

# Effect of Impregnation Temperature on Monoethanolamine-Kenaf Biosorbent for CO<sub>2</sub> Adsorption from Gas Mixture

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## ABSTRACT

*Capturing carbon dioxide (CO<sub>2</sub>) from industrial resources to mitigate the global increase in carbon emissions is a challenging and expensive process, especially when utilizing Carbon Capture and Storage (CCS) technologies. The energy cost of CO<sub>2</sub> capture in post-combustion CCS, employing the absorption method with monoethanolamine (MEA) as the commonly used liquid amine. This study focusing on the adsorption method, which is more energy saving and less corrosive for CO<sub>2</sub> captured, by using modified kenaf biosorbent. Its low density, highly porous and permeability is well matched to be used as adsorbent. This study specifically investigated the influence of impregnation temperature of kenaf modified with a constant concentration of MEA towards the CO<sub>2</sub> adsorption capability at different gas flowrates and pressure bed. From the surface morphology of modified kenaf sorbent, MEA-kenaf impregnated at 55 °C exhibited large pore openings and more structured walls compared to the other temperatures. The kenaf samples were then utilized in IsoSORP gas analyzer for CO<sub>2</sub> adsorption with a mixture of CO<sub>2</sub> - Nitrogen (N<sub>2</sub>) at two different flow rates and various pressure beds. The highest CO<sub>2</sub> adsorption was achieved by the modified kenaf at impregnation temperature of 55 °C (gas flowrate 150 cm<sup>3</sup>/min) and*



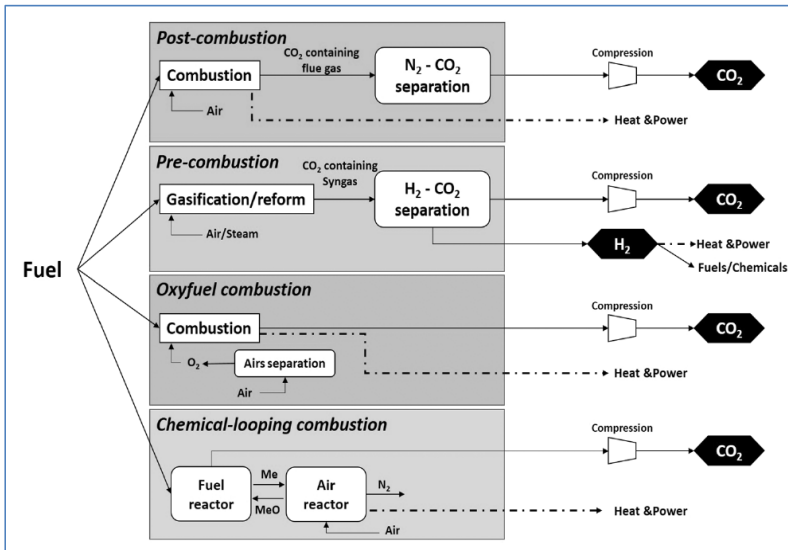
65 °C (gas flowrate 250 cm<sup>3</sup>/min). Apart from that, the adsorption ability shows a proportional increment when the gas pressure flow increased from 10 up to 30 bar. The research findings indicated that increasing the impregnation temperature enhanced the sorbent's pore size and nitrogen content impregnated on the kenaf surface (increase basicity), resulting in a significant improvement in CO<sub>2</sub> adsorption capacity.

*Keywords: Adsorption; Monoethanolamine; Carbon dioxide; Kenaf; Impregnation; Modification*

