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Adsorption of Carbon Monoxide by Jordanian Zeolitic Tuff

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Abstract

The objective of this work was to evaluate Jordanian zeolitic tuff for the adsorption of carbon monoxide. Samples of naturally occurring Jordanian zeolite were collected from southern Jordan at Al-Tafila Government/Al Hala Volcano and physically treated, then evaluated for carbon monoxide adsorption in a fixed-bed column. The effect of pressure, temperature, and zeolite particle size was studied. The adsorption isotherm and the heat of adsorption were obtained. The results indicated that the adsorption isotherm fits well with the Langmuir isotherm, and the saturation adsorption capacity was found to be about 10.1 mg CO/g zeolite. The temperature and zeolite particle sizes were found to have a significant effect on the adsorption. The adsorption loading decreased with increasing temperature and increased with decreasing particle size. An empirical equation for the dependence of adsorption loading on temperature and particle size was derived, and the corresponding heat of adsorption was determined. The heat of adsorption was found to be exothermic below 55.5°C and endothermic above it.

Paper type: Research Paper.

Keywords: Carbon monoxide adsorption, Jordanian natural zeolite, Fixed bed adsorption, Acid gases

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1. Introduction

Carbon monoxide is a poisonous and very corrosive gas (Chenoweth et al., 2021; Ashokkumar et al., 2021), therefore, removing this gas from process streams is essential as a first step in gas treatment processes. Natural gas, Liquefied petroleum gas (LPG), and exhaust vehicular gases are common examples where carbon monoxide needs to be removed. Several methods can be used to remove CO gas. The most common methods are absorption into liquids, adsorption onto solids, and catalytic conversion (Ashokkumar et al., 2021; Luis et al., 2023; Thakur, 2019). Absorption into liquids is the most common method for treating high gas flow rates, while adsorption onto solids is usually used for low flow rates or low carbon monoxide concentrations. Catalytic conversion is usually used in vehicles to convert CO to CO_2 (Luis et al., 2023).

Adsorption is the process by which molecules of gas adhere to the surface of a solid or liquid. It is entirely a surface-based phenomenon, creating a thin film of the adsorbate. Adsorption onto solids has been widely used to clean or capture certain gases such as CO_2, H_2S, NO_2...etc. (Mohammadi et al., 2023). Activated carbon, alumina, zeolite, and other adsorbents have been extensively used for this purpose (Thakur, 2019; Condon, 2019). The objective of this work was to evaluate Jordanian zeolite for the removal of carbon monoxide. Zeolite in Jordan exists in large quantities and in different locations as shown in **Figure 1** (Al Dwairi, 2007). Even though zeolites share the same framework, the attached cation, composition, concentration, and surface area vary across naturally occurring

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types. More than 30 types of zeolite crystals have been found in natural deposits; many can also be synthesized industrially (Auerbach et al., 2003).

Jordanian zeolite exists in large quantities and relatively low prices, therefore, its evaluation for different industrial applications is very important for finding new practical application for this Jordanian mineral (Alnawafleh et. al., 2013). Recently, Jordanian zeolites found successful applications in industry such as water and wastewater treatment (Seader et al., 2016; Saleh, 2022; Aljabarin, 2023), removing poisonous/odor gasses such as H_2S from gas streams (AL Jarrah, 2022a; Liu and Wang, 2017), capturing CO_2 from gas streams (AL Jarrah and Al Dwairi, 2022), air separation (AL Jarrah, 2022b), agriculture (Al Dwairi, 2014), and as a decorative material.

