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Removal of methyl green dye using activated carbon prepared from corn cobs

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Abstract

Dyes released into hydrological systems in textile manufacturing, printing and other dyeing processes are hazardous and toxic to human and aquatic lives. Activated carbons have been remarkably used to treat dye contaminated waste water due to their large surface area and porosity, however regeneration and high cost have limited their applications. This study investigated the use of activated corn cobs (ACC) on the adsorption of methyl green dye from aqueous solution. The raw cobs were collected, crushed into particle size of about 600 µm and modified *in-situ* with KOH to prepare ACC which was characterized using some analytical techniques such as Fourier Transform Infra-Red (FTIR), Energy Dispersive X-ray (EDX) spectroscopy and Scanning Electron Microscopy (SEM). The absorbance of the dye solution was monitored at 620 nm with UV-Visible spectrophotometer. FTIR analysis showed the vibration frequency for C–H, O–H, C=O and C–O stretches at 2950, 3400, 1710, and 1150 cm⁻¹ respectively. SEM results revealed the ACC has a porous surface with heterogeneous pores which became compact after dye adsorption. EDX confirmed the presence of C, O, H and K in the adsorbent. The suitability of the pseudo-first, pseudo second and Elovich kinetic models for the sorption of methyl green onto ACC was examined. The equilibrium data were subjected to Langmuir, Freundlich, Tempkin and Dubinin-Radushkevich isotherm models. The pseudo-second order kinetic model provided the best correlation and was found to be more statistically significant. Langmuir model was found to fit well based on the high values of the coefficient of regression R² and low % standard error values. The monolayer adsorption capacity O_{max} was found to be 85.83 mgg⁻¹. Thermodynamic adsorption processes showed the spontaneous, endothermic and randomness of the systems with free energy change less than zero, enthalpy change (Δ H) of 62.47 kJmol⁻¹ and entropy change (Δ S) of 125.37 Jmol⁻¹K⁻¹.

Keywords: Thermodynamic; Adsorption; Activated corn cobs; Isotherm; Kinetic

1. Introduction

Increase anthropogenic activities as a result of rapid urbanization and industrialization have led to increase environmental pollution with grave consequence in the quality of water available for industrial, agricultural and domestic use. Dyes are highly stable compounds to light, chemical, biological and other exposures[1]. Dyes are basically natural or synthetic organic compounds that can connect themselves to surfaces or fabrics to produce bright and lasting colour [2]. Synthetic dyes are one of the major water pollutants mostly released from various industrial processes such as dyestuff manufacturing, dyeing, printing, textile finishing, etc. Annually, 12 % of the synthetic dyes are discharged into aquatic body from anthropogenic activities. The textile industries account for two thirds of the world's annual production estimated to be 7 x 10⁵ tonnes [3]. Pollution from this source are the major concern for the developing countries because of various factors such as visibility of dyes even in low concentrations; adverse effect on the photosynthetic activities of the aquatic life among others. It is necessary to effectively treat effluents containing dyes due to the environmental and toxicology threats posed to human and aquatics animals. Colour removal is one of the daunting tasks faced by industries, while the development of cost effective and environmentally safe method in dyes adsorption is challenging to researchers. Many processes such as liquid–liquid extraction[4], ozonation, adsorption [5]

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etc, have been adopted to dyes removal in wastewater. However; some of these techniques are inefficient and expensive to treat wastewater containing dyes. Adsorption technique has been found to be a superior separation and purification method because of its easy-nature, low cost, high selectivity and high removal efficiency.

Most countries of the world are agrarians with abundant cellulosic by-products from the production of crops such as groundnuts, millet, soybean rice etc. The natural fiber comes from stalks, leaves and seeds, such as kenaf, sisal, flax, sorghum, wheat and rice [6 - 9]. Natural fiber have been found to be advantageous over the synthetic ones in terms of biodegradability, flammability and non-toxicity [7].Cellulose, a biopolymer is considered a promising natural source that has been extensively explored by researchers for adsorption. Surface modification of cellulose improves its potential in adsorption process, hence, needs to modify the groundnut pods in this work. Also thermal and mechanical resistance may also increase its pollutant adsorption capacity in aqueous and non-aqueous solutions [8].Therefore, this work aimed at investigating the adsorption of methyl green dye from the aqueous solutions using activated corn cobs (ACC) as adsorbent. The kinetic, isotherm and thermodynamic parameters of the adsorption were considered in a batch process.

