Physicochemical Characteristics of Activated Carbon Impregnated with Different Type of Metal Oxide Nanoparticles towards Hydrogen Sulfide Removal

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Abstract. It is highly anticipated to understand the influence of physicochemical characteristics of different types metal oxide nanoparticles impregnated onto palm shell activated carbon (PSAC) adsorbent towards hydrogen sulfide (H\textsubscript{2}S) removal. Alkaline precipitation method was used to synthesis metal oxide nanoparticles (CeO\textsubscript{2}, NiO, CuO, and Fe\textsubscript{2}O\textsubscript{3}). The synthesised metal oxide nanoparticles were then impregnated in the pre-oxidized PSAC adsorbents (Ce/PSAC, Ni/PSAC, Cu/PSAC and Fe/PSAC). The prepared adsorbent was then analysed via Scanning Electron Microscope – Energy Dispersive X-ray (SEM-EDX), Thermogravimetric analysis (TGA), Fourier-transform infrared spectroscopy (FTIR) and H\textsubscript{2}S adsorption analysis. Based on H\textsubscript{2}S adsorption study, different types of metal oxide nanoparticles impregnated on PSAC adsorbent showed different trend of breakthrough time and adsorption capacity. PSAC adsorbent that was modified with metals oxide nanoparticles shows efficient result compared to raw PSAC adsorbent. Among metal oxide nanoparticles, Cu/PSAC adsorbent gives the higher adsorption capacity (86.60 mg H\textsubscript{2}S/g Cu/PSAC) and breakthrough time (80 minutes) compared to the rest of adsorbents.

1. Introduction

Hydrogen Sulphide or commonly known as H\textsubscript{2}S is a colourless, corrosive and highly toxic gas that has offensive rotten egg odour. H\textsubscript{2}S is also a highly flammable gas and also can cause respiratory inhibitor. However, exposed to high concentration H\textsubscript{2}S gas can cause death to a person [1]. It is important to remove H\textsubscript{2}S gas as the gas is highly toxic, which is similar to carbon monoxide in which if contact with the H\textsubscript{2}S gas can cause difficulty in breathing [2]. Based on Department of Environment (DOE) regulation, the limiting value for the H\textsubscript{2}S for chemical and petrochemical industries is 7.5 mg/m\textsuperscript{3} whereas in pulp and paper industries including recycling facilities is 15 mg/m\textsuperscript{3} [3]. Common technologies used for H\textsubscript{2}S removal in industries are: (1) absorption into liquid; (2) adsorption on solid commonly used is activated carbon as adsorbent; (3) biological conversion common process is clause process converting the sulfur compound converted into elemental sulfur by sulphide oxidizing microorganisms with addition of air or oxygen [4]. Among the three technologies, the adsorption on solid has a high adsorption capacity that is recognised to be an energy-efficient technology for H\textsubscript{2}S removal.

Activated carbon (AC) is one of the adsorbents used in the technology in adsorptions on solid. Porous materials with a large surface area are the well-known characteristics that AC had, which are useful for both liquid and gases adsorption. Palm shell is known as endocarp is also been selected to be used as
activated carbon. The PSAC can be applied to the liquid and gas phase in filtration and also in gas adsorption in which to control the pollutant that produced [5]. Several studies related to nanoparticles with other materials such as activated carbon, silica, graphene was used as support [6]. The improvement in adsorption can be achieved by combining the PSAC modified with metal oxide nanoparticles. The unique structure and size of nanoparticle that is as small as 1-100 nm which allow increasing in a surface area [7-8]. AC also has a high surface area, well-developed internal pores structures and high surface reactivity. The addition of metal oxide nanoparticles could enhance the properties of adsorbent.

It is important to synthesis PSAC adsorbent modified with a different type of metal oxides nanoparticles. This is because the different type of element metal oxide nanoparticles provides different characteristics such as in surface area, particle size and adsorbent capacity. Therefore, a study on the physicochemical characteristics of PSAC adsorbent impregnated with different types of metal oxide nanoparticles towards H\textsubscript{2}S removal application is very interesting to be explored.

2. Experimental

2.1. Preparation of Pre-oxidized PSAC

Palm shell activated carbon (PSAC) was used to be modified with metal oxide nanoparticles. The PSAC was sieved into 1 mm of particle size and was dried at 80 °C for 24 hr, to remove excess water. To prepare the pre-oxidized PSAC, it was then immersed in 50% of nitric acid (HNO\textsubscript{3}) for 4 hr. The PSAC was washed with deionised water until the pH closes to neutral. The pre-oxidized PSAC was kept in the oven at 80 °C for 24 hours.

