

Assessment of Metal-Organic Frameworks and Activated Carbon Solutions for Sustainable Hydrogen Gas Storage

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ABSTRACT

The pursuit of sustainable energy sources has elevated hydrogen gas as a potential carbon-neutral fuel with high energy content. However, efficient storage and transportation of hydrogen pose considerable challenges. To address these issues, the utilisation of metal-organic frameworks (MOFs) and activated carbon (AC) for hydrogen storage through adsorption has gained substantial attention. This research article offers a comprehensive assessment of hydrogen storage using these materials. The adsorption mechanism, fundamental to hydrogen storage in MOFs, involves hydrogen molecules adhering to the MOF framework's surface and pores via intermolecular forces. MOFs, comprising metal nodes and organic linkers, present high surface area-to-volume ratios and customizable adsorption properties, making them appealing for gas storage. AC, an amorphous porous carbon form, also displays favourable traits for gas storage.

Introduction

The urgent need for sustainable energy sources has propelled hydrogen gas into the spotlight as a potential carbon-neutral fuel with high energy content. However, efficient storage and safe transportation of hydrogen gas pose significant challenges for its widespread adoption (Abohamzeh et al., 2021). In addressing these issues, the development and use of metal-organic frameworks (MOFs) and activated carbon (AC), both of which share very similar properties, for hydrogen gas storage via adsorption has been an area of huge interest (Wang, 2022).

Hydrogen's versatility as an energy carrier for power generation, industrial processes, and energy storage amplifies the significance of efficient storage methods. Through advancements in hydrogen storage technologies, we accelerate the transition to a greener and more sustainable future, where hydrogen plays a vital role in meeting global energy needs whilst minimising environmental impact.

MOFs are crystalline porous materials constructed from metal 'nodes' and organic molecules known as 'linkers'. MOFs offer advantages such as a high surface area-to-volume ratio, enabling compact storage systems, and tunability to optimise adsorption properties making them promising candidates for gas storage applications since these characteristics enhance hydrogen uptake and storage capacities (Kishore, 2012). Activated carbon, formed by the burning of carbonaceous materials, shares similarly favourable properties with MOFs, namely its highly porous structure (Biswick, 2022).

Importantly, unlike conventional techniques that often require extremely high pressures or low temperatures, MOFs can store hydrogen at significantly lower pressures, typically up to 100 bars (Garcia-Holley et al., 2018). This lowers energy input requirements and enhances safety. Additionally, MOFs' reversible adsorption-desorption behaviour ensures easy release of stored hydrogen.

This research article aims to provide a comprehensive evaluation of hydrogen gas storage via adsorption on metal-organic frameworks and activated carbon.

The Adsorption Mechanism

The process of adsorption plays a critical role in the process of hydrogen gas storage in MOFs. More generally, adsorption refers to the collecting of molecules of a fluid on an adsorbent surface (Raut, 2022). In the context of hydrogen storage, adsorption is the surface phenomenon of hydrogen molecules adhering to the surface and within the pores of the MOF framework. This process occurs primarily through physisorption, which is a non-chemical interaction based on intermolecular forces such as van der Waals.

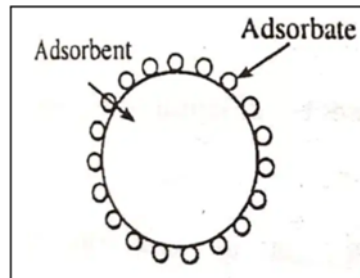


Figure 1. Adsorption (Raut, 2022)

The adsorption mechanism arises since the adsorbent material is composed of molecules that are arranged fairly symmetrically throughout its bulk. However, at the surface, a small residual force exists, and so when the adsorbate comes in contact with the adsorbent, it will experience attraction and will adhere to its surface (Raut, 2022).

Adsorption Isotherm Models

An adsorption isotherm is a graph that represents the variation in the amount of adsorbate adsorbed on the surface of the adsorbent with the change in pressure or concentration at a constant temperature at equilibrium (Raut, 2022).

Adsorption isotherms can be generally categorized into 5 types.

