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Research Letter

Preparation of activated carbon from rice husk for CO_2 adsorption: Isotherm and artificial neural network modelling

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Abstract
A great deal of attention has been paid to the climate change greenhouse effect in recent years. Rice husk porous carbon adsorbent was made using a single step of KOH activation in this investigation. Carbon dioxide adsorption was studied by the generated activated carbon. For adsorption isotherm data, isotherm models such Freundlich, Langmuir, Hill, Temkin, and Dubinin-Rudeshkovich were utilised. Additionally, the capacity to adsorb CO_2 was predicted using artificial neural networks. Trial and error allowed us to choose the architecture with the best regression coefficient and the lowest mean-square error (MSE).

Introduction
Atmospheric CO_2 is currently 416 parts per million (ppm), which is 46.4% over pre-industrial levels. Fossil fuels, which will continue to be the most important source of human energy demand, will contribute to the trend of rising energy usage for a very long period.[1] As CO_2 quantities in the atmosphere increase, it is essential to create technology that can absorb CO_2 effectively.[2] A useful and significant method for reducing the rising CO_2 emissions from the use of fossil fuels is the collection and storage of CO_2 after the combustion of those fuels.[3] Adsorption is a suitable technology among those that have been proposed for the removal of carbon dioxide.[4] Adsorption technology has received a lot of attention recently with regard to preserving the environment and producing sustainable energy.[5] Adsorption utilising the materials extremely porous can show various advantages as carbon dioxide adsorbents, including consumption of lower energy, and has drawn the most interest among the many approaches.[6]

High CO_2 concentrations may be absorbed by a perfect CO_2 adsorbent, which also has great recyclability, is simple to renew, has a high CO_2 selectivity, is inexpensive, and has quick adsorption kinetics.[3,7] Metal–organic frameworks (MOFs), porous carbons and porous polymers are solid adsorbents that selectively absorb carbon dioxide at SATP (25°C, 1 bar), and they may provide a competitive alternative to amine-based solvents. Strong chemical and thermal stability, High pore volume and specific surface area and simple pore-structure manipulation are all characteristics of carbon porous materials.[8]

KOH is one of the primary activators used to create extremely porous carbon. Utilising activated rice husk carbon based on KOH (KOH-AC, 1439 m^2/g), Wang et al. created a low-cost CO_2 adsorbent. Under indoor circumstances, the adsorption rate of RHAC was 2.1 mmol/g at a lower concentration of carbon dioxide (2000–500 ppm).[1] Wang et al. produce carbon using chitosan as a precursor by combining hydrothermal treatment with light KOH activation. Despite having 1249m^2/g surface area, the AC produced from the salt-assisted hydrochar had the maximum CO_2 absorption (4.41 mmol/g). Huang et al. employed KOH in the initial phase of the conversion of garlic peel into hydrochar to synthesise activated carbon materials with high surface area and wide pores. With a surface area of 1262 m^2/g, activated carbon made by treating it with KOH at 800°C and a KOH/hydrochar ratio of 2 shows excellent pore structure.[9] To study the behaviour of CO_2 adsorption, Li et al. created biochars using 70% pine sawdust and 30% sewage sludge by KOH modification. Modified biochars demonstrated greater CO_2 adsorption capabilities than virgin biochars (35.5–42.9 mg/g), ranging from 136.7 to 182.0 mg/g.[10]

The nonlinear statistical model tool known as ANN was created in the 1980s, and today, people are employing it more and more frequently. The process is a useful tool for modelling these processes because it enables the modelling of inputs and outputs in intricate nonlinear systems.[11] To establish the link between variables, such as the dependency of estimated parameters on the adsorption mechanism, models used to explain the adsorption process should meet specific requirements. A great tool for this strategy is ANN, which can carry out mathematical operations for both linear and nonlinear systems.[12] ANN uses a learning mechanism to predict an output from an input. In that it can imitate the activities of the human brain while learning, ANN is similar to the human brain.[13]

Recently, there has been a huge interest in the ANN because of its ability to accurately anticipate complicated physical and chemical phenomena. The ANN model demands more attention to testing, validation, and data points for training and to train an ANN model, the required number of neurons cannot be calculated mathematically due to nonavailability of mathematical calculation. In terms of model accuracy, the ANN algorithm is equally important.

