

Batch Adsorption Study: Sequestration of Cr(VI) ions from Aqueous Solution Utilizing *Boerhavia diffusa*

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In light of the ongoing contamination of water sources by aqueous waste from the electroplating, printing, pharmaceutical, dye and alloy industries, this work describes a new adsorbent for removing chromium ions (Cr(VI)) from a lab-prepared aqueous solution. As an adsorbent, pure and activated form of *Boerhavia diffusa* (abbreviated as RBD and ACBD), also known as Punarnava (in Hindi), was utilized. The adsorbents were characterized for their structure, morphology and thermal analysis through X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM) and thermal gravimetric analysis (TGA). The FTIR spectrum detected various functional groups *viz*. -OH, -CHO, RCO, -CN and C=H, implying strong bonding with metals ions. The amorphous and porous character of the adsorbent was confirmed by SEM and XRD. Consequences of other domains, including adsorbent dose, contact time, pH, concentration and temperature, were also investigated. With a starting concentration of 20 mg/L, pH of 7 (RBD), 5 (ACBD), adsorption dose of 20 mg and temperature of 303 K, the effective adsorption was achieved after 2 h and these were 86.4% and 92.7% in terms of removal efficiency by RBD and ACBD, respectively. The respective monolayer maximum adsorption capacity for RBD and ACBD were 66.28 mg/g and 135.81 mg/g. The Langmuir and pseudo-second order reactions were well-matched the experimental results for RBD and ACBD precisely.

Keywords: Isotherm, Kinetics, Boerhavia diffusa, Adsorption Capacity, Thermodynamics.

INTRODUCTION

The previous decade has shown an exponentially growing trend in the use of heavy metals in various industrial applications, thereby generating a substantial amount of toxic waste. It has been ascertained that even at low concentrations, heavy metals are hazardous and can have lethal impacts on flora and fauna [1]. In particular, chromium metal ions from these toxic wastes are more harmful and have a deeper impact. Chromium ions originate from natural and anthropogenic sources such as minerals beneficiation, mining and effluents from various industries. The natural process of degradation of such toxic heavy metals is a chronic process and their long-term presence is harmful to the ecological balance [2,3]. This critical environmental concern inspires scientists to develop innovative technologies that enable the timely and efficiently removal of harmful components from the environment.

Various researchers have reported diverse technologies for removing heavy metals from these effluents. Biological remediation, membrane filtration, ion-exchange, coagulation, osmosis, adsorption, reverse osmosis, chemical precipitation, photocatalytic degradation, *etc.* are a few methods to name [4-6]. Majority of these methods are complicated to process, generate harmful solid waste and expensive and difficult to maintain due to a lack of raw materials. On the contrary, adsorption is a relatively simple and superior alternative for removing heavy metals from the wastewater [7].

Variety of adsorbents from the natural as well as industrial by products have been developed. Natural adsorbents can be utilized with or without processing or in surface modification, *i.e.* activation, doping, *etc.* Various materials used for the removal of chromium ions (Cr(VI)) are banana peel, papaya peel, mango kernel, ground nut, teff straw and newspaper to name a few [8-13]. Present methodology employ the raw and activated forms of *Boerhavia diffusa* as an adsorbent, which is commonly called "Punarnava" and is a traditional remedy for the stomach infections [14]. Punarnava is a creeping weed perennial plant found abundantly in India and can be pre-

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processed for adsorption with the minimal efforts and cost. The major component of Punarnava biomass is lignin, nucleoside and steroids [15]. The present expermental findings confirm *Boerhavia diffusa* as a potential adsorbent for removal of chromium(VI) from the wastewater.

EXPERIMENTAL

With the cooperation of the Horticulture Department, *Boerhavia diffusa* was obtained from the Deenbandhu Chhotu Ram University of Science and Technology, Murthal (Sonipat) campus. Sulfuric acid and Potassium dichromate were purchased from Merck India Ltd. and Fisher Scientific, respectively. To maintain pH, HCl and NaOH were used and procured from Merck India Ltd. Double distilled water was used throughout the experimentation work.

