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Research Article

Modification & Characterization of Activated Carbon Impregnated with KCl, Na_2S, and KI for Enhancing Mercury (Hg) Removal from Natural Gas

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Abstract

Modified activated carbon (MAC) has been synthesized and characterized to enhance mercury (Hg) removal from natural gas. MAC was modified by impregnating it into KCl, Na_2S, and KI to introduce Cl-, S-, and I- elements. SEM-EDX, FTIR, and SAA were used to characterize the AC and MAC. The isotherm and adsorption capacity were studied using the mercury gas standard. The results of SEM-EDX analysis show that the impregnation method is proven to produce MAC containing elements Cl, S, and I with mass % of 2.78% Cl, 0.76% S, and 39.60% I. The surface area is 421.91 m^2/g, the total pore volume is 0.386825 cc/g, and the average pore size is 1.83369 nm. Group functions are -OH, C=C, C=O, C-O, and vibrations at the wavelength number 617.81 cm^{-1}, which the impregnation agent forms. The mechanism for absorbing mercury gas into MAC follows the Freundlich isotherm model, with a coefficient of determination (R^2) of 0.996. The adsorption capacity on MAC increased 57 times compared to unmodified activated carbon (AC) from 5540.60 to 315730.64 ng/g, with an efficiency maximum of 100%. The MAC has been proven to enhance mercury adsorption from natural gas with an efficiency of 78.6%.

Keywords: Modified activated carbon, isotherm, adsorption capacity, natural gas, mercury

1. INTRODUCTION

Mercury (Hg) is a hazardous and toxic material in the form of heavy metals that are liquid, silver-white in color, and volatile at room temperature, where they are usually in the form of organic and inorganic compounds that are persistent and bio-accumulative. Mercury occurs naturally in nature in several forms, such as metal (elemental mercury), inorganic mercury, and organic mercury [1]. The increasing exploration and production of hydrocarbon compounds has also caused mercury contamination to increase. Starting from health risks, safety, equipment contamination, catalyst damage, toxicity of emissions to the environment, and damage caused by mercury contamination in heat exchangers of LNG (liquified natural gas) plants [2].

Mercury is abundant in natural gas wells with varying levels, and its content needs to be removed to prevent damage to aluminum heat exchangers and other plant equipment. Most natural gas has a small amount of mercury content (trace). The presence of mercury in natural gas, even in small amounts, is considered detrimental because it can cause corrosion to equipment and process facilities in the oil and gas industry [3].

The most common method developed to remove mercury in natural gas is activated carbon adsorption using Fixed-bed reactors (MRUs) [4]. The activated carbon surface can be efficiently modified by chemical impregnation methods to increase the adsorption capacity. According to the adsorbate's nature, activated carbon's surface can be modified by

forming acidic or basic groups [5]. Many adsorbents have been developed commercially, most of which are sulfur-impregnated activated carbon [6,7].

The research data shows that activated carbon impregnated with chloride (Cl) can absorb mercury by 96% [8]. Other studies reached 91.4% with a mercury adsorption capacity of 13.14 mg/g [9]. Mercury absorption capacity for Sulphur-impregnated activated carbon (AC-S) was 21 mg/g, chloride-impregnated activated carbon (AC-Cl) 38 mg/g, and combined impregnated activated carbon (AC-Cl+S) 77 mg/g [10]. The adsorption of iodine-impregnated activated carbon (KI-AC) produced an efficiency of 98-100% [11].

Most of the research involved only one or two impregnation chemicals; studies concerned combined impregnation with three impregnation chemicals. Meanwhile, adsorption using activated carbon impregnated by three impregnates, sulphur (S), chloride (Cl), and iodine (I), offers the potential to enhance mercury removal from natural gas [8], improve the surface properties, and increase the adsorption capacity rather than one or two impregnants [12]. Therefore, this study aims to modify and investigate the characterization of modified activated carbon to promote a combination of groups of Cl, S, and I elements, as well as isotherm and adsorption capacity and its application to natural gas.

