



CO₂ Capture Using Amine-impregnated Activated Carbon from *Jatropha curcas* Shell

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Authors' contributions

All authors were involved in the design of the study, literature searches and statistical analysis. All authors read and approved the final manuscript.

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ABSTRACT

This study is aimed at the synthesis and amine functionalization of *Jatropha curcas* activated carbon. Waste *Jatropha curcas* shell was used to produce the *Jatropha* Activated Carbon (JAC) via chemical activation with Potassium hydroxide (KOH). The JAC was modified with triethanolamine (TEA) to produce (JAC-TEA). The performance of both adsorbents in CO₂ capture study was evaluated in a cylindrical glass column equipped with a digital mass balance. The effect of adsorbent dosage (0.5-1.5 g) and temperature (30-60°C) as a function of time was investigated. The results showed that CO₂ adsorption capacities of the adsorbents increase on amine loading and adsorbent dosage (bed height), while the adsorption capacity decreases with increase in temperature. Adsorption capacity of JAC and JAC-TEA were 66 and 78 mg/g respectively. Crystallinity, morphological structure and surface functional groups of adsorbents were characterized using X-ray diffraction, Scanning electron microscopy and Fourier Transformed Infra red spectrophotometer respectively, while the surface areas and porosity were determined by Brunauer-Emmett-Teller. Both adsorbents had good crystallinity with a well-developed pore structures. The mechanism of CO₂ adsorption onto JAC and JAC-TEA is physisorption and that the adsorbent (JAC-TEA) can be used upto 7 cycles. The results of this study have revealed that a cost-effective high quality porous activated carbon can be prepared from a cheap carbonaceous material like *Jatropha curcas* shell and modified to improve its CO₂ adsorption capacity.

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1. INTRODUCTION

Environmental pollution issues are of great concern globally in this twenty-first century. Presently, the emission of greenhouse gases such as CO₂ into the atmosphere is on the increase. Carbon dioxide emissions results from the burning of fossil fuel. Unfortunately, this additional carbon dioxide to the environment is causing havoc to the fragile balance that the earth manages naturally thereby negatively contributing to climate change and acidification of oceans [1]. The emission of carbon dioxide is also known to originated from a number of other sources which are on the increase daily as the world tends to become more industrialized [2].

According to ref. [3], the Intergovernmental Panel on Climate Change (IPCC) third assessment stated that, the atmospheric concentration of CO₂ had increased globally by about 100 ppm (36%) over the last 250 years which falls within the range bracket of 275-285 ppm to about 379 ppm in 2005. The author added that between 1995 and 2005, the first highest average growth rate of CO₂ concentration (19 ppm increment) in to the atmosphere was recorded and this surpasses records of any decade since direct atmospheric CO₂ concentration measurement began in 1950. Currently as at March 2014, the average CO₂ concentration in the earth's atmosphere has risen to approximately 400 ppm by volume [4], about 21 ppm increment which has superseded previous records.

The most promising means of reducing CO₂ emissions is by post-combustion carbon capture and storage (CCS) methodologies [5]. Post-combustion capture of CO₂ from coal-fire power plant was mostly accomplished using liquid amine scrubbing [6]. Amongst others like cryogenic distillation and membrane purification [7,8]; this is usually based on absorption processes using alkanolamine/water solutions [9] or chilled ammonia. However, this technique suffers from the harmful effects associated with the use of aqueous amine solvents, which includes equipment corrosion, alkanolamine toxicity, solvent loss and high energy consumption [10,11]. These challenges were proposed to be overcome by the use of solid adsorbents [11,12].

Amine-modified solid adsorbents make use of the amine groups grafted or loaded on the

porous materials to adsorb CO₂. CO₂ adsorption on these adsorbents is mostly a chemical process and the adsorption mechanisms mainly include: 1) under dry condition, primary and secondary amines form carbamate with CO₂ and tertiary amine works only as proton acceptor; 2) under humid condition, amines react with CO₂ to produce bicarbonate in the presence of water [13]. The amine-modified adsorbents can be prepared by two methods. One method is to physically impregnate amine into the porous materials, but the amines with low molecular weights (MW) can be leached easily in the adsorption and regeneration processes, resulting in bad reuse performance. Another method is to chemically graft amine onto the porous materials, and the adsorbents are stable in the reuse process. This method have reportedly been used for functionalization of AC from different agricultural wastes [13].