Adsorbent preparation: *Boerhavia diffusa* aggregated from the university campus was washed with hot water (about 40-60 °C) to remove dust and impurities and sun dried. The sun dried material was dried at 110 ± 5 °C in a hot air oven to remove moisture. Then it was ground with a domestic grinder mixer and segregated to different sizes by a sieve shaker in Mechanical Operation Lab. Materials of 300 mesh size were abbreviated as RBD and used for further experimentation and characterization.

A chemical activation process was employed to activate *Boerhavia diffusa* and conc. H₂SO₄ was used for activation. The RBD was soaked with conc. H₂SO₄ for 48 h at room temperature in the ratio of 1:1 (weight by volume). The surface activation was done by placing the material in the furnace at 600 °C for 20 min. Then, the material was neutralized with double distilled water, washed and dried in a hot air oven at 110 ± 5 °C, abbreviated as ACBD.

Characterization: Both adsorbents, RBD and ACBD were studied morphologically and structurally using distinct methodologies. ATR-Fourier transform infrared spectroscopy (FTIR) of Perkin-Elmer Frontier-BSEN60825 was utilized for chemical structure characterization. Each sample was scanned twice in the spectral region of 4000 to 400 cm⁻¹ to record the spectrum. To estimate the crystalline structure at the ambient temperature, the X-ray diffractometer Rigaku Miniflex-600 system was equipped with a Cu tube for generating CuK α radiation (k = 1.5406), as an incident beam in the 20 mode, with voltage 40 KV and current 30 mA. To examine the surface pattern or morphology of the RBD and ACBD, a JEOL-JMS-5600LV FESEM at 20 kV accelerating voltage was utilized. TGA absorption measurements were performed in the 200-800 nm range using a Perkin-Elmer-TGA 550 standard spectrometer.

Adsorption studies: Various adsorption parameters *viz*. contact time, starting concentration, temperature, pH, adsorbent dosages were studied in a 20 mL batch study of adsorption. The isothermal equilibrium behaviour (Langmuir, Freundlich, Temkin and Dubinin-Radushkevich isothermal models) and pseudo-first order, pseudo-second order and intraparticle molecular diffusion models were examined. Adsorption isotherms were computed using initial concentration parameters at constant optimum value for other variables, whereas kinetics

was investigated using time parameters. The removal efficiency was calculated as follows:

Removal (%) =
$$\frac{C_o - C_e}{C_o} \times 100$$
 (1)

Adsorption isotherms: Applying linear Langmuir, Freundlich, Temkin and Dubinin-Redushkvich isotherm models, adsorption isotherms were studied. The model and their linear equation are given below:

Langmuir isotherm:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}$$
(2)

Linear equation:

$$q_{e} = \frac{1}{q_{m}} + \frac{1}{q_{m}K_{L}C_{e}} \text{ or } \frac{C_{e}}{q_{e}} = \left(\frac{1}{q_{m}}\right)C_{e} + \frac{1}{q_{m}K_{L}}$$
 (3)

where q_e = Adsorption capacity at equilibrium (mg/g), C_e = concentration at equilibrium (mg/L), q_m = maximum monolayer adsorption capacity (mg/g), K_L = isotherm constant (L/mg).

Freundlich isotherm:

$$q_e = K_f C_e^n \tag{4}$$

Liner equation:

$$\log q_e = \log K_f + \frac{1}{n} \log C_e$$
(5)

where K_f = approximate adsorption capacity, n = adsorption intensity.

Temkin isotherm:

$$q_{e} = \frac{RT}{b_{T}} \ln \left(A_{T} C_{e} \right)$$
(6)

Liner equation:

$$q_e = B\ln(A_T) + B\ln(C_e)$$
(7)

where B = Constant related to heat of adsorption and is $B = RT/b_T$; $A_T = Temkin$ isotherm equilibrium binding constant (L/g), $b_T = Temkin$ isotherm constant, R = Universal constant, (J/mol).