2. Material and methods

2.1 Materials

Methyl green dye (Fig. 1) were obtained from Merck laboratory, India. Hydrochloric acid (HCl), Urea (CH₄N₂O) and Sodium hydroxide (NaOH) were procured from BDH, London. Other reagents used were of analytical standard.



Figure 1: Structure of Methyl green dye (Basic dye)

2.2 Characterization

Scanning electron microscopy (SEM; Hitachi S4800) equipped with EDAX was used to determine the surface morphology of the adsorbent before and after adsorption, while the EDAX monitored the elemental component of the adsorbent. Organic functionalities were determined by Fourier transform infrared (FTIR) spectroscopy and recorded from 400 to 4000 cm-1 in TENSOR 27 Spectrophotometer (Bruker, Germany) using KBr pellet technique.

2.2.1 Synthesis of activated corn cobs (ACC)

Corn cobs were collected, washed with tap water and rinsed with distilled water to remove dust and impurities. The cobs were air-dried then oven dried at 50°C to constant mass. The dried cobs were pulverized, sieved to obtain particle sizes less than $600\mu m$ and preserved in an air-tight polythene bag to prevent from moisture and made ready for the analysis.

The sieved cobs (50 g) were suspended in 100 ml of 0.1M KOH, 20 ml of 2M urea solution was added as a stabilizer and stirred continuously for 1hr on a magnetic stirrer. The suspension was centrifuged, washed thoroughly with distilled

water till a neutral pH attained. It was then carbonized at 110°C for 2hrs in a vacuum oven at and kept in an air tight container.

2.2.2 Preparation of adsorbate

Methyl green dye (1.0 g) was dissolved in 1litre of distilled water to give a concentration of 1000 mgl⁻¹. The working standard solutions were prepared form the stock solution by serial dilution.

$$C_1V_1 = C_2V_2$$
.....(1)

2.2.3 Adsorption Studies

The batch equilibrium and kinetics adsorption studies were conducted in process in Erlenmeyer flasks containing 25 mL of dye solutions with concentration range between $10 - 50 \text{ mgL}^{-1}$ and 0.1 g of the adsorbent. The contents were placed in a regulated water bath (30 °C) with shaker at 150 rpm, samples were collected at pre-set time intervals. The dye concentrations in aqueous media were determined after the adsorbent was centrifuged by reading the absorbance at 586 nm. The amount of methyl green dye adsorbed (mg/g) by the adsorbent as a function of time (Q_t) and at equilibrium (Q_e) were estimated according to equations 2 and 3 below:

$$Q_{t} = \frac{(C_{o} - C_{t})}{m} \times V \dots (2)$$
$$Q_{e} = \frac{(C_{o} - C_{e})}{m} \times V \dots (3)$$

where C_o , C_t and C_e are the initial, time t and equilibrium concentrations (mg/L) of the dye respectively, V is volume (L) of the solution and m is the mass (g) of the adsorbent.

2.3 Adsorption mechanism and isotherms studies

The mechanisms of adsorption of methyl green dye onto the adsorbent were investigated by subjecting the data from time dependent adsorption to pseudo-first order, pseudo-second order kinetic and intra-particle diffusion models to describe the kinetics of the adsorption process. The mathematical expressions of these models (equations 4 - 7) are as presented in Table 1. All data were analyzed with nonlinear regression analysis method using a program written on Originpro, 2022 software.