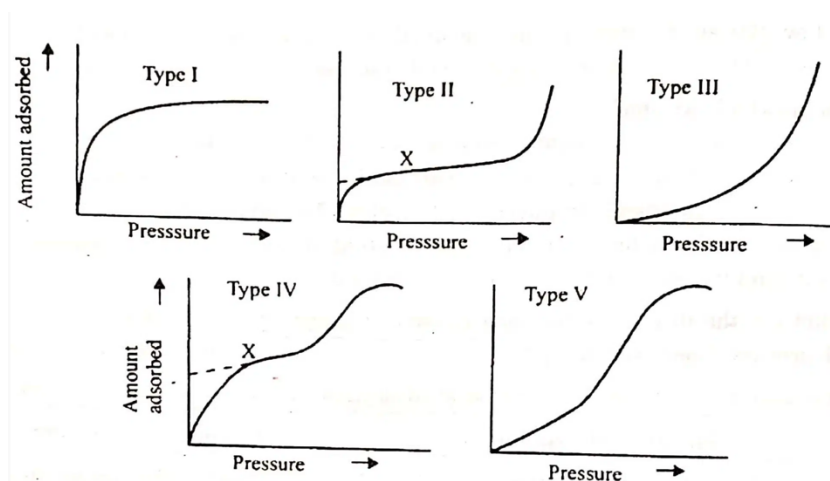


Figure 2. Adsorption Isotherm types (Raut, 2022)

Type I: This isotherm shows a saturation point- the amount of adsorbate adsorbed increases with pressure until no further adsorption can occur. This model also only accounts for monolayer adsorption.

Type II: This type of isotherm generally assumes a varied range of pore sizes, as the graph indicates the formation of the bilayer and trilayer after the monolayer has been deposited.

Type III: This isotherm shows a near-exponential increase in the amount adsorbed against pressure, suggesting that monolayer, bilayer and trilayer formation occurs simultaneously.

Type IV: This isotherm predicts the formation of two layers of adsorbate material on the surface of a mesoporous adsorbent, with the pore sizes being relatively larger than the adsorbate molecular sizes.

Type V: This kind of adsorption isotherm can be observed when there are strong intermolecular attractions between molecules, and the adsorption happens within both pores and capillaries.

There are also many more specific mathematical models formulated by scientists which are used to quantify and explain the relationship between adsorbate concentration and adsorption capacity. Some of the common ones include the Langmuir, Freundlich and BET isotherms. The Langmuir isotherm is described below.

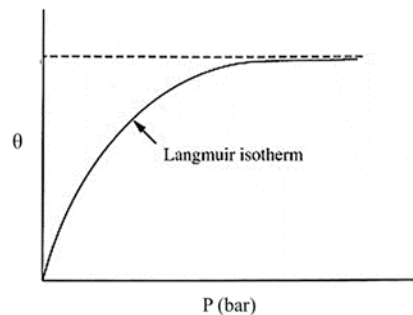


Figure 3. Langmuir, Adsorption Isotherms (Mathew, 2023, as adapted from Doran)

The Langmuir isotherm predicts linear adsorption at low adsorption densities and a maximum surface coverage at higher solute concentrations. It is based on the assumption of monolayer adsorption onto a homogeneous surface, where all adsorption sites are identical and uniform. Once all adsorption sites are occupied, further adsorption cannot occur.

$$\text{Equation 1: } \theta = \frac{Kp}{1+Kp}$$

Where

θ = the fraction of the surface covered by the adsorbed molecule

K= adsorption coefficient

p= pressure

Adsorption Kinetics

Adsorption kinetics, the study of the rate at which adsorption occurs, plays a crucial role in understanding and optimizing adsorption processes for various applications. Early investigations often focused on simple adsorption systems and idealized conditions, yielding classical kinetic models such as the Lagergren first-order, pseudo-second order, and Elovich models (Musah et al. 2021) shown below.

