Use of Bentonite Adsorbent from Ujong Pacu for the Adsorption Free Fatty Acids from Crude Palm Oil

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Abstract

Because free fatty acid content impacts crude palm oil's physical and chemical characteristics, it is a good indicator of the oil's quality. Investigated effective operating parameters include contact time on adsorption and initial free fatty acid concentration. This system was studied using Langmuir and Freundlich isotherms, and the kinetic data was examined using pseudo-first-order and pseudo-second-order models. The findings indicated that increasing FFA concentrations increased the adsorption capacity of free fatty acids using Ujong Pacu bentonite, and it was discovered that CPO weighing 30 g was the ideal weight for FFA adsorption capacity. At equilibrium, the ideal value of adsorption capacity is 502.17 mg/g. While the pseudo-second order model is the model that best describes the adsorption kinetics of the free fatty acid content in Crude, the Freundlich isotherm model best describes the equilibrium data, which indicates the heterogeneous surface of the adsorbent pores and shows that the adsorbate layer formed on the surface of the adsorbent is multilayer. Correlation coefficient (R2) for palm oil (CPO) in the range of 0.946 to 0.9975.

Keywords: Adsorbent, Bentonite, Adsorption, Kinetics

Introduction

Natural resources are in plentiful supply in Indonesia. One of the natural resources that is extensively used in many different sectors, particularly the economic sector, is crude palm oil (CPO). Indonesia has 12.30 million hectares of oil palm land, with CPO production expected to exceed 51.58 million in 2020. The increase is consistent with the demand for CPO exports, which reached 34 million tonnes in 2020, with the remainder consumed domestically (Sinaga & Siahaan, 2015). Crude palm oil and palm kernel oil are the two types of oil that can be produced from palm oil. Free fatty acids, phosphates, pigments, odors, water, and other substances are still present in crude palm oil. Palm oil must be refined, bleached, and deodorized, also known as the CPO refining process, to produce oil of a standard of quality (Nor Shafizah et al., 2022). Free fatty acids (FFA) have an impact on the physical and chemical characteristics of palm oil, which makes them a determinant of its quality. Rancidity caused by the presence of other undesirable components can lower the CPO's quality (If a et al., 2021). Therefore, to maintain and enhance the quality of oil, a fairly efficient and economically beneficial method is required to reduce FFA levels. The FFA content of an oil determines its quality; the oil's poorer quality is indicated by a rancid odor and cloudy color.

There have been numerous studies utilizing a variety of methods to eliminate or reduce FFA levels in CPO. Chemical processing is a commonly employed method for reducing or removing levels of free fatty acids (FFA) in crude palm oil (CPO) (Tan et al., 2009). However, it is worth noting that this approach generates significant quantities of waste and soapstock (Rahayu et al., 2018). In contrast, physical processing methods offer an alternate approach for the elimination or reduction of free fatty acids (FFA). These methods include distillation, membrane filtration, solvent extraction, enzymatic treatment, adsorption, and extraction utilizing adsorbents (Ayu Putranti et al., 2018). Ttilization of adsorbents in the extraction of free fatty acids (FFA) from crude palm oil (CPO) has several benefits, including little oil loss, avoidance of soap contamination, cost-effectiveness, and convenient separation through filtration. Commonly employed adsorbents for the adsorption of free fatty acids include activated carbon, several types of bleaching earth (such as zeolite, kaolin, bentonite, and clay), and adsorbents comprising magnesium silicate. Bentonite has been found to be highly effective in the bleaching process (Ayu Putranti et al., 2018). The optimal condition for this process involves using 2% bentonite in a volume of 200 ml of oil. As a result, the color of the oil closely approximates the yellow shade specified by the Indonesian National Standard (SNI).

In a separate investigation conducted by Syahwandi et al (2019), it was shown that the utilization of ATKS ash as an adsorbent material demonstrated the ability to adsorb free fatty acids (FFAs) from crude palm oil (CPO). The study determined that an optimal adsorbent mass of 0.1 g, along with a 60-minute adsorption duration, resulted in a reduction of FFA levels from 7.321% to 6.297% (Samantaray et al., 2022). Bentonite, an adsorbent commonly found in clay soil, is extensively utilized for the purpose of conjunction in the processing of crude palm oil (CPO). The term "bentonite" is commonly used in commercial contexts to refer to clay that contains the mineral monmorillonite. The primary constituent

Lhokseumawe, Indonesia

of bentonite consists of monmorilonite minerals, which make up around 80% of its composition. These minerals can be represented by the chemical formula [All.67Mg0.33(Na0.33)]Si4O10(OH)2 the compound exhibits a range of colors, including white, yellow, olive green, and bluish brown.

