

Adsorption of Acacia Bark (*Acacia crassicarpa*) on Cd(II) Metal in Solution

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ABSTRACT

Adsorption of Cd(II) metal from aqueous solution using acacia bark powder as adsorbent was performed in this study. This study aims to determine the efficiency and the best adsorption capacity of Acacia bark powder in Cd(II) adsorption with adsorbent dosage and pH parameters. The method used in this study is Cd adsorption (II) batch. Acacia bark powder was characterized using SEM, FTIR and BET to determine surface morphology, powder functional group, powder surface area, pore volume and pore size. Based on research that has been done the best dosage conditions and PH optimization obtained at 3.5 g, pH 8 and adsorption efficiency reached 98.81% and a capacity of 1.3763 mg/g. It is believed that the Acacia bark powder (*Acacia crassicarpa*) has a high ability to adsorb Cd(II) metal at dose and pH variations.

Keywords: Aacacia (Crassicarpa), adsorbent, adsorption, dosage, metal Cd(II), pH.

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INTRODUCTION

Acacia (*Acacia crassicarpa*) is one of the plant species cultivated in Industrial Plantation Forests (HTI) in Indonesia, playing a significant role in the development of the fiber wood industry (Palimbong et al., 2023). The cellulose content in acacia (*Acacia crassicarpa*) bark possesses hydroxyl (–OH) functional groups that have the potential to act as an adsorbent because they are capable of binding metal ions, allowing it to be used in removing heavy metals from wastewater (Aminah et al., 2018).

Adsorption is a series of processes involving the attachment of an adsorbate (pollutant) onto the surface of a solid material (adsorbent). The adsorption process can be influenced by several factors, including the adsorbent dosage and the pH of the solution. The effect of the adsorbent dosage is to determine the optimal amount of adsorbent used during the adsorption process (Siregar et al., 2019). The determination of the degree of acidity (pH) in the adsorption process can affect the charge of the active sites on the adsorbent surface and influence the solubility of metal ions in the solution, making pH a highly



crucial parameter (Rochmah et al., 2017). This is based on the concepts of protonation and deprotonation. At a low pH, the functional groups on the adsorbent surface are protonated and become positively charged or form (H^+) ions, facilitating easier interaction with anionic species or ($-OH$) ions, whereas under alkaline conditions, these groups are deprotonated and become negatively charged, making them highly reactive in forming bonds with metal cations (Nurfriyanti et al., 2017).

Heavy metals can be classified as metals with a density of $\geq 5 \text{ g/cm}^3$. This is related to the characteristics of heavy metals, which are difficult to degrade and can even be adsorbed into the bodies of organisms, such as $Cd(II)$ metal, which belongs to the category of hazardous heavy metals. $Cd(II)$ metal becomes dangerous when its

concentration exceeds the maximum permissible limit for consumption.

This research was conducted to utilize the abundance of acacia bark and the limited use of acacia (*Acacia crassicaarpa*) bark as an adsorbent; therefore, a study entitled the optimization of adsorbent dosage and solution pH on the adsorption of acacia (*Acacia crassicaarpa*) bark powder for $Cd(II)$ metal was carried out. Acacia (*Acacia crassicaarpa*) bark is highly suitable for the removal of color and metals in industrial wastewater because it is easily obtainable and contains higher cellulose levels than several other types of biological waste (Nong et al., 2023). Cellulose has considerable potential as an adsorbent material due to the presence of ($-OH$) groups that can interact with adsorbate components (Kusumawardan et al., 2018).

METHODS

Materials and Instruments

The materials used in this research were acacia (*Acacia crassicaarpa*) bark, cadmium(II) sulfate monohydrate ($CdSO_4 \cdot H_2O$), 0.1 N sodium hydroxide ($NaOH$), 0.1 N hydrochloric acid (HCl), 0.1 N sodium chloride ($NaCl$), demineralized water (H_2O), and Whatman No. 42 filter paper. The instruments utilized included Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) (APHA 3120B), Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy (SEM-EDS) (JED-2300 Analysis Station-JEOL), Fourier Transform Infra-Red (FTIR) (IR Prestige Spectrophotometer Shimadzu), and a Surface Area and Pore Size Analyzer (Quantachrome Quadrasorb-Evo).