2. RESEARCH METHODS

2.1 Material

Chemicals used in this research are commercial activated carbon pro analysis (Merck KGaA No. 1.02186, M=12.01 g/mol, for analysis), sodium sulfide (Merck, M=78.04 g/mol, for analysis), potassium chloride (Merck, M=74.55 g/mol, for analysis), potassium iodide (Merck, M=166.00 g/mol, for analysis), mercury standard box (MB-1 NIC), tedlar bag capacity of 5 L, natural gas from liquefied natural gas (LNG) plant. The instrumentation applied in this research, such as the Fourier-transform infrared spectrometer (FTIR) Thermo Nicolet IS 10, the Scanning Electron Microscope & Electron Dispersive X-Ray (SEM-EDX) JEOL JED-2300 Analysis Station, the Surface Area Analyzer (SAA) St 3 on NOVA touch 4LX Quanta chrome, and the Mercury analyzer NIC- WA5, plays a crucial role in ensuring the reproducibility of our findings, a cornerstone of scientific research.

2.2 Methods

Activated carbon is an adsorbent commonly used to absorb mercury (Hg) in natural gas [2], Adding active groups of Chloride (Cl), Sulphur (S), and Iodide (I) to activated carbon can increase the absorption of mercury in natural gas. This novel research aims to determine the characteristics of modified activated carbon (MAC) using SEM-EDX, FTIR, and SAA. Then, the isotherm and adsorption capacity were determined using the mercury gaseous standard and its application to natural gas from Donggi Senoro LNG (Liquefied natural gas) plant.

2.2.1 Preparation of activated carbon

10 g of commercial activated carbon heated at 120℃ for 3 hours, then cooled again in a desiccator. It is labeled as unmodified activated carbon (AC). The AC was then characterized using SEM-EDX, SAA, and FTIR for the initial characterization.

2.2.2 Modification of activated carbon (MAC)

10 g of commercial activated carbon is soaked using 100 mL of 5% KCl solution at room temperature, stirring at 100 rpm for 12 hours. Then, the KCl-impregnated activated carbon is filtered, and the filtered activated carbon is heated up at 120℃ for 3 hours and then cooled again in a desiccator. It was then soaked using 100 mL of 5% Na_2S solution at room temperature, stirring at 100 rpm for 12 hours [13]. Na_2S-impregnated activated carbon is filtered, and the filtered activated carbon is heated up at 120℃ for 3 hours and then cooled again in a desiccator. After Na_2S impregnation, the activated carbon above is soaked again using 100 mL of 5% KI at room temperature, stirring at 100 rpm for 12 hours. Finally, the activated carbon-impregnated KCl + Na_2S + KI is filtered and heated at 120℃ for 3 hours, then cooled in a desiccator. It is labeled as modified activated carbon (MAC). The MAC was then characterized using SEM-EDX, SAA, and FTIR.

2.2.3 Determination of isotherm and mercury adsorption capacity

The procedure was performed by preparing each 0.010 g of AC & MAC and mercury gas standard with variations 4.992; 24.958; 49.917; 74.875; 99.834; 249.584 ng/L. Then, the adsorption process for all variations was carried out at room temperature and flow of 1 L/min according to Figure 1, and the gas standard being adsorbed was measured by Mercury analyzer WA-5, then calculated % removal efficiency, isotherm, and adsorption capacity. The amount of mercury removal efficiency was calculated following equation (1) [14,15].

$$\%R = \frac{(C_o - C_i)}{C_o} \times 100 \qquad (1)$$

Where %R = removal efficiency, & C_o & C_i = Hg ion concentration before and after adsorption (ng/L).

Langmuir isotherm is shown in the following equation (2) [14,16].

$$\frac{1}{q_e} = \frac{1}{K_L\, q_{max}} x \frac{1}{C_e} + \frac{1}{q_{max}} \quad (2)$$

Where the q_e = amount of adsorbate uptake at equilibrium (mg/g), q_{max} = the maximum adsorption capacity (mg/g), and K_L = Langmuir isotherm constant (L/mg), and C_e = the equilibrium concentration (mg/L).

The separation factor (R_L) is shown in the following equation (3) [17,16].

$$R_L = \frac{1}{1 + C_i \times K_L} \quad (3)$$

Where R_L = Langmuir isotherm separation factor which show the adsorption possibility.

Freundlich isotherm is expressed as the following equation (4) [14,16].

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (4)$$

Where, K_f = Freundlich constant and used to calculate the Freundlich adsorption capacity.

The MAC was then implemented in natural gas, which was taken from the Unit feed gas inlet on the LNG plant into tedlar bags with a capacity of 5 L. Tedlar bags were subjected to adsorption tests using 0.010 g of MAC, as shown in **Figure 1.** The natural gas being adsorbed was then calculated in the %R.