2.2. Synthesis of Metal Oxide Nanoparticles

Nickel nitrate, Ni (NO\textsubscript{3})\textsubscript{2}.6H\textsubscript{2}O (s) (4g, 0.0219 mol) was dissolved in a 20 mL mixture of double distilled water and ethanol (1:1 v/v). The nonionic surfactant, polyvinylpyrrolidone (PVP) (1 g, 0.0001 mol) was dissolved in another 20 mL mixture of water and ethanol (1:1). The resultant mixture was heated to 65°C while stirring. A 5 mL amount of ammonium hydroxide (NH\textsubscript{4}OH) (aq) (6.5 M) was then added drop wise to the above reaction mixture with continued stirring while maintaining the temperature at 65 °C. After the complete addition of the base, the reaction mixture was cooled under room temperature (27 °C), with continuous stirring. The precipitate was centrifuged at 9000 rpm for 15 minutes and as a result, nano Ni (OH) (s) particles was separated. The precipitate was dried in an oven at 50 °C for 24 hr. This procedure was replicated to produce Cu, Fe and Ce nanoparticles [6].

2.3. Impregnation of Metal Oxide Nanoparticles onto PSAC Adsorbent

To synthesis nickel oxide-NPs/PSAC (Ni/PSAC), dried NiOH (1.25 g) was mixed with 25 mL of distilled water and stirred for 30 minutes. The pre-oxidized PSAC (2.5 g) was immersed into the 25 mL solution of NiOH for 1.5 hours while stirring. The solution was filtered, and the precipitate was dried at 80 °C for 24 hr. The NiOH/PSAC was then subjected to calcination at 400 °C with the present of N\textsubscript{2} gas (50 mL/min) for 3 hr. This method was replicated to produce Cu/PSAC (250 °C), Fe/PSAC (600 °C) and Ce/PSAC (250 °C) nanocomposite at different calcination temperature based on TGA results.

2.4. Analysis of Surface Functional Groups

The synthesized metal oxide nanoparticles were analyzed by using SEM-EDX analysis to determine the physical characteristics and elements existing on the adsorbent surface and TGA analysis to investigate the changes of weight over time or temperature. Whereas the impregnated metal oxide nanoparticles onto PSAC were analyzed by using FTIR, to investigate functional groups available after the modification.
2.5. \( \text{H}_2\text{S} \) Adsorption Study

The adsorption activity of the prepared adsorbents was carried out in a fixed bed adsorption column. The schematic diagram is shown in Figure 1. The adsorbents were tested for the adsorption of \( \text{H}_2\text{S} \) as shown in Figure 1, the calculation involves on the adsorption of \( \text{H}_2\text{S} \) are; (1) adsorption capacity, (2) time equivalent to total, (3) mole fraction calculation [9]. The \( \text{H}_2\text{S} \) adsorption was applied on the Ce/PSAC, Cu/PSAC, Ni/PSAC, Fe/PSAC and raw PSAC adsorbents. The reactor was maintained around 30°C ambient temperature while maintaining the flue gas at 3000 ppm (105 mL/min of \( \text{N}_2 \) and 45 mL/min of \( \text{H}_2\text{S} \)). The reactor was insert to 1 g of adsorbent and 0.3 g glass wool to determine the removal of \( \text{H}_2\text{S} \) gas. The molecular weight of \( \text{H}_2\text{S} \) is 34.08 g/mol and the value of \( V_n \) from equation (3) is 22.85 L/mol where \( R = 8.314 \text{ (J/K.mol)} / (\text{m}^3.\text{Pa/K.mol}) \), \( T = 303.15 \text{ K} \) and \( P = 1.10325 \times 10^5 \text{ Pa} \). Therefore, the value of mole fraction of adsorbate, \( y_f \) is 4.4754×10\(^{-3}\) mg/L.

![Figure 1: Schematic diagram of the reactor setup for \( \text{H}_2\text{S} \) adsorption study: (1) gas cylinder, (2) pressure regulator, (3) mass flow control, (4) water bath, (5) humidifier, (6) three way valve, (7) insulated pipeline, (8) pressure gauge, (9) thermocouple, (10) temperature indicator, (11) heating jacket, (12) adsorbent, (13) glass wool, (14) stainless column, (15) bubble soap flow meter, (16) \( \text{H}_2\text{S} \) analyser.](image)

\[
q = \frac{Q_f t_i y_f}{m_c} \quad (1)
\]
\[
q_i = \left[ 1 - \frac{C_i}{C_0} \right] \times \frac{Q_f t_i y_f}{m_c} \quad (2)
\]
\[
V_n = R \frac{T}{P} \quad (3)
\]