Adsorbent Preparation

The acacia bark was washed with clean water and rinsed using distilled water, then cut into small pieces and dried under sunlight until the bark was completely dry.

The acacia bark was crushed using a grinder into a powder form, followed by further drying in an oven at a temperature of $105^\circ C$ for ± 2 days. The obtained powder was sieved using a mesh that allowed it to pass through 100 mesh but retained it at 200 mesh, resulting in a powder size of $100 \text{ mesh} < x < 200 \text{ mesh}$ (where x = particle size). Subsequently, the acacia bark powder was dried in an oven at $105^\circ C$ for 24 hours.

Adsorption Study of $Cd(II)$ Metal by Acacia

The adsorption process was conducted by adding 50 mL of a 100 ppm $CdSO_4 \cdot H_2O$ solution to the acacia bark powder at various adsorbent dosages (0.1, 0.3, 0.5, 0.7, 0.9, 2.5, and 3.5 g). Furthermore, the obtained optimum dosage was mixed into 50 mL of a 100 ppm $CdSO_4 \cdot H_2O$ solution under varying pH conditions (2, 4, 6, 8, and 10). The filtrate obtained from the filtration process was analyzed using Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES).

The adsorption efficiency and adsorption capacity were calculated using Equation (1) and Equation (2):

$$E (\%) = \left(\frac{C_0 - C_e}{C_0} \right) \times 100\% \dots\dots\dots (1)$$

$$Q (\text{mg/g}) = \left(\frac{C_0 - C_e}{W} \right) \times V \dots\dots\dots (2)$$

Determination of the Point of Zero Charge (pHpzc)

A volume of 50 mL of 0.1 N NaCl solution was poured into individual beakers, and the pH of the solution was then adjusted to varying pH levels of 2, 4, 6, 8, and 10 using 0.1 N HCl and 0.1 N NaOH solutions. After the initial pH was measured using a pH meter, the acacia bark powder was added into each beaker according to the optimum dosage that had been obtained.

RESULTS AND DISCUSSION

Adsorbent Characterization: Functional Group Analysis of Acacia Bark Powder

FTIR (Fourier Transform Infra-Red) is a spectroscopic method utilizing an infrared light source, used to determine chemical functional groups within the range of 400 to 4000 cm^{-1} . This analysis aims to identify active functional groups ($-\text{OH}$, $-\text{C}=\text{O}$, $-\text{COOH}$, etc.) and determine the capability of the bark in the metal adsorption process and its interaction with other molecules.

Characterization

Characterization was carried out using Fourier Transform Infra-Red (FTIR) with a scanning range of 400 to 4000 cm^{-1} , Scanning Electron Microscopy-Energy Dispersive X-Ray Spectroscopy (SEM-EDS) at magnifications of 1,000x, 5,000x, and 10,000x on the SEM with an accelerating voltage of 10,000 volts and an emission current of 1.00000 nA, followed by a Surface Area and Pore Size Analyzer, Brunauer-Emmett-Teller (BET) using nitrogen (N) gas, with a molecular weight of 28.013 g/mol, a temperature of 77.35 K, a molecular cross-sectional area of 16.200 $\text{\AA}^2$, and a liquid density of 0.808 g/cm^3 .

Data Analysis

The obtained data were statistically analyzed using Duncan's post hoc test if there were significant differences in the analysis results.

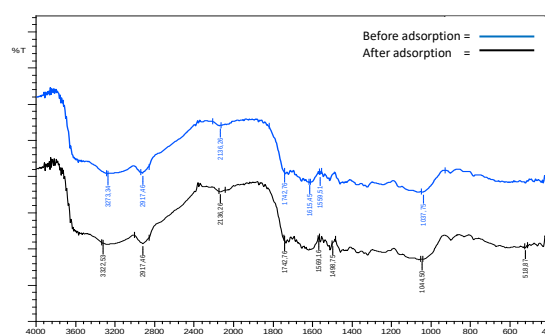


Figure 1. IR spectrum of acacia bark powder

Based on Figure 1, it can be observed that the functional groups of the acacia bark powder before and after Cd adsorption do not differ significantly, which is indicated by a shift in the spectral wavenumbers at the

resulting peaks. The wavenumbers of the FTIR spectral peaks can be seen in Table 1.

