

Research Article

Synthesis of Activated Carbon from Cassava Peeling and Its Applications for Removal of Methylene Blue

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Abstract

The present work aims to increase the value of cassava peelings by its transformation in activated carbon. The activated carbon was prepared by chemical activation using 40% of phosphoric acid solution as activation agent with an impregnation rate equal to 1.5 g of acid/g of precursor. The carbonization was carried out at 400 °C for 90 minutes. Bohem titration and the Lopez-Ramon method were used to determine the chemical characteristics of the prepared activated carbon (ACCP), while surface area of the sample was estimated by methylene blue and iodine index methods. The best kinetic model that describes the adsorption reaction of MB on ACCP activated carbon is the pseudo-second-order model. Adsorption isotherm was also evaluated. Activated carbon (ACCP) has the following characteristics: $pH_{PZC} = 3.9$; Iodine index value equal to 914.4 mg/g and methylene blue index value equal to 643.72 mg/l. ACCP has more acid functions (5.95 meq/g) than basic function (1.3 meq/g). The “batch method” was used for pollutant removal and it reveals that the contact time value is equal to 40 minutes. The influence of activated carbon mass on its methylene blue adsorption capacity was studied. The experimental data are in agreement with the Langmuir and Freundlich isotherm models. The maximum adsorption capacity obtained using Langmuir isotherm model was 75.93 mg/g.

Keywords

Cassava Peeling, Chemical Activation, Activated Carbon, Iodine Index, Methylene Blue Index, Adsorption Isotherms

1. Introduction

Today, with the aim of recovering agri-food waste, several processes for the manufacture of activated carbon based on

carbon-rich plant materials have been developed by many researchers [1-3]. The most widely used methods generally

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Received: 22 January 2024; Accepted: 18 February 2024; Published: 28 February 2024



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have two stages: pyrolysis and activation, which can be chemical or physical, the purpose of which is to enrich the material in carbon matter, create the first pores and develop the porous structure of the material. In Togo, cassava production increases every year. It is estimated at 1,140,200 t by Central Bank of West African States analysts in 2020. An increase of more than 20,000 t compared to 2019, when it was estimated at 1,117,800 t. A year before, in 2018, Togolese farmers produced a volume of 1,089,500 t. The country thus registers approximately 400,000 t of surpluses each year [4]. Cattle are the only way to make use of cassava peelings. However, not all of the peelings are consumed and the rest is dumped into nature. It is in this perspective that we have set the objective of converting cassava peelings into activated carbon. Indeed cassava the flagship food of Togolese cuisine, is used in some recipes such as cassava couscous, fofou but also in the composition of certain craft beers [2].

Once the tubers have been peeled, part of the peelings are discharged into nature, thus constituting waste to be recovered.

Activated carbon is a highly porous, hydrophobic and apolar carbonaceous adsorbent [5] prepared in such a way to have a high degree of porosity which gives it a considerable specific surface. Any organic raw material is a priori likely to be converted into activated carbon. The choice of raw material essentially depends on local supply possibilities. Activated carbons being versatile adsorbents, due to their good adsorption properties [6], are currently used in a large number of water treatment processes to remove organic and inorganic pollutants [7].

2. Experiments

2.1. Preparation of Activated Carbon

Activated carbon (ACCP) was prepared from cassava peelings. The precursor is thoroughly washed with distilled water and then dried at room temperature for 72 hours. The activated carbon was prepared as follows: the precursor was riddled to have particles which diameter was less or equal to 0.8 mm, then, the particles obtained were introduced in phosphoric acid solution of ca. 40% for 16 hours. The ratio of impregnating (Mass of Acid/Mass of precursor) was 1.5. The activated carbon was obtained by carbonization of the impregnated material at 400 °C for 90 minutes in an oven (WC HERAEUS HANAU type RSK 10). The activated carbon obtained is cooled in a desiccator. In order to remove any carbonization residues, the activated carbon is washed in 0.1M hydrochloric acid (HCl) solution, then rinsed several times with distilled water until a constant pH of 6.5 [8]. The washed activated carbon is dried at 105 °C for 4 hours in the oven, then cooled in a desiccator and stored hermetically sealed bottle until the characterization's tests.