Adsorption is a phenomenon characterized by the transfer of adsorbate molecules from the fluid phase to the surface of the adsorbent material. This process continues until the concentration of adsorbate reaches a state of thermodynamic equilibrium. The kinetics of adsorption is dependent on the interaction between the adsorbate and the adsorbent (Suseno et al., 2013). Hence, the objective of this research is to assess the viability of employing bentonite adsorbents sourced from Ujong Pacu, a community in Lhokseumawe, for the removal of free fatty acids (FFA) from crude palm oil (CPO). Additionally, the study aims to comprehend the kinetics of FFA adsorption onto the adsorbents. The data obtained from the experimental results of adsorption equilibrium in this work were analyzed using the Langmuir and Freundlich isotherm models. Additionally, the kinetics of the adsorption process were examined using pseudo-first-order, and pseudo-second-order models.

Literature Review

1. Adsorption

Adsorption refers to the phenomenon wherein a dissolved substance in a solution is attracted and adheres to the surface of an absorbent material, effectively accumulating within the absorbent material. Both of these phenomena frequently occur in conjunction with a process, which is sometimes referred to as sorpsi. The process of adsorption involves the presence of two key components, namely adsorbents and adsorbate (Al-Ghouti & Da'ana, 2020). An adsorbent refers to a solid material that has the ability to selectively take up specific components from a fluid phase. Adsorbents typically employ substances that possess porous structures, enabling the adsorption process to take place within these pores or at specific sites within the particles. Typically, the pores present inside the adsorbent material are characterized by their small size, resulting in a higher inner surface area compared to the outside surface (Nayak et al., 2017).

2. Bentonite

Bentonite is a clay variety characterized by its capacity to adsorb specific things, including poisons, chemical compounds, and particles present in a solution. The efficacy of bentonite as an adsorbent is contingent upon various factors, including the nature of the contaminant to be removed, prevailing environmental circumstances, and the particular applications in question (Rajendran et al., 2022). The utilization of bentonite as an adsorbent often entails the amalgamation of bentonite with a solution or waste stream that contains impurities, afterwards followed by the procedures of separation and purification in order to achieve the intended outcomes. The efficacy and efficiency of bentonite adsorption may exhibit variability contingent upon several aspects, encompassing the specific type of bentonite employed, particle size, pH of the solution, temperature, and concentration of the compounds targeted for adsorption (Resende et al., 2020).

3. Isotherm Adsorption

Isotherm adsorption is a function of the concentration of solutes absorbed in solids to the concentration of the solution. Preparation that can be used to explain isotherm experimental data is studied by Freundlich, Langmuir, and Brunauer, Emmet and Teller (BET). The type of adsorption isotherm can be used to study the liquid and solid phase adsorption mechanism which in general adheres to the type of freundlich and langmuir isotherm. A good adsorbent has a high capacity of adsorption and percentage of absorption (Kyzas, 2012),

4. Adsorption Kinetic Model

The concept of adsorption kinetics pertains to the temporal relationship between the absorption of a material by an adsorbent. The adsorption rate provides insight into the adsorbent's absorption properties with respect to the adsorbate. The determination of the adsorption rate can be achieved by the utilization of the adsorption rate constant (K) and the reaction order derived from an adsorption kinetics model (Hafiyah, 2013). There exist multiple commonly employed adsorption kinetics models that are utilized for the description and analysis of various adsorption phenomena. These models include the first pseudo-order adsorption model, the second pseudo-order adsorption model, the intraparticle adsorption model, and the intrafilm adsorption model (Jean Baptiste et al., 2020).

Materials & Methods

1. Materials and equipment

The necessary materials and equipment for this investigation consist of crude palm oil (CPO) sourced from PT. Syaukath Sejahtera, bentonite purchased from Ujong Pacu Village in Lhoksemawe City, Aceh Province, N-Hexane PA (Merck), and NaOH and HCl 0.1N (Merck).