This paper investigated carbon dioxide adsorption on activated carbon samples, based on the previous explanation regarding natural gases. The study shows how artificial intelligence and neural network modelling may be used to forecast the adsorption capacity of the activated carbon samples stated above using various scope matrixes. With the finest applicable artificial intelligence approaches, the study may give helpful information about unconventional gas and activated carbon preparations for CO_2 adsorption and gas production.

To understand the activated carbons behaviour toward gas sorption in the subsurface, sorption analysis is normally performed traditionally at high-reservoir pressures and temperatures. The goal of doing high adsorption studies is to study supercritical CO_2 sorption and simulate field operational conditions.[14] Most practical research, additionally observations of adsorption analysis using equilibrium isotherm models, which are not always the most accurate. The majority of isotherm models have limitations in terms of pressure, porosity, and even temperature measurement. As a result, there is indeed a fundamental need for a more accurate and larger range of application modelling to improved understanding of the association between selectivity and sorption capacity and the porous medium examined, such as activated carbon.

At up to 200 bar working pressure, CO_2 adsorption analysis is frequently affected by the buoyancy effect, which must be compensated using the equation for other states. As a result, the measurement uncertainty is significantly enhanced.[15] Modifications in isothermal pressure conditions or weight of the sample are commonly used to estimate adsorption capacities, and these parameters and conditions are then correlated to modify the dominating factors and conditions. However, no research has coupled operational characteristics in the field with distribution of pores in a narrow porous medium for increased gas recovery evaluations and innovative uses.

The aim of this work is to examine the effectiveness of AC derived from rice husk as a convenient and cost-effective adsorbent for CO_2 adsorption. Here, an investigation at 273 K and 298 K for carbon dioxide adsorption is reported together with a thorough characterisation of the AC. Scanning electron microscopy (SEM), Fourier transforms infrared (FTIR), and Brunauer–Emmett–Teller (BET) have all been used to examine the characteristics of activated carbon produced chemically by activating carbon with KOH (SEM). For the purposes of this inquiry, the adsorption process was modelled using the Freundlich, Langmuir, Dubinin-Radushkevich (DR), Hill, and Temkin isotherm models. The ANN model was ultimately employed to estimate the adsorption capacity of carbon dioxide.

Materials and methods

Porous carbon synthesis

Rice Husk was collected from local rice mill. KOH is used for chemically activating biomass to produce activated carbon. The most effective and commonly used activator is KOH, as it can create a high total pore volume (especially micropores) and a large specific surface area.[1] For 24 h, the drying process was conducted at 100°C to remove any moisture from the biomass. During the four-hour calcination process, rice husks were heated at different temperatures and under different atmospheric conditions. A temperature of 10°C per minute was programmed, and a gas flow of 40 mL per minute was maintained.[16,17]

Activated carbon is the final product. The sample's powder was produced by crushing. In a single step, activation and carbonisation were applied. The precursor was submerged in an aqueous solution containing KOH for three hours. We impregnated biomass with KOH at a ratio of 1:2. The mixture was cooled after 24 h of heating at 100 degrees. The mixtures were activated for 60 min in a pure N_2 flow at 800°C after being dried for 24 h at 100°C. After being thoroughly cleaned with distilled water three times, the activated carbon was submerged in 1 M HCl and then rinsed once more to remove the chloride ions before being let to dry naturally. The final process was drying the AC for 12 h at 100°C.[18] Figure 1 shows the processes for making activated carbon.

Gas adsorption

A cylindrical reactor illustrated in Fig. S1 was used to examine the CO_2 adsorption capability of activated carbon made from rice husk. There is a cylindrical stainless steel reactor with a grid cell for exposing the CO_2 sample. The device's cylindrical shell is entirely sealed around the AC when it is installed there. The totally sealed cylindrical reactor was filled with 0.5 g of the manufactured porous carbon. Pure CO_2 is introduced into the chamber for 60 min. at 0°C and 25°C with a pressure of 0–1 bar to gauge the amount of gas absorbed.