2.2. Characterization of ACCP

The mass yield, the iodine number, the methylene blue index, the pH at the point of zero charge and the chemical functions of surfaces tests have been carried out to determine some keys characteristics of activated carbon prepared from cassava peelings.

The yield of the synthesized activated carbon was estimated from (1).

$$R(\%) = \frac{\text{mass of coal}}{\text{mass of peelings}} \times 100 \quad (1)$$

The degree of activation called "Burn-off" was also determined by (2)

$$\text{Burn-off}(\%) = \frac{\text{mass of peelings} - \text{mass of coal}}{\text{mass of peelings}} \times 100 \quad (2)$$

The iodine number test was carried out in order to determine the nature of the pores of our activated carbon (ACCP) especially the micropores. 0.2 g of activated charcoal is placed in a 100 ml beaker, to this mass is added 20 ml of the iodine solution (I_2) at 0.02 N. The mixture is stirred for 4 to 5 min then filtered on ashless filter paper. Then, a volume of 10 ml of the filtrate is removed and titrated with a 0.1 N sodium thiosulfate ($Na_2S_2O_3$) solution. The starch paste is used as a final-colored indicator. The iodine value is given by (3) [7]:

$$Q_{I_2} = \frac{[C_0 - (\frac{C_{th} \times V_{th}}{2V_{I_2}})] \times MI_2 \times V_{ads}}{m} \quad (3)$$

Q_{I_2} is the I_2 index adsorption capacity (in $mg\ g^{-1}$),
 C_0 is the initial concentration of the I_2 solution in $mol\ L^{-1}$,
 C_{th} is the concentration of $Na_2S_2O_3$ in $mol\ L^{-1}$,
 V_{th} represents the volume of $Na_2S_2O_3$ at the equivalence in mL,

V_{I_2} represents the volume of iodine dosed in mL,

MI_2 is the molar mass of I_2 in $g\ mol^{-1}$,

V_{ads} is the adsorption volume in mL,

m : the mass of activated carbon used in g.

For the determination of the methylene blue (MB) index which would indicate the presence of mesopores and macropores, the distilled water is used to prepare a stock solution of methylene blue with a concentration of 1.2 g/L. This stock solution was used to prepare, by dilution, a diluted solution with a concentration of 10 mg/L and a series of standard solutions of methylene blue (0.5 mg/l – 2.5 mg/l). The optical densities corresponding to these solutions are monitored with the JENWAY 6705 UV/Vis spectrophotometer at 620 nm. Then, we have put in a 250 ml erlenmeyer flask, 0.1 g of activated carbon and 100 ml of BM solution.

The mixture is stirred for 20 minutes and then filtered. The residual concentration of methylene blue is further determined from the optical density [7]. Thus, the methylene blue index is given by (4):

$$Q_{BM} = \frac{VM(C_i - C_r)}{m} \times 100 \quad (4)$$

Q_{MB} : CA adsorption capacity (in mg/g);

C_i : Initial concentration of the MB solution (in mol/L);

C_r : Residual concentration of the MB solution (in mol/L);

V : Volume of MB solution (in ml);

M : Molar mass of MB;

m : Mass of activated carbon used (in g).

The pH at the point of zero charge (pH_{PZC}) is defined as the pH of the aqueous solution in which the solid exists under a neutral electric potential. The Lopez-Ramon et al. [8], is used to determine the pH_{PZC} of our activated carbon (CA-EDM). Solutions at 0.1 mol/L of NaCl (Fulka) with a pH between 2 and 10 are prepared by adding an aqueous solution of NaOH or HCl. To measure the pH values, we used the JENWAY 3345 pH meter. Then, 0.1 g of activated carbon is brought into contact with 20 mL of each of the solutions contained in stoppered bottles. The suspensions are stirred for 3 days at room temperature. Each solution is then filtered using Whatman filter paper and a new pH measurement is taken. The curve C representing the final pH as a function of initial pH is plotted. The pH_{PZC} then corresponds to the pH of the solution for which the curve C crosses the first bisector.

The surface functions are determined according to the method of Boehm (1966) [8]. It is a classic way for qualifying and quantifying the acid and basic surface groups available at the surface of activated carbons regardless of their formatting.