Table 1. Wavenumbers of acacia bark powder before and after activation, as well as after adsorption.

Functional Group	Wavenumber (cm^{-1})	
	Before Adsorption	After Adsorption
O-H	3273,34	3322,53
C-H	2917,46	2917,46
C-C	2136,26	2136,26
C=O	1742,76	1742,76
C=C	1615,45- 1559,51	1569,16- 1498,75
C-O	1037,75	1044,50
O-Cd	-	518,87

The peak shift in the absorption band from 3273.34 to 3322.53 cm^{-1} indicates the presence of a hydroxyl (O-H) group. The O-H functional group indicates the presence of cellulose content in the acacia bark powder. The peak at the absorption band of 2917.46 cm^{-1} indicates the absorption of an alkyl (C-H) group, which is the

building framework of the lignocellulosic structure. This result is reinforced by the ether (C-O) group in the regions of 1037.75 cm^{-1} and 1044.50 cm^{-1} , which act as the carbon chain links in lignocellulosic compounds (Irawati and Wardani, 2021). The aromatic C=C functional groups at the absorption band peaks of 1615.45–1559.51 cm^{-1} and 1569.16–1498.75 cm^{-1} overlap with the carbonyl (C=O) group from the carboxylic acid (COOH) group, which constitutes the building block of hemicellulose (Irawati and Wardani, 2021). Furthermore, the emergence of a new peak at 518.87 cm^{-1} , identified as the O-Cd group, confirms that the Cd(II) metal has been adsorbed (Mathew et al., 2009).

Surface Morphology and Elemental Content Analysis of the Adsorbent

Surface morphology analysis of the acacia (*Acacia crassicaarpa*) bark powder was conducted to observe the shape, structure, and pore distribution on the powder surface before and after the adsorption of Cd(II) using a Scanning Electron Microscopy (SEM) instrument.

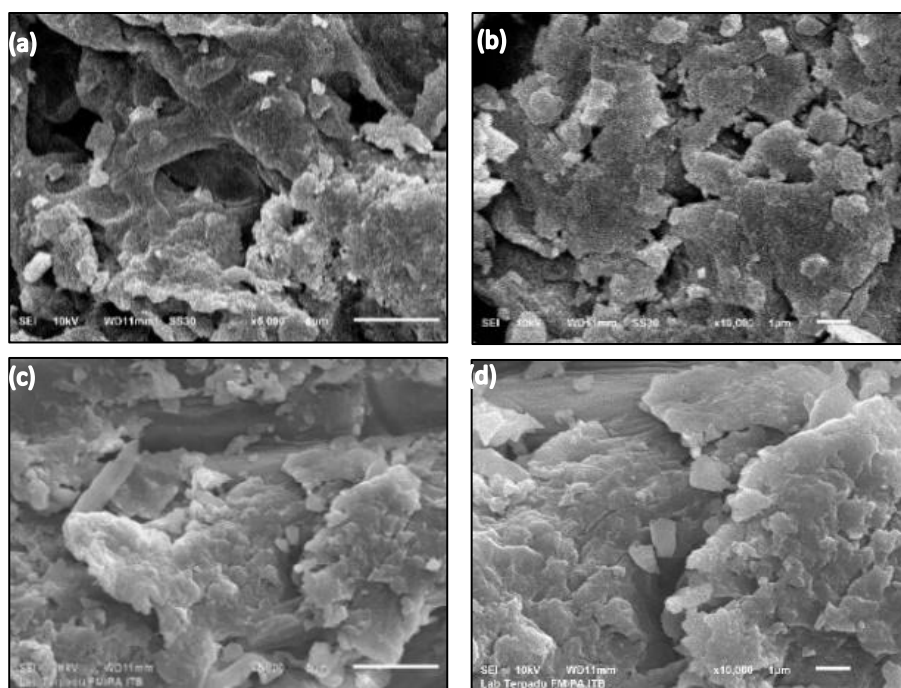


Figure 2. Results of the surface morphology analysis of acacia (*Acacia crassicaarpa*) bark powder using the SEM instrument before adsorption at magnifications of 5,000x (a) and 10,000x (b), and after adsorption at magnifications of 5,000x (c) and 10,000x (d).

