MATHEMATICAL SCIENCES

MULTIVARIABLE NONLINEAR REGRESSION FOR ANALYSING ADSORPTION ISOTHERM MODELS - A CASE STUDY

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https://doi.org/10.5281/zenodo.21105815

Abstract

This paper details the calculation steps, from post-processing experimental data to predicting cooling efficiency of activated carbon (AC)/CO_2-based adsorption system for specified design temperatures. Regression analysis was conducted using advanced Python packages. The results identified the best model to describe adsorption isotherms, with optimal parameters verified using alternative methods implemented via Excel's solver. The study considered a nonlinear equations with two independent variables, specifically Langmuir and T′oth adsorption isotherms, to fit thermogravimetric data representing six equilibrium uptake curves measured at various temperatures and pressures for the CO_2/activated carbon working pair. Physical adsorption is highly relevant for industries releasing low-temperature heat to the environment. The derived model parameters facilitated the estimation of the isosteric heat of adsorption and the development of a pressure-temperatureequilibrium uptake (PTW) diagram, essential for predicting the performance of ideal adsorption cooling cycles. The specific cooling effect (SCE) and coefficient of performance (COP) were determined for selected design temperature conditions.

Keywords: Adsorbent materials, Langmuir isotherm, Nonlinear regression, T′oth isotherm

1. Introduction

Global scientific efforts increasingly focus on developing and promoting carbon-free or carbon-neutral technologies. Also of particular interest are autonomous systems that do not depend on the central power supply. Adsorption refrigerators, which operate waste-free using thermal energy sources such as solar power, are a key component of this movement. Several widely used solid static adsorbents, including activated carbon, zeolites, and metalorganic frameworks, play a crucial role in adsorption cooling systems (ACS). Simultaneously, ongoing research aims to enhance these materials' adsorption capacity and thermal efficiency and optimize the compactness and design of adsorption beds and the overall system performance.

Driving temperature, evaporation temperature, coefficient of performance (COP), and specific cooling power (SCP) are basic parameters for choosing a working adsorbent-refrigerant pair for the ACSs [1]. Let's consider a few working pair cases with application limits and desirable characteristics.

Activated carbon (AC)/methanol is the most common working pair [1]. For example, in [2], the basics of a solar-powered adsorption ice-maker were presented. The authors conclude that the adsorption pair of activated carbon and methanol is preferred more than the water/ammonia pair. The limitation while applying methanol CH_3OH is that the methanol decomposes at 120°C, so the desorbing temperature shouldn't exceed this value. The experimental prototype had a solar refrigeration COP of 0.10-0.12 and produced 4-5 kg of ice per day from the flat-plate collector area of 0.92 m^2. Another effect on methanol decomposition is the alloys used in system construction, as the methanol contacts with it while operating [3]. Aluminum alloys were revealed to have a stronger catalytic effect on the decomposition reaction, so copper alloys are preferred. In such systems, the methanol is in the 0.02-0.2 bar pressure range and at -10°C to 110°C temperature range. In addition, some demonstration systems exhibit very good performance when they are newly built, and performance declines after a year or less of operation. The performance decline is associated with the loss of the vacuum in the system, so re-evacuation, or in other words, repeated vacuuming on an annual basis, is necessary to restore system functionality. The long-term performance deterioration is the main reason behind the cessation of solar refrigerator commercialization. Still, solar-powered refrigeration systems are the most promising technology, with low cost, simple manufacturing, and low maintenance requirements.

Activated carbon (AC)/ammonia (NH_3) pair demonstrates high COP that may reach 0.45 for ice-making and 0.61 for air-conditioning [4]. The highest

SCP (of order 60 – 200 W kg^{-1} for different systems) is achieved with an AC/ammonia pair. Ammonia's thermodynamic properties make it suitable for heating and cooling applications, but it is also a toxic and corrosive substance.

Cryocooler is a refrigerator that can reach temperatures below -153°C (or 120 K). Nitrogen as a refrigerant with ACF (Activated carbon fibers) as an adsorbent is good for cryocooling applications. The adsorption capacity is about 0.75 g g^{-1} at an adsorption temperature of -196°C. The adsorption capacity with operating temperature varies. For typical cooler applications with relatively higher temperatures, nitrogen has a low adsorption capacity. For example, the highest adsorption capacity for the nitrogen/AC pair was 0.00756 g g^{-1} at adsorption temperature and pressure of 30 °C and 100 kPa, respectively.

