Materials Research Proceedings 29 (2023) 109-116 https://doi.org/10.21741/9781644902516-14

# Adsorption of hydrogen sulphide ($H_2S$) using xerogel synthesized from palm kernel shell biochar

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**Keywords:** Palm Kernel Shell Biochar, Xerogel, Hydrogen sulphide, Adsorption

**Abstract:** Xerogel is a typical porous material with a large internal surface area, causing them to have significant gas adsorption. Adsorption performance was investigated to determine the potential contribution of xerogel to removing Hydrogen Sulphide ($H_2S$) in this research. Adsorption is a well-known energy-efficient approach for removing acid gases at low temperatures. $H_2S$ gas harms human health, such as headaches, eye irritation, and loss of smell if exposed to a low concentration. Furthermore, the physical and chemical properties of the raw material and synthesized xerogel were evaluated by various techniques: Fourier Transform Infrared Spectroscopy (FTIR), thermogravimetric Analysis (TGA), and Scan Electron Microscopy (SEM). Results showed that the removal of $H_2S$ increased with increasing adsorbent mass from 3 to 12 g and decreased flow rate from 40 to 26 L/h. The maximum Adsorption capacity of Xerogel for H2S was 27.5 mg/g, and the surface area was 0.2686 $m^2$/g. This research shows the significant potential of using adsorbent materials obtained from waste to absorb $H_2S$.

## Introduction

Various technologies have been established to decrease and restrict $H_2S$ emission, including wet flue gas desulfurization, Adsorption, catalytic reduction [1], and electron beam technology [2]. Adsorption is the most practical option among these technologies because it is cost-effective, efficient, and simple to handle and run. The adsorbents can be recycled for numerous uses by doing the Adsorption, making them cost-effective. There are several examples of solid adsorbent materials that can be adsorbents: zeolites, calcium oxide, activated carbon, metal oxides, hydrogels, xerogels, polymers, and metal-organic frameworks (MOFs) [4]. These adsorbents can be produced from coal, agricultural residues, and other waste biomass. The correct choice of adsorbent materials is a critical step in constructing adsorption systems since the decision significantly impacts the process' economics and performance [5]. The abundance of palm kernel shells obtained by cracking oil palm nuts in Malaysia demonstrates their use as activated carbon precursors. Furthermore, the amount of solid residue from oil palms will cause the problem of waste disposal at the oil palm factory [6, 7, 8].

Presently, the production of porous materials (xerogel) has attracted worldwide attention in the oven-drying process. The oven drying method is much more straightforward compared to other

methods. Xerogel is microporous and can be defined as a low-density biomaterial employed for various promising applications in different fields [9]. Xerogel is produced from gels by replacing a liquid phase, commonly solvent crystals, with a gaseous phase with no ruin of the created porous solid architecture [10, 11, 12]. The significance of Xerogel-based PKS is that it will become an effective and low-cost material widely used globally as an absorbent, not only effective on toxic gases but also in absorbing engine oil and heavy metal removal in wastewater. This research focuses on the synthesis of a new, economical, and versatile product that can serve to adsorb the H2S as an absorbent.

## Methodology

*Material*

This research used palm kernel shell biochar as raw material, $H_2S$ gas, Sodium Alginate, Calcium Carbonate, distilled water, and Glucono Delta Lactone (GDL). Then, the adsorption process used a multi-layered adsorption column.

*Preparation of Xerogel Based on Palm Kernel Shell Biochar*

A magnetic stirrer was used to stir 0.5g of Sodium Alginate into 50 ml of distilled water until the mixture was homogenized. 0.1g CaCO3 is applied to the homogeneous mixture and mixed until homogeneous, followed by 0.5g grind PKS biochar. Then, 0.15g of GDL was applied to the mixture and mixed until homogenized before transferring to the square mould and put in the refrigerator until it was fully gelled. Following that, the hydrogels were removed from the square mould and placed in a petri dish. Oven drying of gels removes the solvent from the colloidal gel to afford a solid dry gel or xerogel. Many types of xerogel dried by oven can lose 50-70% of the original gel's volume with drying. The hydrogel was dried in an oven at 60 ℃ for 48 hours.