The morphology of the acacia bark powder before adsorption was analyzed at magnifications of 5,000x and 10,000x. The results of the surface morphology analysis of the acacia bark powder can be seen in Figure 2. The acacia bark powder before adsorption appeared rough and irregular, with slightly open and hollow pores. After adsorption, the surface morphology of the adsorbent underwent slight changes. The adsorbent surface became more compact, with pores appearing smooth and closed, indicating the successful adsorption of Cd(II) metal.

Table 2. Elemental content of the bark powder adsorbent before and after adsorption

Element	Elemental Composition in the Sample (wt%) Before Adsorption	Elemental Composition in the Sample (wt%) After Adsorption
C	16.88	57.67
N	7.49	24.69
O	67.54	12.88
Al	1.88	0.24
K	2.34	0.26
Ca	1.47	0.51
Mn	0.65	0.43
Fe	1.75	-
Cd	-	0.67

The results indicate that the acacia (*Acacia crassicaarpa*) bark powder before

adsorption contained the elements C, N, O, Al, K, Ca, Mn, and Fe. The dominant elements in the acacia (*Acacia crassicaarpa*) bark powder were O (67.54 wt%) and C (16.88 wt%).

The adsorption process was confirmed by the O element, which experienced a decrease from 67.54% to 12.88%, indicating an interaction with Cd, and was evidenced by the presence of the Cd element with a mass percentage of 0.67% after adsorption. The composition of the C element before adsorption was lower compared to that after adsorption, indicating that the sample was in a powder form without any activation (Khatoon et al., 2018).

Analysis of the Surface Area, Pore Volume, and Pore Diameter of Acacia Bark Powder

The pore characterization of the acacia (*Acacia crassicaarpa*) bark powder was conducted using the Brunauer-Emmett-Teller (BET) method on the adsorbent before adsorption. The Barrett-Joyner-Halenda (BJH) method serves to measure the pore size distribution, specifically within the mesoporous range. Density Functional Theory (DFT) functions to provide more accurate calculation results for the pore diameter (Thommes et al., 2015).

Table 3. Analysis of the surface area, pore volume, and pore diameter of acacia bark powder

Surface Area (m ² /g)	Mesopore Diameter (Å)	Total Pore Volume (cc/g)	Average Pore Diameter (Å)
0.46	- 32.707 (BJH Adsorption) - 31.561 (BJH Desorption) - 33.152-42.426 (DFT)	0.004361	379.007

The results of the characterization above indicate that the acacia bark powder falls into the mesoporous category, with a pore diameter ranging from 32.707 to

42.426 Å. This is also supported by the low surface area data of the adsorbent (0.460 m²/g) for pores < 396.6 Å. This pore size is significantly larger than the diameter of the

Cd(II) ion ($0.97 \text{ \AA}$), thus enabling the metal ions to easily diffuse and bind to the active surface of the adsorbent.

The nitrogen adsorption-desorption isotherm curve in the figure displays a Type IV isotherm with a hysteresis loop, which indicates the presence of a mesoporous structure (2–50 nm) resulting from the phenomenon of capillary condensation.

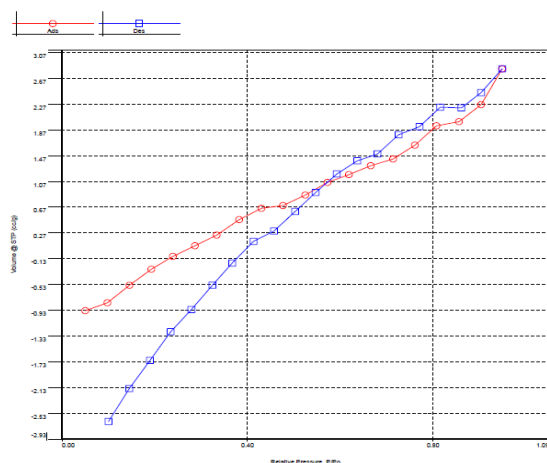


Figure 3. N_2 adsorption-desorption isotherm curve of acacia bark powder

Adsorption of Cd(II) by Acacia (*Acacia crassicarpa*) Bark Powder

Dosage Optimization

The determination of adsorption efficiency and capacity was conducted to establish the optimal conditions for acacia bark powder against Cd(II) using dosage and pH parameters. The adsorption test results of the acacia bark powder for Cd(II) with dosage variations can be seen in Table 4 and the graph in Figure 4.

