ppm; g m<sup>-3</sup>) (Taghipour *et al.*, 2006; Kun, 2005; Moosavi *et al.*, 2005; Oyarzun *et al.*, 2003).

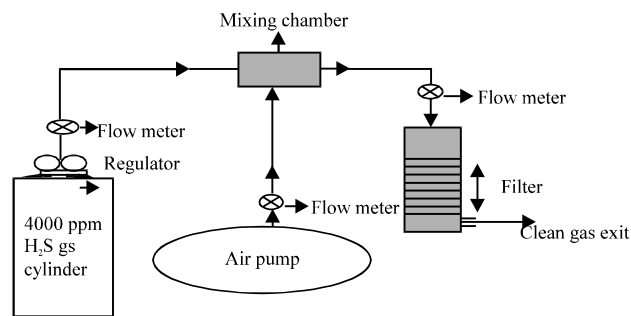


Fig. 1: A schematic flow diagram of the pilot

Brunauer-Emmett-Teller (BET) specific surface area was performed by using a ThermoFinnigan Sorptomatic apparatus using nitrogen adsorption at  $-196^{\circ}\text{C}$ . The surface morphology of the silica was observed under a scanning electron microscope (SEM), using LEO operated at accelerating voltage of 15 kV (Ros *et al.*, 2006).

Independent-samples T-test were conducted to compare Removal Efficiency (RE) and elimination capacity (EC) of hydrogen sulfide in Rice Husk Silica (RHS) and Regenerated Rice Husk Silica (RRHS) packed filter. Preliminary analyses (exploratory data analysis) were performed to ensure no violation of the assumptions of normality.

## RESULTS

The BET value (surface area) for the rice husk silica prepared at  $800^{\circ}\text{C}$  was obtained  $226.3\text{ m}^2\text{ g}^{-1}$ . The result for the chemical composition analysis showed high percentage of  $\text{SiO}_2$  (97.35%). Some of the important physical and chemical characteristics of rice husk silica which was prepared at  $800^{\circ}\text{C}$  such as density, surface area, median pore radius, cumulative pore volume, elemental analysis and chemical compositions are listed in Table 1.

Table 1: Some of the important physical and chemical characteristics of rice husk silica

Characteristics	Unit	Value
<b>Chemical properties</b>		
Analysis of elements		
C	%	$0.05\pm 0.01$
H	%	$0.27\pm 0.01$
N	%	$0.36\pm 0.05$
S	%	$0.03\pm 0.01$
Chemical composition		
$\text{SiO}_2$	%	97.35
$\text{SO}_3$	%	1.66
$\text{K}_2\text{O}$	%	0.43
<b>Physical properties</b>		
Density	$\text{g L}^{-1}$	52
Surface area	$\text{m}^2\text{ g}^{-1}$	226.3
Median pore radius	nm	2.3747
Cumulative pore volume	$\text{cm}^3\text{ g}^{-1}$	0.3078

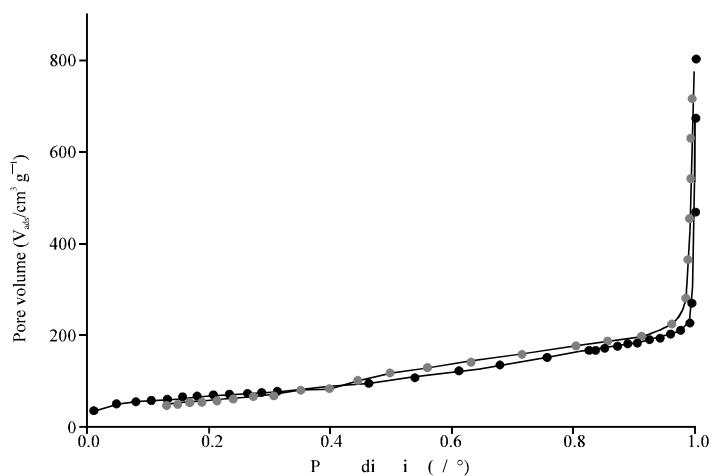


Fig. 2:  $\text{N}_2$  adsorption-desorption isotherms of mesoporous rice husk silica (pore radius size versus pore volume)