Jatropha curcas shell is a cheap carbonaceous material that is abundantly available and can easily be used to prepare high quality microporous carbon with well-developed pore structure. Some important features of *Jatropha curcas* shell that makes it a potential source of activated carbon includes; the shell's external surfaces which is full of cavities and low oxygen groups that suggest high surface area and hydrophobic characteristics [14]. Therefore, this study was designed to take advantage of some of these characteristics to further modify the activated carbon resulting from *Jatropha curcas* shell with the aim of improving its CO₂ adsorption capacity.

2. MATERIALS AND METHODS

2.1 Materials

Microporous activated carbon (JAC) produced from *Jatropha curcas* shell was used. Detail procedure and the properties of the JAC produced can be found in (M Auta et al, Unpublished results) Potassium hydroxide (KOH), Hydrochloric acid (HCl), Triethanolamine (TEA) was purchased from PANLAC Chemicals while CO₂ was supplied by Nigerian Bottling Company CO₂ Production Plant, Kaduna State Nigeria and used without further purification.

2.1.1 Choice of TEA as modifier

TEA is a liquid amine belonging to the tertiary amine group which has proven to have a high

absorption capacity and has been reported by other researcher to have been able to absorb CO₂ in molar ratio 1 : 1 (that is, 1 mole of solvent to absorb 1 mole of CO₂) which is quite high compared to its primary and secondary amine counterparts that falls around 2 : 1 molar ratio [2]; these forms the bases of its choice in this research work as the amine to be used to modify the activated carbon produced, with the aim of improving the adsorption capacity of the activated carbon.

2.2 Synthesis of JAC-TEA

A known quantity (1.9 g) of JAC was placed in a flask containing TEA solution (0.1 g of 99% TEA + 19 g of ethanol stirred for 30 min). The mixture was stirred and heated for 2 hrs at 80°C and then dried at 80°C until the ethanol had volatilized completely [15]. The impregnated sample was designated as JAC-5 indicating that the Amine loading in the composites is 5 wt %. Loadings for 2.5 and 7.5 wt % were also conducted, while JAC-2.5 and JAC-7.5 were designated respectively. JAC-TEA was designated to generally describe all the amine loaded adsorbents that is, JAC-2.5, JAC-5 and JAC-7.5 respectively.

2.3 CO₂ Adsorption Experiment

The experiments were carried out in a cylindrical glass adsorption column with a length of 14 cm and an internal diameter of 1.2 cm. The adsorption column was packed with known amount of adsorbent, weighed on a digital weighing balance before placing it within a temperature-controlled water bath while CO₂ gas was passed through it for some time after which the column was pulled out from the water bath to be re-weighed. The amount of CO₂ captured by the adsorbent was determined by the difference in weight before and after the adsorption process, until the adsorbent was full to its capacity (until no further weight increase). The adsorption process was studied at different bed heights of adsorbent (2.7, 5.4 and 8.1 cm), amine loading on activated carbon (2.5, 5 and 7.5 wt %), adsorption temperature (30, 40, 50 and 60°C). The amount of CO₂ adsorbed (q, mg/g) was determined using equation (1) below.

$$\begin{aligned} \text{Amount of CO}_2 \text{ Adsorbed} \left(q, \frac{\text{mg}}{\text{g}} \right) \\ = \frac{W_t(\text{mg}) - W_0(\text{mg})}{W_0(\text{g})} \end{aligned} \quad (1)$$

Where: W_t is mass of adsorbent at time t
 W_0 is original mass of adsorbent [16].

2.4 Adsorption Isotherm

Adsorption isotherm is a graphical representation of the relationship between the amounts of gas adsorbed by an adsorbent and the equilibrium pressure of the adsorbate at a constant temperature. In this work, the Langmuir [17] and Freundlich [18] isotherm models written in terms of pressure for gas adsorption (Equation 1 and 2) were used to analyze the experimental data obtained.

$$\frac{P}{\left(\frac{x}{m}\right)} = \left(\frac{1}{b} P\right) + \frac{1}{(ab)} \quad (2)$$

$$\log\left(\frac{x}{m}\right) = \log a + \left(\frac{1}{n}\right) \log P \quad (3)$$

where P (mmHg) is the pressure of gas, x (g) is the weight of gas adsorbed, m (g) weight of solid adsorbent, a , b and n are constants whose values depends on the nature of adsorbate, nature of solid adsorbent and temperature and were evaluated from the slopes and intersects of the plots of $P/(x/m)$ against P and $\log(x/m)$ against $\log P$ as shown in Figs. 7 and 8.