1. Pseudo-first order model (Lagergren model)

This model assumes a directly proportional relationship between the rate of adsorption and the remaining concentration of adsorbate molecules, postulating that adsorption occurs primarily through a single-step mechanism with a constant rate coefficient.

$$\text{Equation 2: } \frac{dq}{dt} = k_1(q_e - q)$$

Where

- q_e = equilibrium adsorption capacity
- q = time adsorption capacity
- t = time
- k_1 = rate coefficient for first-order adsorption

2. Pseudo-second order model

The pseudo-second-order kinetic model, which encompasses a wider range of adsorption behaviour operates on the assumption that the rate-limiting step of adsorption involves chemisorption interactions (Hubbe, 2019).

$$\text{Equation 2: } \frac{dq}{dt} = k_2(q_e - q)^2$$

Where

- q_e = equilibrium adsorption capacity
- q = time adsorption capacity
- t = time
- k_2 = rate coefficient for second-order adsorption

3. Elovich model

The Elovich model, which accounts for energetically heterogeneous sorbent surfaces, serves as an extension of the pseudo-second-order kinetic model (Musah et al. 2021). As the adsorbed solute amount increases, the model postulates an exponential decrease in the rate of solute adsorption.

$$\text{Equation 3: } \frac{dq}{dt} = \alpha e^{-\beta q}$$

Where

- α = initial adsorption rate
- q = time adsorption capacity
- β = desorption constant

In recent years, advancements in experimental techniques and computational methods, for example, molecular dynamics simulations, have enabled a deeper exploration of adsorption kinetics at the molecular level. More complex models that consider factors like intra-particle diffusion and multilayer adsorption have also been developed.

Metal-Organic Frameworks & Activated Carbon

MOFs are materials constructed from metal 'nodes' and organic molecules known as 'linkers'. The organic ligands coordinate with the metal clusters to form various structures. These 3D frameworks are highly porous and can be filled with 'guest' molecules. MOFs possess high thermal stability due to the presence of coordination bonds. Due to their exceptional tunability, each MOF type exhibits unique properties and structures, allowing

for the tailoring of materials for specific functionalities and applications. Many MOFs have been synthesised and studied for various applications in the past, from catalysis and gas separation to drug delivery and gas storage (Zhou et al., 2012). A few well-known MOFs include:

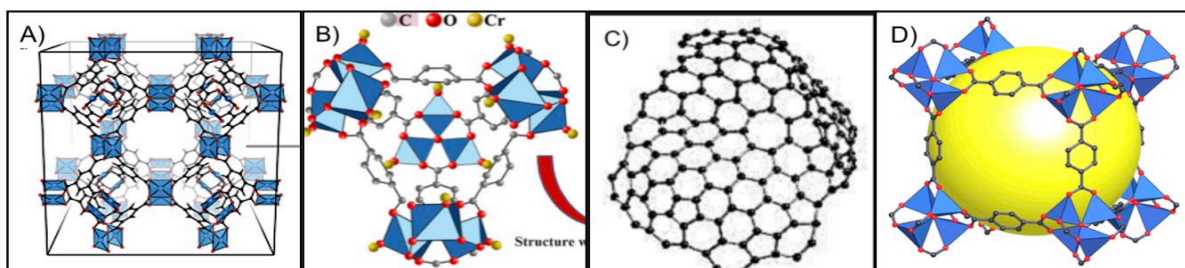


Figure 4. Various MOF Structures (Mathew, 2023, as adapted from Hendon et al., 2015; Jouyandeh et al., 2020; Harris et al., 2010; Sun & Zhou, 2015)

Fig 4A shows the structure of HKUST-1, a MOF composed of Cu ions connected by benzene-1,3,5-tricarboxylate (BTC) organic ligands. Its 3D microporous structure with large cavities and channels provides a large surface area, meaning that this MOF exhibits notable adsorption capacity for hydrogen gas, particularly at moderate pressure.

Fig 4B shows the structure of MIL-101, a MOF composed of Cr ions coordinated with benzene-1,4-dicarboxylate (BDC) organic ligands. It exhibits a large, open framework structure with well-defined mesopores and macropores, resulting in excellent hydrogen properties, with significant capacity at moderate and high pressures. It also possesses very good thermal and chemical stability.

Fig 4C shows the structure of Activated Carbon (AC), sometimes called activated charcoal, which is not a MOF but instead an amorphous highly porous form of carbon (Biswick, 2022). However, it is a subject of research in the field of hydrogen gas storage due to its favourable characteristics. Though its hydrogen uptake via physisorption is relatively low compared to MOFs, this capacity is higher for smaller pores, lower temperatures and higher pressures. It is also low density, easy to regenerate, and of abundance in nature due to biomass.