*Characterization of xerogel*

The chemical structure of the PKS biochar and synthesized xerogel were measured using a Bruker FTIR spectrophotometer following the ASTM standard test method (E1252- 98). The practice covers a spectral range of 4000–450 $cm^{-1}$ according to the ASTM E1252-98. The chemical structure includes the functional group of the biochar, and xerogel was analyzed [14].

To estimate the element content such as moisture content, volatile matter content, carbon content and maximum temperature profile of PKS biochar and synthesized xerogel, the thermogravimetric (TG) method was used by using the Mettler Toledo Thermogravimetric Analyzer [13]. Thermogravimetric analysis is done to study the stability of the extracted xerogel following ASTM standard method D5142-02a. The xerogel biochar and palm kernel shell (PKS) biochar samples were heated up to 950°C with a heating rate of 20°C/min in the presence of air involving a nitrogen atmosphere at 100ml/min flow rate of gas. Then, the sample atmosphere is switched into the air and heated to 1200 °C [15].

SEM has been used to determine the morphologies of the organisms. The sample has been coated with a sheet before being scanned with electron beams with acceleration forces ranging from 10-15 kV to reach 1,000 – 5,000 times resolution. The technique was accompanied by a standard test defined in detail by the American Society for Testing and Content Specification, ASTM E766 – 14 [15].

*Adsorption Test*

The adsorption test was carried out in a multilayer's adsorption column, as shown in Figure 1. The column was made of stainless steel as the hazardous gas was used. The fixed parameters used are 25 ppm inlet concentration of $H_2S$. Then, the adsorption test was started with the different flowrate of $H_2S$ and masses of the adsorbent xerogel to achieve the best Adsorption parameter. The system was run at a temperature of 30°C. Finally, the outlet concentration of $H_2S$ was monitored

continuously using a biogas analyzer (MRU OPTIMA) [20]. The results of the adsorption test were interpreted by analyzing breakthrough time curves and the adsorption capacity, qt (mg/g) of hydrogen sulphide, which was calculated by using Eq. (1) [13]:

$$q_t = \frac{(C_O - C_t)\mathrm{V}}{W} \quad (1)$$

where: qt is adsorption capacity (mg/g), $C_0$ is the initial concentration of $H_2S$ supplied (mg/L), Ce is the concentration of $H_2S$ at any time, V is the volume of the adsorbent (L), and W is the mass of the adsorbent used (g).