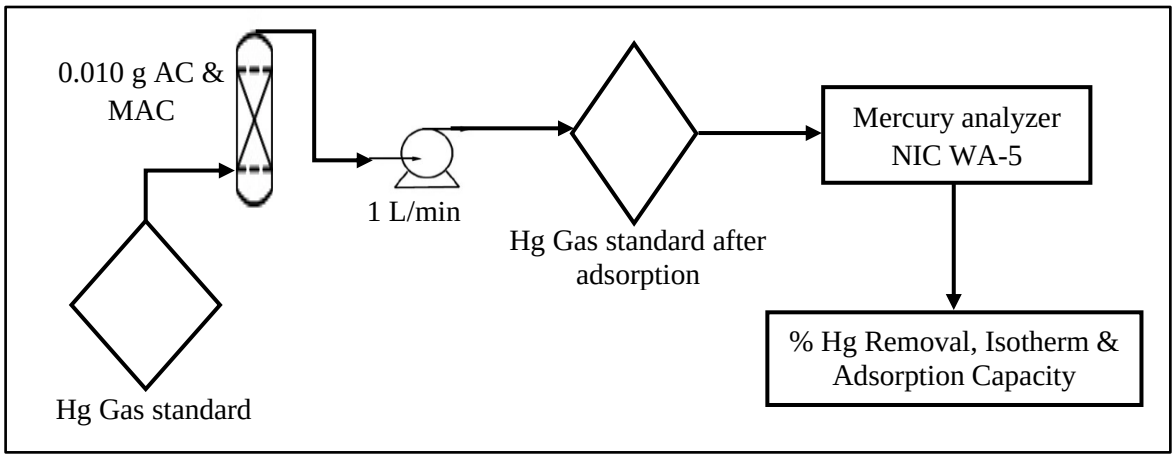

Figure 1. Adsorption scheme for determining isotherm & adsorption capacity

3. RESULTS AND DISCUSSION

3.1 Characterization of Activated Carbon

The activated carbon's surface morphology was examined using SEM-EDX analysis [18]. **Figure 2** displays the SEM data, and **Table 1** displays the EDX results. **Figure 2** shows that the surface of the AC at magnifications of 1000x is rough and shaped like a tunnel, and many small particles cover it. Meanwhile, the MAC has a smoother surface, and the porosity can be seen. Most small particles covering the pore surface have been bound to salt ions during the impregnation process. SEM images for the AC and MAC samples do not exhibit significant differences.

The EDX spectra showed the elements in AC and MAC (**Table 1**). The impregnation process using KCl, Na_2S, and KI has been shown to produce MAC containing the elements Cl, S, and I with a composition of 2.78% Cl; 0.76% S; and 39.60% I; respectively. The percentage of elements varies greatly, a clear indication of the complexity of the process. This variation is caused by sequential impregnation, which is strongly influenced by the binding energy and electron transfer ability/electronegativity of each impregnating agent to activated carbon; therefore, the impregnation agents can replace each other [10,19].

The Cl element of the AC is 0.98%, which is a chemical property probably achieved during the activation process and confirmed by a relatively high mercury removal percentage. This is similar to the results reported by Zhu et al. (2009) [20], who employed commercial activated carbon (NORIT Darco) with a sulphur concentration of 0.83% as the chemical properties.

A surface area analyzer (SAA) is used for assessing the pore size, pore volume, and surface area of a porous material. These characteristics, which are listed in **Table 2**, are crucial to its use. **Table 2** shows that activated carbon surface area and total pore volume decreased after impregnation. It is because impregnation is a process of total saturation of certain substances so that active ion salts will diffuse to fill the pores of activated carbon [21]. Activated carbon will experience a reduction in surface area and porosity size after impregnation and change its physical and chemical properties [22].