The pressure needed to enter the mixing chamber with the CO_2 from the premium cylinder was determined using a pressure gauge and regulator. In addition to the electrical heat trace, a computer also continuously records the reactor's temperature and pressure.

Equation 1 is used to find the activated carbons adsorption capacity.

$$q = \frac{m_{\mathrm{i}} - m_{\mathrm{f}}}{w} = \left(\frac{VM_{\mathrm{w}}}{Rw}\right)\left(\frac{P_{\mathrm{i}}}{Z_{\mathrm{i}}T_{\mathrm{i}}} - \frac{P_{\mathrm{f}}}{Z_{\mathrm{f}}T_{\mathrm{f}}}\right). \tag{1}$$

$$Z = 1 + \frac{BP}{RT}. \tag{2}$$

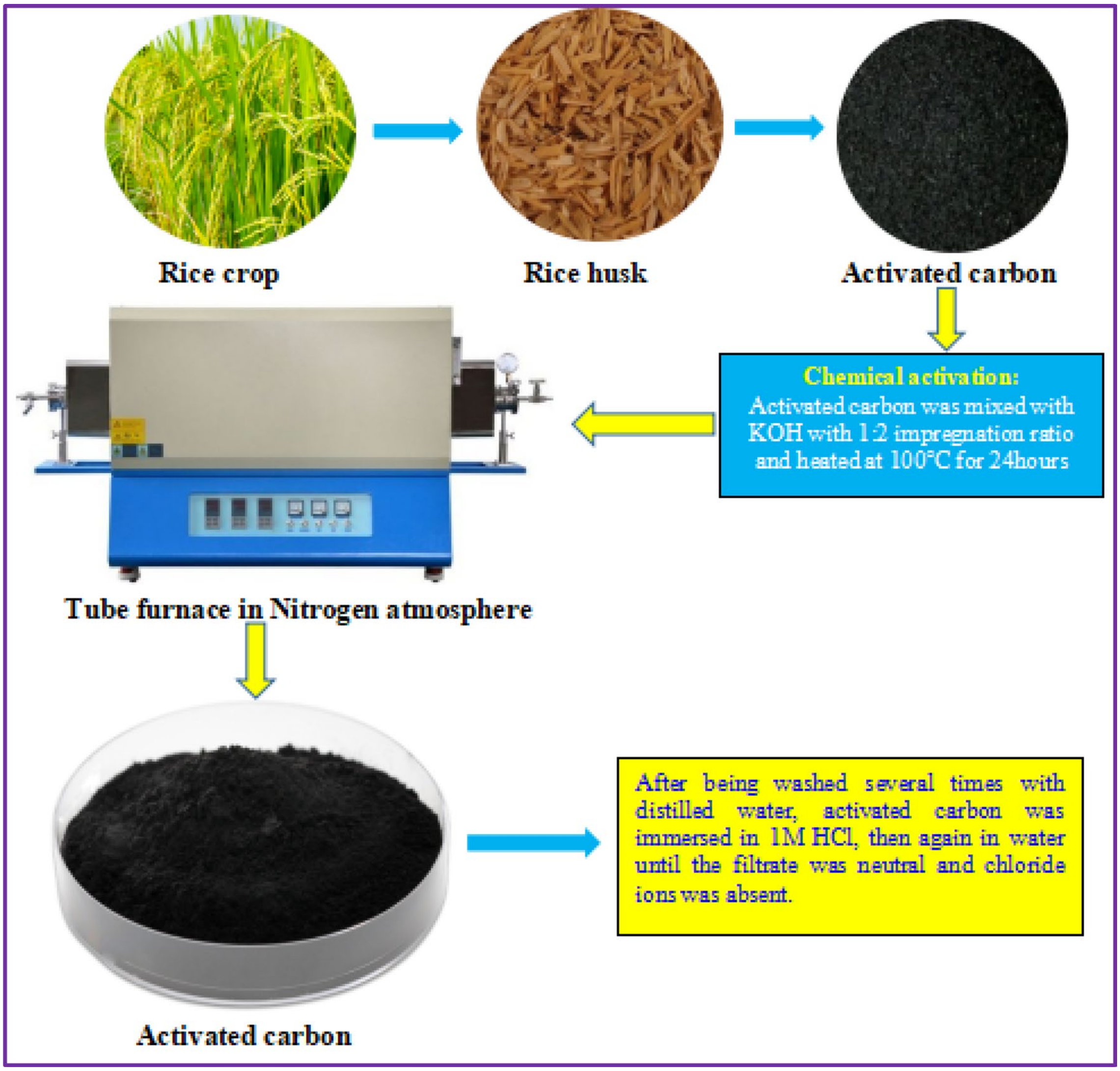


Figure 1. The preparation steps of activated carbon.

The subscripts 'i' denote initial condition and 'f' denote final condition. The variables are temperature (T), stand in for pressure (P), compressibility factor (Z), universal gas constants (R), reactor volume (V), and rice husk adsorbent mass (w). Based on the Virial equation (Eq. 2), the compressibility factor was calculated.[19]

Characterisation

A micrometric ASAP 2020 device captured the porous carbon's pore structure. Samples were degassed to constant weight under dynamic vacuum conditions for 2 h at 393 K in order to prepare them for adsorption–desorption studies. The structure and morphology of activated carbon are examined using a scanning electron microscope (SEM) [model: TESCAN Vega 3]. Functional groups on AC surfaces were found using a Perkin Elmer FTIR spectrometer.

ANN model

The idea behind the neural network is to emulate the actual human nervous system, which processes information in a complex, parallel, and nonlinear manner. A network design based on precise specifications connects the artificial neurons. A neural network (ANN) is a method that uses a sequence of three layers to translate inputs into meaningful outputs (Input layer, hidden layer, and output layer). Data are received by input layers, processed by hidden levels, and finally sent to the output layer. Every piece of data is processed by neurons in accordance with a certain activation function, such as sigmoid, tanh, linear, etc.[20] A weight value corresponds to each link. The neurons in the last layer of the hidden layer output the following equation[15]:

$$h_i = \sigma\left(\sum_{j=1}^{N} W_{ij}x_j + T_i^{hid}\right), \tag{3}$$