2.5 Adsorption Kinetics

The adsorption kinetic data of this work were fitted by the Lagergren pseudo-first-order and pseudo-second-order models [19]. To test the conformity of the experimental work, R^2 values were analyzed [16,20].

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (4)$$

$$\frac{t}{qt} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (5)$$

Where q_e and q_t (mg/g) are the amounts of CO₂ adsorbed at equilibrium and at time t respectively, k_1 (min⁻¹) is the pseudo-first-order rate constant while k_2 is the pseudo-second-order rate constant. k_1 was obtained from the slope of the linear plot of $\ln(q_e - q_t)$ against time while q_e and k_2 were evaluated from the slope and intercept of the linear plot of t/q_t against time. The pseudo-first-order and pseudo-second-order kinetics are presented in Fig. 9 and Fig. 10 respectively.

2.6 Desorption Study

Thermal desorption technique was used to regenerate spent adsorbent. The spent

adsorbent was weighed, introduced into a glass crucible, charged into an oven and heated. It was then removed, cooled and then reweighed. The desorption temperature and time of 100°C and 60 min were used respectively. 10 cycles of adsorption/desorption of CO₂ on adsorbent were conducted [M Auta et al, Unpublished results].

2.7 Characterization of the Activated Carbon

X-ray diffraction (XRD) measurements were carried out on a AXS Bruker advance-8 diffractometer using Cu K α radiation at a scan rate (2 θ) of 0.04° per second recording from 12° to 90°. The accelerating voltage and applied current were 40 kV and 40 mA respectively. The micrographs of the morphological structure of the plain and amine impregnated adsorbent were taken using FEI Quanta 200 Scanning Electron Microscopy (SEM) with accelerating voltage of 20 kV. Brunauer-Emmett-Teller NOVA 4200e Quantachrome NovaWin© 1994-2013 instruments version 11.03 was used to determine the surface area and porosity of the adsorbent. The functional group of the activated carbon was determined using SHIMADZU FTIR-8400S Fourier Transform Infrared Spectrophotometer.

3. RESULTS AND DISCUSSION

3.1 Characterization of the Activated Carbon

3.1.1 XRD

Fig. 1 shows the XRD pattern of JAC and JAC-TEA. The appearance of CaCO₃ compound at 2 θ = 29° revealed the crystalline nature of both adsorbent [21]. The % peak area of the CaCO₃ compound for both JAC and JAC-TEA shows a relatively equal percentage crystallinity. The % peak area of Ca₅(PO₄)₃OH compound at 2 θ = 38° - 48° that carries the hydroxyl group (OH) is

observed to increase for JAC-TEA, this is one of the factors responsible for the increase in CO₂ adsorption capacity of JAC-TEA because –OH-functional groups enhances CO₂ capture [22]. The crystalline structure observed is a tremendous development impacted on the agricultural wastes upon activation [23].

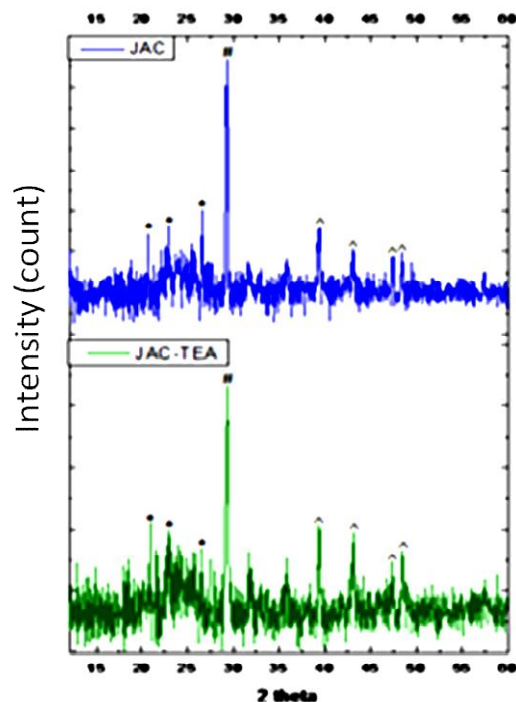


Fig. 1. XRD Pattern of JAC and JAC-TEA Compounds identified; * = C₂CaO₄H₂O; # = CaCO₃ and ^ = Ca₅(PO₄)₃OH.