Fig 4D shows the structure of MOF-5, a MOF Composed of Zn ions and terephthalic acid (TPA) organic ligands. MOF-5 has a cubic framework structure with large, well-defined pores, however, it has moderate hydrogen adsorption properties when compared to other MOFs. It displays the highest capacity at elevated pressures, and powder densification of this material increases volumetric hydrogen uptake.

Methods

Synthesis

For MOFs, synthesis refers to the reaction between metal ions or clusters and organic ligands under specific conditions that creates these crystalline materials. There are a few different methods of synthesis such as mechanochemical, electrochemical, microwave and sonochemical, but in most cases, MOFs are synthesised under solve(hydro)thermal conditions. This refers to the reaction occurring in the presence of a solvent whilst in a closed system, with the temperature being higher than the boiling point of the solvent. As an example, an overview of the synthesis of HKUST-1 utilising the solvothermal method is outlined below:

1. A metal precursor, Copper Nitrate Trihydride, and an organic linker, Trimesic Acid, are placed in the solvent, Ethanol, using the solvothermal synthesis method at 120°C for 12 hours.
2. The mixture is then placed in the centrifuge machine so that the solid powder can be separated.

3. It will then be washed several more times with solvents before the centrifugal machine will be used once more.
4. The powder must then be placed in an oven at 100°C for the drying process so that the remaining solvent can evaporate.
5. Lastly, the activation process is carried out at 150°C in a vacuum oven to remove residual gases and prepare the metal sites for adsorption.

In the case of AC, the synthesis process is vastly different and can vary depending on the precursor it is prepared from. A wide range of carbonaceous materials may be used, such as biomass, agricultural waste, coal or wood. A general overview of the synthesis of activated carbon is outlined below (Bedia et al., 2020):

1. A suitable carbon-rich precursor can be chosen, depending on the desired properties of the AC.
2. There are two types of activation which can follow- physical or chemical.
 - Physical activation:
 - 1) The material is first carbonized at high temperatures (500-900°C) in the absence of oxygen. This process removes any volatile and non-carbon components, leaving behind a material called char.
 - 2) The material is then exposed to an oxidising environment, such as oxygen, carbon dioxide or nitrogen, at elevated temperatures (800-1000°C). This causes the material to expand and form a vast network of interconnected pores.
 - Chemical activation (single thermal step method):
 - 1) Impregnation of the precursor material with an activation agent, common ones being potassium hydroxide (KOH) or zinc chloride (ZnCl₂) occurs at a specific ratio (usually between 0.5 to 5).
 - 2) Depending on the activating agent, the mixture is then heated to a particular activation temperature in an inert gas environment, creating pores and increasing surface area.
 - 3) Lastly, the produced AC is washed thoroughly with water or acid to remove residual chemicals and impurities, before being dried to remove excess moisture.

Characterization

Characterization refers to the process of gathering detailed information and understanding the properties, structure, composition, and behaviour of a material. There are two primary methods of characterization which are employed when analysing the nature of MOFs.



Figure 5. SEM Apparatus (Author's Own) **Figure 6.** TGA Apparatus (Author's Own)

The first is scanning electron microscopy (SEM), an imaging technique used to analyse the surface morphology and topography of materials at a high resolution, providing detailed information about the surface features, particle size, shape, and distribution ("Scanning Electron Microscopy," n.d.). Electrons are generated, accelerated, and directed through lenses and apertures to create a focused beam that strikes the surface of the sample. As the electrons interact with the sample, they produce secondary electrons, backscattered electrons, and X-rays. These signals are collected and processed by one or more detectors to form images displayed on the computer screen.

The second method is thermal gravimetric analysis (TGA), a technique used to study the thermal stability and composition of a material by measuring its weight changes as a function of temperature or time under controlled heating conditions. (Garcia-Herrera & Price, 2020) During TGA, the sample is subjected to a programmed temperature increase, with an inert gas atmosphere being passed over it, and its mass is continuously measured. The changes in mass are attributed to the release or uptake of gases, the loss of volatile components, phase transitions, or thermal decomposition of the sample. By analysing the weight loss/gain patterns, as well as the corresponding temperature at which these changes occur, valuable information about the thermal stability and composition of the sample can be obtained.