Dubinin-Radushkevich (D-R) isotherm:

$$\mathbf{q}_{\mathrm{e}} = (\mathbf{q}_{\mathrm{s}}) \mathrm{e}^{-\mathrm{K}_{\mathrm{ad}}\varepsilon^{2}} \tag{8}$$

Liner equation:

$$\ln q_e = \ln(q_s) - (K_{ad} \in^2)$$
(9)

where
$$\in = \operatorname{RT} \ln \left(1 + \frac{1}{C_e} \right)$$
; q_s = isothermal saturation capacity

(theoretical) (mg/g), $K_{ad} = D-R$ isotherm constant (mol²/KJ²)

Kinetic studies: For studying the kinetics, three models were employed pseudo-first order, pseudo-second order model and intra-particle diffusion models. The empirical and linear equation expression are described below:

Pseudo first order model:

$$\frac{\mathrm{d}q}{\mathrm{d}t} = \mathbf{k}_1(\mathbf{q}_e - \mathbf{q}) \tag{10}$$

Linear equation:

$$\ln(q_{e} - q) = \ln(q_{e}) - k_{1}t$$
 (11)

where $q_e =$ adsorption capacity at equilibrium (mg/g), q = adsorption capacity at time t (mg/g), $k_1 =$ rate constant of adsorption (min⁻¹).

Pseudo-second order model:

$$\frac{\mathrm{d}q}{\mathrm{d}t} = k_2 (q_e - q)^2 \tag{12}$$

Linear equation:

$$\frac{t}{q} = \frac{1}{q_e} t + \frac{1}{k_2 q_e^2}$$
(13)

where $k_2 = rate constant of adsorption (g mg⁻¹min⁻¹).$

Intra-particle diffusion model:

$$\mathbf{q} = \mathbf{K}_{\mathrm{d}} \mathbf{t}^{1/2} + \mathbf{C} \tag{14}$$

where K_d = diffusion constant of adsorption (g mg⁻¹min⁻¹).

Thermodynamic studies: The thermodynamic analysis was carried out to examine the nature of adsorption reaction, *i.e.*, whether the reaction is spontaneous or non-spontaneous. Various parameters like Gibbs free energy (Δ G), entropy (Δ S), enthalpy change (Δ H) were determined for study. Δ G is related to the Langmuir constant (K_L) by following the equation [16].

$$\Delta G = -RT \ln K_{\rm L} \tag{15}$$

It can be rewritten as:

$$\ln K_{L} = \frac{\Delta S}{R} - \frac{\Delta H}{RT}$$
(16)

RESULTS AND DISCUSSION

FT-IR studies: Both adsorbents *viz*. RBD and ACBD were evaluated, ranging between 4000 and 400 cm⁻¹ and the involved functional groups are depicted in Fig. 1a. The -OH (alcoholic) with an organic functional group has a stretched vibration and a strong intensity at 3415 cm⁻¹ and alkanes (C-H) with a carbonyl functional group, have a strong and broad intensity a stretched vibration at 2950 cm⁻¹. Alkynes (-C=C-) with varied intensity and stretch vibration at 2155 cm⁻¹; alkenes (C=C) with variable

intensity and stretch vibration; amide (C=O) with strong intensity and stretch vibration at 1660 cm^{-1} were also observed.

Aromatics with many medium-weak bands and a stretched vibration at 1420 cm⁻¹ were also appeared [17]. The carbonyl group (C=O) with aldehyde and ester stretches at 1750 cm⁻¹ with significant intensity. Alkanes, alkyl halides, amines, acids (-C-H, C-F, C-N, C-O) and esters (C-O) with organic functional groups bending at 895 cm⁻¹ and stretching at 1090 and 1255 cm⁻¹ with moderate to strong bending and stretching, respectively. The bending alkenes with strong intensity peak at 895 cm⁻¹ while alkyl chloride stretches at 670 cm⁻¹ also observed [18].

After activation, the peak of the functional group altered to a modest increase at 3415 to 3610 cm⁻¹ and a decrease at 2922to 2920 cm⁻¹, 1885 to 1750 cm⁻¹ and 895 to 820 cm⁻¹. Additionally, a new peak appeared at 520 cm⁻¹, which may have been caused by aliphatic stretching and aromatic functional group. As shown in Fig. 1, several peaks disappeared following acid activations.

XRD studies: The X-ray diffractograms of RBD and ACBD are shown in Fig. 2. The spectrum shows no any enormous and eminent peak, but shows a broad diffraction background near 22° [19]. These are due to lignocellulose content present in the material. This indicate the material structure is amorphous in nature. These peaks expanded more broadly after the activation of *Boerhavia diffusa i.e.* in the ACBD sample. Hence, ACBD reflects more amorphous than that of RBD by structure.