## INTRODUCTION

Carbon dioxide (CO<sub>2</sub>) emission releases carbon gas into the atmosphere, which contribute to the rapid increment in emission of greenhouse gases (GHGs), hence lead to the critical global climate change. Without rapid action, global temperatures will rise by 1.5 °C by 2030, 2 °C by 2050 as according to the Intergovernmental Panel on Climate Change (IPCC) [1]. The rises in temperature will significantly impact to the sea level rise, floods, wildfires, harsh weather, and the destruction of animal habitat (IPCC) [1]. The usage of energy as an industrial source of fuel has contributed to the expansion of the carbon emission problem, which lead to the implementation of carbon capture and storage (CCS). CCS is one of the practical solutions to mitigate the global increase in carbon emissions by the separation of fossil fuel from energy-related sources and transport to a permanently stored location. The CO<sub>2</sub> captured can be alternatively used in a variety of ways, including enhanced oil recovery (EOR), improving the growth of plants and algae, or as a raw material in the production of fuels, chemicals, or building materials [2]. Figure 1 shows the four approaches of capturing carbon generated from the primary fossil fuel: post-combustion capture, pre-combustion capture, oxy-fuel combustion capture, and chemical-looping combustion capture [3]. Post-combustion capture involves the separation of CO<sub>2</sub> from nitrogen-carbon dioxide mixture as the main constituent of flue gas generated in power plants [4]. The CO<sub>2</sub> is separated from the flue gases produced by the combustion of the primary fuel in air, providing the main advantage of being able to be adapted to any existing plants [5]. While in pre combustion technique, the initial fuel conversion steps are more elaborate and costly than in post-combustion systems. In oxyfuel

combustion systems, the pure oxygen is used instead of air for combustion of the primary fuel to produce a flue gas, which requires the upstream separation of oxygen from air, with a purity of 95–99% oxygen [6]. While in chemical absorption technique, the development of a suitable oxygen carrier offered challenge in transferring oxygen from the air to the fuel by high fuel conversion ratio, high oxygen transport capacity, and good stability [7]. Among all these possible solutions, post-combustion  $\text{CO}_2$  capture is a straightforward approach and forms the basis of the current infrastructure in CCS. The post-combustion captures capable of delivering significant emission reductions from existing large stationary sources, such as power stations and large industrial plants [8].

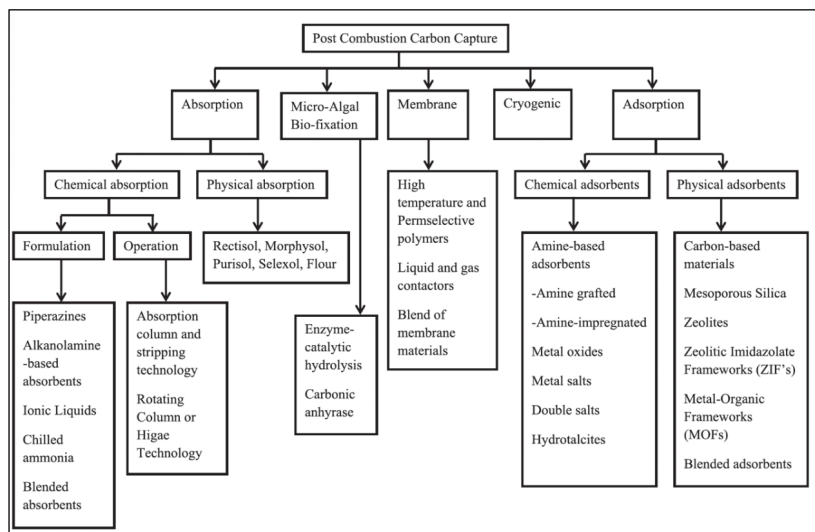


Notes: Taken from Raganati *et al.* [3]

Figure 1: Schematic diagram of CCS.

The highest priorities in the field of CCS are the development of an efficient and cost-effective  $\text{CO}_2$  capture technique, which dependent upon the principle of the capture process [5]. A few post-combustion separation technologies have been reported such as absorption  $\text{CO}_2$  separation, membrane  $\text{CO}_2$  separation, cryogenic  $\text{CO}_2$  separation, micro algal bio-fixation, condensed centrifugal separation and adsorption as shown in Figure 2 [4]. Among these, amine-based absorption is the most mature