Gas Uptake via Adsorption

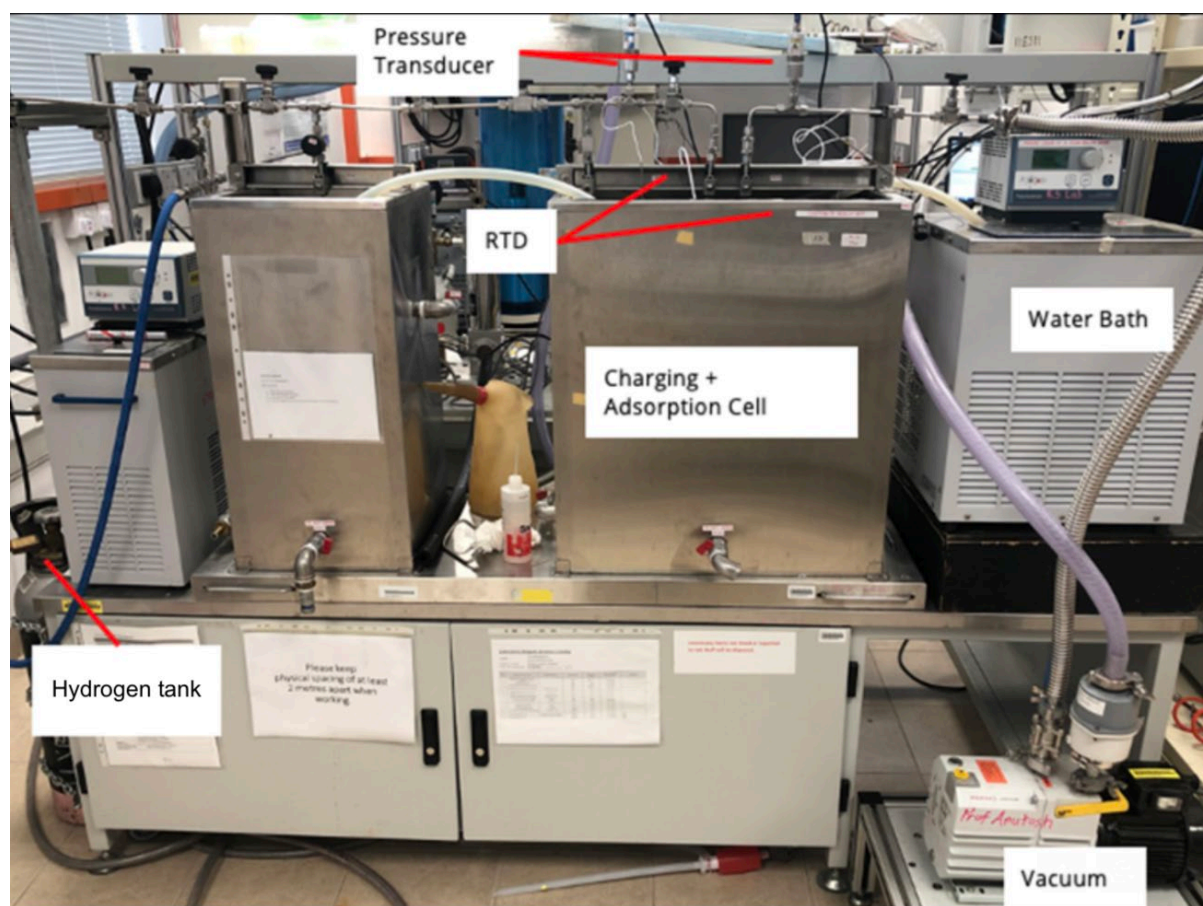


Figure 7. Schematic Diagram of Adsorption apparatus (Mathew, 2023, as adapted from Ng & Chakraborty, 2023)

The general methodology for the gas uptake experiment is outlined below.