2. Prepared adsorbent

The initial impurities present in the zeolite are first removed through a washing process using distilled water, followed by drying in a furnace at a temperature range of 300-400°C for a duration of 4 hours. Subsequently, the zeolite is immersed in a glass beaker containing a 1 N solution and allowed to soak for 4 hours. Finally, the zeolite is subjected to heat in a water bath until it reaches a state of dryness. To achieve a neutral pH or a pH close to 7, zeolite is rinsed with distilled water. The zeolite is subsequently subjected to a drying process within an oven, where it is exposed to a temperature of 1100°C for a duration of 2 to 3 hours (Nor Shafizah et al., 2022).

3. Adsorption Process

The adsorption procedure of free fatty acids (FFA) utilizing the Bentonite Ujong Pacu adsorbent involves manipulating the weight of the crude palm oil (CPO) samples, specifically 10 grams, 15 grams, 20 grams, 25 grams, and 30 grams. Subsequently, the adsorption process of free fatty acids (FFA) was conducted at a temperature of 70 oC for a duration of 4 hours. The levels of free fatty acids were then monitored at 30-minute intervals using the titration of acid-base using a secondary solution of NaOH 0.1 N (Syahwandi et al., 2019).

4. Isotherm Adsorption.

Isothermal adsorption refers to the phenomenon of adsorption occurring under conditions of constant temperature. The Langmuir isotherm model and the Freundlich isotherm model are the most prevalent and extensively employed models for adsorption. The Freundlich isotherm is commonly employed in the evaluation of adsorption processes, specifically when assessing the interaction between adsorbates and adsorbents (Kyzas, 2012). This is accomplished by utilizing the Freundlich equation, which can be expressed as follows:

$$qe = K_F. Ce^{1/n} \tag{1}$$

Where: qe = the amount of substance adsorbed (mg/g); Ce = concentration of solutes, K_F and n = adsorption capacity and intensity of adsorption. While the linear shape can be seen in the equation:

$$logqe = logK_F + 1/nlog Ce$$
(2)

The Langmuir isotherm is employed for the assessment of adsorption phenomena between an adsorbate and an adsorbent. This evaluation is conducted by utilizing the Langmuir equation, which is expressed as follows:

$$qe = qm. KL \frac{ce}{1 + KL.Ce}$$
(3)

While the linear shape can be seen in the equation:

$$\frac{Ce}{qe} = \frac{1}{qm.KL} + \frac{Ce}{qm} \tag{4}$$

Where: qm = Maximum adsorption capacity (mg/g; KL = adsorption constant langmuir (l/mg) The qm and KL constants can be determined by analyzing the intersection and slope of the linear relationship between Ce/qe and Ce.

5. Kinetic Adsorption Model

The analysis of the adsorption kinetics of phosphorus onto soil adsorbents involved the utilization of the pseudo-firstorder and pseudo-second-order models models (Jean Baptiste et al., 2020). The statistical criteria employed to assess the adequacy of the models in representing the experimental data were the coefficients of determination (r²). The pseudo-firstorder kinetic model, presented by Lagergren, is expressed by Equation (5):

$$\frac{dq_t}{dt} = k_1(qe - qt) \tag{5}$$

Integrating Equation (5) between the limits from initial conditions qt = 0 at t = 0 to qt = qt at time t yields:

$$qt = qe[1 - \exp\left(-k_1t\right)] \tag{6}$$

where k_1 is the rate constant of adsorption (g/min), qe is the amount of FFA adsorbed at equilibrium (mg/g), and qt is the amount of FFA adsorbed at time t (mg/g).

Ho's pseudo-second-order kinetic model is expressed by Equation (7)

$$\frac{dq_t}{dt} = k_2 (qe - qt)^2 \tag{7}$$

For initial conditions qt = 0 at t = 0 the integrated form of Equation (7) is:

$$qt = \frac{q_{\tilde{e}}k_2t}{1+q_{e}k_2t} \tag{8}$$

where k_2 is the rate constant of adsorption (g/(mg.min)), qe is the amount of FFA adsorbed at equilibrium (mg/g), and qt is the amount of FFA adsorbed at time t (mg/g).