option for post-combustion. However, this technique presents significant drawbacks when applied to post-combustion processes such as the high energy demand for sorbent regeneration, corrosion issues, amine losses through evaporation, chemical degradation of amines in the presence of oxygen, and environmental concerns regarding amine disposal [9-10]. Adsorption, relying on the ability of gases (the adsorbate) to be adsorbed on a solid surface (the adsorbent), which can be subsequently regenerated, has been receiving great research interest because of its favorable characteristics over all the other alternative solutions [11]. Adsorption occurs on the surface while absorption entails the whole material volume. Adsorption may take place physically; this will involve weak van der Waals forces (physi-sorption). It may take place chemically, which will involve covalent bonding (chemi-sorption) and it may occur due to electrostatic attraction [4]. Several important factors in adsorption include (i) ease of regeneration of adsorbed CO<sub>2</sub> [11], (ii) adsorbent's durability [12], (iii) CO<sub>2</sub> selectivity by adsorbent [3], (iv) adsorption capacity [13] and, (v) adsorbent stability after frequent adsorption/desorption cycle [14]. However, it is important to point out that other aspects, affecting the performance and overall cost of the CO<sub>2</sub> adsorption process, must be considered for the adsorption to become one of the leading capture techniques. Even though this gas separation technology is mature in several large-scale industrial applications, its application to post-combustion CO<sub>2</sub> capture still needs very intense research effort to develop, beyond suitable materials [15]. The conventional adsorbents used for adsorption process are relatively expensive, such as activated carbon, zeolite, Metal Organic Framework (MOF) [16]. Consequently, there is a growing interest on the development of low-cost adsorbents which include biological materials and industrial by-product wastes [16-17]. These factors underscore the need for further exploration and optimization of adsorbents for efficient post-combustion capture.



Notes : Taken from Ben-Mansour *et al.* [4].

**Figure 2: Post-combustion process in CCS.**

Thus, the significant role of bio sorbents in the post-combustion adsorption phase as a way of overcoming the overall cost impact in post combustion, become the focus of this research work. Numerous studies have demonstrated that peanut husks [18], sugarcane bagasse pulp [19], banana peels [20], corn cob [21], rice husk [22], and potato peel can be utilized to produce inexpensive adsorbents for the removal of pollutants [23-24]. Adsorbents derived from kenaf plant are selected due to its abundance in Malaysia and its CO<sub>2</sub> capturing capabilities. Due to its low density, highly porous, and cheaper adsorbent than other natural-based materials such as palm shell, wood pulp, coconut, and corn cob, kenaf is well-matched to be used as adsorbent [25]. Notably, kenaf can be harnessed as a natural sorbent and integrated into the post-combustion system of CCS, which stands as one of the most promising and extensively utilized methods for carbon capture. Recently, modification of the kenaf surface to improve its properties is needed as to increase CO<sub>2</sub> adsorption capability. Therefore, this study is aimed to study the ability of kenaf biosorbent with modification of amine impregnated to enhance CO<sub>2</sub> adsorption. Specifically, the investigation is focused on the impregnation temperature of kenaf modified-MEA effect on

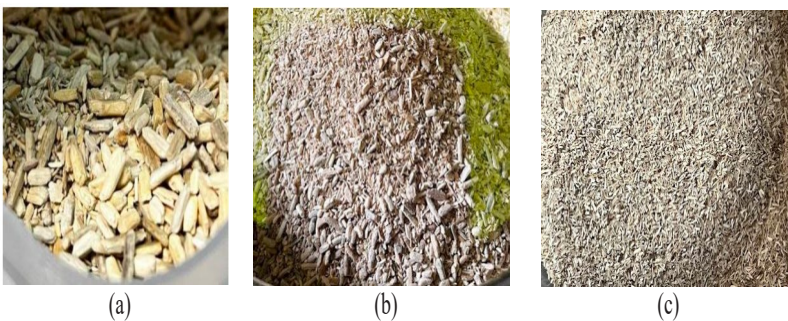
the structural properties towards CO<sub>2</sub> adsorption enhancement capability. Apart from that, the study also focused on the gas flowrate and pressure condition effects towards the CO<sub>2</sub> adsorption capability.

## MATERIALS AND METHOD

In overall, the experimental procedures involve chemically modifying on kenaf sorbent through the wetness impregnation method and analyzing the structural and elemental composition of the samples using Field Emission Scanning Electron Microscopy (FESEM) equipped with Energy Dispersive X-ray (EDX). After the structural characterization, the CO<sub>2</sub> adsorption tests were carried out using the IsoSORP Gas Analyzer.