1. Ensure that the adsorption chamber and system are empty of any gas. This is achieved by desorbing the chamber at an elevated temperature to remove any adsorbed gas. Then, allow sufficient time for the system to cool down, ideally overnight.
2. Calibrate the system using helium. Helium is commonly used as a calibration gas in adsorption measurements because it is a non-adsorbable, inert gas that allows for accurate determination of the system dead volume, V_1 .
3. Now start the gas uptake experiment with the desired gas at a pressure of 1 bar.
4. Open the valve to release it into the charging cell. Measure pressure, P_1 , and temperature, T_1 , on a data logger.
5. Wait for the temperature to stop fluctuating, returning to around 25°C.
6. Open the valve to release the gas from the charging cell into the adsorption chamber. Again, measure pressure, P_2 , and temperature, T_2 .
7. Apply the formula $\frac{P_1V_1}{T_1} = \frac{P_2V_2}{T_2}$ to solve for V_2 .
8. Apply the ideal gas law ($PV = mRT$), where $V=V_2$ to calculate the mass of gas adsorbed, m , in the adsorption chamber.
9. Repeat steps 4-8 at increasing pressures (2 bar, 3 bar, up to 6 bar, as applicable) to investigate the adsorption isotherm across the pressure range.
10. Sum up the masses of gas adsorbed across each pressure trial to obtain the total adsorbed gas quantity.

Results and Discussion

Adsorption Uptake

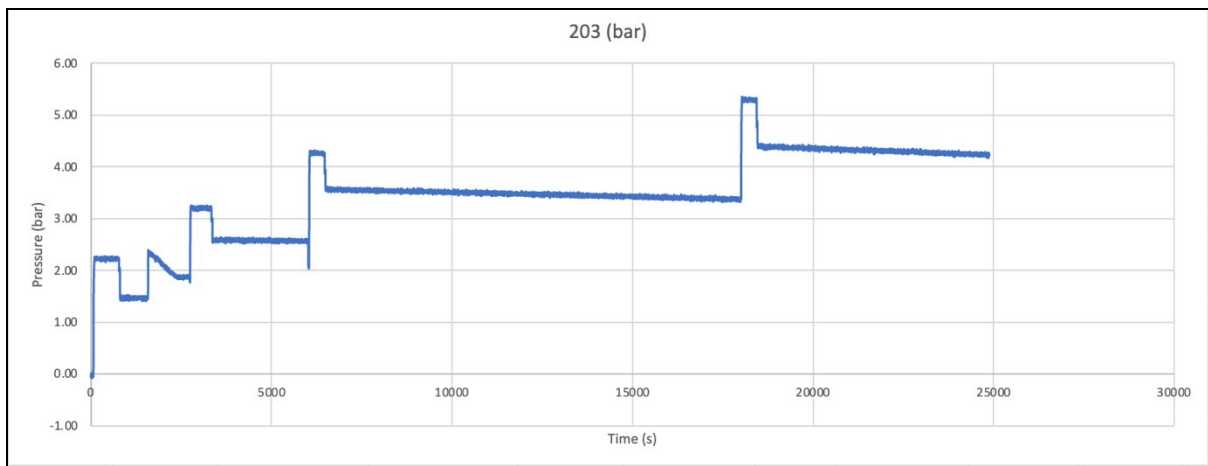


Figure 8. Pressure (bar) against Time (s) (Ng & Chakraborty, 2023)

This data is largely just used for calculation purposes but can be used to better understand the relationship between fluctuations in pressure over time. The graph starts with the initial pressure of the gas in the system, which is typically close to zero or the pressure of the background gas. As the hydrogen gas is first introduced, the pressure in the system increases rapidly from the initial pressure, and this step usually shows a sharp, near-vertical increase in pressure. After introducing the gas, the system is allowed to equilibrate, and the pressure may decrease slightly as the gas molecules initially adsorb onto the surface. As the adsorption progresses, the

pressure decreases, indicating that the gas is being taken up by the sample material. The rate of pressure decrease depends on the adsorption kinetics. The pressure eventually reaches a plateau, indicating that the adsorption process has reached equilibrium. This process is reflected by each of the square-shaped steps on the graph. The pressure of the entire system is then increased, for example from 2 bar to 3 bar, and this is then repeated. From the graph, it appears that as the pressure is incremented to higher levels, it takes a shorter period of time for the pressure to initially equilibrate, and a significantly longer amount of time at a plateau as the number of available adsorption sites on the material's surface becomes limited. The deviation in the graph seen at about 1500s can be attributed to an experimental step that occurs when opening the valve for the gas to flow.