where N = number of input neurons, σ = activation function, W = weights, T^{hid} = threshold terms of the hidden neurons, x_j = inputs of the input neurons and.

A feed-forward radial base function (RBF) with a 2:10:1 architecture was used to simulate the CO_2 adsorption characteristics using activated carbon made from rice husk, as shown in Fig. S2. For the training sample, the data from the 38 data (70% training) and the 16 data (16% validation and 16% testing) were utilised, respectively. The buried layer neurons were activated by using the Tan-Sigmoid transfer function (tansig). Pressure and temperature were also used as input variables in the current investigation. A process response or output variable was formed by adsorption on the AC (The amount of CO_2 adsorbed). With the use of several designs and between 2 and 20 neurons, the ideal hidden layer of the ANN model was discovered. The statistical metric known as mean-square errors (MSE) is used to train ANN. The estimated MSE values are shown against the quantity of neurons in the hidden layer, as seen in Fig. S3. To obtain the least amount of mistake, 10 neurons in the intermediate layer were specifically chosen. Equations 4 and 5 were used, respectively, to determine MSE & correlation coefficient (R^2).[21]

$$\mathrm{MSE} = \frac{1}{N}\sum_{i=1}^{N}\left(Y_{\mathrm{predicted}} - Y_{\mathrm{real}}\right)^2, \quad (4)$$

$$R^2 = \sum_{i=1}^{N}\frac{\left(Y_{\mathrm{predicted}} - Y_{\mathrm{real}}\right)^2}{\left(Y_{\mathrm{predicted}} - Y_{\mathrm{mean}}\right)^2}. \quad (5)$$

Results and discussion

Pore size distribution and N_2 adsorption–desorption

On rice husk-derived activated carbons, CO_2 adsorption tests were performed at STP from 0–1.0 relative pressure (P/P_0). The progressive absorption of nitrogen at 77 K is shown in Fig. 2(a). The curve showed a high rise of 500 cm^3/g at low relative pressures ($P/P_0 < 0.02$) followed by a second fast rise up to 0.85 bar. The curvature then progressively grew until it reached 1 bar. These discoveries led to the conclusion that the material had micropores, which could be seen in the pattern of the pore size distribution [Fig. 2(b)]. There seems to be a slight hysteresis between absorption and desorption as a result of the big spaces trapping gas. The result was that the SBET volume and the volume of the micropores were 2458 m^2/g and 0.76 cm^3/g, respectively. This substance is more valuable than the majority of porous carbons.