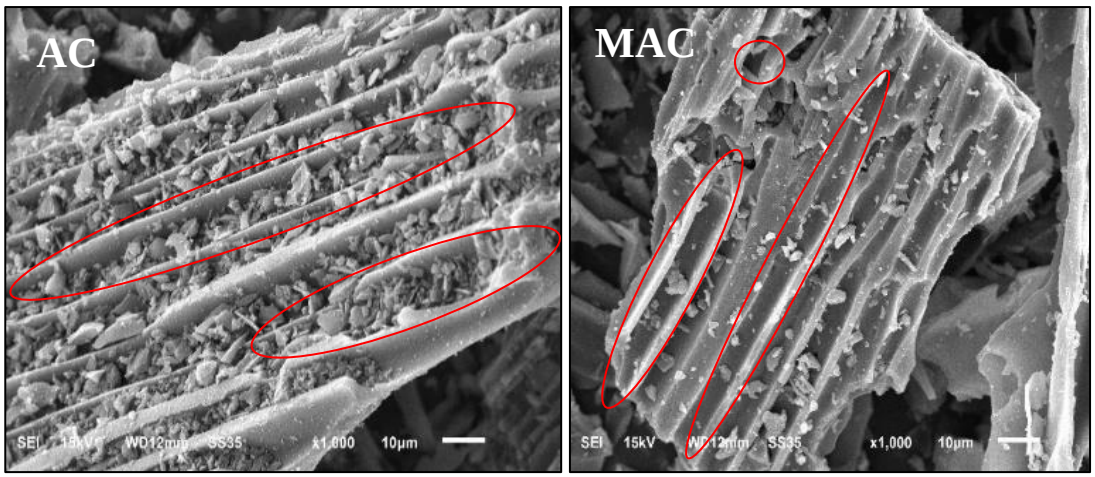

Figure 2. Morphology of unmodified (AC) & modified activated carbon (MAC)

Table 1. EDX result of unmodified activated carbon (AC) & modified activated carbon (MAC)

Adsorbent	Elements (%mass)						
	C	O	Cl	S	I	Na	K
AC	72.12	26.91	0.98	-	-	-	-
MAC	37.68	8.80	2.78	0.76	39.60	3.75	6.64

Table 2. SAA analysis result of unmodified activated

No	Adsorbent	Surface area (m^2/g)	Total pore Volume (cc/g)	Average pore size (nm)
1	AC	708.28	0.568061	1.60406
2	MAC	421.91	0.386825	1.83369

The International Union of Pure and Applied Chemistry defines the pore size classification as micropore < 2 nm, mesopore 2-50 nm, and macropore > 50 nm. This classification is the basis for the pore size classification scheme [23]. This research led to the classification of AC and MAC as micropores.

As seen in **Figure 3**, adsorption and desorption isotherms were also evaluated using nitrogen at 77.35 K. The adsorbed gas at a relative pressure (P/Po), where P is the gas's vapor pressure, and Po is the adsorbent's saturation pressure, was described using the adsorption isotherms.

Figure 3 shows AC and MAC have similar isotherm curves type II, monolayer-multilayer adsorption. From the beginning of the curve to the middle of the isotherm, it is almost linear, often used to indicate the stage where monolayer coverage has been completed, and multilayer adsorption will begin soon [24,25,26]. The isotherm model is relevant to the mercury adsorption isotherm results found in **Figures 6 & 7** that show the mercury adsorption process on AC and MAC occurs in a complex monolayer and multilayer manner.

FTIR analysis is to identify functional groups found in activated carbon. FTIR scanning using KBr with a measurement range of 4000 – 400 cm^{-1} (**Figure 4**). **Figure 4** shows the results of the FTIR spectrum, which are similar between AC and MAC. The absorption area at wavenumbers 3421.44 and 3417.57 cm^{-1} indicate the existence of hydroxyl groups (-OH) obtained from the existence of water molecules in activated carbon; this can be seen from the shape of the high and asymmetric peak at the beginning of the absorption area of wavenumbers indicating the presence of hydrogen bonds[20,27].

At the wavenumbers 1563.56 and 1561.53 cm^{-1} as aromatic rings (C=C)[20,28] ; the weak peak at 1382.74 and 1383.28 cm^{-1} can be associated with oxygen functional groups, C=O, or in-plane vibration of -OH (carboxylic-carbonate groups)[27, 28]. Wavenumbers 1126.79 & 1101.57 cm^{-1} as C-O (lactonic, phenolic, ether groups, etc) [20,28,29].