At the equivalence we have:

$$C_e \times V_e = C \times V \quad \text{from where} \quad C = \frac{C_e \times V_e}{V} \quad (5)$$

$$n = (C_i - C) \times V_i \quad (6)$$

n : quantity of groups per gram of activated carbon,

V_e : volume of HCl or NaOH poured at equivalence,

C_e : concentration of the HCl or NaOH solution,

V : volume of the solution to be titrated,

C : concentration of the solution to be titrated,

V_i : volume of the initial solution,

C_i : concentration of the initial solution.

2.3. Application of ACCP in Model Compound Removal

Methylene blue, a cationic dye, is used as a model pollutant

to test ACCP efficiency in adsorbing medium-sized organic pollutants [9].

The methylene blue adsorption experiments are carried out using the "batch method" in 200 ml erlenmeyer flasks, at ambient temperature (28 ± 2 °C.) and at a stirring speed of 250 rpm. The effects of contact time, ACCP mass and initial MB concentration were studied. First of all, we carried out initial experiments to assess a needed time to reach equilibrium, Then, the solutions are centrifuged and analyzed to determine the residual concentration of pollutant. The maximum adsorption capacity Q_e (mg/g) of methylene blue was calculated by (7).

$$Q_e = \frac{(C_0 - C_e) \times V}{m} \quad (7)$$

Where C_0 and C_e in mg/L represent respectively the concentrations of MB at the initial time (t_0) and at equilibrium (t_e) in the solution, V the volume of the solution and m the mass of ACCP.

1) Influence of contact time

The experimental procedure followed consists in bringing into contact a mass of 0.1 g of activated carbon in a flask containing 200 mL of the methylene blue solution at a concentration of 100 mg/l. The mixture is placed under magnetic stirring at 250 rpm. At regular time intervals, samples were taken and the colored solution was separated from the adsorbent by centrifugation. The absorbance of the supernatant solution was then measured.

2) Influence of the mass of the activated carbon

The method consists in using variable masses of activated carbon (100 – 500 mg), with equal volumes (100 ml) of MB solution at the same concentration (100 mg/L) and at natural pH of the solution. The mixture is stirred under the same conditions for a time greater than the equilibrium time determined with above experiments. After stirring, the suspension is centrifuged for 2 min at 2500 rpm; then the related absorbance of centrifugate is measured.

3) Influence of the initial concentration of methylene blue

The same mass of activated carbon (100 mg) was introduced into several erlenmeyer flasks containing the same volume (100 mL) of methylene blue solution of different concentrations (100, 200, 300 mg/L) at natural pH of the solution. The obtained mixed solution is treated as above.

3. Results and Discussion

3.1. Mass Yield

The mass yield of the ACCP activated carbon is 56.3%. This result is close to that of Zyoud *et al.* who obtained a mass yield of 54% for an activated carbon made from olive kernel at 450 °C [9]. From this value of the mass yield (56.3%), a

burn off of 43.7% is deduced; a value of less than 50%, which suggests that CA-EDM has micropores so could adsorb small molecules such as those responsible for tastes and odors as pointed out by some authors [6, 10, 11].

3.2. Iodine Value

The iodine index is a parameter which gives an indication of the microporosity of activated carbons. The iodine number Q_{I_2} obtained for the activated carbon produced in this work is 914.14 mg/L. The value found was between 600 and 1100 mg/g suggesting that the carbon used in this study has the capacity to adsorb small molecules [12].

3.3. Methylene Blue Index

The methylene blue index value for ACCP activated carbon is 643.72 mg/g. This value of the methylene blue index suggests that in addition to the micropores confirmed by the value of the iodine index, ACCP carbon could also have meso and macropores. Ousmaila Sanda Mamane et al, on the other hand, obtained methylene blue indices of 523.61 mg/g and 506.97 mg/g respectively on activated carbons prepared by chemical means (H_3PO_4 à 25%) from the shells of *Balanites Egypitiaca* and *Zizyphus Mauritiana* cores [7].