Table 1 Kinetic models for the adsorption studies of methyl green dye

Name	Model
Pseudo-first order	$Q_t = Q_e \left(1 - e^{-k_1 t}\right) \dots 4$
	Q_e and Q_t are the amounts (mg g ⁻¹) of dye adsorbed per unit mass of adsorbent at equilibrium time and time t, respectively, while k_1 (min ⁻¹) is the rate constant for the pseudo-first order kinetics
Pseudo-second order	$Q_{t} = \frac{k_{2}Q_{e}^{2}t}{1 + k_{2}Q_{e}t} \qquad5$
	Q_e and Q_t are the amounts (mg) of dye adsorbed per unit mass of adsorbent at equilibrium time and time t, respectively, while $k_2(g mg^{-1} min^{-1})$ is the rate constant for the pseudo-second order kinetics
Elovich	$Q_t = \frac{1}{\beta} \ln(\alpha \beta * t) \dots 6$
	where α (mg g ⁻¹) is the initial adsorption rate constant and the parameter β (g mg ⁻¹ min ⁻¹) is related to the extent of surface coverage and activation energy for chemisorptions

Intraparticle diffusion	$Q_t = K_{id} t^{0.5} + Ci$ 7
	where <i>K_{id}</i> is the intraparticle diffusion rate constant (mg mg ⁻¹ min ^{-0.5}), and C is a constant (mg mg ⁻¹) which gives information about the thickness of boundary layer

Adsorption equilibrium data were also subjected to the Langmuir (1916), Freundlich (1906), Tempkin and Pyzhev (1940), and Dubinin and Radushkevich, (1947) isotherms models (represented by equations 8 - 11). The isotherm parameters were obtained by the least square fit method as earlier mentioned. The mathematical expressions of these isotherm models (equations 10 - 13) are as presented in Table 2.

Table 2 Isotherm models applied for the adsorption of studies of CV and MB

Name	Model
Langmuir	$Q_{eq} = \frac{Q_{\max}bC_e}{1+bC_e} \dots 8$
	Q_{eq} and Q_{max} are the amounts (mg/g) of dye adsorbed per unit mass of adsorbent and maximum adsorption capacity at equilibrium, respectively, C_e is the equilibrium concentration of adsorbate, while <i>b</i> (L mg ⁻¹) Langmuir constant.
Freundlich	$Q_{ra} = K_F C_r^{\frac{1}{n}}$
	- eq I e
	K_F (mg g ⁻¹) (L mg ⁻¹) ^{1/n} is a rough estimation of adsorption capacity of the adsorbent, 1/n is the adsorption intensity.
Tempkin	$Q_e = \frac{RT}{b_T} \ln a_T C_e \dots \dots$
	R (J molK ⁻¹) is the gas constant, T (K) is absolute temperature, a_T (mg L ⁻¹) is the binding constant and b_T (L g ⁻¹) is related to the heat of adsorption
Dubinin– Radushkevich	$Q_e = Q_s e^{-\beta \varepsilon^2} \dots 11$
	Q_s (mg g ⁻¹) is the saturation capacity, β (mol J) ² is a constant relation to adsorption energy while
	ε is related to the mean free energy of adsorption and given $\varepsilon = RT * \ln\left(1 + \frac{1}{C_e}\right)$

2.4 Thermodynamic parameters

The thermodynamic parameters, ΔG° , ΔH° and ΔS° explain the feasibility, spontaneity and the nature of adsorbateadsorbent interactions during the adsorption process (Adeogun et al., 2016). Their values were obtained from the temperature dependent equilibrium study by viewing the process at equilibrium using the notation below:

The equilibrium constant in term of the adsorbate (C_e), adsorbent dosage (m) and adsorbed quantity (Q_e) could be written as:

The van't Hoff plot, $(lnK_D \text{ versus } 1/T)$ for the adsorption process gives the slope and intercept from which thermodynamic parameters were obtained.

3. Results and discussion

3.1 FTIR characterization of the ACC

Fourier transform infra red (FTIR) technique was used to detect the functional groups present in ACC and to identify the ones involved in the adsorption of methyl green dye. The IR spectrum of ACC after the dye adsorption shown in Figure 2.The prominent band at 3150 cm⁻¹ was assigned to sp³ C – H of the cellulose in the adsorbent. The stretching vibration frequencies at 3450 and 3470 cm⁻¹ were assigned to 0 – H group in the adsorbent. The bands at 1730 and 1720 cm⁻¹ indicated v (C = O) of the adsorbent. Meanwhile, the symmetric stretch at 1600 and 1620 cm⁻¹ were assigned to C = C groups of the aromatic compounds, suggesting the functional groups interaction between the adsorbent and dye molecules [38]. The band at 1120 cm⁻¹ was assigned to the C–O group. The changes in FTIR spectrum confirmed the effect of modification in activated corn cobs with mostly bonded v(O-H) which explained the inter and intra-molecular hydrogen bonding of the polymeric compounds (macromolecular association). This is in agreement with O-H vibration frequency observed in cellulose and lignin, thus showing the presence of free hydroxyl groups on the modified adsorbent [24]. The prominent shift was due to the effect of activation in the adsorbent (ACC) indicating its usefulness in the adsorption processes [34].