AC/R134a pair has the highest adsorption capacity of 2 g g^{-1} at adsorption temperature and pressure of 30 °C and 800 kPa. The boiling temperature of R134a is (-26.55 °C). R-134a is a hydrofluorocarbon CF_3CH_2F with insignificant ODP (ozone depletion potential) and a relatively lower GWP (global warming potential). The gas is non-flammable and primarily used in household refrigerators with conventional vapor-compression cycles.

AC/CO_2 CO2 pair is about a relatively high-pressure system. The highest adsorption capacity at 30 °C and 100 kPa is only 0.0844 g g^{-1}, so the higher working pressures preferred, say at 4586 kPa and 25 °C the adsorption capacity was 1.135 g g^{-1} [1]. The thermodynamics performance of the CO_2/activated carbon (Maxsorb III-based composite) pair is of order COP = 0.16 for the system with the evaporator temperature T_{evap} = 5°C and the condenser temperature T_{cond} = 25°C [5]. CO_2 is a non-toxic and non-flammable natural refrigerant. Maxsorb III is a highly recognized material for good thermal properties and high capacity among activated carbon samples.

A new class of microporous materials, PCPs (porous coordination polymers) and MOFs (metal-organic frameworks) have recently become available as alternatives to AC. There is another class of carbon-based adsorbents. In [6], the authors examined one of them - nitrogen-enriched nanostructured carbon (UFZ-700). Unlike activated carbon, this type of carbon can be derived using inexpensive, widely available sources. MOFs show a huge surface area and large pore volume. The surface areas of typical MOFs range from 1000 to 10,000 $m^2 g^{-1}$, which can exceed those of traditional porous materials such as zeolites and carbons. Also, the average pore size is tunable due to a much wider variety of chemical compositions. Table 1 illustrates some common microporous materials for comparison.

Thus, the main pros and cons of popular adsorbent-adsorbate pairs for ACSs are listed above. Additionally, a brief summary of the necessary features is provided when choosing a working pair based on the intended installation environment for the system as well as the target temperature in the cooled chamber. A number of factors that have a long-term cumulative impact on the refrigerator's operation are also covered, including toxicity, corrosion, leak proofness, vacuum tightness, maximum adsorption capacity deterioration, explosiveness risks due to high working pressures, etc.

There is endless room for improvement in the various heating and cooling systems designed for different purposes. Such systems usually contain many parts that can be considered and studied separately. The system performance can be enhanced by employing multi-beds and a complex piping network. Advancing the cycles from single to two-bed configurations may improve the COP by at least 35% and, at most, even double [4]. In addition, new materials are developed by combining or/and compressing various existing materials to improve the compactness and characteristics of adsorption beds in thermal systems.

The current paper describes the calculation steps from the post-processing of experimental data to predicting the cooling efficiency of ACS. Part of the regression analysis was performed using emerging capabilities of Python packages. Alternative methods were implemented using the Excel solver. Based on the previous work done by various authors, this paper also presents an approach to calculating the cooling efficiency parameters, SCE, and COP. The cooling cycle performance of any solid-gas adsorption system is normally studied using the PTW (pressure-temperature-concentration) diagram [7]. The derived model parameters allowed the development of a PTW-graph diagram, essential for predicting the performance of ideal adsorption cooling cycles. Thermodynamic analysis of CO_2/AC-based cooling systems is well discussed in [8]. Another motivation for research work is that the established isotherm model is also used in simulation studies of the dynamics of the adsorption process. Here are some examples of simulation studies [9, 10].

Table 1.

Porous properties of different types of microporous materials

Adsorbent	SSA [$m^2\ g^{-1}$]	Pore volume [$cm^3\ g^{-1}$]	Average pore diameter, d_{ave} [nm]	Thermal stable up to	References
MOF-NU-1000	2320	1.4	0.8-3.1		[11]
MOF-5	1077	0.49	1.8	400 °C	[12]
ZIF-8	1700	0.662	1.2	550 °C	[13, 14]
Activated carbon Maxsorb III	3140	1.7	2.008		[15]
Activated carbon fiber ACFC-1500	1500	0.62	1.69		[16]
Zeolite Na-P1CS	53.5	0.13	11.1		[17]
Natural zeolite ZN	27	0.1	17		[18]