3.4. pH at Point of Zero Load

The zero-charge point pH of activated carbon (ACCP) is 3.9 as shown in Figure 1. The ACCP then has an acid character reflecting the relatively large number of acid sites introduced during the chemical activation. Indeed: If the pH of the solution is lower than the pH_{pzc} , the surface functional groups of the adsorbent will be protonated by an excess of H^+ protons of the solution and the support becomes an attractor of

negatively charged adsorbate. On the other hand, if the pH of the solution is higher than the pH_{pzc} , the surface functional groups of the adsorbent will be deprotonated by the presence of the OH^- ions of the solution and the support becomes a positively charged adsorbate attractor. This promotes the adsorption of cationic dyes and the increase of electrostatic forces between the negative charge of the adsorbent and the positive charge of the dye [13].

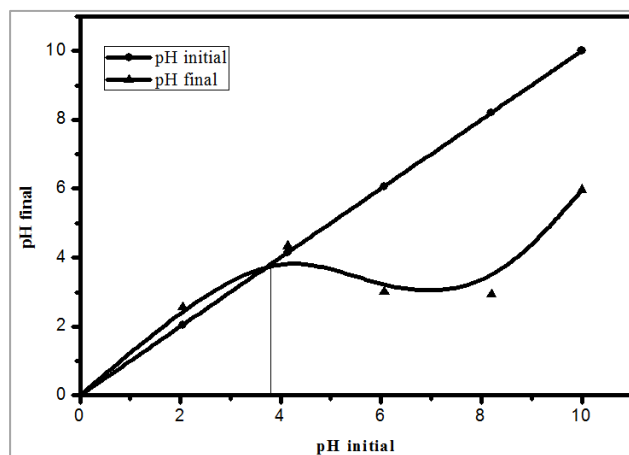


Figure 1. ACCP Activated carbon PZC pH determination.

3.5. Surface Chemical Functions

To determine the quantities of basic and acid groups on the surface of the ACCP carbon, the measurements were carried out according to the Boehm method. The results of the titrations determining the number of surface chemical functions are presented in Table 1.

Table 1. Surface functions of ACCP activated carbon.

	Acids (meq/g CA)					Basics (meq/g CA)
	Carboxylic	Lactone	Phenol	Carbonyl	Total acid	Basic total
Activated carbon ACCP	0.75	0.34	3.26	1.6	5.95	1.3

The number of sites per g is determined by multiplying the number of equivalents per g of activated carbon by Avogadro's number N . The results are recorded in Table 2.

Table 2. Number of sites $\times 10^{20}$ /g of ACCP activated carbon.

	Acids		Basics			
	Carboxylic	Lactone	Phenol	Carbonyl	Total acid	Basic total
Activated carbon (ACCP)	4.52	2.05	19.63	9.64	35.83	7.83

The analysis of these results shows the predominance of the acid functions compared to the basic functions. These results are in agreement with the pH_{PZC} and this can be explained by the strength of the H_3PO_4 activator at 40%; H_3PO_4 is a strong oxidant which can oxidize surface carbon atoms, causing them to lose electrons and become positively charged [14]. This acid character is mainly due to the phenolic (54.79%) and carbonyl (26.90%) functions.

3.6. Influence of Contact Time

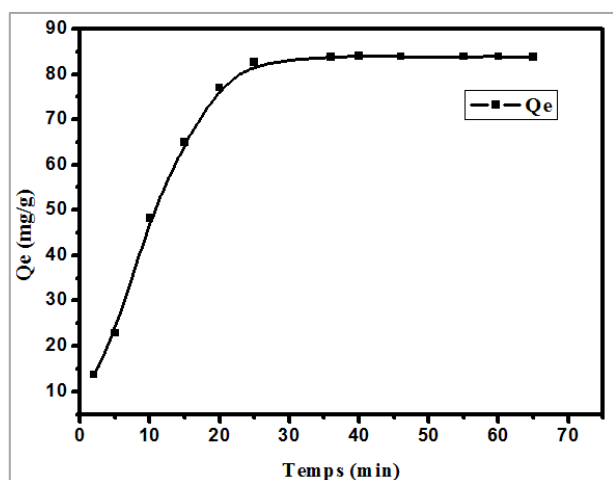


Figure 2. Kinetics of adsorption of methylene blue on activated carbon ($C = 100 \text{ mg/L}$, $V = 100 \text{ mL}$, natural pH , $m_{\text{CA}} = 0.1 \text{ g}$ at 250 rpm).