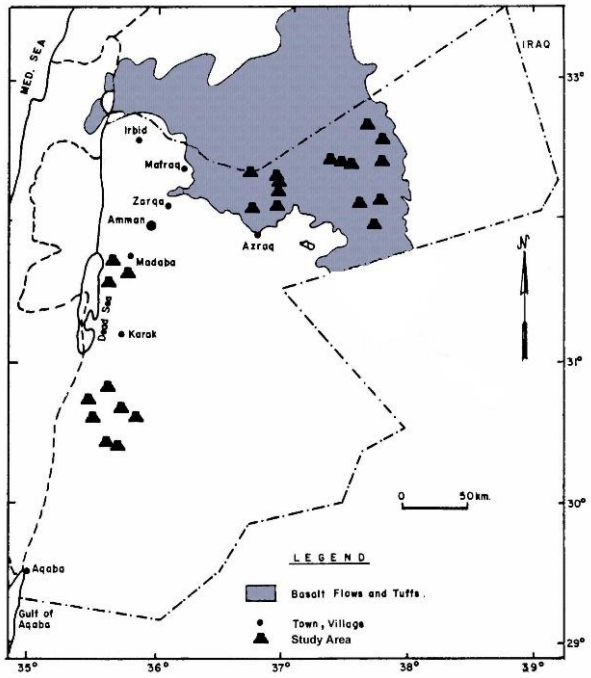

Fig. 1. Main Locations of Zeolite in Jordan. Reproduced from R. Al Dwairi, Characterization of the Jordanian zeolitic tuff and its potential use in Khirbet es Samra wastewater treatment plant, PhD Thesis, University of Jordan, 2007, with permission.

Pure zeolites can be defined as a crystalline microporous material composed of hydrated aluminosilicates with a composition described as shown in **Figure 2** (AL Jarrah, 2022c).

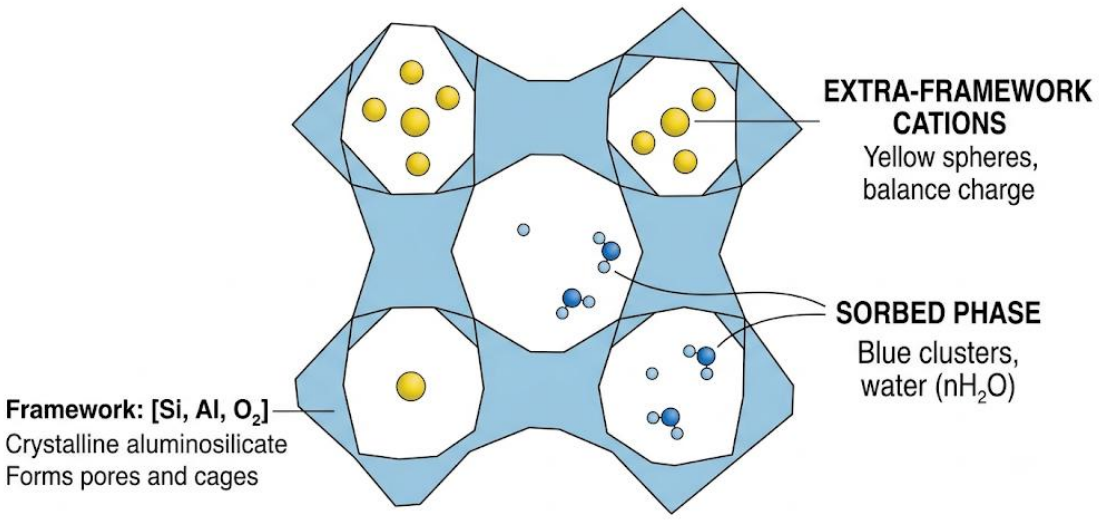

Fig. 2. Schematic Presentation of Zeolite Framework.

The extra framework cations are exchangeable ions that can be replaced by other ions as, for example, in ion exchange technique used in water treatment. In addition, because of the high surface Volcano of zeolites due to microporosity, some components can adsorb onto the surface of zeolites (Van der Waal Forces) as in the adsorption of CO onto zeolite in this work.

Absorption of carbon monoxide has been evaluated by many researchers for different types of zeolites over wide temperature and pressures ranges (Davarpanah et al., 2020; Ko et al., 2020; Feng et al., 2020; Ma et al., 2023). On the other hand, Jordanian zeolites have not been yet evaluated for adsorption of carbon monoxide. The composition of zeolite in Jordan changes slightly from place to place. **Figure 3** shows the average composition of southern Jordanian zeolite (Al Dwairi 2007).