### Materials

Kenaf chips core without outer periphery bark (kenaf bast), with the size of 3 mm to 6 mm as shown in Figure 3 was obtained from the National Kenaf and Tobacco Board (LTKN), Kelantan were used in this study. These kenaf chips were grinded with and sieved to a size of 250 µm to 500 µm. The size of 250-500 µm were selected in this study in reference to the previous research [17], where kenaf with particle size of 300–499 µm produced clear and noticeable wall construction with porous and smooth surface compared to other particle sizes.



**Figure 3: Raw kenaf chips from National Kenaf and Tobacco Board (LTKN), Kelantan at different sizes; (a) 3-6 mm, (b) grinded, and (c) sieved to 250-500 µm.**

## Method of Amine-Impregnation with Kenaf Sorbent

Before amine impregnation process, the kenaf raw with 250  $\mu\text{m}$  to 500  $\mu\text{m}$  in sizes was soaked inside methanol solvent with a mass ratio of raw kenaf to solvent of 1:20 to prepare the alcoholic solution. The 1:20 mixture was stirred with a magnetic stirrer at a speed of 600 rpm for 20 minutes. The purpose of this step was to ensure consistent amine loading on the kenaf surface and to improve the drying process of the kenaf samples. After kenaf was soaked in methanol solvent, the resulting mixture was carefully filtered through a fine mesh filter to separate the kenaf-methanol particles from the solvent. Once the filtration process was completed, the collected kenaf-methanol were set aside to air-dry naturally over the course of an entire night, allowing for the gradual evaporation of any remaining traces of excess solvent and ensuring the preservation of the modified kenaf's structural integrity. For the impregnation procedure, an alcoholic amine solution with a concentration of 200 wt.% was prepared. The MEA was combined with methanol solvent at a mass ratio of 2:1 (MEA to methanol solvent). The mixture underwent stirring for 20 minutes using a magnetic stirrer at a speed of 600 rpm. To investigate the impact of temperature, the air-dried kenaf-methanol sample was immersed in the alcoholic amine solution at temperatures of 45 °C, 55 °C, and 65 °C. Stirring of the mixture was maintained at a speed of 600 rpm for a duration of 5 hours. Subsequently, the impregnated kenaf was subjected to purging with an API filter press to eliminate the alcoholic amine, followed by overnight drying at ambient pressure and temperature.

## Structural Characterization Procedures

Field Emission Scanning Electron Microscopy with Energy Dispersive X-Ray Spectroscopy (FESEM-EDX) was used to analyse the structure and elemental composition of the raw and modified kenaf samples. The samples were conducted at Pusat Instrumentasi dan Perkhidmatan Sains UMS, PIPSUMS. The magnification chosen for FESEM analysis was X1000 and since the samples were organic material, it was coated with 100% t-butyl alcohol for 15 minutes [26]. The results gained from the structural characterization were then compared with the data from the previous research conducted by Zaini [25] and Hazimi [27]. The comparison was performed to differentiate the effect of impregnation temperature on the pore

size. This research involves examining the shape, form, and the pore size, as well as determining the elemental constituents present in the samples. Through these analyses, valuable insights can be obtained regarding the properties and potential applications of raw kenaf and amine-modified kenaf in CO<sub>2</sub> adsorption studies.

## CO<sub>2</sub> Adsorption Process

The IsoSORP gravimetric analyzer was utilized to analyze the adsorption of CO<sub>2</sub> in both raw and modified MEA-kenaf samples (45 °C, 55 °C, and 65 °C). The experimental work was conducted at Malaysia-Japan International Institute of Technology (MJIIT), UTM at Kuala Lumpur, where the IsoSORP gravimetric analyzer is located as shown in Figure 4. The gas mixture consisting of 30% CO<sub>2</sub> and 70% N<sub>2</sub> was supplied at different flow rates (150 and 250 cm<sup>3</sup>/min) and pressure beds ranging from 1 to 30 bar.



Figure 4: IsoSORP Gravimetric Rubotherm 2125 2B/277/3185 series at MJIIT, UTM.