The highest intensity of the C-O peak may result from the sequential impregnation process, which includes repeated heating and cooling at 120 °C. This process leads to the formation of numerous oxides on the surface. According to Boehm (2001) and Figueiredo (1988), the surface oxides decompose to

CO_2 and CO during heating at high temperatures. This process produces highly reactive sites that remain on the activated carbon surface, and whic have a free-radical character. After cooling to room temperature, they can react with oxygen (air) or moisture (water vapor), forming new surface oxides [29,30]. In the FTIR spectrum of MAC, there is a vibration at wavenumbers 617.81 cm^{-1}, which AC does not exist. These are the consequences of KCl, Na_2S, and KI used as the impregnation agents. These are the consequences of using KCl, Na_2S, and KI as the impregnation agents. These results are similar to those obtained in the research of Cai et al. (2014) [31], which produced a new absorption area in KI and KBr-impregnated activated carbon, namely in the absorption area of wavenumbers 793 – 453 cm^{-1} and also similar to the research of Daiem et al. (2020) [32], which exhibit a relatively weak absorption area at nearly 670 cm^{-1} as the result of using $ZnCl_2$ as an activation agent. It was shown that KCl, Na_2S, and KI impregnated resulted in additional functional groups. These functional groups can act as electron donors or acceptors during mercury sorption on the MAC surface, depending on the type of surface functional group interactions [20,33]. A schematic illustration of the functional groups is shown in **Figure 5**.

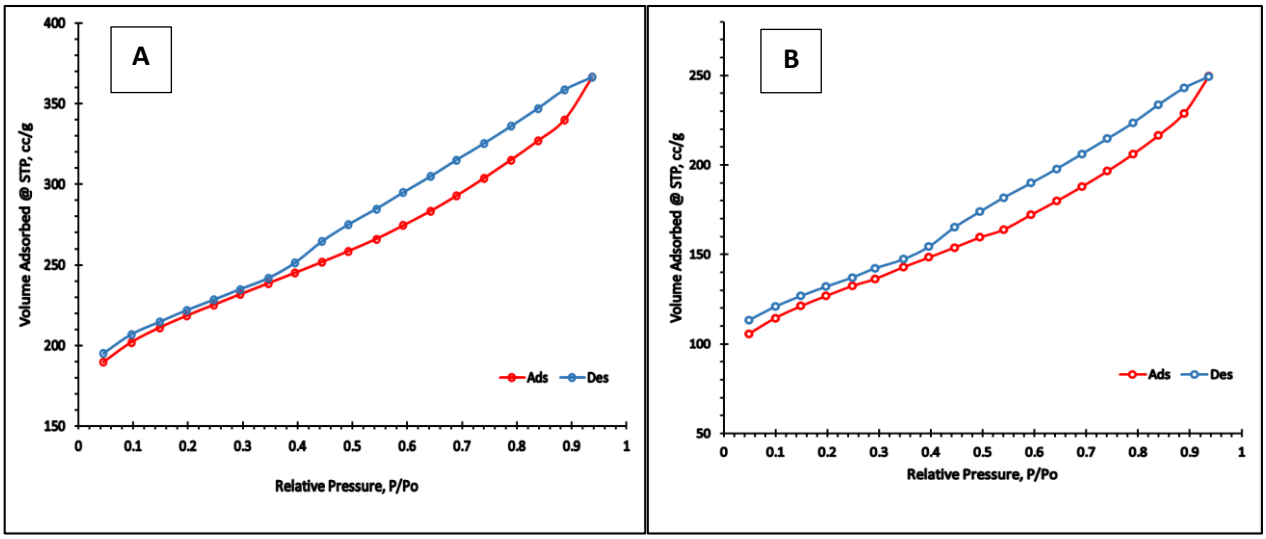

Figure 3. Isotherm curve of AC (A) & MAC (B) measured using nitrogen at 77.35 K at relative pressure (P/Po)