2. Adsorption isotherm models

Quoting the work [19], the Langmuir isotherm model is the most straightforward and practical for both physical and chemical adsorptions. Pressure *p* and temperature *T* are two independent variables of Langmuir Equation 1 and To´th Equation 2 isotherm models. Equilibrium adsorption *C* is the dependent variable, and is the amount adsorbed [g g^{-1}]. The Langmuir model has three fitting parameters, while the To´th model has four. The list of correlated parameters or regression features is shown in Table 2, Table 3. Theoretically, at zero pressures, absorption is zero; therefore, zero points are added to the calculation.

$$\frac{C}{C_0} = \frac{b_0 \exp\left(\frac{E}{RT}\right) p}{1 + b_0 \exp\left(\frac{E}{RT}\right) p} \quad (1)$$

$$\frac{C}{C_0} = \frac{b_0 \exp\left(\frac{E}{RT}\right) p}{\left(1 + \left(b_0 \exp\left(\frac{E}{RT}\right) p\right)^t\right)^{1/t}} \quad (2)$$

The coefficient of determination (R^2), Equation 3, and the root-meansquare deviation (RMSD), Equation 4, are used as a measure of prediction error to compare both methods.

$$R^2 = 1 - \frac{\sum_{i=1}^{n} (y_i - \hat{y}_i)^2}{\sum_{i=1}^{n} (y_i - \bar{y})^2} \quad (3)$$

$$RMSD = \sqrt{\frac{\sum_{i=1}^{n} (\hat{y}_i - y_i)^2}{n}} \cdot 100\% \quad (4)$$

The experimental data represents 6 equilibrium uptake curves measured with varying temperatures and pressures for activated carbon/carbon dioxide working pair, see Figure 1.

Python (Version 3.11.5) is used for calculations. Optimal parameters of the isotherm models were obtained using two alternative methods. The calculation results in Table 2, Table 3 revealed that the To´th model better describes adsorption isotherms with the lowest root mean square deviation of 0.441%. The surface plot illustrated in Figure 2. The side view demonstrated in Figure 3 shows fitted adsorption isotherms and experimental data.

Table 2.

Optimal parameters of To´th isotherm obtained using Method 1: Python curvefit and Method 2: Excel Solver

Estimated parameters	**Unit**	**Value Method 1**	**Value Method 2**
Saturated amount adsorbed, C_0	[g g^{-1}]	3.625	2.6307
Isosteric heat of adsorption, E	[kJ mol^{-1}]	18.27	19.024
Equilibrium constant, b_0	[kPa^{-1}]	2.112E-07	1.887E-07
Heterogeneity parameter, t	[-]	0.4107	0.4774
RMSD	[%]	0.441	0.5635
Coefficient of determination, R^2	[-]	0.99964	0.99940

Table 3.

Optimal parameters of Langmuir isotherm obtained using Method 1 – Python curve-fit, Method 2 – Excel Solver

Estimated parameters	Unit	Value Method 1	Value Method 2
Saturated amount adsorbed, C_0	[g g^{-1}]	1.274	1.1357
Isosteric heat of adsorption, E	[kJ mol^{-1}]	18.652	20.722
Equilibrium constant, b_0	[kPa^{-1}]	2.096E-07	1.217E-07
RMSD	[%]	1.426	1.877
Coefficient of determination, R^2	[-]	0.99630	0.99357