Prior to the adsorption process, a pre-treatment method was applied to remove any moisture or previously adsorbed CO<sub>2</sub>. This involved subjecting the adsorbent to a temperature of 80 °C under vacuum conditions for 2 hours until a constant weight loss was achieved. Buoyancy measurements were conducted on fresh and regenerated samples using purified N<sub>2</sub> gas at



a temperature of 30 °C and pressure ranging from vacuum conditions to 30 bar. The samples were placed in the sample crucible on the magnetic suspension balance as shown in Figure 5. To replicate the composition of flue gas emitted from power plants, the gas mixture of 30% CO<sub>2</sub> and 70% N<sub>2</sub> was selected for adsorption measurements. The samples were evacuated for 30 to 60 minutes before the gas was introduced. The pressure was incrementally varied from 1 to 30 bar, with a gas flowrate of 150 cm<sup>3</sup>/min at a temperature of 30 °C. Equilibrium sorption was reached within approximately 50 minutes for each pressure level. Desorption was conducted at 30 bars by depressurizing the sample to a vacuum and heating it to 80 °C for 2 hours until no further weight loss was observed. The buoyancy and adsorption measurements were performed to initiate another adsorption cycle. The entire process was repeated three times, and average values were recorded. The same procedures were applied for the second gas flowrate of 250 cm<sup>3</sup>/min for all four samples (raw and impregnated kenaf).

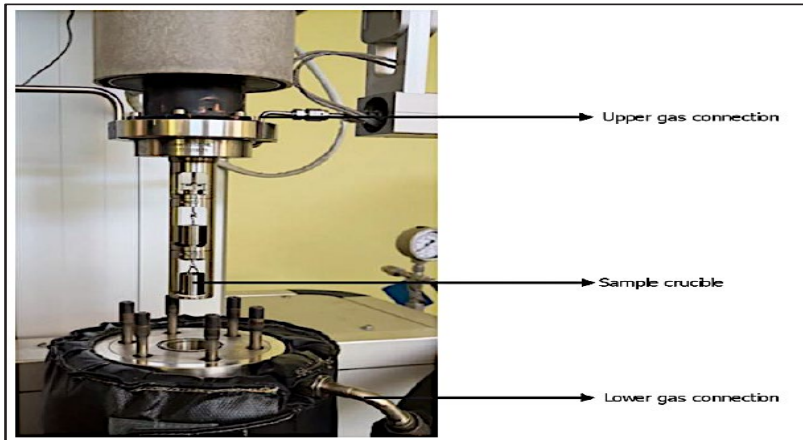


Figure 5: Magnetic suspension balance for buoyancy measurement.

The CO<sub>2</sub> adsorption capacity was calculated using the CO<sub>2</sub>/N<sub>2</sub> selectivity obtained from previous studies, and the the adsorption capacity of the samples were compared with Zaini [25] and Nurhidayah *et al.* [28]. The selectivity equation shown in Equation (1) to calculate the CO<sub>2</sub> adsorbed from the gas mixture of CO<sub>2</sub>/N<sub>2</sub>.

$$\text{Selectivity} = \frac{v_{\text{CO}_2}/v_{\text{N}_2}}{P_{\text{CO}_2}/P_{\text{N}_2}} \quad (1)$$

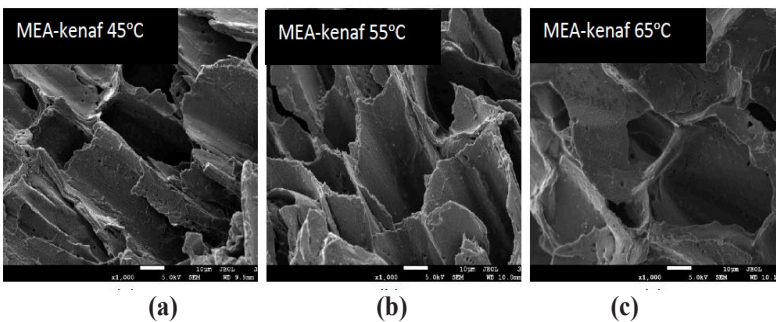
where  $v_{CO_2}$  and  $v_{N_2}$  are the adsorption capacities (mg/g) of the respective gas. The  $PCO_2$  and  $PN_2$  is the partial pressure partial pressure of  $CO_2$  and  $N_2$ , respectively.