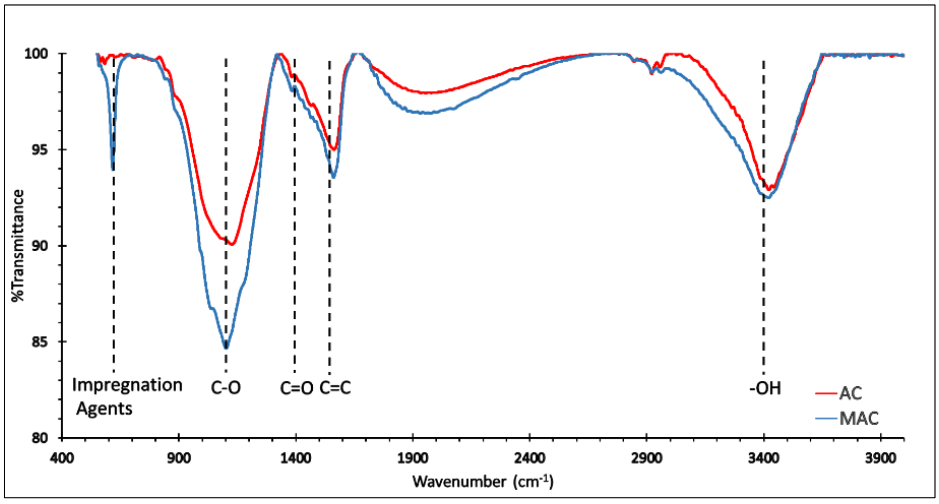

Figure 4. FTIR spectra of unmodified (AC) & modified activated carbon (MAC)

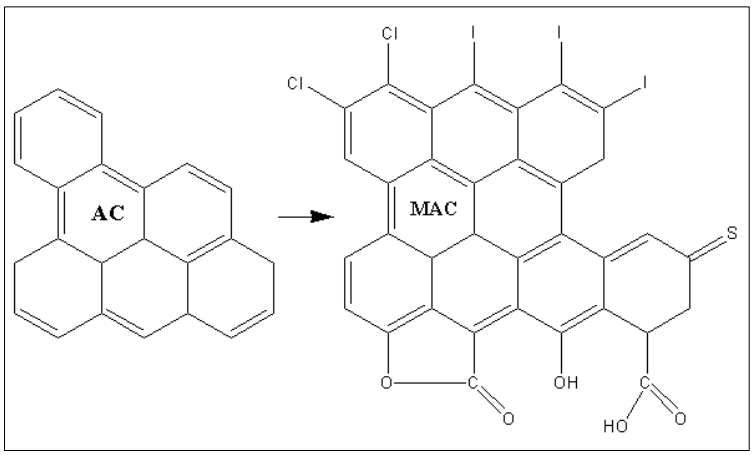

Figure 5. Schematic illustration of unmodified (AC) & modified activated carbon (MAC) functionalization

3.2 Adsorption test, isotherm and mercury adsorption capacity

An adsorption test using standard gas aims to see how the adsorbent can be modified before and after modification to improve the efficiency of mercury gas absorption at various concentrations. It was obtained that the efficiency of mercury removal of MAC increased significantly to 100 %; this is inversely proportional to AC, which experienced a decrease in efficiency from 98.31% to 43.40%, at a concentration of 4.992 ng/L to 249.584 ng/L, as in **Table 3**. According to the other research by Jan et al. (2017)[34], activated carbon impregnated with KI, HCl, and Sulphur increased mercury absorption efficiency to 100%, while unimpregnated activated carbon only reached 60-80%. The results by Rosmayati (2012)[8], showed that activated carbon impregnated with chloride (Cl) could absorb 96% mercury (Hg). In another study Sano et al. (2017)[10], impregnation using K_2S and $CaCl_2$ can increase absorption efficiency up to 50-80 times compared to unimpregnated activated carbon. These results prove that activated carbon impregnated with KCl, Na_2S, and KI can increase the absorption efficiency of mercury gas up to 100%.

The results of determining the isotherm adsorption on AC are in **Figure 6**. Whereas, the isotherm adsorption on MAC are in **Figure 7**. **Table 4** presents the overall isotherm parameters of mercury adsorption on AC and MAC. The Freundlich isotherm model describes the mechanism of mercury gas absorption into adsorbents in both AC and MAC. It can be seen from the Langmuir coefficient of determination (R^2) of 0.986 and 0.990. Meanwhile, Freundlich has 0.987 and 0.996 for AC and MAC adsorbents, demonstrating that both AC and MAC exhibit similar Type II isotherms, indicative of monolayer-multilayer adsorption. This observation highlights the relevance of the Freundlich adsorption isotherm over the Langmuir model, as stated in **Figure 3**.

The way to find out whether the experimental data follows the Langmuir or Freundlich equilibrium model can be stated in the value of the coefficient of determination (R^2); if the plot results are close to 1, then the experimental data follows the equilibrium model [16]. Other evidence states that the mercury adsorption process does not follow the Langmuir isotherm model which is the separation factor (R_L) value, which is more than 1 [35,36]. The R_L value which is within the range of 0-1 suggests that the adsorption is favorable Langmuir isotherm model [37].