Morphological studies: SEM images represents the morphology of RBD and ACBD adsorbents at different optical magnifications (Fig. 3a-b) illustrates the structure's significant porosity and roughness, which helps the metal ions to attach with adsorbent [20]. Moreover, it also demonstrated that neither of the adsorbents have a distinct structure. The highlighted regions correspond to the aggregated and irregularly shaped carbon particles. Images are taken at the scale of 10 μ m decipted that the cavities at the surface were around 1-3 μ m, whereas at the same scale ACBD images show the large number of holes and pores with small in size *i.e.* less than 1 μ m. The EDS results revealed the different ions as Cl, Ca, Na, *etc.* Cr(VI) can easily





Fig. 2. (a) SEM image of RBD and (b) SEM image of ACBD





attached with these ions to form $CrCl_6$ in ionic compound *e.g.* calcium chromate and sodium chromate.

Effect of parameters variations: Parameters variation and effect on adsorption removal efficiency and adsorption capacity given in Fig. 4 as (a) initial concentration (b) contact time (c) pH (d) adsorbent dose and (e) temperature. The analysis of contact time taken from 20 to 140 min with a 20 min interval. As time of contact increases adsorption increases till the saturation occurs. Maximum adsorption reached at the time of 120 min as the total surface area and pore size filled with adsorbate. The effective removal of Cr(VI) ions increases with time from 68.9% to 86.4% for RBD, but if we evaluate these terms for ACBD; originates efficiency increment was 52.4% to 92.7%. During the adsorption process the remaining parameters remains constant that the optimal values of initial concentration was 20 mg/L, adsorbent dose 20 mg, time 120 min and volume of sample during whole batch process remains 20 mL. If we increase the concentration from 20 mg/L to 100 mg/L with interval of 10 mg/L, the adsorption efficiency decreases from 86.4% to 55.3% of raw Boerhavia diffusa (RBD) and activated carbon of Boerhavia diffusa (ACBD) represents the percent removal from 92.7% to 83.8% as the concentration increase from 20 to 100 mg/L. The decrement in efficiency was due to increment in concentration and amount of RBD and ACBD remains constant i.e. 1 g/L (20 mg) for 20 mL sample. However, higher amount of Cr(VI) ions attached more with the adsorbent as multilayer adsorption occurs, so adsorption capacity increases.

The study of effect of doses carried out from 2 mg to 20 mg for prescribed volume of sample. If we increase the mass of an adsorbents *i.e.* RBD or ACBD as presumed adsorption efficiency increased from 19.4% to 86.4% for RBD and 19.3% to 92.7% for ACBD. The reason is attributed due to an incre-

ment in the surface area and the pores as quantity of adsorbents increases but the concentration of the adsorbate *i.e.* Cr(VI) ions remains unchanged.

The heavy metal ions adsorption greatly affected by the pH of sample, as shown in Fig. 4d. At higher pH, the adsorption efficiency increases from 72.5% to 86.4% at 3 to 7 pH for RBD and 72.5% to 92.7% at 3 to 5 pH for ACBD. On further increasing the pH (pH > 7, pH > 5), the H⁺ ions resist the active sites to adsorb the Cr(VI) ions due to the repulsive forces and thus thereby adsorption decreases. This is because of adsorption reach to the alkaline region; the chemical such as H₂SO₄, HCl, NaOH might forms Cr(OH) and Cr(OH)₂ by partial hydrolysis. Due to complex attachment of Cr(VI) ions precipitates with the hydroxide of chromium formed, hence the pH for further experiment was optimized as pH 7 for RBD and pH 5 for ACBD [21,22]. The adsorption efficiency increases with increase in temperature but increment in temperature does not enhance the prospective standards. This was occured due to the breakup of bonds and formation of new bonds. But as the temperature stability of adsorbents decreases, an increment is not more efficient.

Adsorption isotherms: The results of various isotherms of Langmuir, Freundlich, Temkin and Dubinin-Radushkevich are shown in Fig. 5a-d, respectively. The data for adsorption isotherm was derived from the experiment of change in initial concentration which was performed from 20 to 100 mg/L. However, the maximum adsorption reached at 20 mg/L, after which no change was observed in ion removal *i.e.* adsorption saturated at this concentration. In order to find out the best suitable isotherm, the regression coefficient R² was calculated which indicates the values between 0 to 1 [17]. The other values of parameters are given in Table-1.