## RESULTS AND DISCUSSION

The structural characterization of raw and MEA-modified kenaf (45 °C, 55 °C and 65 °C) were analysed through the result obtained from the FESEM-EDX test. The  $CO_2$  adsorption capacity results obtained from IsoSORP Gravimetric Gas Analyser were evaluated for the raw and modified kenaf at different gas flowrate of 150 and 250  $cm^3/min$  with different pressure (1-30 bar). The overall result was evaluated based on the raw and modified kenaf.

### Structural Characterization

The composition and surface modifications of kenaf using amines were examined in this study through FESEM. The aim was to analyse any changes in kenaf's morphology after modification under different impregnation temperatures. The results revealed structural alterations during the modification process, providing insights into the effects of temperature on the modified kenaf surface as shown in Figure 6.



**Figure 6: FESEM microscopy of MEA-modified kenaf sample at different impregnation temperature (a) 45 °C, (b) 55 °C, and (c) 65 °C (magnification: 1000x).**

The pore size, structural integrity, and wall thickness varied depending on the impregnation temperature. For example, MEA-kenaf impregnated at

65 °C had larger pore size and thinner walls compared to those at 45 °C and 55 °C. However, the higher temperature caused partial destruction of the thin walls, potentially impacting the loading of amine groups on the kenaf surface. Comparatively, MEA-kenaf impregnated at 55 °C exhibited the most favourable morphology, with large pore openings but more structured walls compared to the other temperatures. The findings demonstrate that increasing the impregnation temperature leads to an enlargement of the pore size while simultaneously thinning the cylindrical wall. Thus, higher impregnation temperatures result in larger pores and thinner cylindrical walls in the sorbent's structure [25]. The amine loadings at 200 wt.% were analysed using Energy Dispersive X-ray (EDX) at different impregnation temperatures (45 °C, 55 °C, and 65 °C) to examine the elemental composition of the samples. The focus was on Carbon (C), Oxygen (O), and Nitrogen (N) elements to determine the nitrogen percentage, which reflects the basicity of the MEA-modified kenaf sorbent. The percentage for each element is shown in Table 1.

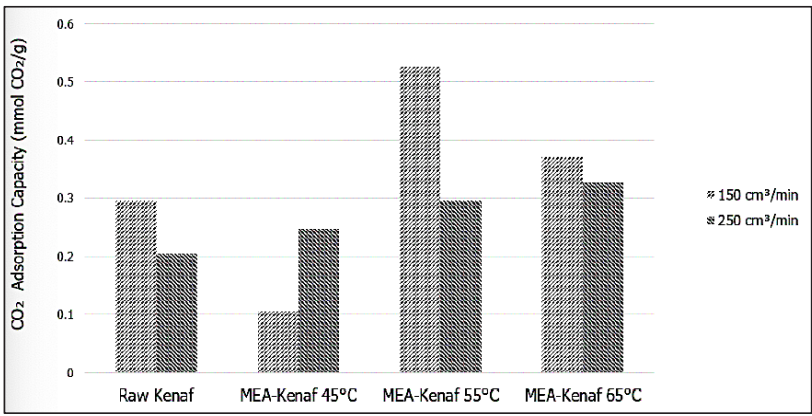
**Table 1: Elemental Composition Percentage of Raw and Impregnated Kenaf Core Adsorbent**

Sample	Elemental composition (%)		
	Carbon	Oxygen	Nitrogen
Raw Kenaf	51.06	48.94	0.00
Methanol-Kenaf	52.20	47.80	0.00
MEA-Kenaf @ 45 °C	55.10	32.10	12.60
MEA-Kenaf @ 55 °C	59.80	30.10	9.40
MEA-Kenaf @ 65 °C	56.40	31.10	11.90

Based on the data presented in the table, the carbon percentage of the amine-modified kenaf sorbent generally increases with rising temperature. However, an interesting observation is that at a temperature of 65 °C, there is a sudden decrease in the carbon percentage. Additionally, the nitrogen content of the sorbent follows a similar trend, increasing with higher temperatures. However, it exhibits fluctuations at 55 °C and then resumes an upward trend at 65 °C. The findings highlight a direct relationship between temperature and the carbon and nitrogen content of the amine-modified kenaf sorbent. As the temperature increases, there is a corresponding increase in both carbon and nitrogen percentages, indicating a potential link between temperature and the sorbent's composition.