Scanning electron microscopic analysis (SEM)

A scanning electron microscope (SEM) operating at 25 kV was used to analyse activated carbon made from rice husk. As observed in the SEM picture (Fig. S4), AC usually consists of a structure that is rather smooth with irregular holes on the surface.

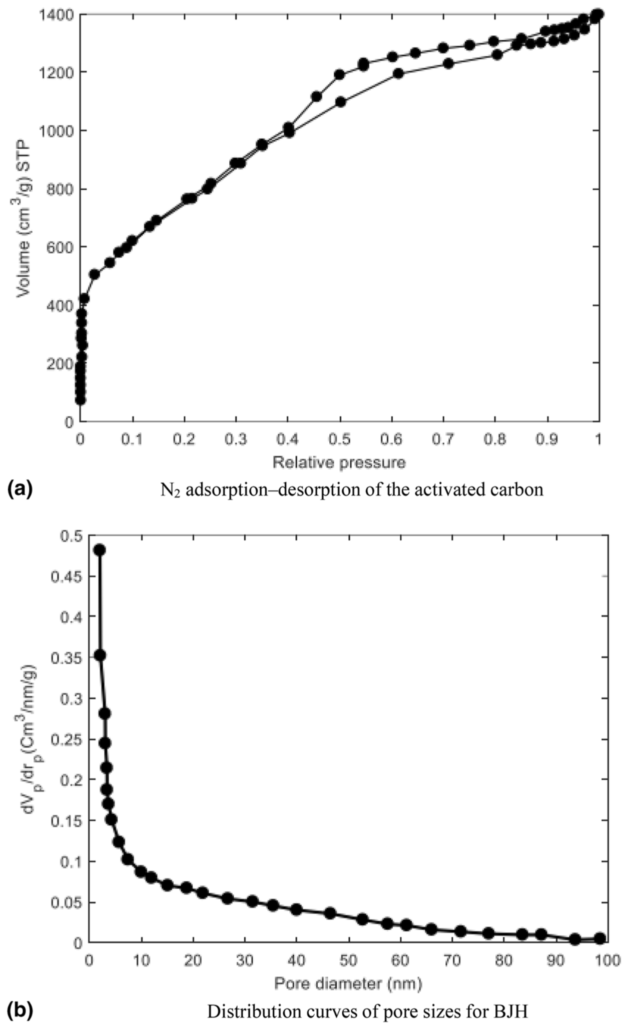


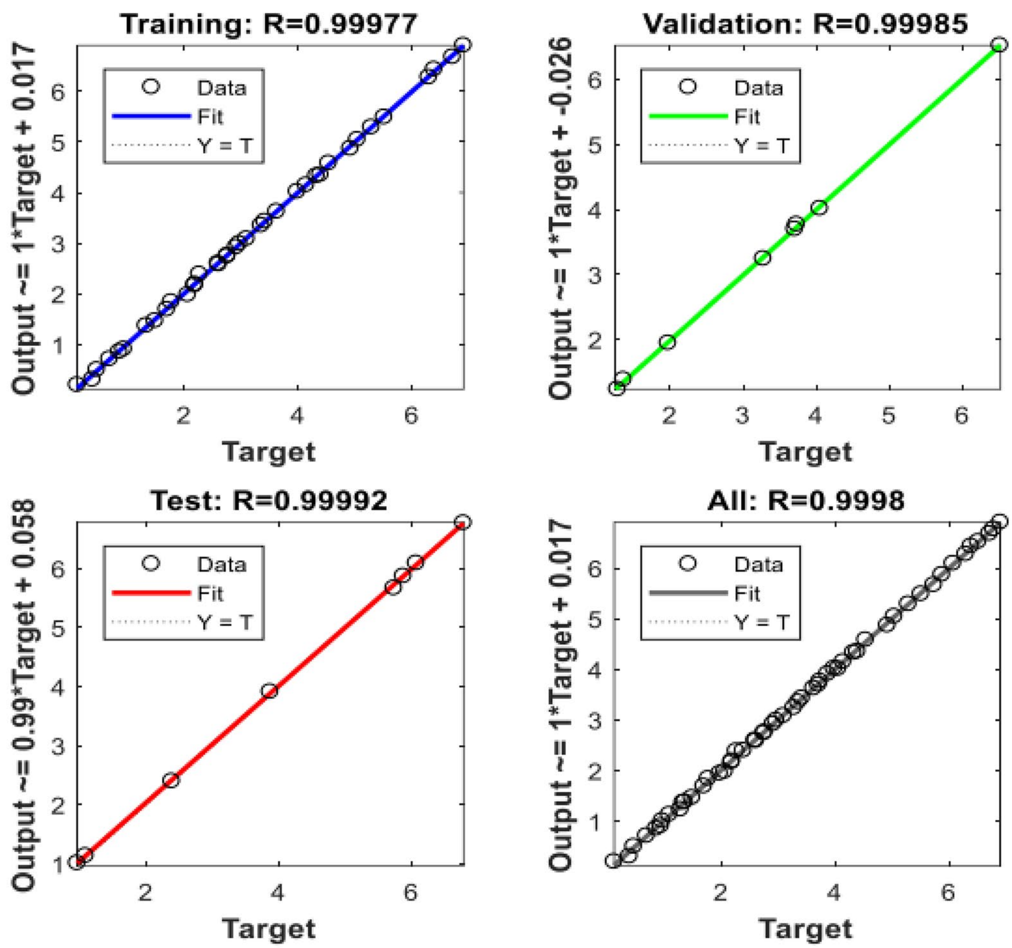
Figure 2. (a) N2 adsorption–desorption of the activated carbon. (b) Distribution curves of pore sizes for BJH.

Fourier transform infrared analysis (FTIR)