Fig. 4. Effect of (a) initial concentration, (b) time, (c) pH, (d) dosage and (e) temperature



Fig. 5. (a) Langmuir isotherm model, (b), Freundlich isotherm model, (c) Temkin isotherm model and (d) Dubinin-Radushkevich isotherm model

TABLE-1						
THE CALCULATED VALUES OF						
LAN	NGMUIR, FREUN	DLICH, TEMKIN	AND			
DUBINI	IN-RADUSHKEV	ICH ISOTHERM	MODELS			
Adsorption		Cr(VI)	Cr(VI) removal			
isotherm	Parameters	removal by	by ACBD			
models		RBD (pH = 7)	(pH = 5)			
	\mathbb{R}^2	0.99814	0.99295			
Longmuir	q _{max} (mg/g)	66.2897	135.813			
Langmuir	K _L (L/mg)	0.11775	0.01058			
	R _L	0.2296	0.4522			
	\mathbb{R}^2	0.97361	0.98675			
Freundlich	n	2.39026	1.54342			
	K_{f} (mg/g)	12.544	15.583			
	\mathbb{R}^2	0.99469	0.98332			
Temkin	B (J/mol)	14.3881	28.9441			
	b _T	175.0851	87.035			
	$A_{T}(L/g)$	1.174956	1.10284			
Dubinin- Redushkvich	R ²	0.82244	0.841			
	K_{ad} (mol ² /kJ ²)	1.7797×10^{-6}	8.3949×10^{-7}			
	$q_s(mg/g)$	46.37498	65.8448			

For removal of Cr(VI) by RBD and ACBD the Langmuir isotherm model found best fit as values of R² are 0.99814 and 0.99295, respectively, which is near to linearity than other.

Langmuir isotherm gives the maximum adsorption capacity of an adsorbent which was 66.29 mg/g and 135.81 mg/g for RBD and ACBD, respectively. The value of Langmuir coefficient (K_L) show the affinity to binding sites had the value 0.11775 L/mg and 0.01058 L/mg for RBD and ACBD. For both RBD and ACBD, the separation factor (R_L) values were 0.2296 and 0.4522 that indicates the adsorption is favourable for Cr(VI) ion removal.

The Freundlich isotherm is required for heterogeneous surfaces and provide the information about the multilayer adsorption. The value of n is greater than one *i.e.* 2.39026 and 1.54342 for RBD and ACBD, respectively which show the favourability and physical nature of adsorption. The values (for RBD and ACBD) from Temkin Isotherm determined are $A_T = 1.174956 L/g$, 1.10284 L/g, B = 14.3881 J/mol, 28.9441 J/mol, $b_T = 175.0851$, 87.035 and indicated the sign of heat of adsorption, which represents the physical adsorption. The D-R isotherm model employed to determine whether the adsorption is physical or chemical. The value of mean free energy E *i.e.* greater than 8 that means adsorption is chemical as well as physical [18].

Kinetic studies: The residence time for batch process of adsorption is the most important parameter for study of kinetics

of adsorption. This observation is investigated at different time intervals *i.e.* from 20 min to 140 min with a interval of 20 min. The equilibrium condition reaches after 120 min for the Cr(VI) ion removal by RBD and ACBD. The data computed is given in Table-2 and plots for RBD and ACBD are shown in Fig. 6. The correlation coefficient (0.9942 and 0.99841 for RBD and ACBD) show that pseudo-second order is best fitted, which means the adsorption process is happened due to the chemical reaction. The adsorption process occurs by the sharing of electron by Cr(VI) ions and forms the bond with the surface of RBD and ACBD. The intra particle diffusion model provide that the mechanism of process is of more than one, because it does not passes through origin and complex [18].