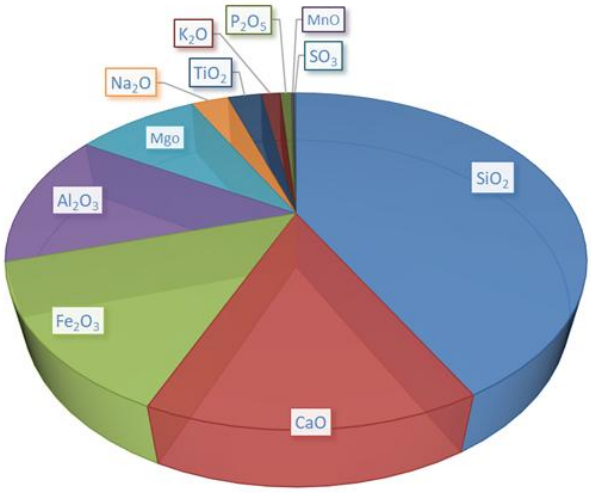

Fig. 3. Composition of Southern Jordanian Zeolite. Reproduced from R. Al Dwairi, Characterization of the Jordanian zeolitic tuff and its potential use in Khirbet es Samra wastewater treatment plant, PhD Thesis, University of Jordan, 2007, with permission.

1.1 Characteristics of Jordanian zeolite

The Southern Jordanian zeolite exists mainly at Tafila/ of Al Hala Volcano. Zeolite exists from the surface down to a few meters' depth. It also exists in piles and spreads chunks. Zeolitic tuff from Al Hala Volcano is a hydrous aluminum silicate and is mostly found as a weathered white material filling the pyroclastic vesicles of volcanic rocks (Al Dwairi 2014). **Figure 4** shows a photo of crushed zeolite taken from that area. **Figure 5** shows a microscopic and an electron microscope image of zeolite. **Tables 1** and **2** present the physical properties and chemical composition, respectively, based on three samples collected from different locations, as reported in the work by Al Dwairi (2014). **Table 2** clearly shows that the composition is almost identical across locations.

Fig. 4. Course and Fine Crushed Zeolite from Tafila/ Al Hala (Aljabarin, 2023).

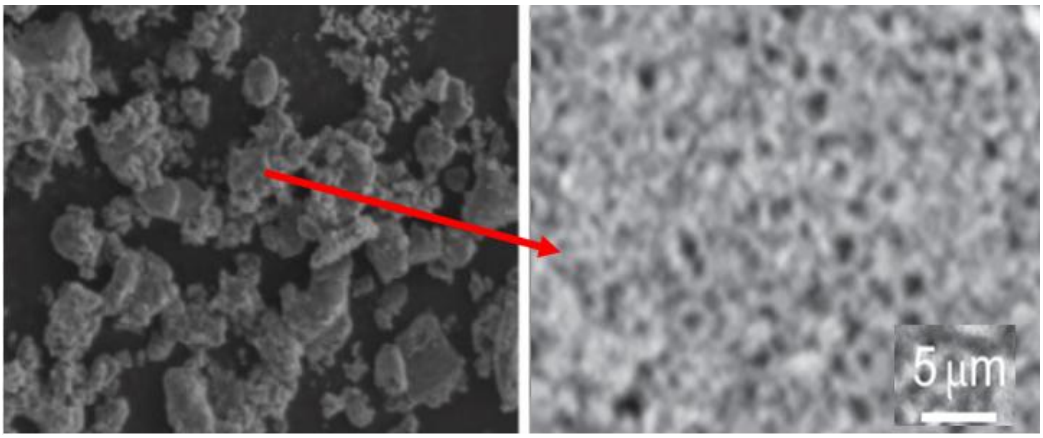

Fig. 5. Microscopic Pictures of Jordanian Zeolite Collected from Tafila/ Al Hala.

Table 1. Properties of southern Jordanian zeolite at Al Tafila/ Al Hala.

Property	Value
Density	2.1 kg/ m^3
Porosity	49%
Specific surface area	470 m^2/g
Pore Diameter	6.2 A^o
Solubility	Insoluble
Melting point	>1,600°C
Hardness	4.1 Moh's
Color	Red

Table 2. Composition of the Collected Samples from Tafila/ Al Hala. Adapted from [Al Dwairi, 2014], with permission from Jordan Journal of Earth and Environmental Sciences.