3.1.2 Scanning Electron Microscopic Analysis (SEM)

The SEM images of plain adsorbent (JAC), modified adsorbent (JAC-TEA) and spent adsorbent (JAC-A) are presented in Fig. 2a-2c.

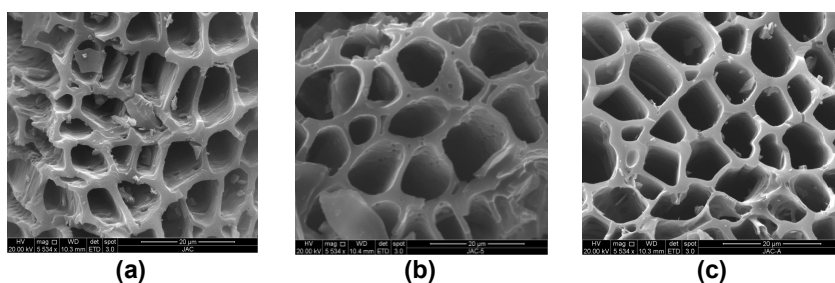


Fig. 2. SEM images of (a) JAC (b) JAC-TEA and (c) JAC-A Image magnification: x5534

Fig. 2a shows the morphological structure of JAC. It shows a well developed pore structure of a regular average dimensions as revealed by XRD pattern, while the pore structure of JAC-TEA shows a less regular dimensions due to TEA impregnated on its surface as shown in Fig. 2b. Fig. 2c is the morphological structure of a spent adsorbent JAC-A; the whitish appearance there signifies the presence of CO₂ adsorbed on the surface of the adsorbent.

3.1.3 Brunauer-Emmett-Teller (BET)

The BET analysis of JAC surface area and porosity gave surface area of 689.41 m²/g and pore volume of 0.35 cm³/g (M Auta et al. Unpublished results) which fall within the range of 0.2-0.6 m³/g for activated carbons with large pore volumes [24]. The nitrogen adsorption isotherm plotted suggested that the adsorbent is microporous in nature which is of the type I isotherm according to IUPAC classification [24]. Therefore, the large pore volume coupled with the microporous nature of the activated carbon produced from *Jatropha curcas* shell in this work can be said to be highly favourable for CO₂ adsorption. The significant surface areas recorded is an added advantage since the larger the surface area of an activated carbon, the better its adsorption capacity. In M Auta et al. (Unpublished results), it was shown that JAC has

higher specific surface area than the activated carbons (AC-EFB, AC-CNS) [25], Synthesized from oil palm empty fruit bunch and coconut shells under the same experimental conditions.

3.1.4 FT-IR

In Fig. 3, the FT-IR spectra show that the plain JAC adsorbent seems to possess the entire surface functions of JAC-TEA adsorbent but with lower intensity. 3300-3500 bands assigned to amine N-H stretch and 3750-4000 band assigned to O-H stretch of hydroxyl group are predicted to enhance CO₂ adsorption in microporous carbon materials [22]. JAC-TEA adsorbent also possesses extra oxygen – containing functional groups at 4019.79 and 4341.91 assigned to O-H vibration which is also an advantage property for JAC-TEA adsorbent as compared to JAC adsorbent.

3.2 CO₂ Adsorption Studies

3.2.1 Effect of Bed height

Bed heights 2.7, 5.4 and 8.1 cm which are equivalent to 0.5, 1 and 1.5 g respectively of adsorbent dosage were investigated. The effects of bed height of adsorbent on the amount of CO₂ adsorbed were presented in Fig. 4.