3.2 SEM and EDAX analysis

The SEM images of raw corn cobs and activated corn cobs (ACC) are shown in Fig 3. It was deduced that the ACC became compacted after adsorbed with methyl green dye. Several large pores in a rod shape were shown on the surface as compared with raw cobs. The open pores on ACC indicated an effective activation process of the adsorbent which enhances adsorption of dye [25]. The EDX analysis (Fig.4) shown a high percentage of carbon in the ACC adsorbent which make it suitable and effective adsorbent.



С

Figure 3 SEM analysis of activated corn cobs before and after adsorption of methyl green dye (a) modified cobs (b) adsorbed with dye (c) adsorbed with dye at 50 °C (d) raw corn cobs



Figure 4 EDAX of ACC: (a) Modified corn cobs (b) adsorbed with MG dye

3.3 Adsorbent Dosage Study

The adsorbent dosage increased from 0.05 - 1.0g proportionally increases its removal efficiency ranging 92.5 - 98.6% as presented in Figure 5, which could be attributed to the increase in adsorption sites of the ACC adsorbent [26]. The reduction of the removal efficiency noted on further increase in the adsorbent dosage resulted from particle interactions that collapsed the active sites of the adsorbent [27].



Figure 5 Adsorbent dosage against % removal of MG dye

3.4 Solution pH study of the adsorption of methyl green dye

The removal efficiency of the dye increases with the pH of the media increase as presented in Figure 6. pH affects the chemical properties of the dye solution, the adsorbent surface charge, as well as interactions in the media [28]. The maximum 95.45% removal efficiency was obtained for the dye solution at pH 7.8. The acidic pH lowers the adsorption of the adsorbate because of its competition with the hydrogen ion on the available adsorption sites [29].



Figure 6 The Plot of solution pH against methyl green % adsorbed

3.5 Effect of initial concentration and contact time

Removal of methyl green dye from aqueous solution depends on the initial dye concentration and contact time. The quantity of dye removed increased proportionally as initial concentration increases. The dye was rapidly removed in the first 10 minutes, maintained for the next 30 minutes then continued steadily until equilibrium. Adsorption capacities

at equilibrium increases from 5.45 to 16.89 mg/g as the initial concentrations increases from 10 to 50 mg/L as shown in figure 7 below.



Figure 7 Effect of initial concentration with time

3.6 Adsorption Kinetics

The meaning of adsorption kinetics is a function of elaborate analysis of time dependent adsorption data. The process was characterized by mechanisms such as; chemical interactions between the adsorbent (ACC) and adsorbate (methyl green dye), mass transfer of the adsorbate into and within the adsorbent or combination of these mechanisms, hence combination of models were required to elucidate the mechanism of adsorption [32, 39]. The pseudo first-order, pseudo second-order as well as Elovich and intra-particle kinetic models were used to examine the kinetics of adsorption data which are depicted in Figure 8 while the parameters for these fits are presented in Table 3.

By considering the values of the regression coefficients R^2 for the models, error function analyses and the closeness of the Q_e determined experimentally with the theoretical values showed that pseudo- second order kinetic model was much favoured, suggesting physical interactions between the adsorbent and adsorbate [30]. The Elovich kinetic model fit (Figure 9) agreed with the experimental data (Table 3) and showed that $R^2 > 0.90$, the values β_{el} indicate the available site for adsorption decreases with increase dye concentrations [34-36]. The positive values of these constants confirmed the model, hence Elovich model properly explained the initial kinetics of adsorption of the dye onto the adsorbent as previously reported in literature [31].