## CO<sub>2</sub> Adsorption Capability

The main objective was to measure the CO<sub>2</sub> adsorption capacity of chemically modified kenaf, specifically when exposed to a gas mixture consisting of 30% carbon dioxide and 70% nitrogen gas. This analysis primarily focuses on comparing various MEA-kenaf sorbents with the impact of impregnation temperature (45 °C, 55 °C, and 65 °C) on the CO<sub>2</sub> adsorption capacity at two different gas flow rate, and elevated pressure conditions (ranging from 1 to 30 bar). Figure 7 shows the result of CO<sub>2</sub> adsorption capacity at different flowrate while Figure 8 shows effects of the pressure bed on CO<sub>2</sub> adsorption capacity for each raw and modified kenaf samples.



**Figure 7: CO<sub>2</sub> adsorption capacity at different flowrate for each raw and modified kenaf samples.**

Based on result obtained in Figure 7, it was observed that at higher flow rate of 250 cm<sup>3</sup>/min, the CO<sub>2</sub> adsorption capacity consistently increased from raw kenaf to amine-modified kenaf. However, at a flow rate of 150 cm<sup>3</sup>/min, the highest adsorption capacity was recorded at MEA-kenaf 55 °C, showing a sudden rise compared to other samples. From the past research conducted by Nurhidayah *et al.* [28], within the range of 100 to 300 cm<sup>3</sup>/min, the adsorption capacity initially decreased and become increasing as the flow rate increased further. At the impregnation temperature of 45 °C, the adsorption capacity of MEA- kenaf increased with higher gas flow rates. Analysis using FESEM-EDX showed that kenaf had structured cylindrical walls with higher nitrogen concentration at 45 °C, leading to improved

adsorption efficiency. At the elevated impregnated temperatures (55 °C and 65 °C) and higher flow rates (150 to 250 cm<sup>3</sup>/min), the adsorption capacity decreased due to increased amine vapor pressure and potential amine losses [29]. Consequently, this phenomenon may cause a decrease in the adsorption capacity at elevated temperatures when increasing flowrates are supplied, specifically at 55 °C and 65 °C. However, the adsorption capacity values at higher temperatures still depicts the higher values, hence this proves that the amine and methanol losses to the surroundings does not directly affect the adsorption performance of the sorbent. Fewer amine loadings would lead to reduced basicity and consequently less CO<sub>2</sub> adsorption. Basicity plays a vital role as it enhances CO<sub>2</sub> adsorption [30].

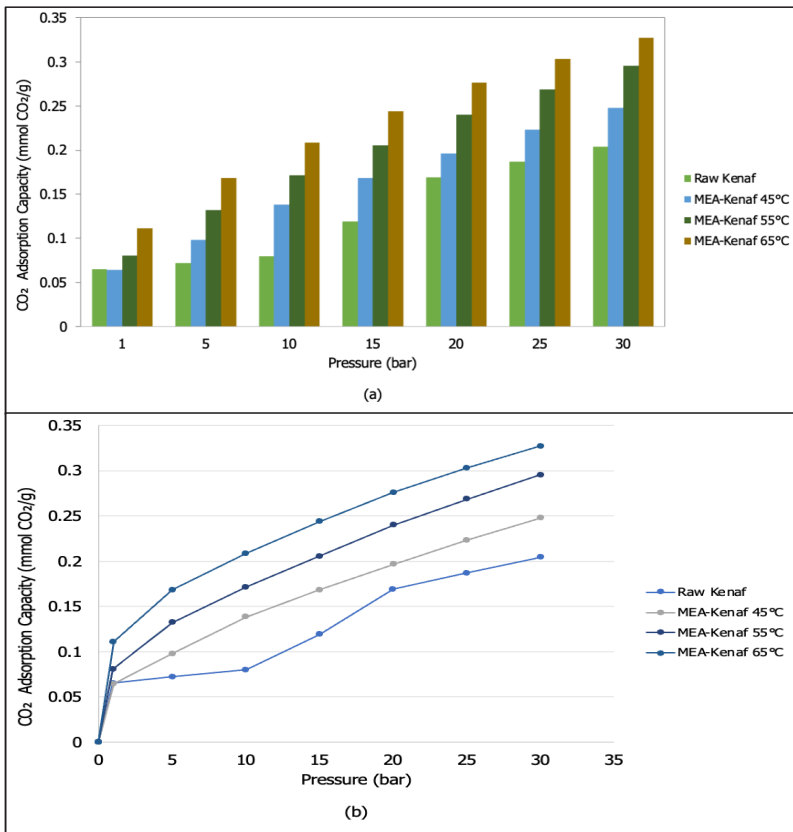


Figure 8: CO<sub>2</sub> adsorption capacity at different pressure bed; (a) bar graph, (b) line chart.