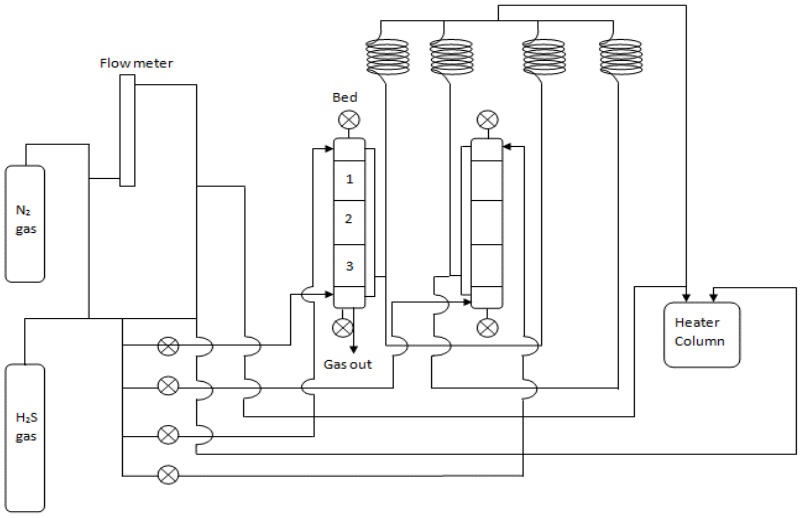


*Figure 1: Schematic diagram of Multilayers Adsorption Column.*

## Results and discussion

*Characterization of Raw Material and Xerogel*

Figure 2 shows the FTIR spectrum of (a) palm kernel shell (PKS) biochar and (b) xerogel biochar for comparison. Broad characteristic peaks demonstrate the presence of O-H stretching at 3356.82 $cm^{-1}$ for PKS biochar and 3333.76 $cm^{-1}$ for xerogel biochar in the spectra and show hydrogen-bonded hydroxyl group (O-H) in which identical results were reported by [16]. Ma et al. (2015) [17] reported that the C-H stretching vibration with a wavelength of 2942.51 $cm^{-1}$ was observed for PKS biochar and 2917.64-2887.73 $cm^{-1}$ for xerogel, showing the alkane and aldehyde group. Functional group C=C stretching was observed on 1637 $cm^{-1}$ for PKS biochar, and 1616.98 $cm^{-1}$ indicated the presence of an aromatics compound. Aromatic ester is found at bending vibration 1314.05 $cm^{-1}$ in xerogel [18]. The bending vibration around 1466 $cm^{-1}$ represents the existence of the biochar C-C and C-H (alkane) groups. A peak at 1100 cm-1- 1029 $cm^{-1}$ shows the presence of the S= O group in xerogel. Figure 6 illustrates the PKS and xerogel FTIR analysis [17].

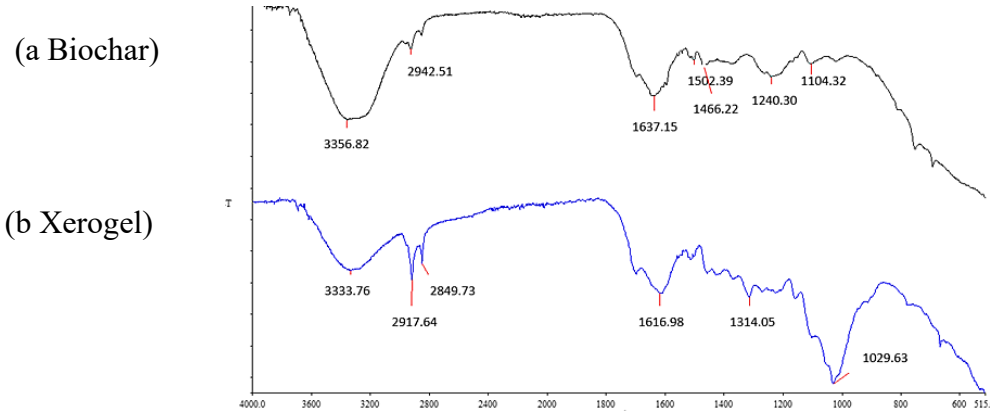


*Figure 2: PKS and xerogel FTIR analysis.*

Figure 3 illustrates PKS and xerogel TGA analysis. The TGA curve was divided into three sections to evaluate the sample's thermal stability. The first stage is the mass loss of 10.9% (2.2 mg) up to 144.55°C due to moisture removal from the PKS biochar sample. Meanwhile, mass loss of 7.03% (1.4 mg) was found in the first step of the TGA curve of xerogel biochar at around 146.64°. This mass loss is mainly attributed to the physical Adsorption of water in the xerogel at the first step of the TGA curve [18]. The mass loss at the second step is 52.1% (10.42 mg). A significant mass loss in the xerogel sample is higher than PKS biochar in step 2 due to the chemically bound water from the weight reduction that was attributed to chemically bonded water from the sol-gel manufacturing technique [17].