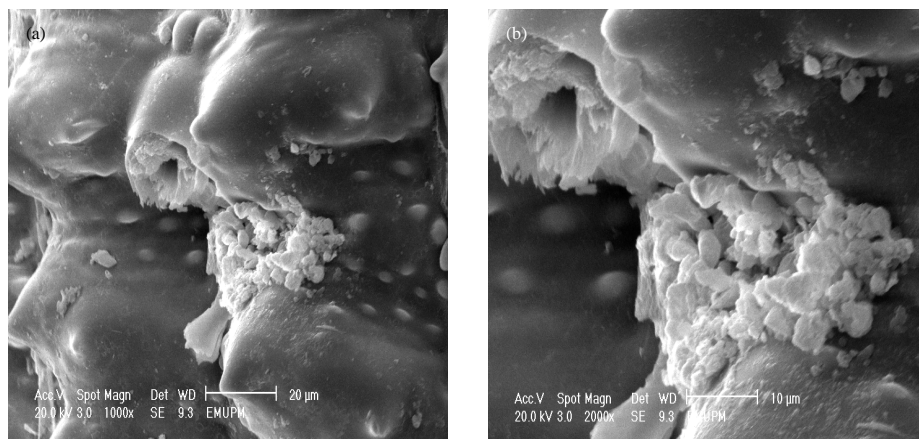


Fig. 3: Scanning Electron Microscope (SEM) Images of Rice Husk Silica Incinerated at 800°C with magnification of (a) 1000 and (b) 2000, respectively.

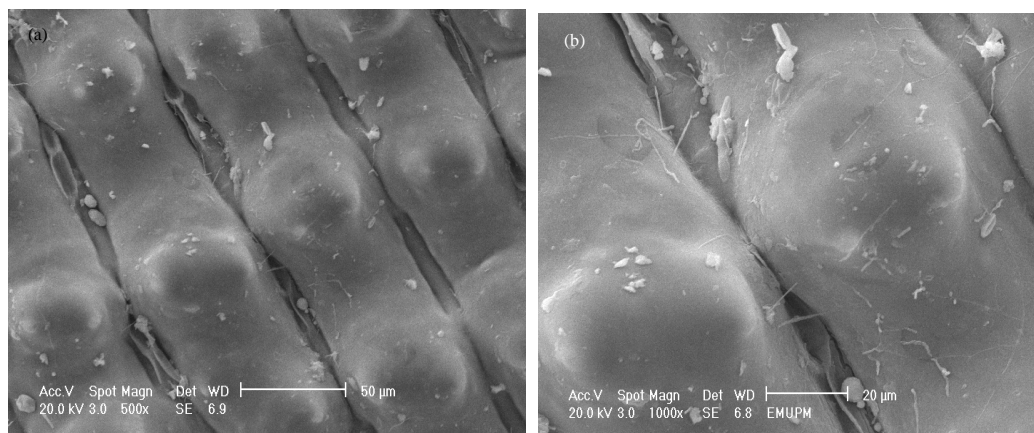


Fig. 4: Scanning Electron Microscope (SEM) Images of Raw Rice Husk with Magnification of (c) 500 and (d) 1000, respectively

Moreover, the silica showed to be mesoporous with a median pore radius of 2.4 nm. Figure 2 shows the pore radius size (nm) versus pore volume ( $\text{cm}^3 \text{g}^{-1}$ ).

Scanning electron microscope (SEM) morphology of rice husk silica prepared at 800°C with magnification of (a) 1000 and (b) 2000, are shown in Fig. 3. On the other hand, the SEM image of raw rice husk with magnification of (c) 500 and (d) 1000 are shown in Fig. 4 in order to compare of the silica shape and pores with raw rice husk.

Removal efficiency (%), removal capacity ( $\text{g m}^{-3} \text{h}^{-1}$ ) and pressure drop ( $\text{mm H}_2\text{O}$ ) were used to investigate the performance of the filter. Figure 5 shows the removal efficiency versus operating time and different inlet concentration of  $\text{H}_2\text{S}$  in the physical filter packed with rice husk silica and regenerated rice husk silica prepared at 800°C. At a loading rate of  $0.98 \text{ g H}_2\text{S m}^{-3} \text{ h}^{-1}$  and  $15 \text{ mg m}^{-3}$  inlet concentrations of  $\text{H}_2\text{S}$ , greater than 98 and 99% of removal efficiency was observed in the filter packed with RHS and RRHS, respectively. This result showed both RHS and RRHS have high removal efficiency at low concentration.

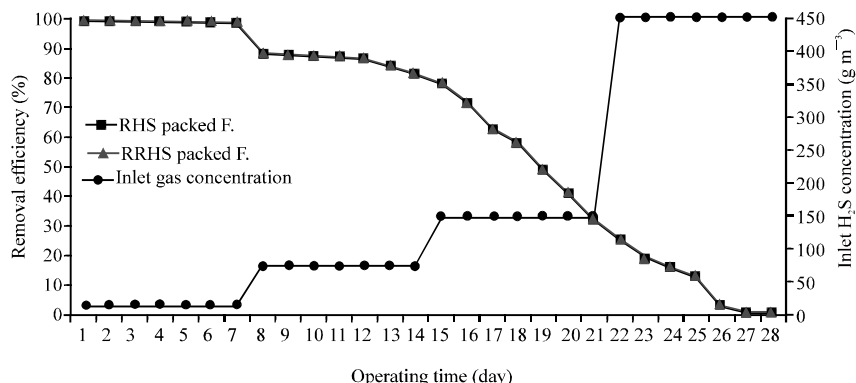


Fig. 5: Removal efficiency Vs operating time and different inlet concentration of H<sub>2</sub>S in the filter packed with Rice Husk Silica (RHS) and Regenerated Rice Husk Silica (RRHS)