Another piece of evidence can be seen from the adsorption intensity (n) in the Freundlich isotherm. If the value of $1/n$ is in the range of $0.1 < 1/n < 1$, then the Freundlich isotherm is favorable [17]. The Freundlich approach is that the adsorbent surface is heterogeneous, and adsorption forms multiple layers. It allows the adsorbate to move freely until the adsorption process occurs in many layers.

The Freundlich isotherm n value for modified activated carbon is lower than the unmodified activated carbon. These data suggest that adsorption on modified carbon is advantageous, but not as efficient as unmodified activated carbon, particularly at low adsorbate concentrations. These results are consistent with those of Zhu et al. (2019), who found that the Freundlich model suited the data better than the Langmuir model, particularly at higher mercury concentrations. It suggested that there might be differences in the Hg sorption mechanisms by MAC at low and high concentrations, most likely due to heterogeneous interactions between various functional groups [20].

Table 3. Adsorption test at various concentration

No.	Initial concentration Hg (ng/L)	Residual concentration AC (ng/L)	Residual concentration MAC (ng/L)	Hg Removal AC (%)	Hg Removal MAC (%)
1	4.992	0.084	0.000	98.31	100.00
2	24.958	1.923	0.005	92.29	99.98
3	49.917	9.096	0.012	81.78	99.98
4	74.875	17.338	0.021	76.84	99.97
5	99.834	38.215	0.040	61.72	99.96
6	249.584	141.277	0.112	43.40	99.96

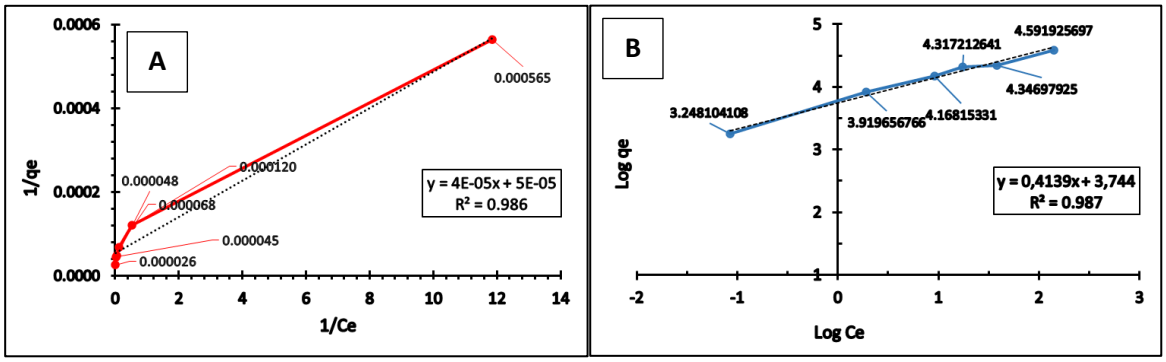

Figure 6. Langmuir (A) & Freundlich (B) isotherm of mercury adsorption on unmodified activated carbon (AC)

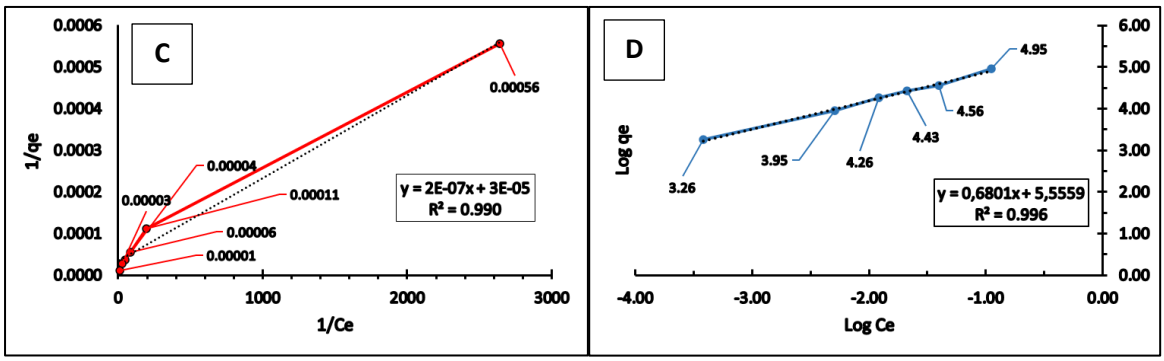

Figure 7. Langmuir (C) & Freundlich (D) isotherm of mercury adsorption on modified activated carbon (MAC)