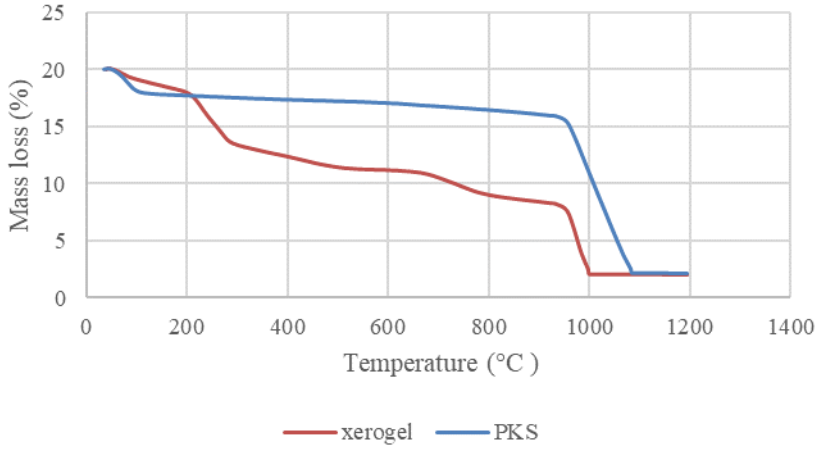


*Figure 3: TGA analysis*

After switching the atmosphere to oxidizing atmosphere combustion of air at 950°C, a remarkable mass loss of 68.5% (13.7 mg) is shown at 938.16°C, and a mass loss of 30.96% (6.2 mg) is shown at between 950-1000°C for PKS biochar and xerogel biochar, respectively. The final peak at 950ºC represents carbon thermal breakdown, and the remaining curve represents ash content [18].

The SEM analysis of PKS biochar and xerogel showed in Figure 4. The surface structure of the xerogel and palm kernel shell (PKS) biochar samples is very irregular and fibrous, with limited regular structuring. In contrast to the surface of PKS biochar, the surface of xerogel had a highly complex network and a rough surface.

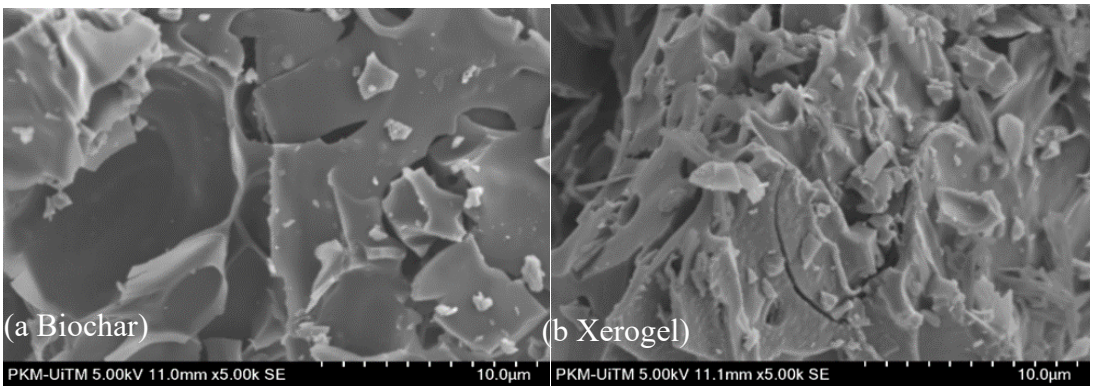


*Figure 4: SEM Analysis (a) biochar, (b) xerogel*

Based on xerogel SEM, the presence of hydrogel has clogged the pores and created more unstable holes due to polymerization. As an outcome of this situation, we predicted that the hydrogel's ability to swell in the presence of a hydrophilic liquid would assist in removing impurities trapped in the pores and holes of the material [19].

**Adsorption Test**

*Effect of Mass*

Figure 5 shows the breakthrough curve for the effect of the mass of adsorbent in the removal of $H_2S$. The mass of the adsorbent ranging from 3 g to 12 g, was analyzed for the adsorption capacity test. The test was run at a temperature of 30 °C and a flow rate of 30 L/h.