For the pressure bed variation effect as shown in Figure 8, it was found that as the pressure increased, the adsorption capacity of CO<sub>2</sub> also increased. The CO<sub>2</sub> adsorption capacity was increased proportionally with the gas pressure supplied from 1 to 30 bar at a constant gas flowrate of 250 cm<sup>3</sup>/min. This shows that at the higher flowrate, the CO<sub>2</sub> adsorption was increasing uniformly for the increment of impregnated temperature with pressure rise. According to Le-Minh *et al.* [31], it has been observed that an increase in adsorbate partial pressure results in a corresponding increase in adsorption capacity. They explained that this connection is attributed to the proportional relationship between adsorbate (gas molecules) partial pressure and the overall pressure within the system. Supporting this concept, Thomas and Crittenden [32] mentioned that this rise in adsorption capacity occurs because molecules are more densely packed together, leading to a higher likelihood of contact with the available adsorption sites. When the partial pressure of the adsorbate increases, the molecules become more densely packed, leading to a higher chance of successful adsorption onto the adsorbent material. This understanding of how adsorbate partial pressure influences adsorption capacity is crucial for the design and optimization of adsorption processes in different applications, especially in environmental engineering. Therefore, it is evident that as pressure increases, the adsorption of CO<sub>2</sub> also increases. The pressure and impregnation temperature are interconnected factors that significantly impact CO<sub>2</sub> adsorption capacity. A higher impregnation temperature expands the sorbent's pore size, providing more surface area for gas molecules interaction with the kenaf. When combined with higher pressure, this drives more gas molecules into the kenaf sorbent's pores, enhancing adsorption capacity. However, it is crucial to balance both temperature and pressure to avoid potential drawbacks. Extremely high temperatures can cause structural changes, affecting sorbent performance, while excessive pressure may lead to mechanical damage and reduced adsorption capacity.

## CONCLUSION

By utilizing MEA as the amine agent and investigating the impact of impregnation temperature, the study highlights the significance of temperature in enhancing the amine modification process and optimizing CO<sub>2</sub> adsorption capacity. The highest CO<sub>2</sub> adsorption around 0.53 mmol

CO<sub>2</sub>/g was achieved by the modified kenaf at impregnation temperature of 55 °C with gas flowrate 150 cm<sup>3</sup>/min and about 0.33 mmol CO<sub>2</sub>/g at 65 °C with gas flowrate 250 cm<sup>3</sup>/min. Apart from that, the adsorption ability shows a proportional increment when the gas pressure flow increased from 10 up to 30 bar. The result shows that the pressure and impregnation temperature are interconnected factors that significantly impact CO<sub>2</sub> adsorption capacity. It is crucial to balance both temperature and pressure to avoid potential drawbacks. Extremely high temperatures can cause structural changes, affecting sorbent performance, while excessive pressure may lead to mechanical damage. Future research should focus on further exploring the effects of elevated temperatures (100-175 °C) and flowrates (350-450 cm<sup>3</sup>/min) to determine their influence on the adsorption process. These investigations will provide a deeper understanding of the relationship between temperature, flowrate, and the adsorption capabilities of amine-modified kenaf, advancing its potential as an effective sorbent material for carbon capture applications. The abundance of kenaf in Malaysia, coupled with the availability of MEA amine, makes it an advantageous choice for reducing costs and promoting sustainability in the implementation of CCS technologies within industries. Such measures are crucial for effectively reducing carbon emissions and achieving the goal of Net Zero Emission (NZE) by 2050, as outlined in the commitments made during the Paris Agreement. Consequently, incorporating kenaf as a CO<sub>2</sub> adsorbent holds great promise for supporting sustainable and cost-effective CCS implementation in various industrial sectors.

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