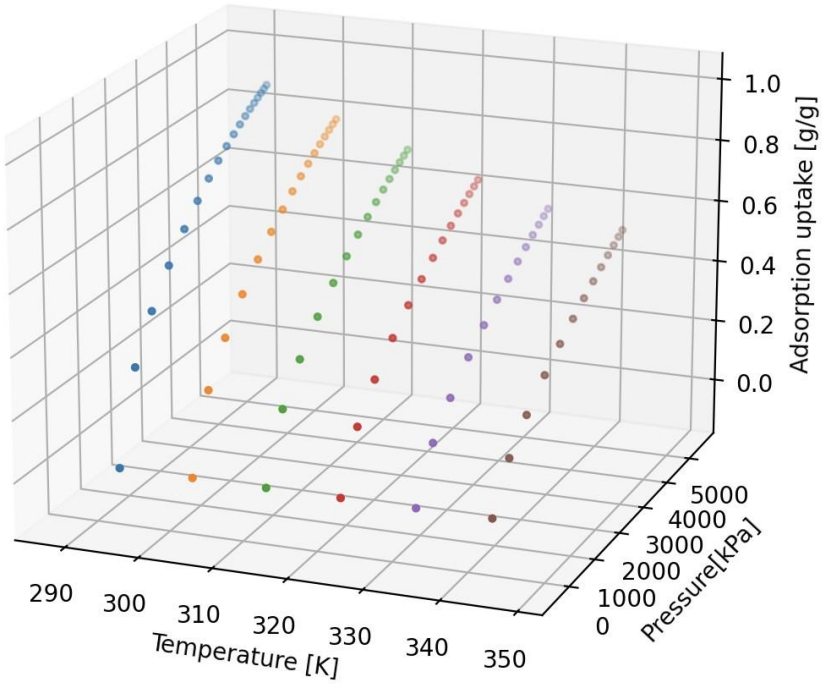

Fig. 1. Equilibrium uptake data for activated carbon/CO_2 pair

3. Clapeyron diagram of ideal solid-adsorption cycle

The PTW graph shows the thermodynamic relation between equilibrium pressure, adsorption temperature, and adsorption uptake (see Figure 4). The graph is essential for predicting the ideal adsorption cooling cycle performance, so to say, to calculate SCE and COP. Thermodynamic analysis of AC/CO_2-based cooling systems is well discussed in [8]. Let's consider the case of temperatures closer to those taken in the paper. The waste heat or solar energy as a heat source is supplied at 80°C, and the heat sink temperature is 30°C (maximum and minimum temperatures in the collector).

The evaporator temperature is T_{evap} = 5°C, the condenser temperature

T_{cond} = 25°C, respectively, the evaporator pressure P_{evap} = 3,9695 *MPa*, and the condenser pressure P_{cond} = 6,4342 *MPa*. The amount of adsorbate per adsorption cooling cycle varies from W_{max} to W_{min}. W_{max} (adsorption) is measured as a function of P_{evap} (which is the saturation pressure of the cooling load temperature T_{evap}), and the heat sink temperature T_{ads} = 30°C