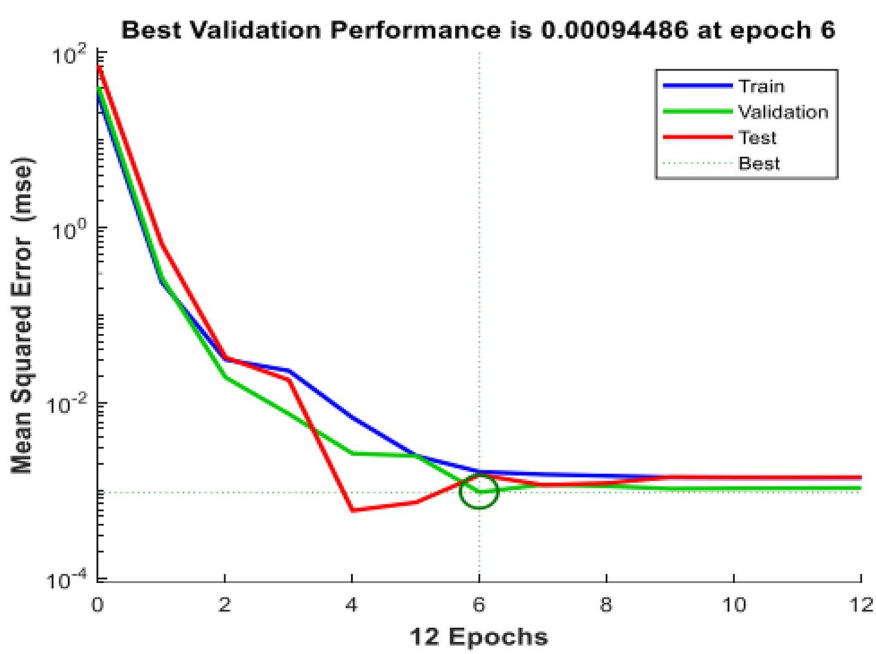
The adsorbent's FTIR spectrum is crucial for determining if the appropriate functional groups and bonds are present. The activated carbon made from rice husk may be seen in Figure S5 FTIR spectrum. hydroxyl group stretching in the presence of -OH evidenced by the peak at 3430 cm^{-1} is caused by. Methoxyl group molecular vibrations may be observed at a wavelength of 2924 cm^{-1}, which is related to the symmetric and asymmetric vibrations of C–H. Vibrations from the C–O are said to have a peak at 1057 cm^{-1}.[18]