Sample No.	SiO_2	Na_2O	Fe_2O_3	MgO	Al_2O_3	K_2O	CaO	MnO	TiO_2	P_2O_3	CO_2
1	41.70	0.521	15.50	6.67	15.60	0.94	7.62	0.199	3.28	0.70	6.80
2	40.80	0.670	15.80	7.23	16.90	0.80	6.70	0.210	3.17	0.90	6.10
3	39.60	0.340	16.01	6.89	16.17	0.82	8.10	0.220	3.50	0.81	6.90
Average	40.70	0.510	15.77	6.93	16.223	0.853	7.473	0.210	3.32	0.803	6.60

2. Materials and Methods

2.1 Samples Preparation and Treatment

Three samples were collected from different locations, washed with potable water, dried, and then chemically analyzed as shown in **Table 2**. The samples were well mixed, crushed in a small jaw crusher, milled in a small ball mill, and then screened to the required size. To remove any adsorbed materials, such as CO_2 (as shown in Table 2), and water moisture from the pores of the zeolite, the samples were heated to about 100°C for 3 hours and then cooled in air. The samples are now ready for evaluation.

2.2 Experimental Setup

Figure 6 shows the experimental apparatus used to evaluate zeolite for the adsorption of carbon monoxide. A compressed carbon monoxide gas obtained from 99.9 % pure gas cylinder was fed to the fixed bed of zeolite. The bed is 40 cm in length and 10 cm in diameter, containing 6.6 kg of zeolitic tuff. The bed temperature was controlled via a water path with an electrical coil. The pressure was measured by a pressure gauge. The flow rate was controlled by manipulating valves 1 and 2. Carbon monoxide (99.9 % purity) was supplied from a standard industrial gas cylinder with a nominal fill pressure of 150–200 bar at 20 °C. A two-stage pressure regulator

reduced the pressure to the desired experimental set-point (0.5–6.0 bar, absolute) before admission to the adsorption bed. Bed pressure was monitored by the inline pressure gauge shown in **Figure 6**.

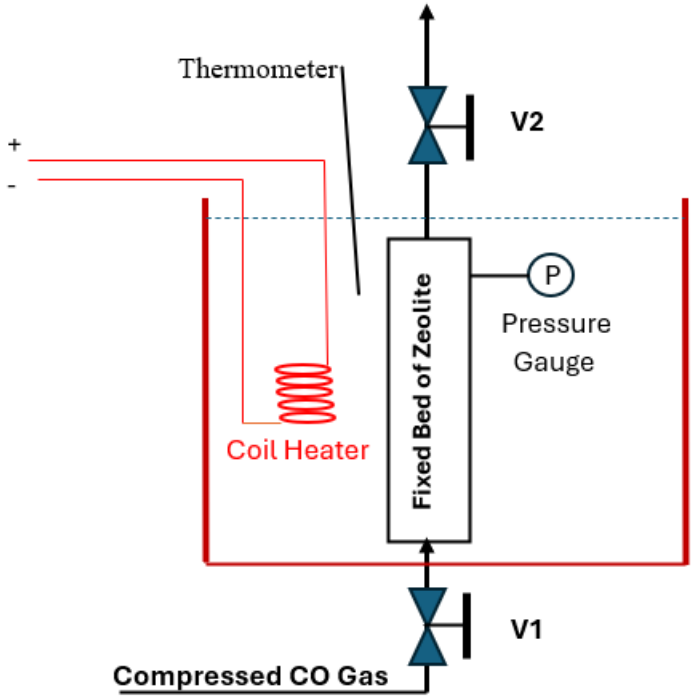

Fig. 6. Experimental Batch Apparatus.

From the measured initial and final pressures, the adsorbed number of moles of carbon monoxide can be calculated from the ideal gas law as:

$$P_i V = n_i RT \quad (1)$$

$$P_f V = n_f RT \quad (2)$$

where P is the absolute pressure (Pa); V is the volume of the gas (m^3); n is the number of moles; R is the universal gas constant (8.314 kJ/(kmol.K)); T is the absolute temperature (K); i is the initial state; and f is the final state.

Therefore, the number of moles adsorbed is:

$$\Delta n = n_i - n_f = \frac{V(P_i - P_f)}{RT} \quad (3)$$

and the loading is:

$$q = \frac{MW.\Delta n}{m} = MW.\frac{V(P_i - P_f)}{mRT} \quad (4)$$

where q is the equilibrium loading, MW is the molecular weight, and m is the mass of zeolite in the adsorption column. The volume of gas V was measured using water by the volumetric method. The volume occupied by the adsorbed CO itself was accounted for using an adsorbed-phase density of 0.79 g/cm³. The dead volume in the tubing, valves, and gauge connections is about 10–15 cm³. Temperature was kept steady by a water jacket held within ±0.5 °C of the target, and each pressure step was allowed to settle for 30 minutes for the finest particles and 4 hours for the coarsest. Equilibrium was accepted only when the pressure was stable enough that the rate of change dropped below 0.001 bar per minute. All pressures fed into Equations (1) through (4) are absolute values. At high loadings, the CO molecules within the zeolite pores occupy real space that must be accounted for. At the maximum capacity reported (10.1 mg CO/g zeolite), this adsorbed layer occupies about 84 cm³ — roughly 4–5% of the empty bed volume. That is big enough to affect the answer, so the number was recalculated over and over until it settled within 0.1%.

The waiting time for equilibrium also varies significantly with particle size. The biggest particles (6.146 mm) need up to 4 hours for the gas to fully penetrate and stabilize. The smallest ones (0.081 mm) are done in just 30 minutes.

3. Results and Discussion

Figure 7 shows the adsorption loading versus carbon monoxide pressure at different temperatures ranging from 22°C to 90°C at a zeolite particle size of 200/pan mesh size (d = 0.081 mm). From the shape of the curves, it can be directly concluded that the adsorption

isotherm takes the shape of Langmuir adsorption isotherm. In addition, the figure clearly shows that the adsorption loading decreases as the temperature increases, which is expected for adsorption. The dependency of adsorption on temperature is significant, increasing the temperature from 22ºC to 90ºC results in decreasing the adsorption loading about 94% which is considerably high. Above 90ºC, the adsorption becomes negligible. Therefore, Jordan zeolite is not recommended for high temperature applications.

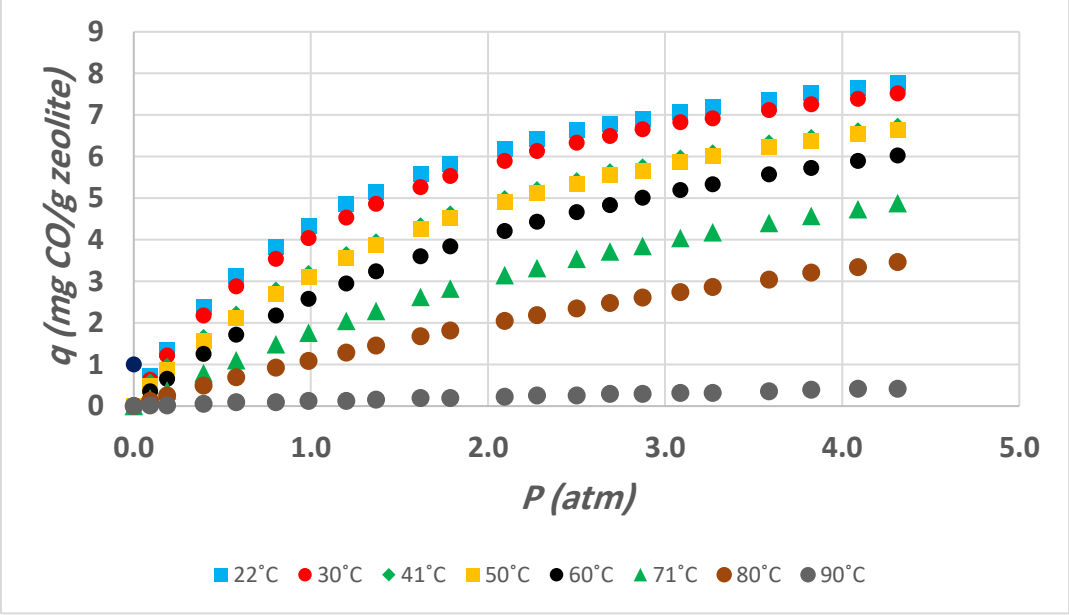

Fig. 7. Adsorption Isotherms at Different Temperatures for Zeolite Particles Size of 0.081 mm (200/pan mesh size).

Figure 8 shows the adsorption isotherms at 22ºC (room temperature) for different zeolites particles sizes. The figure clearly shows that the adsorption depends on particles size. As the zeolite particles size decreases the adsorption significantly increases. Decreasing the size from about 6 mm to 0.081 mm results in increasing the adsorption by about 62%. Therefore, the smaller zeolite particles size is the better for adsorption of CO onto Jordanian zeolite. This is expected because as the particle's size decreases, the surface area increases. Also, since pure zeolite is hardener than any impurities exist in the naturally occurring zeolite (Al Dwairi, 2014; Dwairi, 1987; Ibrahim, 1996), when zeolites sizes reduced by milling and washed by water these impurities are dissolved and washed out by the water leaving zeolite purer which in turn increasing the adsorption.