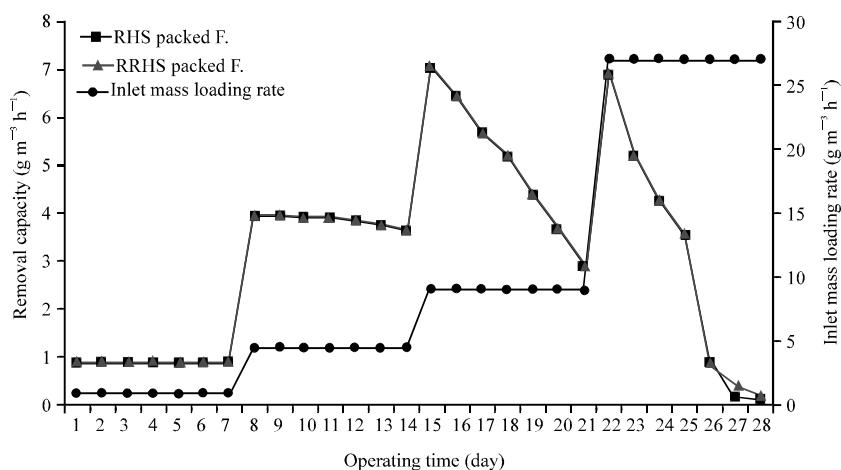


Fig. 6: Removal capacities Vs operating time and different inlet concentration of H<sub>2</sub>S in the filter packed with Rice Husk Silica (RHS) and Regenerated Rice Husk Silica (RRHS).

The maximum removal capacity obtained in the two packed filters with RHS and RRHS was  $7.02 \text{ g m}^{-3} \text{ h}^{-1}$  at a loading rate of  $9.0 \text{ g m}^{-3} \text{ h}^{-1}$  and  $150 \text{ mg m}^{-3}$  inlet concentrations of H<sub>2</sub>S. The result showed regenerated rice husk silica had the same removal capacity as rice husk silica (Fig. 6).

Figure 7 shows the result of pressure drop versus operating time and different inlet concentration of hydrogen sulfide. As Roshani *et al.* (2005) explain that any increase in pressure drop will add to the operating cost of the filter as odorous air therefore it must be supplied to a greater pressure in order to achieve the same flow rate. In this study, pressure drop was undetectable amount (zero) versus different operating times and different inlet mass loading rates. The amount of pressure drop was one of the important factors to judge about filter's performance to removal of gas pollutants.

There was not a significant difference in the removal efficiency of hydrogen sulfide (with  $15 \text{ mg m}^{-3}$  inlet concentration) for RHS packed filter ( $M = 98.30$ ,  $SD = 0.08$ ) and RRHS packed filter ( $M = 98.67$ ,  $SD = 0.41$ );  $t(6.47) = 2.36$ ,  $p = 0.53$ . Moreover, there was not a significant difference in the removal efficiency of hydrogen sulfide (with  $75 \text{ mg m}^{-3}$  inlet concentration)

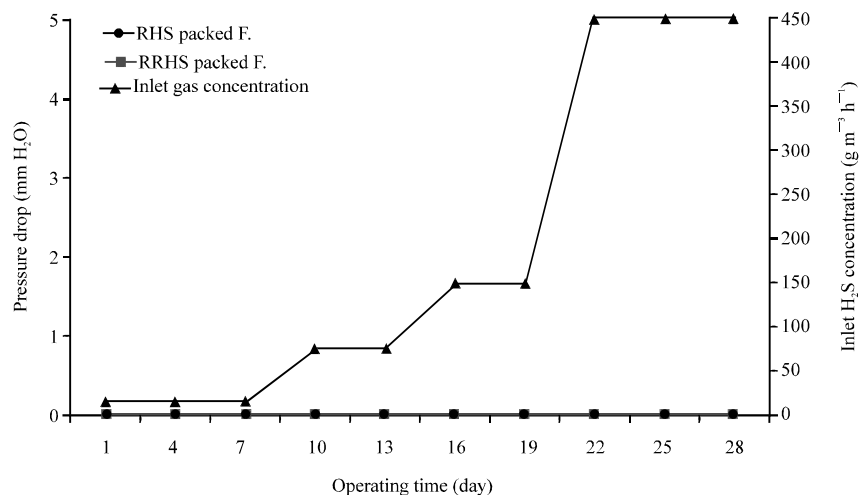


Fig. 7: Pressure drop vs operating time and different inlet concentration of H<sub>2</sub>S in the filter packed with Rice Husk Silica (RHS) and Regenerated Rice Husk Silica (RRHS)