Modelling of CO2 adsorption by ANN

To determine the adsorption capacity the Neural Network Toolbox from MATLAB 2017a is used. The artificial neural network used in the current study has two inputs, a hidden layer with 10 neurons inside of it, and one target output. Best correlation coefficient and lowest error(MSE) architecture were ultimately chosen after much trial and error. Figure 3(a)



(a) ANN regression data for training, test and validation steps



(b) The validation performance of the best ANN

Figure 3. (a) ANN regression data for training, test, and validation steps. (b) The validation performance of the best ANN.

displays the outcomes of the experiment and the CO_2-adsorbed output values of the neural network model. The determination of linear coefficient for entire set of data, according to the findings, was 0.9998. From the modelling data, ANN model can accurately estimate CO_2 adsorption on rice husk-activated carbon. The network obtains the greatest degree of validation according to the performance graph [Fig. 3(b)]. The outcomes of the ANN model for forecasting ANN weights are shown in Table AI based on the CO_2 adsorption network's optimal weight.[22]

The effect of temperature and pressure

Figure S6 depicts a CO_2 adsorption isotherm at 273 K and 298 K. The activation of rice husk-based activated carbon is significantly influenced by the isotherm. Figure S6 shows how as the adsorption temperature rises, the quantity of CO_2 that can be absorbed declines. Because adsorption is exothermic, its quantity reduces with rising temperature. It rises with pressure. The carbon dioxide adsorption comparison by several activated carbons are listed in Table S1. The present activated carbon from rice husk virtually performs the same as the others and reveals the outstanding carbon dioxide adsorption at 273 K and 298 K when the observed carbon dioxide adsorption capacity is compared to that reported in previous research, according to the comparison. The findings of this study can be utilised to create new rice husk-activated carbon that is efficient in absorbing CO_2.

Isotherm modelling

The adsorption rate of carbon dioxide depends on gas pressure. Figure 4 shows adsorption curve of carbon dioxide at atmospheric pressure and at temperature 273 and 298 K. The lower binding strengths of the carbon dioxide adsorbate and the Rice Husk-activated carbon can be used to explain why CO_2 adsorption capacity declines with increasing temperature in Fig. 4.[23] Additionally, the CO_2 adsorption capacity decreases at high temperatures, indicating an exothermic sorption process and the presence of physical sorption. Unlike chemisorption, which is mediated by a strong van der Waals force, physical adsorption relies on a weaker van der Waals force, which tends to break down as temperature rises.[23,24] Table S2 predicts the isotherm constants and associated R^2 values for the CO_2 adsorption using the regression approach.

The Hill isotherm is the best match for the adsorbent at 273 K based on the R^2 values in Table S2. (Fig. 4). Based on R^2 values, Table S2 demonstrates that Freundlich and Hill isotherms (Fig. 4) are the best-fit models for the adsorption of activated carbon from rice husk at 298 K. According to the Freundlich model, carbon dioxide adsorption on the surface of activated carbons is multilayered rather than limited to a monolayer.[23] The Hill model is frequently used to describe how various species attach to homogenous surfaces.[25] Endothermic desorption is more beneficial at high temperatures, which lowers CO_2 adsorption capacity. According to Table S2's Freundlich constant, n, the CO_2 adsorption will be successful. The Dubinin-Radushkevich and Temkin isotherms are also helpful in providing information on A_T (heat of adsorption) and adsorption mean free energy(E). These are the energy parameters. The CO_2 adsorption process is entirely physical, as demonstrated by energy parameter values of 3 and 4 kJ/mol.[26]