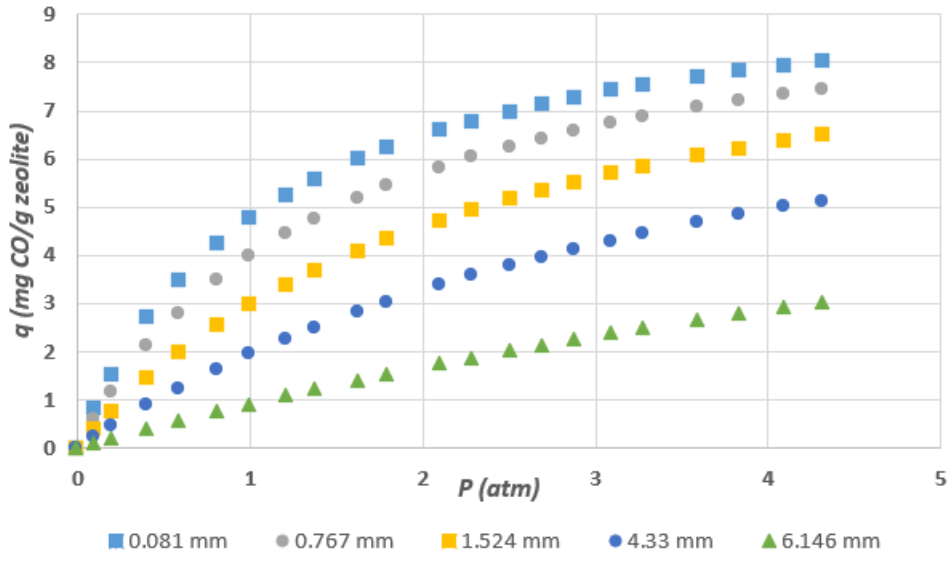

Fig. 8. Adsorption Isotherms at 22ºC for Different Zeolite Particles Sizes.

The Langmuir adsorption isotherm is given by (Seader et al., 2016):

$$q = \frac{k\, q_m\, P}{1 + k\, P} \quad (5)$$

where q_m is the maximum loading corresponding to complete surface coverage and k is the adsorption equilibrium constant. Equation 5 can be arranged in the following form,

$$\frac{P}{q} = \frac{1}{q_m} P + \frac{1}{q_m k} \quad (6)$$

Therefore, by plotting *P/q versus P, the* slope is $1/q_m$ and the intercept is $1/q_m\, k$, from which q_m and k are obtained. **Figure 9** shows a plot of Equation 6 for measurements at 22°C and 0.081 mm particle size. The figure also shows the equation of best fit with R^2 on the plot.

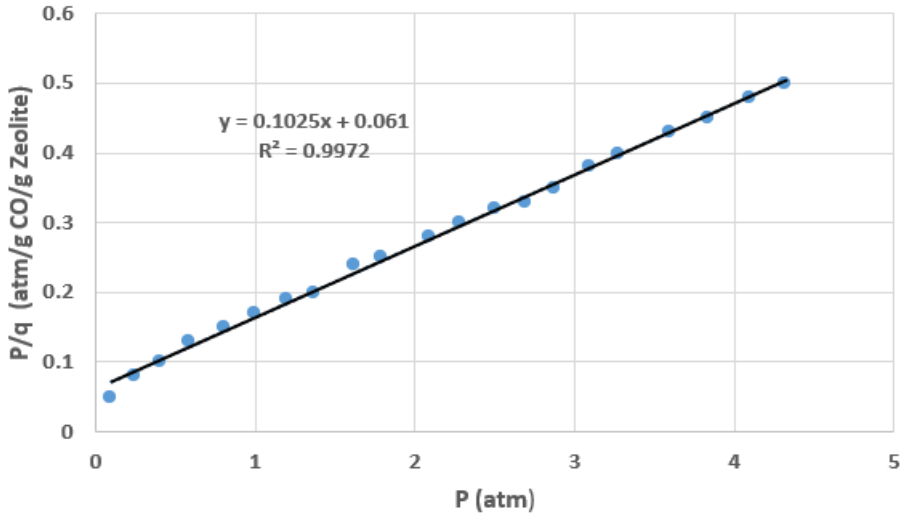

Fig. 9. Curve fitting of Experimental Adsorption Isotherm Data at 22°C and 0.081 mm Particles Size.