rs

	C ₀ (mg/L)	10	20	30	40	50
First order	Q _e (exp) (mg/g)	8.365	16.206	22.372	24.94	24.967
	Q_e (cal) (mg/g)		16.148	22.104	24.151	24.226
	k1 (mins ⁻¹)	0.025	0.019	0.025	0.032	0.047
	R ²	0.998	0.998	0.999	0.998	0.999
	Adj. R ²	0.989	0.976	0.993	0.967	0.989
	% SSE	0.002	0.001	0.004	0.011	0.001
Second order	Q_e (cal) (mg/g)	10.224	20.463	26.783	28.512	28.411
	k ₂	0.002	0.001	0.001	0.001	0.002
	R ²	0.991	0.997	0.998	0.998	0.999
	Adj. R ²	0.989	0.992	0.991	0.969	0.991

	%SSE	0.078	0.093	0.07	0.051	0.049
Elovich	α (mg/gmin ⁻¹)	0.541	0.899	1.523	2.111	3.403
	β _{el} (g/mg)	0.445	0.233	0.177	0.166	0.177
	R ²	0.993	0.997	0.996	0.996	0.996
	Adj. R ²	0.989	0.918	0.992	0.987	0.986
	% SSE	0.065	0.0875	0.092	0.061	0.054
Intra particle diffusion	$K_{id 1}(mg/g/g/min^{1/2})$	1.182	1.893	3.108	4.425	5.919
	$K_{id 2}$ (mg/g/g/min ^{1/2})	0.364	0.873	1.014	0.959	0.845
	C1 (mg/g)	0.3135	-0.4197	-0.486	-0.7075	0.6835
	C ₂ (mg/g)	3.256	3.68	7.847	11.163	14.02
	R ²	0.953	0.966	0.978	0.985	0.981
	Adj. R ²	0.887	0.899	0.911	0.897	0.958
	% SSE	0.037	0.087	0.085	0.091	0.056









Figure 8 Kinetic model plots (a) Pseudo-first-order (b) Second order (c) Elovich model (d) Intra-particle diffusion

3.7 Adsorption isotherm

Adsorption isotherms described the phenomenon governing the release or mobility of a substance from the aqueous media to a solid-phase at a constant temperature and pH, the interpretations of these models are critical to the overall improvement of adsorption mechanism pathways and effective design of adsorption system [41]. The adsorption isotherm models predicted to confirm the adsorption of methyl green dye onto ACC are shown in Figure 10 and parameters (Table 4). The isotherm parameters revealed that the entire isotherms model investigated fit very well with the data at temperature 40 °C and correlation coefficient R² values in the order of Langmuir > Freundlich > Tempkin > Dubinin-Radushkevich, with maximum adsorption capacities (Q_{max}) of 85.83 mgg⁻¹. At temperature 30° C, Langmuir > Freudlich = Tempkin > Dubinin-Radushkevich and 56.75 mgg⁻¹ adsorption capacities. The R_L values is less than one, indicated a favourable adsorption. Freundlich parameters confirmed the heterogeneity nature of the surface of adsorbent, ¹/n value of < 1 indicates a normal Langmuir isotherm, otherwise cooperative adsorption [33]. The Tempkin isotherm parameters and the R² values showed favourable fits for the dye, to imply that adsorption process is characterized by uniform distribution of binding energies. The mean free energies obtained for the adsorption of the dye is 1.45 kJ/mol, confirming the physisorption adsorption as suggested by the kinetic fit [34].

	Table 4 Adsor	ption isotherms	parameters at 30	and 40 °C
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Isotherms	Parameters	Values	
		30°C	40°C
Langmuir	$Q_{max} (mgg^{-1})$	56.75	85.83
	b (L mg ⁻¹)	0.392	0.265
	RL	0.176	0.094
	R ²	0.997	0.999
Freundlich	K_F (g mg ⁻¹ min ^{-1/n})	7.620	5.460
	1/n	0.472	0.351
	R ²	0.986	0.993
Tempkin	bт	227.358	536.684
	a _T (L mg ⁻¹)	3.604	4.744

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	R ²	0.990	0.996
Dubinin-Radushkevich	Q_s (mgg ⁻¹)	22.130	16.329
	$\beta x 10^7 (mol J^{-1})^2$	5.79	1.26
	E(kJ mol ⁻¹)	1.914	1.456
	R ²	0.984	0.987