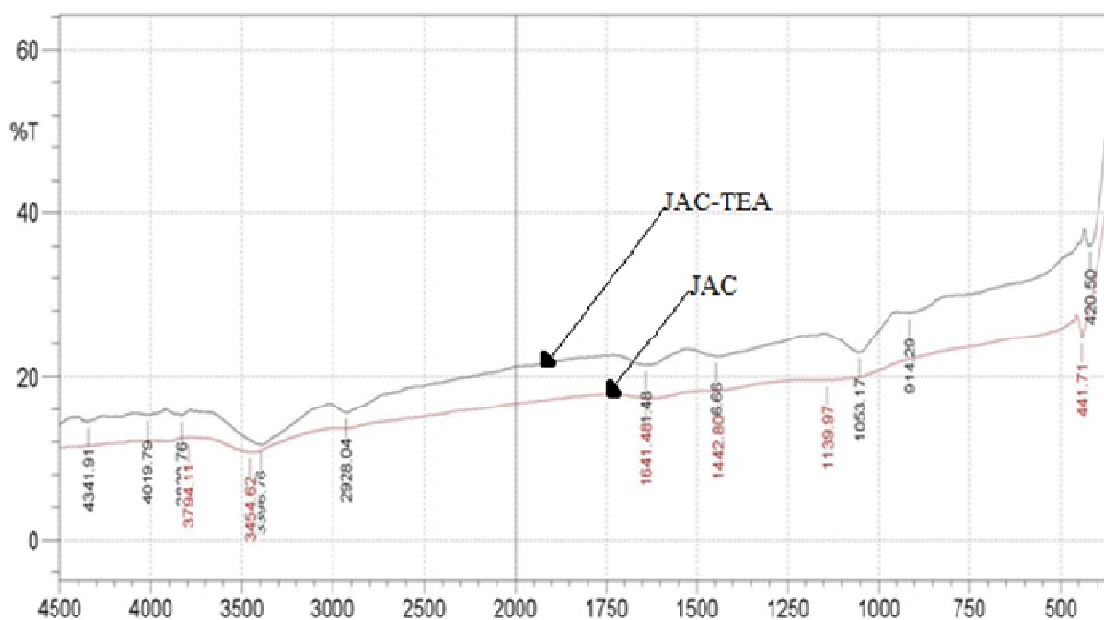


Fig. 3. FTIR Spectra of JAC and JAC-TEA

It was observed that an increase in the bed height of adsorbent leads to a corresponding increase in the adsorption capacity of CO₂. Bed heights of 2.7 cm and 5.4 cm of JAC adsorbent captures 36 mg/g and 66 mg/g of CO₂ respectively. The increase in the amount of CO₂ adsorbed was due to the increase in the active sites of the adsorbent, thus improving interaction between CO₂ molecules and adsorbent active sites. This observation shows appreciable consistency with the report of [26].

3.2.2 Effect of amine loading on activated carbon

The effect of amine loading (2.5, 5, and 7.5 w%) on CO₂ adsorption of the JAC is presented in Fig. 5.

From the concentration range studied, it was observed that adsorption capacity increased with

increase in amine loading. This observation is similar to that of the other researchers who also reported an increase in CO₂ uptake with corresponding amine loading increase [15]. The JAC-5 adsorbent gave higher CO₂ adsorption capacity of 78 mg/g as compare to the plain adsorbent JAC which was 66mg/g and JAC-7.5 which was 62 mg/g (Fig. 5).

3.2.3 Effect of adsorption temperature

Adsorption was tested under the temperature range between 30 – 60°C; this range covers the post-combustion flue gas temperature range of 35 – 45°C [9]. The results are presented in Fig. 6.

The adsorption capacity of the adsorbent JAC was observed to be decreasing as the temperature increases from 30°C - 60°C. This is a peculiar phenomenon in physisorption.