By repeating the curve fitting for every temperature and particles size, the experimental data of **Figures 8** and **9** was best fitted into Equation 5 as follows:

$$k = (1 - a\, T)(1 - b\, d) \quad (7)$$

where $q_m = 10.1\ \frac{mg\ CO}{g\ zeolite}$;

a, and b are the empirical coefficients for CO; $a = 0.018^o C^{-1}$; and $b = 0.15\ mm^{-1}$.

Over the temperature range of 22-90°C and particle size of 0.081-6.146 mm. **Table 3** shows a comparison of the maximum loading obtained in this work compared to literature values. Jordanian natural zeolite is very competitive to other natural zeolites. Moreover, the data shows that using zeolite for adsorption of carbon monoxide is an effective way of removing carbon monoxide from gas streams. The maximum loading q_m is in the order of 10 mg/g zeolite, this value considered excellent in adsorption processes (Seader et al. 2016).

Table 3. Maximum Loading of Jordanian Zeolite in Comparison with Other Zeolites.

Adsorbent	q_m (mg CO/ g Adsorbent)	Reference
Jordanian Zeolite/Tafila-Al Hala	10.1	This Work
Italian natural zeolite	8.5	Caputo and Pepe, 2007
Australian natural zeolite	11-23	Wahono et al., 2021
Chilean natural zeolite	9.8	Alejandro-Martín et al., 2019
Croatian natural zeolite	11.2	Cavallo et al., 2023
Turkish natural zeolite	5-14.5	Narin et al., 2004
Deutschland natural zeolite	2-23	Fuss et al., 2021
Serbian zeolite	12	Ivanenko et al., 2020
Chinese natural zeolite	10.5	Chen et al., 2018
Norton Z-90 0H zeolite	10.1	Egerton and Stone, 1970

The heat of adsorption can be calculated from Clausius-Clapeyron equation as follows (Smith et al., 2017)

$$\frac{d\ lnP}{dT} = \frac{-\Delta H_{ads}}{R\, T^2} \quad (8)$$

where ΔH_{ads} is the heat of adsorption [kJ/mol], or equivalently Equation 8 can be written as

$$\frac{dP}{dT} = -\frac{P}{R\,T^2}\Delta H_{ads} \quad (9)$$

Rearranging Equation 9 obtain

$$\Delta H_{ads} = -\frac{R\,T^2}{P}\frac{dP}{dT} \quad (10)$$

and rearranging Equation 5 obtain

$$P = \left(\frac{1}{\frac{q_m}{q}-1}\right)\frac{1}{k} \quad (11)$$

From Equations 9 and 11 obtain

$$\frac{dP}{dT} = \left(\frac{1}{\frac{q_m}{q}-1}\right)\left(\frac{-1}{k^2}\right)\frac{dk}{dT} \quad (12)$$

again, from Equation 5

$$\frac{1}{k} = \left(\frac{q_m}{q}-1\right)P \quad (13)$$

and from Equation 7

$$\frac{dk}{dT} = -a(1-bd) \quad (14)$$

Substitution of Equations 13 and 14 into Equation 12 obtain.

$$\frac{dP}{dT} = \frac{a}{(1-a\,T)}P \quad (15)$$

Substitution of Equation 15 into Equation 10, obtain

$$-\frac{\Delta H_{ads}}{R} = \frac{a\,(T+273.15)^2}{(1-a\,T)} \quad (16)$$

Substitution the value of a = 0.018 in the above equation, obtain the heat of adsorption for Jordanian zeolite over the temperature range 22 - 90°C,

$$-\frac{\Delta H_{ads}}{R} = \frac{0.018\,(T+273.15)^2}{(1-0.018\,T)} \quad (17)$$

Figure 10 shows the heat of adsorption versus temperature (Equation 17). Up to T=1/0.018 = 55.5°C adsorption process is exothermic and after 55.5°C the adsorption process becomes endothermic. As it is known, adsorption process is always exothermic process and endothermic adsorption is not possible, but this is true for at least adsorption of carbon monoxide onto Jordanian zeolite. No sufficient study in the literature was found for the heat of adsorption of carbon monoxide onto zeolite at high temperature. However, this phenomenon could be explained due to, probably, sort of endothermic reaction between carbon monoxide and some constitute or impurities of the zeolite.