For the adsorption capacity calculation (1), the \( y_f \) represented the mole fraction of adsorbate, \( Q_f \) represents volumetric feed flowrate, \( m_c \) represent mass of adsorbent used, \( y_i \) is gas mol fraction based on 3000 ppm of \( \text{H}_2\text{S} \) and \( t_i \) represent time equivalent to the total.
3. Results and Discussion

3.1. Thermal Analysis of Metal Oxide Nanoparticles

The metals oxide, PVP and raw PSAC were analysed using thermogravimetric analysis (TGA) with ramp rate of 10 °C/min to 800 °C in order to analyse chemical states of the active species. Figure 2 shows the TGA analysis of synthesised metals oxide nanoparticles. The metals oxide possessed first peak around 150 °C, which most probably due to moisture trapped in the structure of the adsorbents which losses the weight around 150 °C [10]. The weight loss around 450 °C in the TGA profile of the metal hydroxides can be justified as the decomposition of the surfactant of the PVP. The decomposition of the PVP can confirmed in Figure 2 TGA analysis shows the decomposition of PVP around 450 °C.

![Figure 2: TGA analysis of metal hydroxide, raw PSAC and PVP](image)

The weight loss for the Fe(OH)_3 at 26-350 °C indicates the decomposition of Fe(OH) through the loss of -OHs with Fe_2O_3 nanoparticles formation [11]. The weight loss for Ce(OH)_3 around 150 °C shows the loss of OHs with the formation of CeO_2 nanoparticles [10]. The Ni(OH)_2 weight loss indicate from 25 - 210 °C shows that the first weight loss was evaporation of water molecules and the second weight loss was from 210-400 °C was due to thermal decomposition of Ni(OH)_2 to NiO nanoparticles [12]. The Cu(OH)_2 indicates three step of decomposition. Temperature ranging 35-166 °C was caused by water evaporation. The temperature ranging 166-200 °C was the decomposition of organic compound and the temperature ranging from 200-260 °C was the decomposition of OHs from the Cu(OH)_2 which will be form CuO nanoparticles [13]. Therefore, from the TGA analysis, the calcine temperature of each metals hydroxides was proven to form metals oxide nanoparticles. The calcine temperature for Fe(OH)_3 was at 600 °C, Ce(OH)_3 was at 250 °C, Ni(OH)_2 was at 450 °C, and Cu(OH)_2 was at 300 °C.
3.2. Physical Characteristics of Metal Oxide Nanoparticles

The scanning electron microscope (SEM) analysis was used to provide high-resolution image of the samples, while energy-dispersive X-ray (EDX) analysis was carried out to generate information on the chemical composition of the synthesized metal oxide nanoparticles (CuO, CeO$_2$, NiO, and Fe$_2$O$_3$). The EDX analysis generated the elements that are present as well as their distribution in the metal oxide nanoparticles. Figure 3 shows the result of SEM analysis of CeO$_2$, CuO, Fe$_2$O$_3$, and NiO nanoparticles.

![SEM images of CeO$_2$, CuO, Fe$_2$O$_3$, and NiO nanoparticles.](image)

Figure 3: SEM analysis of (a) CeO$_2$ nanoparticles (b) CuO nanoparticles (c) Fe$_2$O$_3$ nanoparticles (d) NiO nanoparticles.