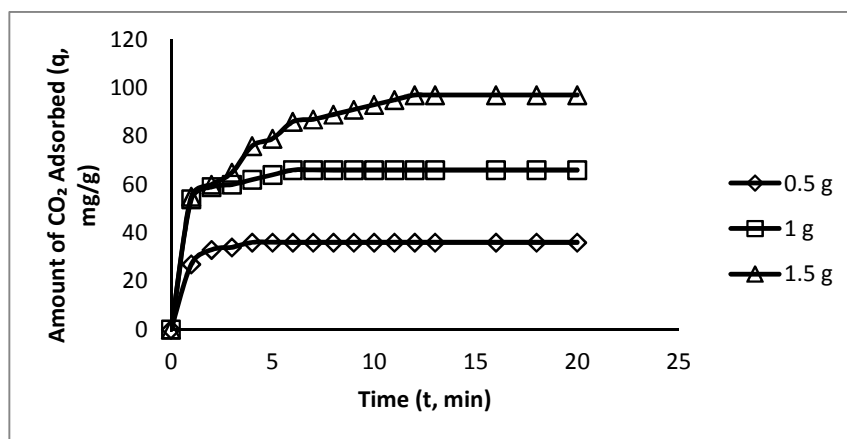


Fig. 4. Effect of adsorbent dosage (bed height) on the amount of CO₂ adsorbed

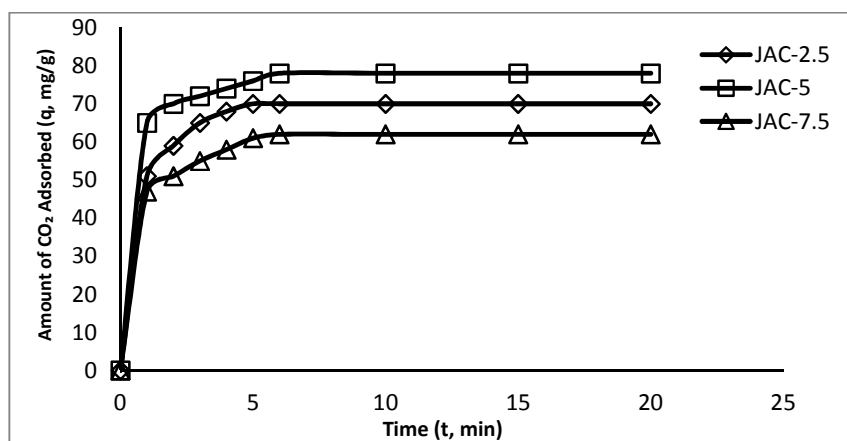


Fig. 5. Effect of amine loading on activated carbon on the amount of CO₂ adsorbed

Reference [27] reported that physisorption process involves high surface adsorption energy and molecule diffusion at elevated temperatures, which results in instability of the adsorbed gas on the surface of activated carbon and consequently, desorption process will occur. In physisorption unlike chemisorptions only weak van der waals forces exist between the adsorbed molecules of CO₂ and carbon surfaces; any increase in temperature easily breaks this bond that was why a decrease in CO₂ capacity was observed as temperature increases. This shows that the adsorption is more controlled by the physical characteristics of the adsorbent [28].

3.3 CO₂ Adsorption Isotherms

Langmuir and Freundlich Adsorption Isotherms for JAC and JAC-TEA at room temperature (28 °C) were presented in Figs. 7 and 8. It was from

this Figures that data presented in Table 1 were generated.

From Table 1, it can be observed that the experimental data was best fitted into the Freundlich isotherm model for both JAC and JAC-TEA since their correlation values (R²) of 0.9962 and 0.9945 respectively was closer to unity than the correlation coefficient values found in Langmuir isotherm model [29]. Freundlich isotherm model has been reported to be easy to fit adsorption data [30]. Failure of the experimental data to conform to Langmuir isotherm model could be due to its failure to conform to Langmuir assumption, which states that, at maximum adsorption, only a monolayer is formed and that molecules of adsorbate do not deposit on the other already adsorbed molecules of adsorbate, only on the free surface of adsorbent [17].