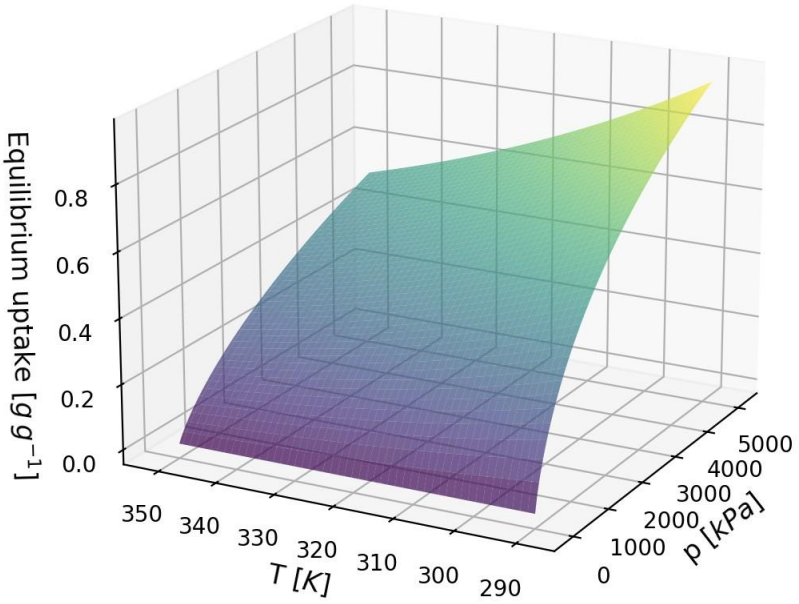

Fig. 2. Surface plot using the To´th model

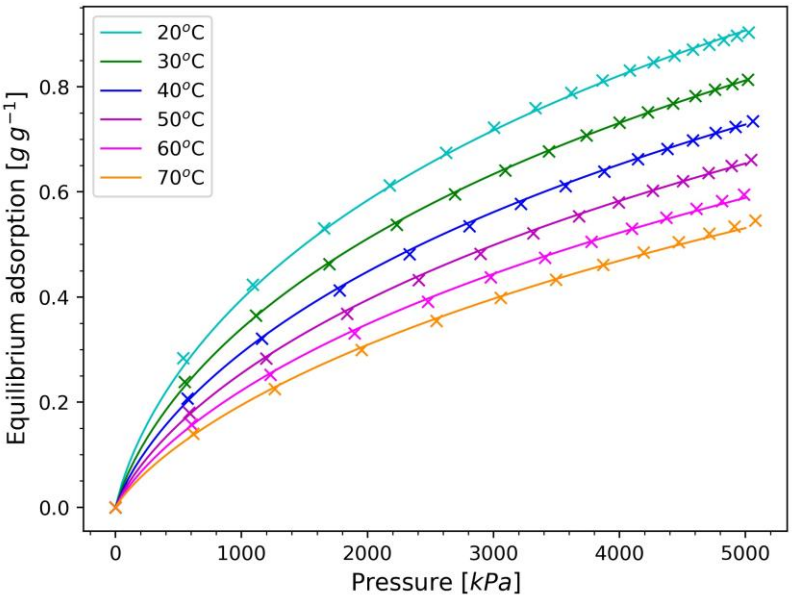

Fig. 3. To´th isotherm model along with the experimental absolute adsorption uptake.

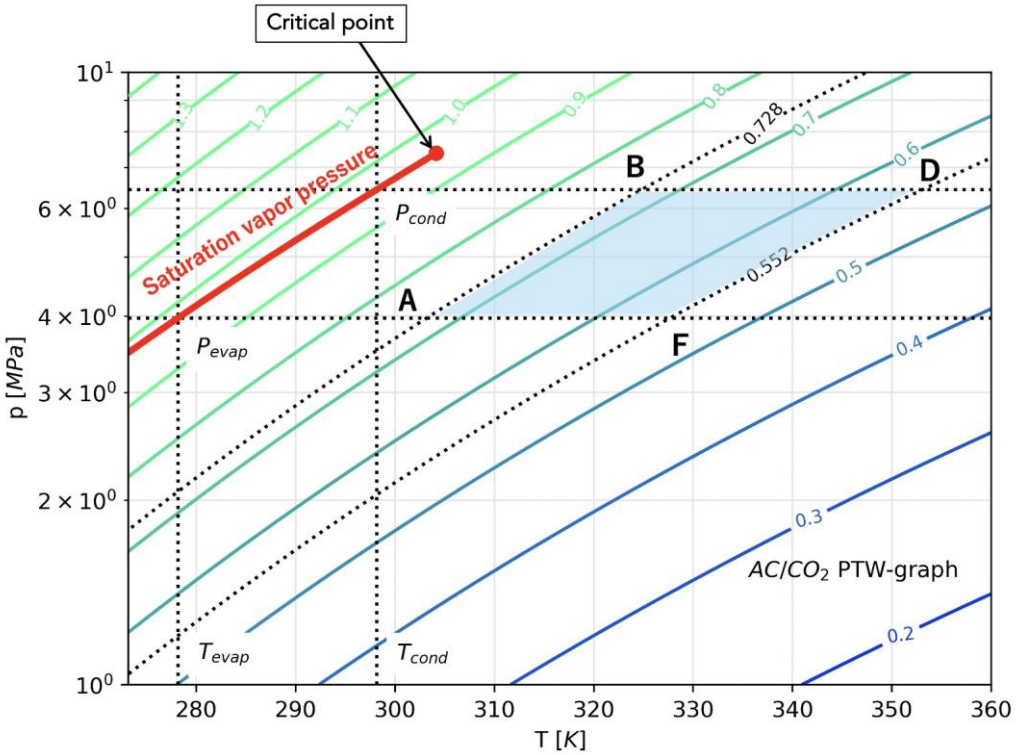

Fig. 4. PTW-graph for AC/CO_2 working pair

[20, 21]. By analogy, W_{min} (desorption) is found as a function of P_{cond} (which is the saturation pressure of condenser temperature T_{cond} = 25°C), and the heat source temperature T_{des}= 80°C. Maximum and minimum uptakes during the working cycle are W_{max} = 0,7282gg^{-1} and W_{min} = 0,5517gg^{-1}. Carbon dioxide has a critical temperature of 31,1°C (or 304,13K) and a critical pressure of 7,377MPa.

$$W\text{max} = f(p\text{sat}(T\text{evap}), T\text{ads}) \quad (5)$$

$$W\text{min} = f(p\text{sat}(T\text{cond}), T\text{des}) \quad (6)$$

The enthalpy of vaporization, also known as the latent heat of vaporization for carbon dioxide at 3.9695MPa and at the boiling point of T_{evap} = 5°C is $\Delta H_{vap} = 2.94 \times 10^5 J\,kg^{-1}$ [22]. The specific heat capacity of the adsorbent (composite AC) was taken as a constant for the range of temperature utilized, $c_{p,AC}$ = 0.8212$J\ g^{-1}\ K^{-1}$. Latent heat of desorption (theoretically, should be the same as the heat of adsorption, E), H_{des} = 18.27$kgmol^{-1}$. The specific heat capacity of refrigerant (liquid CO_2), $c_{p,ref}$ is taken as a function of temperature [7, 22].