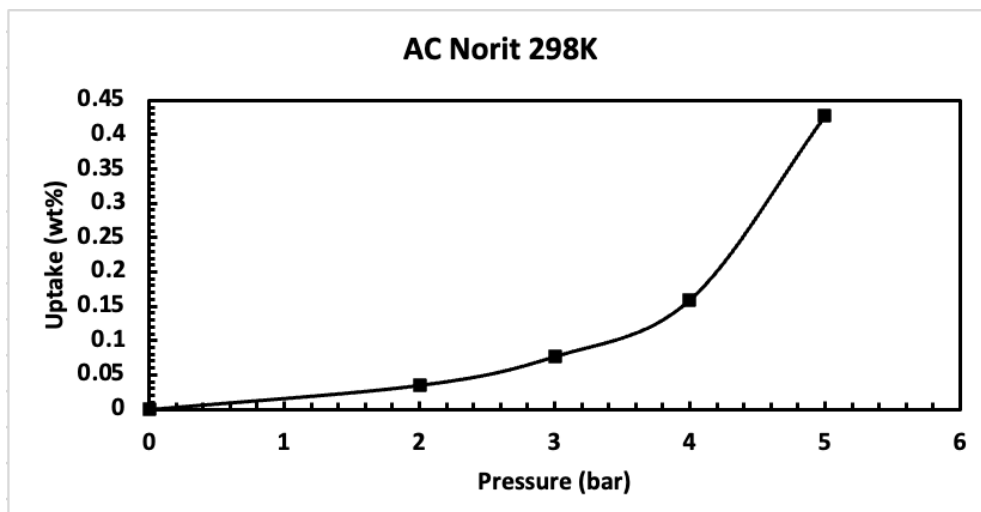


Figure 9: Uptake (wt %) against Pressure (bar) (Ng & Chakraborty, 2023)

This graph of hydrogen gas uptake (wt %) of a type of activated carbon as pressure increases is an adsorption isotherm created from experimental data. The shape of the graph produced, near-exponential growth with one main increment of uptake in the form of linear increase, which may suggest that the adsorption isotherm can be categorised as a Type III isotherm. Therefore, this graph of hydrogen gas adsorption onto activated carbon appears to support the Type III isotherm at lower pressures, however, further investigation must be conducted to confirm this reaction. From the graph, there seems to be no saturation point in the form of a plateau within the range of pressure measured, so the maximum adsorption capacity can not be determined. Extending the adsorption behaviour to higher pressures may be able to provide more information regarding the uptake capacity of this activated carbon. However, it is known that the maximum hydrogen uptake from this graph is 0.43 wt% at 5 bar and 298 K.

Conclusion

In conclusion, the study of hydrogen gas storage via adsorption on metal-organic frameworks (MOFs)/activated carbon presents a promising avenue for addressing the urgent need for sustainable energy sources. The adsorption mechanism primarily involves physisorption, where hydrogen molecules adhere to the surface and pores of the MOF framework through intermolecular forces. MOFs offer high surface area-to-volume ratios and possess tunable adsorption properties, making them attractive candidates for compact and efficient gas storage systems.

However, challenges such as the low hydrogen uptake capacity of some MOFs, requiring careful handling and storage, and their stability and durability under high-pressure conditions need to be addressed. Additionally, compatibility issues with existing hydrogen storage infrastructure pose obstacles to widespread adoption on an industrial scale, so assessing the feasibility of MOFs is an important consideration.

In the case of AC, its porous nature allows it to store a significant amount of hydrogen gas, more specifically 0.43 wt% at 5 bar and 298 K.

AC offers impressive adsorption capabilities, making it effective when storing gas in its pores. It is also extremely versatile, finding applications in various industries like water treatment, air purification, and pharmaceuticals. However, many MOFs can achieve much higher hydrogen adsorption capacities relative to AC (Li et al., 2018). Furthermore, the synthesis process can be energy-intensive, and disposal of spent AC can pose environmental concerns if not managed properly. Therefore, these concerns must also be addressed through further research.

Ultimately, efficient hydrogen storage is essential for the successful implementation of hydrogen as a clean source of energy. Through advancements in MOF and AC-based hydrogen storage technologies, we can progress towards a more sustainable future.

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