Results and Discussion

1. The Effect of CPO Weight on FFA Levels

Free fatty acids (FFAs) are acidic compounds that are liberated through the process of hydrolysis of lipids. High-quality palm oil is characterized by a low free fatty acid (FFA) content and a high bleaching power. During storage, it is crucial to ensure that both the FFA level and the bleaching power remain stable over an extended period without significant alterations. The acid-base titration method was employed to conduct an analysis of free fatty acid levels. The fundamental premise underlying the study of free fatty acid (FFA) content involves the determination of the FFA content as a weight

percentage (w/w) of the total fruit content in palm oil (CPO), with the molecular weight of the FFA being 256 (specifically referring to palmitic acid).



Figure 1. Relationship between the weight of crude palm oil and the quantity of free fatty acid

According to the data presented in Figure 1, it can be observed that the lowest concentration of free fatty acids (FFA) is found at a CPO weight of 10 grams, resulting in an FFA percentage of 0.1891%. Conversely, the highest concentration of FFA is observed at a CPO weight of 30 grams, yielding an FFA percentage of 0.4332%. The analysis data reveals that the average free fatty acid (FFA) level in crude palm oil (CPO) obtained from storage tanks is as follows: 3.44%, 3.54%, 3.64%, and 3.68%, with an overall average of 3.57%. Based on the acquired data, it can be asserted that there is a daily increment in free fatty acid (FFA) levels. However, it is noteworthy that these levels remain within the quality benchmarks established by PKS Tandun, specifically falling between the range of 3.00 to 4.5%. Elevated concentrations of free fatty acids have been found to adversely impact the overall quality of crude palm oil (CPO). Specifically, these high levels can induce rancidity, resulting in an undesirable taste, discoloration, and a reduction in oil output. In order to mitigate the levels of free fatty acids (FFA), it is imperative to implement preventative measures at the earliest stages of the production process, starting from the harvesting phase and continuing through the storing phase prior to marketing (Syahwandi et al., 2019).

2. Effect of FFA Concentration

Figure 2 depicts the curve illustrating the adsorption process of FFA with varying amounts of palm oil (CPO), employing Ujong Pacu bentonite as the adsorbent. The adsorption efficiency exhibited a notable increase, rising from 75% to 93%, when the FFA concentration reached 4,332.47 mg/L. This increase in adsorption efficiency was found to be directly proportional to the adsorption capacity, which continued to rise from 177.24 (mg/g) to 502.17 (mg/g). According to Miranda et al (2016) desirable adsorbent is characterized by its elevated adsorption capacity and efficiency. Bentonite is composed of montmorillonite, a mineral with a significant interlayer space size that enables it to possess a high adsorption capacity. Consequently, it serves as an efficient adsorbent for the adsorption of free fatty acids (Ayu Putranti et al., 2018).

The utilization of an adsorbent mass of 2 g, characterized by a particle size of 200 mesh, demonstrates a highly effective absorption of free fatty acids (FFA) from crude palm oil (CPO) at various concentrations. The experimental findings at a concentration of 10 grams of CPO yielded an initial free fatty acid (FFA) value of 1,890.54 milligrams per liter (mg/L), along with an adsorption capacity (Qe) value of 177.24 milligrams per gram (mg/g) and an absorption efficiency (EP) of 75%. A concentration of 15 grams of CPO yielded an initial FFA value of 3,150.89 milligrams per liter (mg/L), along with an adsorption capacity (Qe) value of 334.78 milligrams per gram (mg/g) and an absorption efficiency (EP) of 85%. A solution containing 20 grams of CPO was used to determine the initial FFA concentration, which was found to be 3,544.75 milligrams per liter. The adsorption capacity (Qe) of the CPO was measured to be 398.78 milligrams per gram, and the absorption efficiency was determined to be 90%. A concentration of 25 grams of CPO yielded an initial FFA value of 3,781.07 milligrams per liter, along with an adsorption capacity (Qe) value of 4,332.47 milligrams per liter (mg/L). The adsorption capacity (Qe) of the CPO was determined to be 502.17 milligrams per gram (mg/g), with an absorption efficiency (EP) of 92.73%.