The adsorbed quantity increases with the reaction time following three slopes (Figure 2) initially the quantity adsorbed increases rapidly in the first 20 minutes due to extra-particle diffusion (external diffusion) of the particles of the adsorbate, characterized by the transfer of the solute through the surface of ACCP, then a second which is slow, represented by the tangential part of the curve, this stage characterizes an intra-particle diffusion of the material during which there is transfer of particles of the adsorbate through the internal surface and the external surface of ACCP and finally a last which remains constant after 40 minutes, indicating a state of

equilibrium. This shows that the dye adsorption equilibrium by the ACCP activated carbon is reached around 40 minutes, with an abatement rate of 82.62%. Compared with 5 min according to the work carried out by A. TALIDI et al [15] for the adsorption of MB on pyrophyllite; and 60 min on bio-sorbent prepared from Algerian date pits according to the work of KHELIFI et al [16]. Ibrahim et al found 70 min and 100 min for phenol adsorption respectively on activated carbons made from shea cake and cotton cake [17].

3.7. Influence of the Mass of Activated Carbon

The reduction of methylene blue is strong when the mass of activated carbon is large (Figure 3). This can be elucidated by the rise in the mass of the adsorbent, which augments the specific surface area, consequently enhancing the number of available adsorption sites, thereby resulting in an increase in the quantity of adsorbed dye. This has already been reported by many authors [16-18].

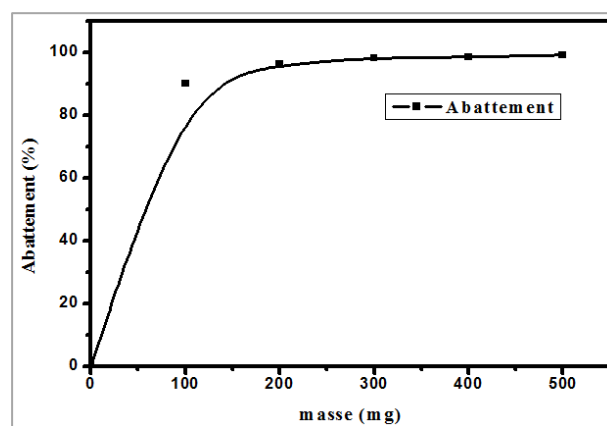


Figure 3. Influence of the mass of activated carbon on the elimination of MB ($C = 100 \text{ mg/L}$, $V = 100 \text{ mL}$, natural pH).

3.8. Influence of the Initial Concentration of Methylene Blue

The percentage of elimination decreases with the increase in the initial concentration of MB, with orders of magnitude of

82.62%, 59.96% and 41.04% respectively for the concentrations 100, 200 and 300 mg/L. This could be due to the presence of a large number of molecules which will diffuse towards the sites on the surface of the adsorbent. These results imply that the initial concentration of methylene blue has a significant effect on the retention capacity of the adsorbent, therefore the adsorption depends on the initial concentration of the adsorbate.

3.9. Adsorption Kinetics of Methylene Blue

Pseudo-first-order and pseudo-second-order kinetic laws were used in this study. These equations are respectively:

$$\ln(q_e - q) = \ln q_e - k_1 t \quad (8)$$

$$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} \cdot t \quad (9)$$

q_e and q (mg/g) representing the quantities of MB adsorbed respectively at equilibrium and at a time t ,

k_1 (min^{-1}), k_2 (mg/g.min) kinetic constants of the reaction of adsorption.

Pseudo-first-order kinetics

The plot of $\ln(q_e - q)$ against time t for activated carbon (ACCP) gives a linear form (figure not shown). The value of k_1 was calculated from the slope of this line, and q_e , cal from the ordinate at the origin.

The kinetic constant of the pseudo-first order adsorption reaction K_1 , the quantity of MB adsorbed at equilibrium q_e , and the correlation coefficient R_1^2 are given in Table 3. The results show that the experimental q_e is not in agreement with the q_e calculated from the linear form of the pseudo-first order kinetics, and the correlation coefficient obtained is very low. Therefore, the pseudo-first-order kinetic model is not well suited to describe the adsorption of methylene blue on ACCP activated carbon.

Table 3. Pseudo-first order rate constants for adsorption of MB on ACCP.