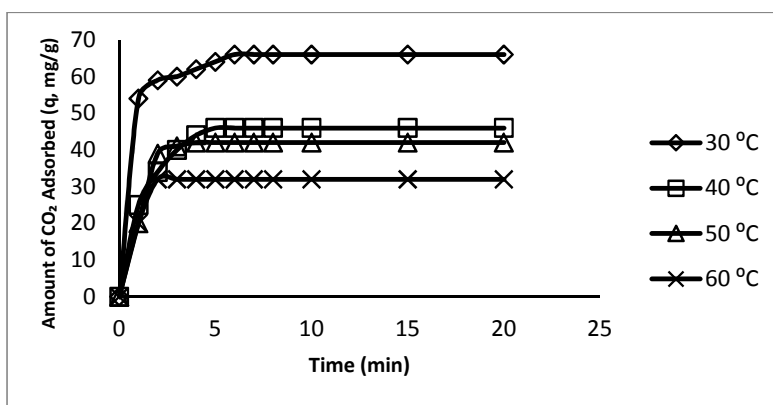


Fig. 6. Effect of adsorption temperature on the amount of CO₂ adsorbed

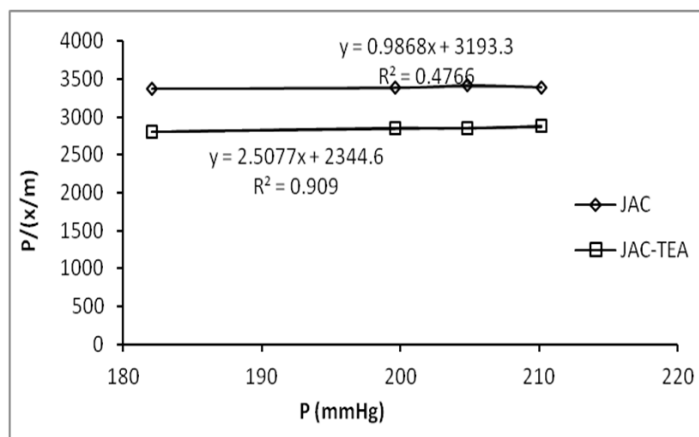


Fig. 7. Langmuir adsorption isotherms for JAC and JAC-TEA at room temperature (28°C)

3.4 Adsorption Kinetics

From the linearized Lagergren pseudo-first-order and pseudo-second-order models presented in Equation (4) and (5) respectively, k_1 was obtained from the slope of the linear plot of $\ln(q_e$

$- q_t)$ against time while q_e and k_2 were evaluated from the slope and intercept of the linear plot of t/q_t against time. The pseudo-first-order and pseudo-second-order kinetics are presented in Figs. 9 and 10 respectively which were further used to generate data presented in Table 2.

Table 1. Parameters obtained from adsorption isotherms for JAC and JAC-TEA

Adsorbent	Langmuir isotherm model			Freundlich isotherm model		
	a (mmHg ⁻¹)	b (g/g)	R ²	a (g/g)	1/n	R ²
JAC	3.10×10^{-4}	1.01	0.4766	3.97×10^{-4}	0.944	0.9962
JAC-TEA	1.07×10^{-3}	0.3988	0.909	6.28×10^{-4}	0.8912	0.9945

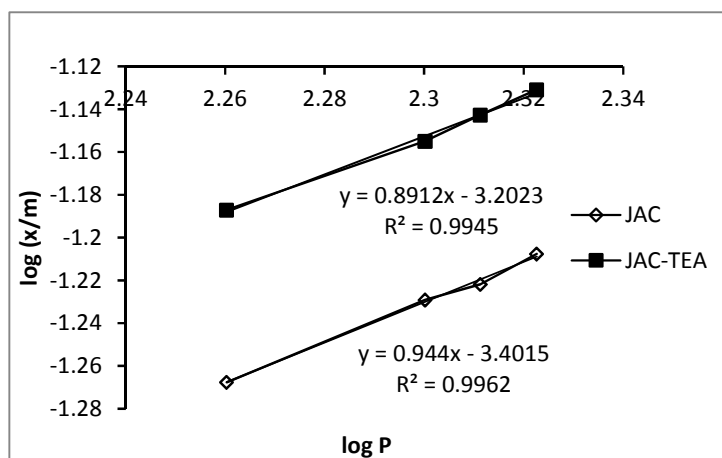


Fig. 8. Freundlich adsorption isotherms for JAC and JAC-TEA at room temperature (28°C)