Table 4. Results of the adsorbent dosage optimization of acacia (*Acacia crassicarpa*) bark powder for Cd(II) metal

Dosage (g)	Adsorption Efficiency (%)	Adsorption Capacity (mg/g)
	E	Q
0,1	$90,34 \pm 0,21$	$44,6731 \pm 0,10^g$
0,3	$92,52 \pm 0,78$	$15,2513 \pm 0,13^f$
0,5	$92,92 \pm 0,56$	$9,1900 \pm 0,06^e$

0,7	$93,15 \pm 1,06$	$6,5809 \pm 0,08^d$
0,9	$93,50 \pm 1,53$	$5,1372 \pm 0,09^c$
2,5	$94,52 \pm 0,29$	$1,8696 \pm 0,00^b$
3,5	$96,76 \pm 0,10$	$1,3671 \pm 0,00^a$

Based on the results of Duncan's test, variations in adsorbent dosage demonstrated a significant difference in adsorption capacity. The optimal dosage of acacia bark powder for Cd(II) occurred at 3.5 g, yielding an adsorption efficiency of 96.76% and an adsorption capacity of 1.3671 mg/g. The adsorbent dosage of 3.5 g was selected as the optimum condition because it produced the highest adsorption efficiency of 96.76%, indicating the most effective adsorbate removal capability from the solution. This was despite the 0.1 g dosage exhibiting a higher adsorption capacity of 44.6731 mg/g. Therefore, the determination of the optimum condition was based on the adsorption efficiency value, as this parameter represents the level of adsorbate removal from the solution achieved by the adsorbent.

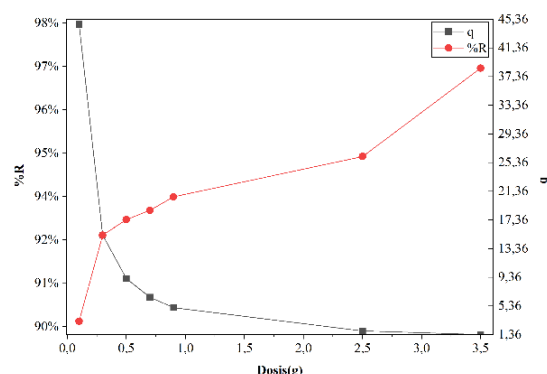


Figure 4. The relationship between adsorbent dosage and the adsorption capacity and efficiency of Cd(II) metal.

The increase in the amount of adsorbent used leads to an increase in adsorption efficiency due to the greater number of active sites available on the adsorbent surface. However, the adsorption capacity exhibits a decreasing trend as the adsorbent dosage increases. This occurs because the adsorption capacity is expressed as the amount of bound metal ions per unit mass of the adsorbent; the use of a large

quantity of adsorbent causes the distribution of metal ions in the solution to become relatively small compared to the amount of adsorbent, leaving many active sites suboptimally occupied. An increase in the adsorbent dosage lowers the metal ion-to-adsorbent ratio, resulting in the majority of active sites not being optimally filled. This condition induces a shift in the adsorption equilibrium and results in a decrease in the adsorption capacity per unit mass of the adsorbent (Sy et al., 2018).

pH Optimization

The pH_{pzc} value was obtained from the line intersecting the x-axis, with a pH_{pzc} value at the point of 4.7. The best efficiency and capacity obtained at a dosage of 3.5 g were 96.76% and 1.3671 mg/g, respectively. After determining the optimal dosage, the evaluation of the pH parameter was continued to achieve adsorption optimization. The adsorption results of the acacia bark powder for Cd(II) under varying pH conditions can be seen in Table 5 and the graph in Figure 5.