C_0 (mg/l)	q_e , exp (mg/g)	q_e , Cal (mg/g)	K_1	R_1^2
100	34,95	110,02	0,5882	0,3435

Pseudo-second-order kinetics

The plot of t/q versus time for the ACCP activated carbon gives the linear form (Figure not shown). The values of K_2 and q_e , Cal were determined respectively from the slope and the ordinate at the origin of this straight line.

The kinetic constant of the pseudo-second order adsorption reaction k_2 , the amount of BM adsorbed at equilibrium q_e , cal and the correlation coefficient R_2^2 are given in Table 4.

The correlation coefficient R_2^2 is greater than the correlation coefficient R_1^2 , moreover the experimental value of q_e is much closer to the value of q_e calculated from the linear form of the pseudo-second order. Therefore, The pseudo-second-order kinetic model provides a better description of the kinetics of the methylene blue adsorption reaction on ACCP.

Table 4. Pseudo-second order rate constants for adsorption of MB on ACCP.

C_0 (mg/l)	q_e , exp (mg/g)	q_e , cal (mg/g)	K_2	R_2^2
100	34,95	41,67	0,0048	0,9376

3.10. Study of Adsorption Isotherms

Adsorption isotherms are crucial for determining the maximum capacities and identifying the specific type of adsorption expected to take place. They are obtained by the

graphic representation of $Q_e = f(C_e)$ where Q_e and C_e are respectively the quantity of MB adsorbed per g of activated carbon and the equilibrium concentration of MB. The experiment is carried out at a temperature of $28^\circ\text{C} \pm 2$.

The experimental result obtained indicates that the adsorption isotherm of methylene blue is of the L type (Langmuir

isotherm) but is also close to the H type (Freundlich isotherm) according to the classification of Giles et al. [19].

The main parameters characterizing the Langmuir models and the Freundlich model are recorded in Table 5.

Table 5. Values of adsorption parameters Q_0 , R_L , n and K_F of MB.

FREUNDLICH			LANGMUIR		
N	K _F	R ²	Q ₀	R _L	R ²
1,68	21,79	0,98	75,93	0,025	0,91

The value of R_L indicates the type of the isotherm, either being favorable ($0 < R_L < 1$), unfavorable ($R_L > 1$), linear ($R_L = 1$) or irreversible ($R_L = 0$) [20]. For this study the value of R_L turned out to be 0.025; this suggests that the adsorption process is favorable to the concentration of MB used. The correlation coefficient R^2 (0.91) of the Langmuir model is close to 1. This high correlation between the adsorption data and the Langmuir model indicates the homogeneous nature of the surface of ACCP [20].

The adsorption capacity calculated by the Freundlich model ($K_F = 75.93$ mg/g) indicates that the ACCP activated carbon has an average affinity with respect to methylene blue. Finally, the value of the adsorption intensity ($n=1.68$) of methylene blue calculated satisfies the heterogeneity condition ($1 < n < 10$).

Analysis of the results shows that the adsorption isotherm of methylene blue on ACCP activated carbon obeys both the Freundlich and Langmuir adsorption isotherms, as indicated by the high values of the correlation coefficients ($R^2 > 0.90$).

4. Conclusion

This study show that activated carbon prepared from cassava peeling can be use used for efficient methylene blue removal from water. This activated carbon has high iodine index (914.4 mg/g) and methylene blue index (643.72 mg/g) which would imply that this activated carbon is capable of adsorbing small and medium molecules. The Bohem chemical analysis showed the presence of various functional groups on the surface of activated carbons.

Equilibrium and kinetic investigations were conducted to examine the adsorption of methylene blue from aqueous solutions onto activated carbon ACCP. The adsorption kinetics can be regarded as pseudo second order. Both of Freundlich and Langmuir equations showed a somewhat better fit for adsorption of methylene blue using ACCP activated carbon. On basis of the studies reported, we can conclude that cassava peelings could be valued as a precursor for the synthesis of activated carbon for the removal of dyes from aqueous solution.

Abbreviations

ACCP: Activated charcoal made from cassava peelings,
CA: Activated Carbon,
MB: Methylene Blue,
pH_{PZC}: pH at Point of Zero Load.

Conflicts of Interest

The authors declare no conflicts of interest.

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