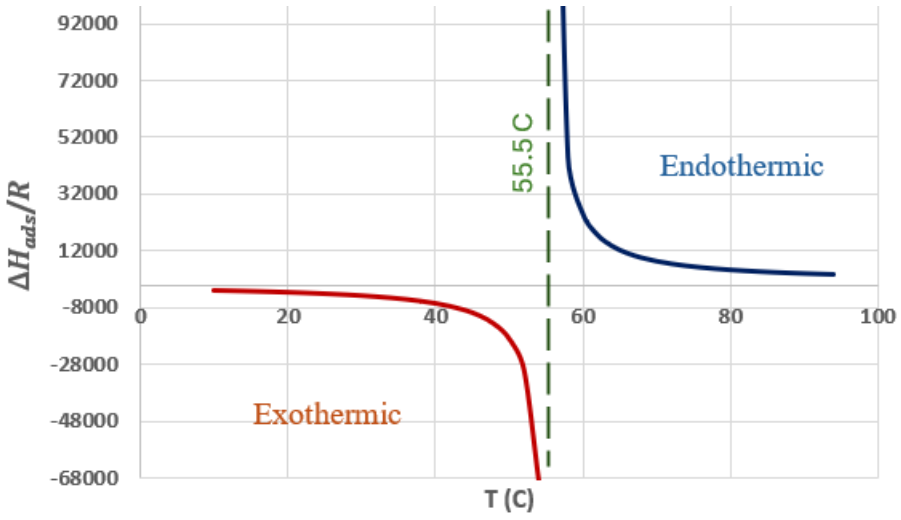

Fig. 10. Heat of Adsorption of CO Based on Jordanian Zeolite by using Equation (17).

4. Conclusion

In this work Jordanian natural zeolite collected from Tafila government/ Al Hala area was evaluated for adsorption of carbon monoxide. The maximum adsorption capacity was experimentally measured and compared with other literature's values and found to be competitive or even in some cases exceeding most other zeolites for adsorption of carbon monoxide (9.8 mg CO/g zeolite, Alejandro-Martín et al., 2019). The maximum adsorption capacity of carbon monoxide by Jordanian zeolite was found to be about 10.1 mg CO/g zeolite which is significantly high for the Jordanian zeolites to be considered for carbon monoxide adsorption at low concentration. The adsorption isotherm of carbon monoxide by Jordanian zeolite was found to obey Langmuir adsorption isotherm and fitted with 99.7% accuracy. The effect of temperature and zeolite particles size on adsorption was studied and found to have a significant effect on adsorption. As the temperature increased, the adsorption loading significantly decreased, while as the particle's sizes decreased the adsorption significantly enhanced and therefore, fine zeolite particle sizes are recommended for the adsorption (D=0.081 mm has the maximum adsorption). The heat of adsorption was found to be exothermic below about 55.5°C, while endothermic above 55.5°C. This was explained due to the presence of some endothermic reaction between zeolite or impurities in zeolite with carbon monoxide. Adsorption of other gasses such as (H_2S, NO_2, SO_2 …etc.,) by Jordanian zeolite are recommended for future study to find more solutions for removing these harmful gasses from gas streams and find new applications for Jordanian zeolite.

CRediT Author Contribution Statement

N. Aljabarin: Conceptualization, Investigation, Data Curation, Writing – Original Draft

Declaration Statements

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Conflicts of Interest
The author declares no conflict of interest.

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Nomenclature

Symbol	Description	Unit
ΔH	Heat of adsorption	kJ/mol
m	Mass of zeolite	kg
MW	Molecular weight of carbon nonoxide	Kg/kmol
n	Number of moles	Moles
P	Pressure	Pa
Q	Loading	mg CO / g zeolite
R	Universal gas constant	kJ/(kmol.K)
T	Temperature	K
V	Volume	m^3

Subscripts
ads: adsorption
f: final
i: initial
m: maximum

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