Thermodynamic studies: For thermodynamic study, the adsorption was employed at different temperature (293, 303, 313, 323 and 333 K) and the parameters like Gibbs free energy (Δ G), change in entropy (Δ S), change in enthalpy (Δ H) values

are shown in Table-3. The negative value of Gibbs free energy indicates the feasibility of adsorption and spontaneous in nature. Increasing values with temperature showed that the removal efficiency of Cr(VI) is also increased. The positive values of change in enthalpy (Δ H) provided that the adsorption is endothermic. This mean energy are required to remove the ions of heavy metals. The endothermic reaction also employed the adsorption is physical in nature. The values of enthalpy change were 8.641 and 6.867 kJ/mol. The value of change in entropy indicated that a reduction in the randomness as increasing in temperature because of negative in sign [17].

Comparative studies: Comparison efficiency of RBD and ACBD with other adsorbents towards the removal of Cr(IV) is shown in Table-4. The data indicated that *Boerhavia diffusa* in the activated form is found to be better adsorbent as compared to other reported adsorbents towards the removal of Cr(IV) ions.

	TABLE-2	
THE CALCULATED VALUES OF PSEUDO FIRST ORDI	ER, PSEUDO SECOND ORDER AND INT	TRA-PARTICLE DIFFUSION MODELS
Dennile Contractor	Development enter	Takan mential differsion

Matelione	Pseudo first order			Pseudo second order			Intra-particle diffusion		
(adsorbent)	\mathbb{R}^2	$K_1 (min^{-1})$	q _e (mg/g)	\mathbb{R}^2	K ₂ (g/mg min)	q _e (mg/g)	\mathbb{R}^2	K _{id} (g/mg min ^{0.5})	C (mg/g)
Cr(VI) (RBD)	0.96984	0.02065	5.8116	0.99795	0.005746	18.3587	0.98754	0.50515	11.5214
Cr(VI) (ACBD)	0.99330	0.02224	12.9246	0.99603	0.001795	21.9010	0.97305	1.13238	5.84921



Fig. 6. (a) Pseudo first order model, (b) pseudo second order model, (c) intraparticle diffusion model and (d) van't Hoff model equation

TABLE-3 THERMODYNAMIC DATA OF DIFFERENT PARAMETERS								
				Thermodynam	ic parameters			
(adsorbent) R ²	\mathbf{R}^2	R^2 $\Delta H (kJ/mol)$	$\Delta S (J/mol K)$ ·	$-\Delta G (kJ/mol)$				
	К			293 K	303 K	313 K	323 K	333 K
Cr(VI) (RBD)	0.765094	8.641	42.8197	3.629	4.656	4.901	5.130	5.488
Cr(VI) (ACBD)	0.646347	6.867	45.0656	6.619	6.401	7.209	7.692	8.268

TABLE-4 COMPARATIVE DATA OF MAXIMUM ADSORPTION CAPACITY OF DIFFERENT ADSORBENTS TOWARDS Cr(VI) ION

Adsorbents	Max. adsorption capacities (mg/g)	Ref.
Chitosan	35.6	[8]
Single-walled carbon nanotubes	20.3	[8]
Multi-walled carbon nanotubes	2.48	[8]
Powered activated carbon	46.90	[8]
Teff straw	3.51	[9]
Graft copolymerization banana peel	6.17	[10]
Activated carbon prepared from mango	7.80	[11]
kernel		
Papaya peel	7.16	[12]
Avocado kernel seed	10.8	[12]
Juniperus procera sawdust	16.03	[12]
Modified ground nuts	131	[13]
Low cost dolomite	8.38	[23]
Un-oxidized peanut carbon	43.29	[24]
Oxidized peanut carbon	44.05	[24]
Treated newspaper	59.88	[25]
RBD	66.28	Present
ACBD	135.81	study

Conclusion

The adsorption behaviour of Cr(VI) ions on raw *Boerhavia diffusa* (RBD) and activated carbon of *Boerhavia diffusa* (ACBD) was investigated successfully using various isotherm models and kinetic models. Activation was done by conc. H₂SO₄, which improves the adsorption efficiency towards the ions. The Langmuir isotherm model provided the best fit and the maximum monolayer adsorption capacities of both adsorbents RBD and ACBD were determined to be 66.28 mg/g and 135.81 mg/g, respectively. The kinetic adsorption data are better described by Lagergren's model of pseudo-second order. The present adsorbent is found to be superior for the removal of chromium(IV) ions and its performance improves upon activation.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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