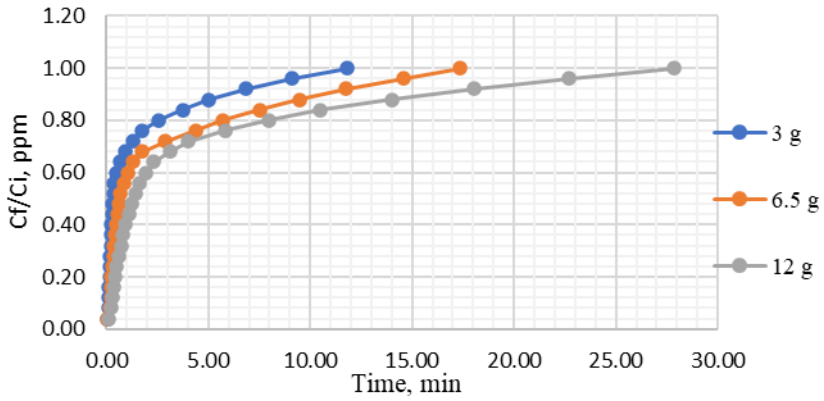


*Figure 5: Breakthrough Curve for Different Mass of Adsorbent*

Based on Figure 2, the 3 g of adsorbents need around 11 minutes for the $H_2S$ fully adsorb. Next, the 6.5 g of adsorbents need 17 minutes to adsorb $H_2S$, which is longer than before. Meanwhile, for the 12 g of adsorbents, it takes the longest time to adsorb H2S, which is 28 minutes fully. The same results were reported by [16] for removing $H_2S$ gas using palm kernel shell-activated carbon.

*Effect of Flowrate*

Figure 6 shows the breakthrough curve for the effect of flow rate in the removal of $H_2S$, which ranges from 26 L/h to 40 L/h. The test was run at a temperature of 30°C and the mass of the adsorbent at 6.5 g.

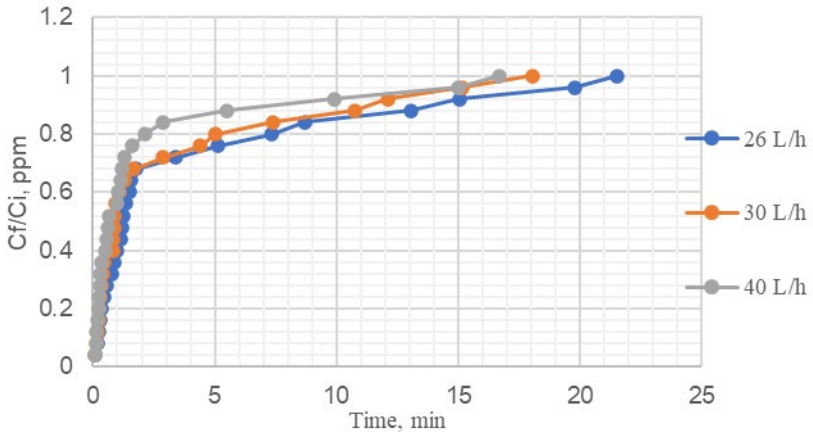


*Figure 6: Breakthrough Curve for Different Flowrate*

Based on the Figure 6, with the flow rate of 26 L/h, it took 22 min for the $H_2S$ fully adsorb, which is the longest time to adsorb $H_2S$. The flow rate of 30 L/h needs 18 minutes, which is shorter than before. However, at the flow rate of 40 L/h, it took the fastest time to adsorb $H_2S$, which was 17 minutes. When the flow rate was increased, the breakthrough curve gradient became steeper, and the breakthrough time became shorter. This phenomenon was explained by [19], that the gas stream had lesser contact time when the flow rate was higher.

## Conclusion

This experiment showed the great capability of the PKS biochar xerogel for removing $H_2S$ using a multilayer adsorption column. The breakthrough time for different parameters such as the mass of adsorbent and $H_2S$ flowrate were investigated. It can be concluded that the lower mass has a shorter breakthrough time. However, the higher flowrate of $H_2S$ gave a shorter breakthrough time. The 3 g of adsorbent and the 40 L/h of $H_2S$ are the parameter for the shorter breakthrough time, but 12 g of adsorbent and 26 L/h flow rate are the optimum parameters for the high adsorption capacity. Based on characterizations, the modified xerogel palm kernel shell biochar can be used as a low-cost adsorbent for adsorption in industries.

## Acknowledgement

The authors would like to thank the Universiti Teknologi MARA (UiTM) for the financial support. The research is conducted at the School of Chemical Engineering, College of Engineering, UiTM, under the support of KEPU grant (600- RMC/KEPU 5/3 (017/2021).

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