Figure 2. Effect of FFA Concentration on Absorption Capacity and Absorption Efficiency

3. Isotherm Adsorption

Isothermal adsorption refers to the phenomenon of solute adsorption onto solid surfaces under conditions of constant temperature and pressure. Figures 3a and 3b depict the graphs illustrating the FFA adsorption isotherms utilizing Ujong Pacu Bentonite. The observed discrepancy in linearization between the Langmuir and Freundlich equation tests is evident from Figures 3.a and 3.b. The Langmuir equation exhibits a coefficient of determination (R2) value of 0.9683, while the Freundlich equation demonstrates a higher R2 value of 0.9992. The Langmuir isotherm was linearized, resulting in the determination of slope and intercept values from the equation Y = 0.00007x + 0.08190. In equation (2.2), the variable b represents the Langmuir isotherm constant, which is measured in units of L/mg. Additionally, Qo denotes the maximum absorption capacity of the monolayer, expressed in units of mg/g. The linearization of the Freundlich equation was performed, resulting in the equation y = 1.2686x - 4.3938, where y represents the dependent variable and x represents the independent variable. The slope and intercept values obtained from this linearization process were utilized in the equation 2.4.





The R2 values obtained for both the Langmuir and Freundlich models exceed 96%, indicating that these models are suitable for describing the adsorption of free fatty acids (FFA) on crude palm oil (CPO) employing bentonite as an adsorbent. The obtained R2 value suggests that the Freundlich equation is a better appropriate model for describing the adsorption process of Free Fatty Acids (FFA) using Ujong Pacu Bentonite adsorbent in Lhokseumawe. The Freundlich isotherm characterizes the concurrence between the empirical data and the model, demonstrating its superior suitability compared to the Langmuir model. The discovered correlation coefficient value of 0.9992 indicates that the absorption model investigation exhibits a multilayer phenomenon, occurring across many layers. The Freundlich Model yielded a KF value of 0.00003815 and a n value of 1.2718. Table 1 displays the parameter values for the linear regression of the Langmuir and Freudlich equations.

Table 1. Adsorption equilibrium of the Langmuir and Freundlich models								
Equilibrium Adsorption	Persamaan linear	Konstanta		R ²				
Langmuir	qe/Ce = 0,00007 - 0,08190 qe	b (L/mg) Qo	0,08190 0,0009	0.9683				
Freundlich	Ln Qe = -7,8714 + 1,2718 ln Ce	K _F 1/n n	0,00003815 1,2718 0,7863	0.9992				

4. Adsorption Capacity

The results of the FFA adsorption procedure utilizing bentonite are presented in Figure 6, illustrating the adsorption capacity and efficiency. The adsorption capacity refers to the quantity of adsorbate that is deposited on the surface of the adsorbent. This accumulation occurs in order to achieve optimal circumstances throughout the adsorption process, namely in the case of bentonite when the maximum capacity is reached. The data presented in Figure 4 demonstrates that there is a positive correlation between contact time and the rate of FFA removal by bentonite. This suggests that the adsorption capacity of FFA tends to increase with longer contact times, albeit not to a substantial extent.



Figure 4. The relationship between absorption capacity and time

The research trends observed align with the findings published in previous studies conducted by (Purwasasmita et al., 2015). Cowan et al. (2012) reported that the adsorption capacity of FFA is dependent on the duration of contact, as evidenced by the findings that the inclusion of 1% lipase and 2.0% (w/w) glycerol after 14 hours of contact time resulted in a reduction of FFA from 4.98% to 1.73%, or a 65.26% decrease. According to Purwasasmita et al. (2015), the adsorption capacity of the CPO FFA was found to increase with longer contact time. The adsorption capacity of FFA CPO reached its maximum value during a contact time of 240 minutes. Salman et al. (2011) found that there is a positive correlation between the duration of the contact between the solution and the adsorbent and the quantity of adsorbate that is adsorbed onto the surface of the adsorbent. Riyadi et al. (2016) also observed a similar pattern, whereby the duration of contact had an impact on the adsorption capacity of FFA. Specifically, it was noted that a longer contact time resulted in a higher adsorption capacity of the adsorbent.

5. Kinetic Adsorption

During the process of adsorption, molecules (referred to as adsorbate) undergo a transfer from the liquid phase to the surface of the adsorbent material. The investigation of the kinetics of adsorption is crucial for elucidating the underlying adsorption mechanism and evaluating the efficacy of the adsorption process. In this study, the investigation of adsorption kinetics involved the utilization of three distinct kinetic models: the pseudo first order and pseudo second order models. These models were employed to assess the adsorption process of free fatty acids (FFA) onto bentonite and to determine the reaction rate constant (k) (Jean Baptiste et al., 2020).