for RHS packed filter ( $M = 85.53$ ,  $SD = 2.45$ ) and RRHS packed filter ( $M = 85.42$ ,  $SD = 2.55$ );  $t(12) = 0.08$ ,  $p = 0.93$ . Also, There was not a significant difference in the elimination capacity of hydrogen sulfide (with  $15 \text{ mg m}^{-3}$  inlet concentration) for RHS packed filter ( $M = 0.882$ ,  $SD = 0.010$ ) and RRHS packed filter ( $M = 0.881$ ,  $SD = 0.007$ );  $t(12) = -0.289$ ,  $p = 0.778$ . Moreover, there was not a significant difference in the elimination capacity of hydrogen sulfide (with  $75 \text{ mg m}^{-3}$  inlet concentration) for RHS packed filter ( $M = 3.84$ ,  $SD = 0.10$ ) and RRHS packed filter ( $M = 3.84$ ,  $SD = 0.11$ );  $t(12) = 0.001$ ,  $p = 1.000$ ).

Chou *et al.* (1986) studied the removal of hydrogen sulfide from biogas by silica gel. They reported that hydrogen sulfide could be selectively and completely removed from the biogas until the silica gel became saturated. They used commercial silica gel as packing material in an adsorbed tower.

## DISCUSSION

This study showed that silica prepared from rice husk at  $800^{\circ}\text{C}$  temperatures after acid leaching has a mesoporous structure with high surface area ( $226.3 \text{ m}^2 \text{ g}^{-1}$ ) and median pore radius of  $2.3747 \text{ nm}$ . Moreover the chemical composition of the silica showed a high percentage of  $\text{SiO}_2$  (97.35%). This finding is in agreement with Mittal (1997) report which described "rice husk ash is one of the most silica rich raw materials containing about 90-98% silica (after complete combustion)". Application of rice husk silica to removal of hydrogen sulfide showed high removal efficiency for low concentration (greater than 98% with  $15 \text{ mg m}^{-3}$  and greater than 85% with  $75 \text{ mg m}^{-3}$  of hydrogen sulfide inlet concentration). The removal efficiency reduced gradually duration of operating time and different inlet concentration of hydrogen sulfide. As a result, elimination capacity reduced to 0.43% after 28 days of operating time and  $450 \text{ mg m}^{-3}$  of hydrogen sulfide inlet concentration. Regenerated rice husk silica (RRHS) showed greater than 99.0% removal efficiency at  $15 \text{ mg m}^{-3}$  of hydrogen sulfide inlet concentration. The results found in RRHS packed filter are the same as RHS packed filter, where the removal efficiency reduced at the high inlet concentration.



The advantages of the application of rice husk silica include: I) by using the rice husk silica, there will be no pressure drop over the time unlike in the biological filters. In this study, after 28 days of operating time, as well as different inlet concentrations of hydrogen sulfide (from 15 to 450 mg m<sup>-3</sup>), the pressure drop did not increase in the RHS and RRHS packed filter (the pressure drop was 0.0 mm H<sub>2</sub>O during the operating time). McNevin and Barford (2000) reported an increasing pressure drop from less than 500 to greater than 2500 Pa after 3 months of continuous operating time. Meanwhile, the maximum pressure drop of 18 mm H<sub>2</sub>O was reported in the study by Roshani *et al.* (2005) who evaluated the performance of biofiltration in the removal of H<sub>2</sub>S from gas stream. The amount of the pressure drop is one of the important factors to determine the filter's performance in the removal of gas pollutants. For this, Roshani *et al.* (2005) explained that "any increase in pressure drop adds to the operating cost of the biofilter as odorous air must be supplied at a greater pressure to achieve the same flow rate"; II) there will be no acclimatization time incurred as in the biological filters. Cho *et al.* (2000) mentioned that "in general, when a biofilter was inoculated with a mixed culture such as activated sludge, the biofilter reaches equilibrium after 6 days to 2 weeks". Meanwhile, Sercu *et al.* (2005) reported an acclimatization period of 13 days to reach the maximum removal efficiency of hydrogen sulphide. In addition, Rehman *et al.* (2009) reported an acclimatization period of than 10 days for the removal of hydrogen sulfide in the biofilter; III) after the saturation process, rice husk silica can be regenerated by incineration.

## CONCLUSION

Ramirez *et al.* (2009) described the concentrations of hydrogen sulfide in gas emissions are usually very dilute and traditional physical-chemical technologies such as incineration, adsorption or chemical scrubbing tend to be costly. Based on the findings of the present study, rice husk silica could be used as a packing material to removal of H<sub>2</sub>S at low concentration of malodorous stream.

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