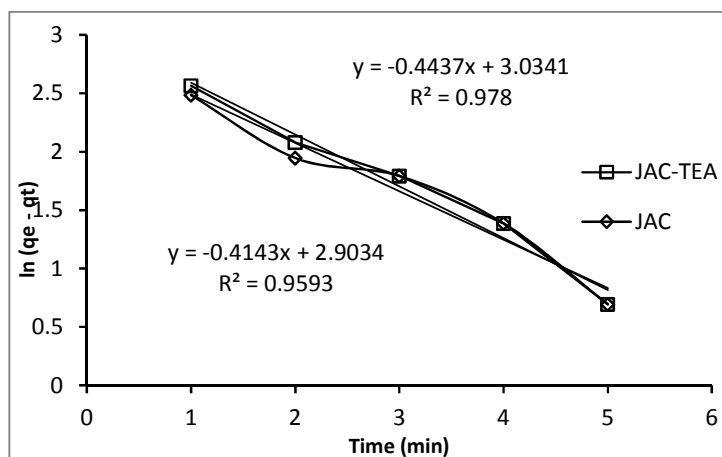


Fig. 9. Pseudo-first-order kinetics for CO₂ adsorption onto JAC and JAC-TEA

Table 2. Parameters for CO₂ adsorption kinetics on JAC and JAC-TEA

Adsorbent	Pseudo-first-order kinetics			Pseudo-second-order kinetics		
	q _e (mg g ⁻¹)	k ₁ (min ⁻¹)	R ²	q _e (mg g ⁻¹)	k ₂ (min ⁻¹)	R ²
JAC	18.24	0.414	0.9593	66.67	0.0549	0.999
JAC-TEA	20.78	0.4437	0.978	79.37	0.0496	0.9994

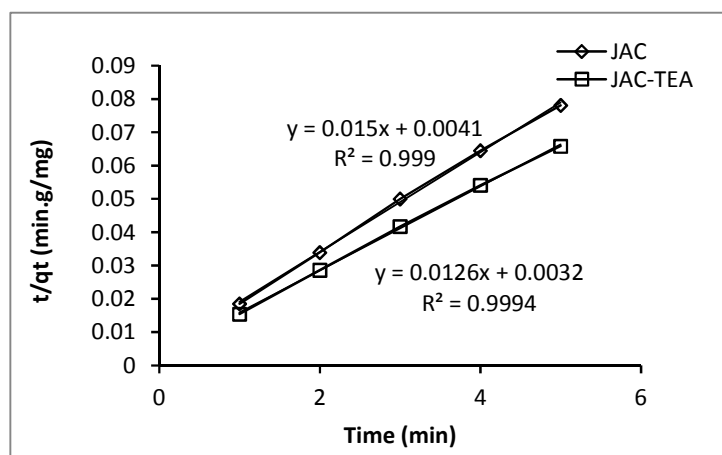


Fig. 10. Pseudo-second-order kinetics for CO₂ adsorption onto JAC and JAC-TEA

Evaluating the correlation coefficient (R^2) obtained in both pseudo-first-order and pseudo-second-order kinetics models for the adsorption of CO₂ onto JAC and JAC-TEA, it can be clearly observed that the correlation coefficients of 0.999 and 0.9994 obtained from pseudo-second-order kinetic model indicates that these systems conformed more to pseudo-second-order kinetics model [28]. The significance of its conformity can be observed from the q_e values of pseudo-second-order model, which shows the actual CO₂ adsorption capacity of the adsorbents as compared to the q_e values of pseudo-first-order model [16]. The resulting parameters of both models for the systems studied are presented in Table 2.

3.5 Desorption Study

The desorption temperature and time (100°C, 60 min) used was similar to the adsorbent pre-treatment temperature and time; this method is in line with other report by researchers who were able to successfully carry out a desorption study with the temperature used for pre-treatment [26]. JAC-TEA adsorbent was able to withstand 7 cycles of adsorption/desorption of CO₂ onto it which reveals the stability of the JAC.

4. CONCLUSION

Activated carbon from *Jatropha curcas* shell was produced, modified with TEA, after which CO₂ adsorption studies were carried out on both plain (JAC) and modified (JAC-TEA) adsorbents. The surface nature of JAC was changed after the modification, which made JAC-TEA adsorb a higher amount of CO₂ as compared to JAC. The

CO₂ adsorption capacities of both adsorbents were observed to increase on bed height increase and decrease on increase in temperature. The mechanism of CO₂ adsorption on JAC and JAC-TEA were mainly physisorption possessing low activation energy which is suitable for desorption. The results of this study shows that a high quality porous activated carbon that can be used to mitigate CO₂ emission could be produced from a cheap raw material like *Jatropha curcas* shell, an agricultural waste which suggests a possible cost-effective sorbent for CO₂ capture.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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