Figure 9 Isotherm plots of Qe (mgg-1) against Ce (mgL-1) at (a) 30 °C (b) 40 °C (c) 50 °C (d) 45 °C

3.8 Adsorption Thermodynamic Study

The van't Hoff plot (Fig.12) for the adsorption of methyl green dye using ACC and the thermodynamic parameters (Tab.12) showing enthalpy change (Δ H) and entropy change (Δ S) had positive values of 62.48 KJmol⁻¹ and 135.27Jmol⁻¹K respectively, hence, endothermic adsorption process and this indicate that some amount of heat was consumed to transfer dye ions from aqueous solution to the active site of ACC adsorbent. The positive value of Δ S illustrate an increase in the degree of randomness of the system with changes in the hydration of the adsorbed dye ions [37]. The negative values of Gibb's energy indicated the spontaneity of the adsorption process and the decrease of the values with increasing temperature indicated more efficient adsorption at higher temperatures.

3.9 Thermodynamic Analysis

The thermodynamic parameters, ΔG° , ΔH° and ΔS° explain the feasibility, spontaneity and the nature of adsorbateadsorbent interactions during the adsorption process[38]. The equilibrium constant in term of the adsorbate (C_e), adsorbent dosage (m) and adsorbed quantity (Q_e) could be written as:

$$K = \frac{Q_e}{C_e * m} \tag{25}$$

Where k is the equilibrium adsorption constant[39]

T is the temperature in Kelvin, other parameters had already been discussed.

$$\Delta G = -RT \ln K \dots (27)$$

R is the gas constant and equal to 8.314 kJmol⁻¹

$$\ln k = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \dots (28)$$

The van't Hoff plot, (In K_D versus 1/T) for the adsorption process gives the slope and intercept from which thermodynamic parameters were obtained.

$$C = \frac{\Delta S}{R} \dots (29)$$
$$m = \frac{\Delta H}{R} \dots (30)$$

Table 5 Thermodynamic Parameters	s for the adsorption of dye
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Temp (K)	<i>InK</i> _d	ΔG	ΔΗ	ΔS	R ²
		(kJ mol ⁻¹)	(kJ mol ⁻¹)	(J mol ⁻¹ K ⁻¹)	
308.15	1.282	-3.431	62.48	135.27	0.985
313.15	1.705	-4.317			
318.15	1.785	-5.204			
323.15	2.404	-6.091			

In k = equilibrium constant, $\Delta G = Change in Gibb's$ free energy, $\Delta H = Enthalpy change$;; $\Delta S = Entropy change$, $R^2 = Correlation coefficient$.

The van't Hoff plot (Fig.10) for the adsorption of MG dye were obtained using equations as previously described [2] and the thermodynamic parameters (Table 5) depicts enthalpy (Δ H) and entropy change (Δ S) of positive values of 62.48

KJmol⁻¹ and 135.27Jmol⁻¹K respectively. This illustrated endothermic adsorption process and revealed that heat absorbed when dye ions transported to the active sites of the adsorbent. The positive value of Δ S indicates an increase in the degree of randomness of the system with changes in the hydration of the adsorbed dye ions [40]. The negative values of Gibb's energy indicated the spontaneity of the adsorption process and the decrease of the values with increasing temperature indicated more efficient adsorption at higher temperatures.



Figure 10 Van't Hoff plot of lnK against 1/T

4. Conclusion

Activated carbon derived from corn cobs biomass was prepared and applied to adsorb methyl green dye from aqueous solution. The treatment adopted batch adsorption method to consider some parameters; contact time, initial dye concentration, solution pH, adsorbent dosage and temperature. The kinetics of adsorption of the dye was best explained with pseudo second-order kinetic while Langmuir isotherm model fitted the equilibrium data with monolayer adsorption capacity of 85.83 mg/g. The models showed that the predicted models are suitable for the prediction of the adsorption process with $R^2 = 0.998$. The thermodynamic parameters proved that the adsorption process was feasible, spontaneous, endothermic and random in nature.

Compliance with ethical standards

Disclosure of conflict of interest

No conflict of interest to be disclosed.

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