The Clapeyron diagram of the ideal solid-adsorption cycle consists of 4 stages, see Figure 4:

- 1. A→B Isosteric pre-heating
- 2. B→D Isobaric desorption
- 3. D→F Isosteric pre-cooling
- 4. F→A Isobaric adsorption.

The specific cooling power (SCP) has a unit of [W kg^{-1}] and is calculated differently; do not confuse it with the SCE. SCP is the ratio of cooling energy to the product of adsorbent mass and cycle time [23]. The specific cooling effect (SCE) is given by Equation 7 and found to be 44.7 kJ kg^{-1}. The SCE is the same as the total energy output Q_C in some other papers, both are used equally.

$$SCE = (W_{max} - W_{min}) \left[\Delta H_{vap} - \int_{T_{evap}}^{T_{ads}} c_{p,ref}\, dT \right] \quad (7)$$

The system efficiency, coefficient of performance:

$$COP = \frac{Q_{\mathrm{C}}}{Q_{\mathrm{T}}} \tag{8}$$

$$Q_{\mathrm{T}} = Q_{\mathrm{A-B}} + Q_{\mathrm{B-D}} \tag{9}$$

$$Q_{\mathrm{A-B}} = \int_{T_{\mathrm{A}}}^{T_{\mathrm{B}}} c_{p,\mathrm{ads}}\, dT + W_{\max} \int_{T_{\mathrm{A}}}^{T_{\mathrm{B}}} c_{p,\mathrm{ref}}\, dT \tag{10}$$

$$Q_{\mathrm{B-D}} = \int_{T_{\mathrm{B}}}^{T_{\mathrm{D}}} c_{p,\mathrm{ads}}\, dT + \int_{T_{\mathrm{B}}}^{T_{\mathrm{D}}} W \cdot c_{p,\mathrm{ref}}\, dT + (W_{\max} - W_{\min})\, H_{\mathrm{des}} \tag{11}$$

COP value is found to be 0.27.

Table 4.

CO_2 gas-specific isobar heat capacity vs. temperature and pressure

	Temperature T [degC]	Temperature T [K]	Pressure p [MPa]	Specific Heat c_{p} [kJ kg^{-1} K^{-1}]
A	30	303.15	3.9695	1.3380405
B	51.6	324.75	6.4342	1.6562389
D	80	353.15	6.4342	1.3050357
F	54.57	327.72	3.9695	1.1640829

4. Conclusions

The present study evaluates the performance efficiency of the novel material in an adsorption refrigerator based on the results of the TGA experiment. It specifically examines the case of carbon dioxide and activated carbon as a working pair.

Firstly, the equilibrium uptake parameters were correlated using a multivariable nonlinear regression method and two alternative calculation tools. The study results are in agreement with each other, and the results are given in the literature on similar systems. The choice of loss function or inner hyperparameter set may cause negligible deviations. The best fit was achieved when using Python curve-fit, with RMSD of 0.441% for To´th isotherm.

Secondly, performance metrics, such as specific cooling effect and coefficient of performance, are discussed in relation to prior research and projected for selected operating temperatures. SCE and COP are predicted to be 44.7 kJ kg^{-1} and 0.27, respectively. The PTW diagram for the composite AC/CO_2 pair is constructed. The stages of the ideal cooling cycle are described in detail to better understand the fundamentals of solid-gas ACS's operation principle. Thus, the study details the calculation of cooling efficiency, encompassing the progression from post-processing experimental data to forecasting outcomes for specific system design temperatures.

Thirdly, the paper illustrates the potential for more precise computations with the help of available and modern tools. Most engineers and researchers have encountered similar optimization problems at some point or another, and Python packages for resolving them are rapidly evolving. It is noteworthy to mention the significance of the completed review on adsorption-based working pairs and related features during use. The present results are relevant as a reference for future related studies.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgement

The author (BB) wishes to acknowledge the financial support received from JSC Center for International Programs – Bolashaq, and the research was performed on scope of 500 scholars fellowship program.

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