The SEM analysis was used for morphological study of nanoparticle of CeO$_2$. Figure 3 (a) shows the SEM image of CeO$_2$ prepared in alkaline precipitation method. The image shows agglomerated of the nanoparticles. Besides, a spherical-like shaped of the nanoparticle with clumped distribution are slightly visible through the SEM analysis [14]. The average size of the CeO$_2$ was about 66.55 nm. Figure 3 (b) shows the SEM image of CuO nanoparticles. The surface morphology of the synthesized CuO nanoparticles flakes shaped structure with average size of 91.26 nm [15]. Besides, SEM analysis image sample of Fe$_2$O$_3$ nanoparticles was recorded as shown in Figure 3 (c). The surface morphology of the Fe$_2$O$_3$ nanoparticles is average crystalline size with average size 37.17 nm [16]. Lastly, Figure 3 (d) shows the image of NiO nanoparticles. The results indicate a spherical shape and agglomeration process [17]. The average size of the NiO nanoparticle observed from the SEM image is 91.46 nm.
Energy-dispersive X-ray (EDX) analysis of CeO₂ prepared by alkaline precipitation was shown in Figure 4 (a) which confirms the Ce and O with weight percent. The EDX shows the peak of Ce contains 63.77 % weight and the O contains 36.23 % of weight while the atomic percentage of Ce was 16.73 % and the O has 83.27 % in the CeO₂ nanoparticle sample. Besides, the CuO nanoparticles was evaluated in the EDX analysis shows in Figure 4 (b) and found to have 48.85 % of weight of Cu and 51.15 % of weight of O. The atomic percentage of the CuO in theoretical the Cu will be 50 % and O will be 50 % and the experiment found to have 80.62 % of O and 19.38 % of Cu. This is because the EDX analysis can only select one area. Thus the amount of Cu and O in different area gives different values in the CuO nanoparticles [13]. Next, Figure 4 (c) shows the EDX of synthesized Fe₂O₃ nanoparticles that peak shows around 0.5 and 0.7 KeV related to the binding energies [18]. The EDX confirms the presence of Fe and O atoms in nanoparticles. The atomic percentage obtained from EDX quantification was 32.72 % of Fe and 67.28 % of O. The weight percentage of the Fe was 62.93 % and O was 37.07 %. Lastly, Figure 4 (d) shows the EDX spectrum of prepared NiO nanoparticles. The EDX pattern has indicated the Ni and O atom with a percentage of Ni of 21.24 % and O of 78.76 %. The weight percentage of NiO nanoparticles contain 50.26 % of O and 49.74 % of Ni.

3.3. Functional Groups Available on Impregnated PSAC Adsorbent

The Fourier-transform infrared spectroscopy (FTIR) was used to identify the organic, polymeric and inorganic material in the metals oxide nanoparticles impregnated with palm shell activated carbon (PSAC). In this experiment, the metal oxide nanoparticles impregnated with PSAC were analysed using FTIR analysis. The samples that undergo the analysis were raw PSAC, Ce/PSAC, Cu/PSAC, Fe/PSAC,
and Ni/PSAC. Figure 5 shows an analysis of FTIR of sample raw PSAC, Ce/PSAC, Cu/PSAC, Ni/PSAC and Fe/PSAC. The FTIR analysis shows the peak at 3910 cm\(^{-1}\), 3748 cm\(^{-1}\), 1750 cm\(^{-1}\), 1473 cm\(^{-1}\), 1099 cm\(^{-1}\) and 488 cm\(^{-1}\) of the sample raw PSAC, Ce/PSAC, Cu/PSAC, Ni/PSAC and Fe/PSAC.

![FTIR analysis on raw PSAC and metals oxide nanoparticles/PSAC](image)

Figure 5: FTIR analysis on raw PSAC and metals oxide nanoparticles/PSAC

The first peak is at shows the present of graphite group (C) [19]. The peak 3748 cm\(^{-1}\) shows the present of hydroxyl group (O–H) of adsorbed H\(_2\)O molecules [20-23]. The bands at about 1750 cm\(^{-1}\) in wavenumber show highly conjugated of (C=O) axial deformation, such as highly conjugated C=O stretching or C–O stretching of carboxyl groups [21-22]. The band at 1473 cm\(^{-1}\) shows the conjugated with (C–OH) alcohol group in aromatic rings of the samples [23]. The band at 1099 cm\(^{-1}\) shows the location of strong (C–O) groups [23-24]. The FTIR analysis shows a shoulder peak 488 cm\(^{-1}\) indicates the a few free element of sample which are the Ce-O\(_2\) in Ce/PSAC, Cu-O in Cu/PSAC, Ni-O in Ni/PSAC, and Fe\(_2\)O\(_3\) in Fe/PSAC [19, 23].