Table 4. Parameters of Langmuir & Freundlich isotherm parameters on mercury adsorption

Adsorbent	Langmuir			Freundlich			
	q_{max} (ng/g)	R_L	R^2	K_f (ng/g)	n	$1/n$	R^2
AC	16029.25	>1	0.986	5540.60	2.3322	0.429	0.987
MAC	24888.04	>1	0.990	315730.64	1.5198	0.658	0.996

The adsorption of Hg^0 on the surface of the adsorbent can be classified into two types of absorption: physical adsorption and chemical adsorption. Adsorption of Hg^0 through physical adsorption is reversible, occurring at low temperatures. On the other hand, chemical absorption is associated with specific activation energy and occurs along with increasing temperature.

Physical adsorption involves the surface through weak bonds, such as van der Waals forces as attractive forces between an adsorbent (solid) and an adsorbate (gas molecules), whereas chemical adsorption is the attractive forces between the adsorbent and the adsorbate in the form of chemical bonds formed within a single-molecule layer [38].

The predicted adsorption capacity of an adsorbent can be determined using the Langmuir and Freundlich isotherm equations. The q_{max} value indicates the Langmuir isotherm adsorption capacity. The K_f value indicates the Freundlich isotherm adsorption capacity [39]. The results showed that the adsorption capacity of MAC increased 57 times

compared to the unmodified activated carbon (AC), from 5540.60 to 315730.64 ng/g.

3.3 Mercury (Hg) adsorption on natural gas

Modified activated carbon (MAC) was tested for mercury absorption on natural gas obtained from the LNG (Liquefied Natural Gas) plant. The adsorption results are presented in **Table 5**.

It is worth noting that using MAC led to a significant decrease in mercury concentration from 1.38 to 0.30 ng/nm^3, with a remarkable 78.6% removal. This reduction can be attributed to the binding of mercury from natural gas to the active sites of Cl, S, and I ions obtained from the modification process [40,2]. The larger size of iodide ions, in the order of I > Cl > S, suggests a higher surface exposure, leading to more significant interaction with mercury and, thus, more effective removal. According to Zhu et al. (2009), MAC's mechanism of mercury removal may involve electrostatic interactions, mercury surface reduction, and ion exchange, in which mercury combines with surface halogen to produce mercury halides.

The percentage of mercury removal may increase due to the characteristics of the natural gas sample from the LNG plant used in this study, which has a very small mercury content. The mercury concentration in the natural gas samples after MAC absorption had reached the minimum instrument detection limit of the mercury analyzer [41]. It proves that MAC impregnated with KCl, Na_2S, and KI can reduce mercury content in natural gas.

Table 5. Mercury (Hg) adsorption test on natural gas

No.	Description	Hg Concentration (ng/nm^3)			Average (ng/nm^3)	Hg Removal (%)
		I	II	III		
1	Natural Gas Initial Concentration	1.48	1.32	1.35	1.38	78.6
2	Natural Gas after adsorption on MAC	0.37	0.26	0.26	0.30	

4. CONCLUSIONS

The impregnation process using KCl, Na_2S, and KI has been proven to produce modified activated carbon (MAC) containing Cl, S, and I elements. The surface area is 421.91 m^2/g, the total pore volume is 0.386825 cc/g, and the average pore size is 1.83369 nm. It has three groups: OH, C=C, C=O, and C-O. There is a vibration at a wavenumber of 617.81 cm^{-1}; these are ions from the impregnation of KCl, Na_2S, and KI. The mechanism of mercury gas absorption into impregnated activated carbon (MAC) follows the Freundlich isotherm model. The adsorption capacity on MAC increased 57 times compared to unmodified activated carbon (AC), from 5540.60 to 315730.64 ng/g. MAC has been proven to reduce mercury concentration from natural gas by 78.6% removal. This study showcased a new method for modifying activated carbon with multiple functional groups to synthesize a more effective carbon-based adsorbent for enhanced mercury removal from natural gas.

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