Table 5. Results of the solution pH optimization of acacia bark powder for Cd(II) metal

pH	Adsorption Efficiency (%) E	Adsorption Capacity (mg/g) Q
2	92,45±0,29 ^a	1,3010±0,06 ^a
4	97,08±0,18 ^b	1,3661±0,00 ^b
6	97,74±0,07 ^c	1,3754±0,00 ^c
8	98,81±0,12^c	1,3763±0,00^c
10	98,21±0,19 ^d	1,3822±0,06 ^d

The results of Duncan's test indicated that variations in the solution pH after contact with the adsorbent resulted in significant differences in adsorption efficiency and capacity, except at pH 6 and 8, where no significant differences were observed. The optimum pH value of the acacia (*Acacia crassicarpa*) bark powder for Cd(II) occurred at pH 8, with an adsorption efficiency of 98.81% and an adsorption capacity of 1.3763 mg/g.

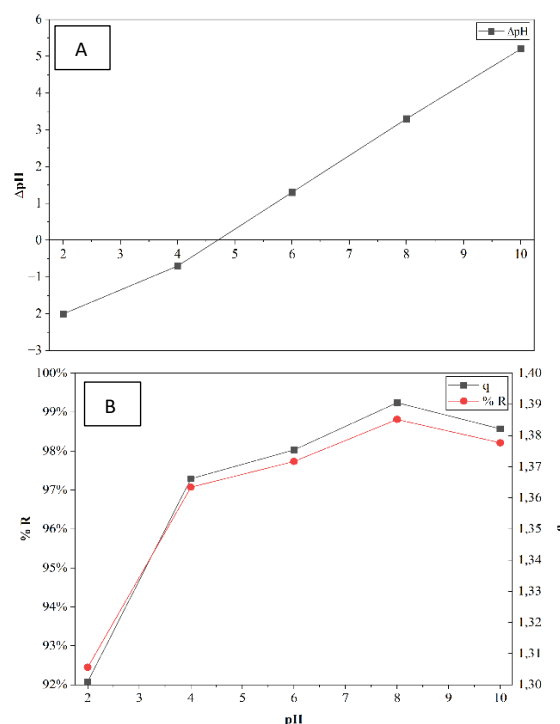


Figure 5. The pH_{pzc} value of acacia bark powder (A) and (B) the relationship between solution pH and the adsorption capacity and efficiency of Cd(II) metal.

At acidic pH levels or below pH 5, the adsorbent becomes protonated, resulting in electrostatic repulsion towards Cd^{2+} ions and preventing the adsorbent from achieving maximum adsorption. The adsorption of Cd^{2+} ions increased at pH 6 and 8. This is due to a reduction in competition between H^+ ions and positively charged metal ions (Cd^{2+}) on the adsorbent surface, resulting in decreased repulsion towards Cd^{2+} ions and enhancing the occurrence of the metal ion adsorption process onto the adsorbent (Wijaya and Ita, 2015).

At low pH values, the material surface is positively charged; therefore, the interactive force is primarily an electrostatic force. In addition, if the concentration of H^+ ions is high, competition with metal cations during the adsorption process causes a decrease in efficiency. When the pH is higher than the pH_{pzc} (4.7), the adsorption efficiency and capacity increase. A high pH value indicates that the adsorbent surface is negatively charged, but if the pH is too high,

the solution becomes alkaline, making more OH^- ions available. The combination of Cd^{2+} ions with abundant OH^- ions can lead to the precipitation of $\text{Cd}(\text{OH})_2$ (Nguyen et al., 2020).

The adsorption efficiency and capacity values at pH 8 exhibited an increase in the adsorption percentage. This is due to the deprotonation of the adsorbent's functional groups, which produces a negative charge, thereby strengthening the interaction with $\text{Cd}(\text{II})$ ions. Conversely, the adsorption

percentage at pH 10 experienced a slight decrease caused by the formation of $\text{Cd}(\text{OH})_2$ precipitates, which are difficult to be adsorbed by the hydroxyl groups on the adsorbent. Under higher pH conditions, Cd^{2+} ions react with OH^- ions to form precipitates that can cover the adsorbent surface and reduce adsorption effectiveness (Sihotang, 2021).

CONCLUSION

Acacia (*Acacia crassicaarpa*) bark powder influences the adsorption efficiency and capacity for the adsorption of $\text{Cd}(\text{II})$ metal, with the optimal adsorption

conditions achieved at a dosage of 3.5 g and a pH of 8, yielding an adsorption efficiency of 98.81% and an adsorption capacity of 1.3763 mg/g for $\text{Cd}(\text{II})$.

EXPRESSION OF GRATITUDE

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