Figures 5, depict the kinetics of bentonite, namely the pseudo first order and pseudo second order models, under different weight conditions of palm oil. The kinetic parameter values pertaining to the first-order pseudo reaction are presented in Table 2. The experiment used three different concentrations of CPO: 10 g, 20 g, and 30 g. The corresponding R2 values obtained were 0.9560, 0.7886, and 0.8755, respectively. The relatively low R2 value associated with the pseudo-first-order model suggests a lack of agreement between the kinetic model and the experimental results. Figure 6 illustrates that the adsorption kinetics of this model are more favorable for lower concentrations.



Figure 5. kinetic adsorption models can be visualized by plotting the ln (qe-qt) versus time. a) pseudo-first-order and b) pseudo-first-order

Table 2 presents the pseudo second order kinetic parameters for the adsorption of free fatty acids (FFA) on bentonite, with varying weights of crude palm oil (CPO) at 10 gr, 20 gr, and 30 gr. The data presented in Table 2 illustrates the relationship between the dependent variable, the reciprocal of quantum efficiency (1/qe), and the independent variable, the reciprocal of temperature (1/t). The experiment involved measuring the Linear Regression value (R2) for different concentrations of CPO solution. A concentration of 10 g resulted in an R2 value of 0.946, while a concentration of 20 g yielded an R2 value of 0.9975. Finally, a concentration of 30 g resulted in an R2 value of 0.9777.

A higher coefficient of determination (R2) indicates that the pseudo second order model is more appropriate for fitting the experimental data on adsorption capacity and equilibrium. The pseudo second order experimental model exhibits a good fit with the regression parameters. It may be inferred that the rate-determining phase of the pseudo-second-order model has undergone chemisorption, which involves the interaction of valence forces through the sharing or exchanging of electrons between the adsorbent and the adsorbate. Bentonite has -Si-O- groups that effectively engage in Vander Waals interactions with the carboxylic groups found in free fatty acids (Nayak et al., 2017).

Table 2. I focesses determine the pseudo-inst-order and pseudo-inst-order models computation parameters.							
Kinetics Adsorption	R ²	k_2	k_1	Qcalc	Qexp	CPO	
models		(g mg-1 min-1)	(m1n-1)	(mg/g)	(mg/g)	(gr)	
pseudo-first-order	0,956	-	0,0103	124,6363	118,1583	10	
	0,7886	-	0,0196	724,8031	354,4750	20	
	0,8755	-	0,0181	400,1342	256,0097	30	
pseudo-second- order	0,946	2E-05	-	232,558	118,1583	10	
	0,9975	1,3E-07	-	3.333,33	354,4750	20	
	0,9777	8,6E-07	-	1.428,57	256,0097	30	

Table 2. Processes determine the pseudo-first-order and pseudo-first-order models computation parameters

Conclusions

The Ujong Pacu bentonite sample is characterized by the presence of montmorillonite, a mineral known for its effectiveness at assisting FFA absorption experiments. This study investigates the impact of varying concentrations of CPO weight (10, 15, 20, 25, and 30 grams) on the adsorption capacity. The obtained results indicate that the adsorption capacity values were 177.24 mg/g, 334.79 mg/g, 398.79 mg/g, 431.28 mg/g, and 502.18 mg/g, respectively. The optimal absorption condition for bentonite at equilibrium is achieved when a weight of 30 g of CPO is used, resulting in an absorption efficiency of 92.73%. The Freundlich adsorption isotherm equation was utilized to assess the absorption capability of free fatty acids (FFA) on crude palm oil (CPO) using Bentonite Ujong Pacu. The obtained correlation coefficient (R2) was determined to be 0.9992. The adsorption capacity of CPO was determined for different weights of the material. Specifically, for 10 g of CPO, the adsorption capacity (Qt) was found to be 118.16 mg/g. Similarly, for 20 g of CPO, the adsorption capacity was measured to be 354.48 mg/g, while for 30 g of CPO, the adsorption capacity was determined to be 256.01 mg/g. The kinetics model for FFA adsorption as proposed by CPO adheres to a pseudo second-order model, with R2 values ranging from 0.946 to 0.9975.

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