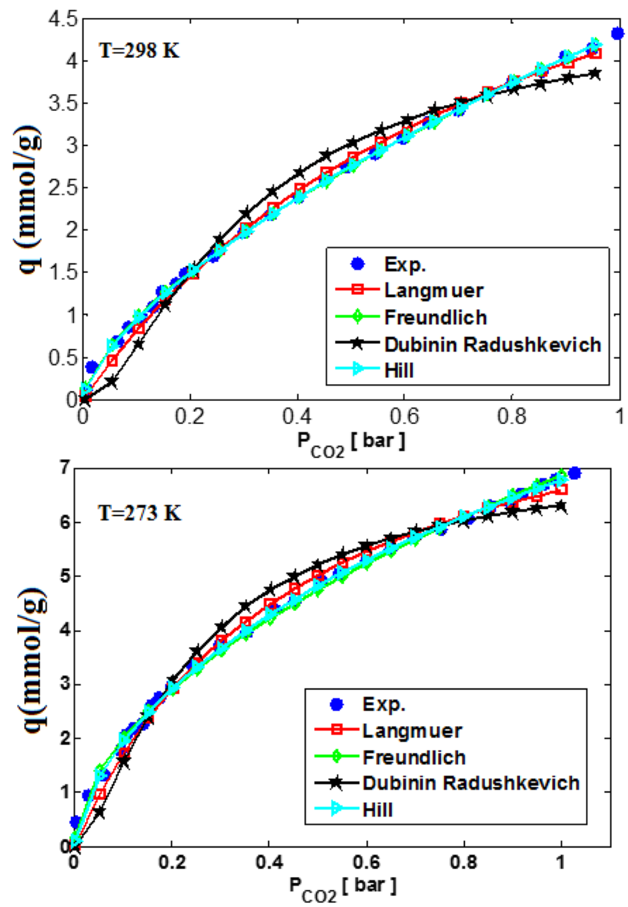


Figure 4. Experimental data and isotherm models at 273 K and 298 K for CO2 adsorption.

Conclusion

KOH effectively activated and carbonised a carbon produced from biomass to obtain significant CO_2 adsorption capacity. At a holding temperature of 60 min, the activation was carried out above 800°C. Using scanning electron microscopy (SEM), Fourier transform infrared (FTIR), and Brunauer–Emmett–Teller (BET), the structure and characteristics of AC have been investigated. The total pore volume and BET surface area 2458 m^2/g and 0.76 cm^3/g respectively. With a strong CO_2 absorption of 6.76 mmol/g at 0°C and 1 pressure, this carbon exhibits. Lower binding strengths between the adsorbate and the activated carbon might be the cause of the declining CO_2 adsorption capability with rising temperature. At 289 k and 1 bar, AC-KOH had an adsorption capacity of 4.31 mmol/g. Based on R^2 values, Freundlich and Hill isotherms are the best-fit models for the activated carbon adsorbent at 298 K. The Hill isotherm is the model that fits the adsorbent at 298 K the best. Results indicate that the linear correlation coefficient for the entire set of data was 0.9998. The ANN outcomes demonstrated how well the neural network model could forecast CO_2 adsorption on activated carbon. The models' predictions of process performance were largely accurate, according to their findings.

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Data availability

The datasets generated and analysed during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of interest

On behalf of all the authors, the corresponding author states that there is no conflict of interest.

Appendix

See Table AI.

Table AI. The weights of RBF network with Levenberg–Marquardt algorithm.

Neuron	1	2	3	4	5	6	7	8	9	10
1W{1, 1}	−0.95205	−4.6498	−4.2967	−4.3362	−3.4764	−4.0559	−3.7245	1.1675	−2.4699	0.7053
	5.263	−0.65388	0.79438	−1.265	−3.5654	−0.59199	2.89	4.5297	−1.8386	−4.5716
b{1}	3.585	2.9496	2.56	1.1842	−0.26483	−0.79994	−1.1087	2.0934	−4.8113	4.2445
LW{1, 1}	−1.6906	0.45472	−0.08057	−0.06529	−1.9462	−0.1839	−0.67141	−0.22446	−0.92397	−0.21734
b{2}	−1.1291									

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1557/s43579-022-00262-w.

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