3.4. \(H_2S\) Adsorption Study
Figure 6 shows the result of \(H_2S\) adsorption of 1 g raw PSAC, Ce/PSAC, Cu/PSAC, Ni/PSAC, and Fe/PSAC adsorbents. Table 1 shows the breakthrough capacity and adsorption capacity of the \(H_2S\) by using raw and impregnated PSAC. Equation 5 below shows the total adsorption capacity and the total adsorption value is 48.45 mg \(H_2S/g\) Ce/PSAC.
It was observed that the breakthrough time of the Ce/PSAC adsorbent is at 4 minutes and increasing until it reaches equilibrium around 226 minutes. The adsorption capacity was calculated where the volumetric flowrate, $Q_f$ is 150 mL/min (total of 105 mL/min of H$_2$S gas and 45 of mL/min of N$_2$ gas) and mass of the adsorbent, $m_a$ is 1 g. The time equivalent to the breakthrough, $t_b$ was set to 0.05 $C/C_0$ in 6 minutes. Therefore, breakthrough capacity of Ce/PSAC is 4.03 mg H$_2$S/g Ce/PSAC and its total adsorption capacity is 48.45 mg H$_2$S/g Ce/PSAC. The breakthrough time of the Cu/PSAC adsorbent was observed is at 80 minutes and increasing until it reaches steady state around 515 minutes. The time equivalent to the total is around 0.05 $C/C_0$ in 129 minutes. Therefore, adsorption capacity of Cu/PSAC is 86.60 mg H$_2$S/g Cu/PSAC, while total adsorption capacity is 213.67 mg H$_2$S/g Cu/PSAC. It was observed that the breakthrough time of the Ni/PSAC adsorbent is around 9 minutes. At around 9 minutes of time, the $C/C_0$ started to raise until it reaches the steady state around 361 minutes. The breakthrough capacity was calculated at 13.5 minutes is 9.06 mg H$_2$S/g Ni/PSAC, while the total adsorption capacity of Ni/PSAC is 67.01 mg H$_2$S/g Ni/PSAC. For Fe/PSAC adsorbent, it can be observed as the beginning of the 10 minutes the breakthrough are started and around 120 minutes the graph begins to become

![Figure 6: Breakthrough curves of H$_2$S adsorption by using raw and impregnated PSAC adsorbents](image.png)

| Samples   | Breakthrough capacity, mg H$_2$S/g Adsorbent | Total adsorption capacity, mg H$_2$S/g Adsorbent |
|-----------|---------------------------------------------|-----------------------------------------------|
| Ce/PSAC   | 4.03                                        | 48.45                                         |
| Cu/PSAC   | 86.60                                       | 213.67                                        |
| Ni/PSAC   | 9.06                                        | 67.01                                         |
| Fe/PSAC   | 11.08                                       | 40.63                                         |
| raw PSAC  | 2.85                                        | 22.16                                         |
steady because it already reached the adsorbent limit. From Fig. 6, when the C/C₀ is at 0.05, the time taken was around 16.5 minutes. Therefore, the calculated breakthrough capacity value is 11.08 mg H₂S/g Fe/PSAC. Thus, the total adsorption capacity can be calculated which the value is 40.63 mg H₂S/g Fe/PSAC. The application of H₂S adsorption on the raw PSAC adsorbent was done in order to compare the efficiency of the impregnated PSAC adsorbent with metal oxide nanoparticles. Compared to the impregnated PSAC, the raw PSAC adsorbent resolved very quickly in the breakthrough time, which was around 3 minutes and it started to rise and reach steady state at 143 minutes.

4. Conclusions
In conclusion, the metal oxide nanoparticles that were synthesised using alkaline precipitation method resulting in metals hydroxides. The metal hydroxides nanoparticles undergo TGA, SEM and EDX analysis. The TGA analysis shows the temperature needed to calcine for the metals hydroxides for the decomposition of impurities. The TGA analysis gives the result of Fe(OH)₃ was at 600 °C, Ce(OH)₃ was at 250 °C, Ni(OH)₂ was at 450 °C, and Cu(OH)₂ was at 300 °C. The EDX shows the result of the weight and the atomic percentage of the metals oxide nanoparticles which major consist of metals element and O element. The SEM shows the morphology of the metal oxide nanoparticles and average size (Fe₂O₃ = 37.17 nm, CeO₂ = 66.55 nm, NiO = 91.46 nm, CuO = 91.26 nm). It shows that the metal oxides are present of nanoparticle which is the size average should be <100 nm in size. The metals hydroxides were than impregnated with peroxidised PSAC and calcined to produce M-O/PSAC nanoparticles adsorbent. FTIR shows the presence of (C–OH) alcohol group, (C=O) carboxyl groups, (O–H) hydroxyl group, (C=O) carbonyl group and (M-O) group in the metals oxide nanoparticles impregnated with PSAC adsorbent (MO/PSAC). Lastly, the application of H₂S removal was applied to the MO/PSAC adsorbent. From these parameter studies, Fe₂O₃/PSAC, CeO₂/PSAC, NiO/PSAC, and CuO/PSAC adsorbent is more promising than without metal oxide nanoparticle which is the raw PSAC adsorbent itself. From the MO/PSAC adsorbent, the Cu/PSAC adsorbent has the best adsorption capacity among the MO/PSAC.

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Acknowledgement
The authors are grateful for the financial supports by FRGS grant (600-IRMI/FRGS 5/3 (022/2019)) and facilities provided by Universiti Teknologi MARA (UiTM) Cawangan Pulau Pinang in the accomplishment of this research work.